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**Using a water treatment residual and compost  
co-amendment as a sustainable soil improvement  
technology to enhance flood holding capacity**

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A thesis submitted in fulfilment of the requirements for the degree of Doctor of  
Philosophy

School of Engineering and Computing Sciences Durham University

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## **Abstract**

The recycling of clean wastes, such as those from the treatment of drinking water, has gained importance on the environmental agenda due to rising costs of landfill disposal and movement towards a 'zero' waste economy. More than one third of the globe's soils are degraded and as such policies towards determining soil health parameters and reversing destruction of the globe's most valuable non-renewable source are at the forefront of environmental debate. This thesis questions the opportunity for water treatment residual (WTR) to be used as a beneficial material for the co-amendment of soil with compost to improve the soil's flood holding capacity (Kerr *et al.*, 2016), which includes functions such as the water holding capacity, hydraulic conductivity, soil structure and shear strength. Currently, water treatment residual is typically sent to landfill for disposal, but this research shows that the reuse of WTR as a co-amendment is able to improve the flood holding capacity of soils. This research crosses the boundary between geotechnical and geoenvironmental and provides a holistic approach to quantifying a soil from both perspectives.

Iron based water treatment residual from Northumbrian Water Ltd was used in both laboratory and field trials to establish the effect of single WTR and a compost and WTR co-amendment on the water holding capacity (the gravimetric water content, volumetric water content, volume change of samples i.e. swelling and shrinkage), and the effect of amendment on the erosional resistance, hydraulic conductivity and shear strength compared to a control soil. A series of four trials were conducted to develop and establish a novel method to determine the water holding capacity, supplemented by standard geotechnical methods to determine the flood holding capacity. The use of x-ray computed tomography has provided accompanying information on the morphology of dried WTR and changes in the internal characteristics of amended soil between a dry and wet state. The amendment application rate ranges from 10 – 50%.

Experiments have shown that the single amendment of WTR, compared to a control soil, yields significant increases in the hydraulic conductivity (by up to a factor of 28), increases the shear strength of soils at low testing pressure (25 kPa) by 129%, increases the maximum gravimetric water content by up to 13.7%, and

improves swelling by up to 12% (but only at the highest amendment rate, 30%), increases the maximum void ratio when saturated by 11%, and reduces shrinkage by maintaining porosity by 14%. However the application of WTR as a single amendment has implications for the chemical health of the soil as it is highly effective at immobilising phosphorous as and such cannot not effectively be used as a soil amendment. The single application of compost yielded significant improvement in the water holding capacity (improving gravimetric water content by up to 34.7%, increasing the sample volume by up to 83.3%, and increased the void ratio by 8.2%), however this application reduces the hydraulic conductivity by up to 84.5% and the shear strength by 3% compared to the control soil.

Co-amendment using compost and WTR (in two forms, air dried 80% solids and wet at 20% solids, as produced from water treatment works) improved the flood holding capacity of soils by retaining the structural improvements of amendment using WTR and the water holding capacity improvements of compost. Compared to the control soil, for co-amended soils the gravimetric water content was improved by up to 25%, the volume increased by up to 51.7%, experienced 13% less shrinkage and an 11.5% increase in maximum void ratio. The hydraulic conductivity was also improved by up to 475%, and shear strength was increased at both low and high testing pressures by to 53.8%.

Taking into account these effects of co-amendment on essential soil functions that determines a soil's flood holding capacity (maximum gravimetric water content, volume change, resistance against shrinkage, void ratio (porosity), hydraulic conductivity and shear strength), the economical and environmental sustainability issues, the co-amendment of soil using compost and WTR may provide a solution to both recycling clean waste product and improving the quality of soil.

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## **V. List of Abbreviations**

BD – bulk density

$D_d$  – dry density

DETS – Derwentside Environmental Testing Services

FHC – flood holding capacity

GWC – gravimetric water content

LOI – loss on ignition

NWL – Northumbrian Water Ltd

OM – organic matter

VWI – volumetric water content

WHC – water holding capacity

WTR – Water Treatment Residual

WTR<sub>d</sub> – air dried WTR (80% solids)

WTR<sub>w</sub> – unprocessed WTR (20% solids)

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## **Statement of Copyright**

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## 1. Introduction

There is currently a disconnect between geotechnical and geoenvironmental (soil science) research, with respect to the fundamental philosophies, definitions and the viewpoint on soils. The geotechnical world sees the presence of organic matter and processes of volume change in soils as a fundamental flaw in its use as a material (Franklin *et al.*, 1973), however in geoenvironmental engineering the emphasis on preserving soil functions by maintaining organic matter for its long-term sustainability is forefront (Quotes B- E). This thesis focuses on developing research at the boundary of geotechnical and geoenvironmental work, in a world that splits up soils, water and organic matter, by researching soil with a holistic view of changes in soil function when soils are flooded. The water holding capacity, permeability characteristics, shear strength and erosional resistance of soils are components are critical for the health of our ecosystems, however they are seldom viewed simultaneously to assess a soil's potential to withstand degrading events such as flooding.

*"It is generally accepted that the presence of organic matter in soils acts to the detriment of their engineering qualities"*

Quote A Franklin *et al.* (1973)

*"A nation that destroys its soils is a nation that destroys itself"*

Quote B Roosevelt (1937) in Lal *et al.* (2007)

*"There is a need to increase the volume of pore space a soil can retain under a given load through soil or crop management or the use of soil amendments"*

Quote C Angers *et al.* (1987)

*"there is great potential in managing the soil to increase its organic matter as a means of alleviating the problem of soil compaction"*

Quote D Ohu *et al.* (1985)

*"There is limited appreciation of the role of organic matter in influencing the compatibility of agricultural soils"*

Quote E Soane (1990)

In light of increasing threats to soil health and movement towards a 'zero waste' economy, the work in this thesis assesses the impact of recycling waste materials

from the clean drinking water industry in combination with compost to amend soils. This is discussed in reference to the concept of 'flood holding capacity', defined by Kerr *et al.* (2016) as "*the ability or capacity of a soil to take up and store flood water upon submersion without significant soil erosion or loss of shear strength and to resist the detrimental impacts of flooding on soil structure and critical eco- service functions*". A discussion of flood holding capacity assesses the implications of using a combination of physical metrics - volumetric water content, permeability and shear strength in order to characterize how soils respond to flood and drought conditions. We do this in order to both understand how we may better assess soil function and how we might maintain and even enhance soil function with the use of soil improvement technologies. This is very timely for two reasons. Firstly, since the UK Government's Sustainable Soils Alliance has just launched a call in August 2018 for to define soil health and secondly the UN has just announced that soil health underpins all the UK's Sustainable Development Goals.

### **1.1 Threats to soil**

Soil is a highly valuable natural non-renewable resource, and fundamental to life on Earth. It is arguably the most important natural resource on earth due to the wide range of eco-system functions that it performs as part of the water, nitrogen and carbon cycles, where soil organic matter generates and regulates every ecosystem service that sustains life on earth (Lal, 2003). The complex matrix of soil allows it to function as Earth's largest environmental filter, providing an enormous carbon sink, providing a habitat for plants and organisms while recycling nutrients and filtering harmful materials. The structural characteristics of soil render it somewhat like a sponge, with the ability to hold, transmit water and regulate its movement, which provides a natural flood defence by mitigating extremes in precipitation. However, one third of soil across the globe is moderately to highly degraded (FAO & ITPS, 2015), where degradation can be defined as a decline in soil function (Lal, 2009), split into three aspects: physical, chemical and biological (Dexter, 2004). Physically degraded soil has reduced structural ability and as a result is more susceptible to erosion, compaction and reduced water infiltration, which increase the likelihood of flooding. Chemical degradation typically involves acidification, nutrient depletion, greater concentrations of heavy metals, and contamination from industry, which affect the productivity of the soil. Biological degradation of soil is typically

characterised by a loss in soil organic carbon, increased green house gas emissions, loss of plant and micro-organism biodiversity (Lal, 2009). There are a multitude of implications of degradation, which include the loss of food yield and security, the loss of critical functions such as carbon and water storage and increased risk to impacts from flooding and erosion. Although this thesis focuses on the physical characteristics of soil and its interaction with water, the importance of the biological and chemical functions of soil cannot be underestimated. Often once the processes of soil degradation begin, a downward spiral ensues if there is no intervention in destructive anthropogenic processes that accelerate soil degradation. Anthrosols, i.e. a soil that has been heavily modified by human activities occupy a very small percentage of the earth's surface (0.0004%), but are becoming larger with continued influence of society on soils (FAO Soils Group, 2000)

Mbagwu & Obi (2003) and Biancalani *et al.* (2012) suggest that soil erosion is the most prevalent mechanism of soil degradation worldwide, affecting approximately 85% of land. Soil erosion is in fact a natural geologic process, however accelerated soil erosion as the result of anthropogenic influences is a negative process and destroys the resource of soil far faster than it can ever recover (Lal, 2003 & 2015). Rozanov *et al.* (1990) state that more soil has been lost in the last 10,000 years than is currently available for agricultural use. A common reference on the magnitude of erosion is to Oldeman (1994) who states that water erosion affects 1094 million ha of land across the globe, which represents approximately 12% of the land used by mankind. Many assessments of soil erosion are lacking in quantitative, unbiased and reliable data due to the difficulty in timely and accurate detection of soil erosion (Lal, 2003; Obalum *et al.*, 2017), and the lack of a universal definition of 'soil erosion'. Readers are directed to Lal (2003) and Lal (2009) which provide thorough reviews of global erosion trends and processes and an overview of soil quality and management of soil degradation, however the fundamental message is that we are unsustainably degrading our most valuable natural resource, which has both tangible and intangible effects on the productivity and functions of our soils.

In the UK, degradation of soil typically occurs through erosion, compaction, soil contamination and loss of nutrients including organic matter (Hamza & Anderson, 2005; Van Oost *et al.*, 2007). Research at Cranfield University has suggested that soil

degradation costs the England & Wales economy an estimated £1.2 billion per year (Graves *et al.*, 2015), however Environmental Audit Committee (2017) suggest that we are still complacent to degradation. According to UK Climate Projections (Murphy *et al.*, 2009), as a result of climate change the UK is likely to experience hotter drier summers and warmer wetter winters, with increased likelihood of extreme weather events. The KPMG suggest that the cost of flooding in the UK could rise to £6 billion of which £2 billion included flood defence repairs, higher renewal insurance costs, and loss of agriculture yield (Hershey, 2016, [1]). The need to preserve soils so that they are less vulnerable to these extremes and may be able to help mitigate their effects, is vital. Although the economic effects of soil degradation are tangible for processes directly dependent on soil such as crop yield, the impact of soil degradation also includes increased risk of flooding due to detrimental changes in soil structure and water holding capacity as a result of compaction and the loss of soil organic matter.

## **1.2 Soil health policy**

At the World Soils Conference (2018), the UN announced that all sustainable development goals are reliant on healthy soils (UN News, 2018 [2]). The importance of protecting soil and remediating soils degraded by human activity (anthrosols) is slowly being better recognised by governments and local authorities, i.e. bodies that may be able to insight and regulate long term change in how we treat soils. Soil quality and soil sustainability are key words that have appeared at important global environmental agendas, in Agenda 21 (section 2) of the Rio Summit (UNCED, 1992), at the UN Framework Convention on Climate Change (UNFCCC, 1992), and in Articles 3.3 & 3.4 of the Kyoto protocol (UNFCCC, 1997), where attempts to quantify the level of degradation of the globe's soils have been made. Sustainability is defined as "*development that meets the needs of the present without compromising the ability of future generations to meet their own needs*" (Brutland *et al.*, 1987), however the term quality is much more difficult to define, and the reason for on-going debate on how to best measuring improvements. Soil quality is a concept with varying perceptions, due to a historic lack of definition or legislation unlike legislated determinations of water or air quality. Karlen *et al.* (1997) suggest that at the most basic level, soil quality is the capacity of a soil to function, where function refers to the dynamic nature of soil encompassing three critical components; sustained

biological productivity, environmental quality and plant/animal health. The concept of 'soil quality' can be adapted to the particular use of that soil, i.e. for agriculture, remediation of waste, recreation or for the development of urban areas, hence why an interdisciplinary approach is needed to assess the soil in context of its application. Commonly the organic matter content is used as an indicator of the soil quality due its vital role in many functions of the soil (Obalum *et al.*, 2017) and the relative ease of measurement, however the single measurement of soil parameter is not wholly sufficient to determine the total response of a soil, such as degree of soil erosion, to a given perturbation, such as flooding (Dexter & Czyz, 2000; Karlen *et al.*, 1997; Acton & Gregorich, 1995; USDA-NRCS, 1996; Öztaş 2002).

The concept of soil quality is suggested by Karlen *et al.* (1997) to be simultaneously redundant and impossible as 'everyone' knows what makes a good soil however due to the heterogeneity in soil orders, quantifying the natural differences is a challenging task. To provide guidelines and targets to determine soil health, parameters to indicate a soil's quality are required. The difficulty with trying to implement soil policy across the globe is the lack of access to evidence needed for the implementation of policy (clear cause-effect links), the long-term nature of the processes of soil change (which may take decades before detection) and a disconnection between the needs of urbanising human societies and the needs of soil (FAO, 2015 [3]). The FAO (2015) suggest that there are four critical priorities to maintain soil as a sustainable resource and avoid further degradation of our planet's most important resource.

- Sustainable soil management: minimising further degradation to provide food security
- Stabilisation or increase of global soil organic matter stores and identification of SOC improving strategies and implementation.
- Stabilisation or reduction of global nitrogen and phosphorous fertilizer use in areas close to the limits of total fixation, and increase in areas of nutrient deficiency.
- Improvement in condition of the soil and our ability to observe and monitor these changes through improvements in knowledge on soils

In the UK a Soil Strategy for England was published in 2009. Recent events such as the 2013/2014 floods that cost an estimated £1.3 billion in damages have placed soils firmly on the policy agenda (Environment Agency, 2016 [7]). In October 2017, the Sustainable Soils Alliance (SSA [4]) was launched at a parliamentary reception where MP Michael Gove stated that soils are the UK's most valuable source and suggested that improving soil health would be at the heart of future policy (referring to DEFRA's A Green Future: Our 25 Year Plan to Improve the Environment, 2018). This statement describes the newfound governmental/policy maker attention that soil is rightfully gaining (Krzywoszynska, 2017 [8]). The event was attended by 200 experts and leaders, which represent various parts of the soil communities in the UK (academics, farmers, industries) in addition to MPs, from which four distinct tasks or areas requiring political attention were concluded:

- A regulatory framework to promote best practise and deter harmful soil management practise
- A viable system for the monitoring and evaluation of the quality of our soils
- A robust compliance system of economic incentives balanced with regulatory measures
- Investment in training, education and public communication, and a career path for farming as a profession

A number of key statistics stated in this report are particularly prevalent in this piece of research, and highlight the magnitude of the issue of soil degradation in the UK;

- The contribution of damaged soils to flooding events is estimated to be £233 m per year (Securing UK Soil Health, 2015 [9]).
- Compaction threatens 35% of Europe's soil and contributes to flooding (Soil: worth standing your ground for, 2011 [10]).
- The UK has lost 84% of its fertile topsoil since 1850, with erosion continuing at a rate of 1-3 cm/year (The Committee on Climate Change report, 2015 [11]).

- The central estimate for annual (quantifiable) costs of soil degradation in England & Wales is £1.2 bn, linked to loss of organic content of soils (47%), compaction (39%) and erosion (12%) (Graves *et al.*, 2015).
- 300,000 Ha of UK soils are contaminated with PTEs (Environmental Audit Committee Report on Soil Health, 2016 [12]).
- UK soils store over 10 billion tonnes of carbon in the form of organic matter (The Welsh Government State of Natural Resources Report, 2017 [13]), which is 50 x the UK annual GHG emissions.

As a result of increasing concern over the health of UK soils, a number of policies and targets within long-term frameworks have recently emerged. An example of this positive change towards safe guarding soils is the ‘new farming rules for water’ (DEFRA, 2018), introduced to standardise good farming practices and protect water quality, which requires land managers to test their soils every five years and take measures to sustain soil and prevent pollution from runoff or soil erosion into local water bodies. The remit of the legislation, enforced by the Environment Agency, includes ammonia pollution from the application of fertilizers, and the effects of cultivation and irrigation methods employed.

Currently the UK follows DEFRA’s 2011 strategy (Safeguarding our soils: A strategy for England [14]), with a headline target that by 2030, all soils in England will be managed sustainably and degradation threats tackled successfully. The strategy states that spreading recycling material to land is important for increasing soil organic matter while diverting suitable materials from landfill. The UK government has committed to the 4per1000 initiative, which aims to increase soil organic matter by 0.4% each year (UN Climate Change Convention, Paris 2015 [15]). Although ambitious, if each nation were to achieve this goal, 75% of the global annual greenhouse emissions would be offset and would contribute to the limitation of global temperature increase (<2 °C) beyond which the IPCC indicates that the effects of climate change would be significant [5].

### **1.3 Engineering soils; soil amendment and waste recycling**

The previous section discussed the wider implications of soil degradation across the globe and with specific reference to the UK and emerging policy on how to best improve our soils which includes increasing organic matter content of soils. However, this emerging policy does not cover the important research area of how we can optimise specific soil functions such as flood resilience. This thesis focuses on engineering soils by adding mineral and organic amendments in order to optimise 3 soil functions, the water holding capacity (the maximum volume of water a soil can hold), the hydraulic conductivity (the speed at which water can move through the soil), and the shear strength and erosional resistance (which determines how well a soil can resist the effects of shearing and erosional forces).

The economic costs of flooding are readily measurable when considering the effects on buildings, infrastructure and knock on impacts to businesses after an event (e.g. £1.3 billion for recent floods in 2013/2014). However, both the role of soils in being able to help mitigate these flooding events through water storage and vice versa the role of flooding in having a long-term deleterious effect on soil health (soil is often literally washed away) has not been studied. In an increasingly urbanised environment, soils could provide significant mitigation to flooding should their quality be maintained or improved, such that they are able to store and transmit water while retaining their structure and resistance to erosion. The water holding capacity of a soil, and therefore the resilience to flood and drought is dependent on the organic matter content, where just 1% mass increase in organic carbon can yield a volumetric water increase of 1.16% (Minasny & McBratney, 2018). However, soil is routinely degraded by the permanent removal of organic matter through agricultural practices. For example, in 2014, 30 million tonnes (out of a total of 100MT) of organic wastes produced from land were not returned to the land meaning that carbon levels are falling (House of Lords, 2014 [6]). This vicious cycle of soil degradation can be broken if organic matter is replaced and stabilised at sustainable rates.

Previous work at Durham University (ROBUST) has shown that minerals such as manganese oxides are able to stabilise organic carbon in sediments (although the mechanism is not fully characterised). Further work at Durham has shown that the

mineral waste Water Treatment Residuals (abbreviated as 'WTR', from clean water treatment) when added to contaminated soil can immobilise heavy metal pollution (McCann *et al.*, 2015 and 2018) and although this includes the immobilisation of phosphorous (which is detrimental for plants), this issue is mitigated if sufficient supplementary P is added with the co-amendment of compost. Although initial positive effects on the soil quality in the laboratory have been recorded, the long-term benefits or wide-scale field application of recycling waste WTR minerals into soil are currently not quantified due to the relative novelty of research into its effect and slow rate of measurable soil change. Restrictive legislation in the UK on the use of landfill has pushed the agenda of recycling waste by industry to new heights, and companies such as Northumbrian Water Ltd (NWL) are working hard to explore disposal avenues that close the loop on waste production by recycling organic wastes back to land, under the guidance of EA regulations.

#### **1.4 Thesis outline**

The effects of adding compost and WTR as single amendments to soil are well characterised in a geochemical context. However there is no known research that assesses the effect of co-application on the flood holding capacity of soils, which includes an holistic analysis of the water holding capacity, hydraulic properties and shear strength of soils amended with co-applications of compost and WTR. This work aims to provide information for water companies on how they may best manage their WTR mineral waste. Currently the disposal criteria for WTR are based on the presence of heavy metals and nutrient values, and WTR may only be spread to land should the concentration of these in soils match acceptable levels. In effect, companies such as NWL may spread the WTR on land providing it is not of detriment to the soil. This thesis provides an investigation into the WTR produced in the NE of the UK (by NWL), and although the makeup of WTRs is highly dependent on the region in which clean water is treated, this research has global implications. The flood resilience of soils is investigated, but the ability of WTRs to increase drought resilience is important in developing countries such as South Africa, that have larger extremes in flood and drought scenarios, therefore this work may provide insight in how to best improve both drought and flood resilience by reusing WTR wastes in conjunction with organic amendments.

The aim of this thesis was to establish the effects of adding water treatment residual and compost to soil, with respect to the water holding capacity, hydraulic conductivity and shear strength, and hence establish if using WTR with compost can improve soil function, rather than just being sent to landfill. An assessment of soil's 'flood holding capacity' was achieved by completing the following specific objectives:

- 1) Development of novel water holding capacity experiments to assess the maximum gravimetric and volumetric water content of amended and unamended soils over at least one wetting and drying sequence.
- 2) Erosional resistance testing of amended and unamended soils through fall cone penetrometer and a newly developed 'Veitch' method.
- 3) Assessment of hydraulic conductivity of amended and unamended soils using a triaxial cell apparatus
- 4) Assessment of shear strength properties of amended and unamended soils using a triaxial cell apparatus
- 5) Analysis of amended and unamended soils using X-ray Computed tomography, in order to understand the effect of amendment on soil structure.
- 6) Field trial application of the co-amendment at Weetslade Country Park.

This thesis is structured in the following way:

Chapter 2 provides an introduction to important soil relationships relevant to the flood holding capacity of soils (e.g. soil organic matter and aggregation) and important characteristics of soil structure, before exploring current limitations of soil analysis, with particular reference to the way in we measure water in soil.

Chapter 3 summarises how water treatment residual is produced, stored and disposed of, with particular reference to the operation of Northumbrian Water Ltd, and subsequent characterisation of WTR and a discussion on implications for use as a soil amendment.

Chapter 4 characterises materials used in the thesis, the methods used (with the exception of XRCT) including the development of a new methodology for water holding capacity trials, erosional resistance testing and triaxial testing.

Chapter 5 subsequently provides the analysis and discussion obtained from the testing outlined in Chapter 4.

Chapter 6 provides a head-to-toe introduction of the theory, discussion of methods and analysis of results obtained from using x-ray computed tomography on amended soils. In addition, there is a micro-scale analysis of air-dried WTR.

Chapter 7 discusses a field trial using the co-amendment conducted at Weetslade Country Park, Northumberland.

Chapter 8 summarises the major findings and provides a summary.

## 2. Fundamentals of Soils

In a geotechnical content, the word soil refers to the unbonded mineral matter formed due to weathering, where the term 'topsoil' means the highly organic upper layers of a soil's profile in which plants grow, in which water fluctuates above the water table and has variable characteristics based on water content and compression (Powrie, 2004). Soils are a complex, unique and irreplaceable essential resource formed under the influence of plants, micro-organisms, soil biota, water and air from a parent material due to physical, chemical and biological weathering processes (Breemen & Buurman, 2002). They provide a substrate for plant growth, biochemical cycling of water, and elements such as carbon and nitrogen. They form a critical subsystem of many ecosystems. Soils take up to a millennium to form from a relatively inert geological substrate; practises such as abusive agricultural management, land clearing and reclamation, erosion (natural and anthropogenic), salinization, desertification, and the use of land for industry and housing are destroying soil faster than it can ever form. At the most fundamental level, soil matter is a three phase matrix of solid, liquid and gas (Richards, 1965) formed from the interaction of weathering and biological activity upon a parent material (igneous/metamorphic/sedimentary rocks); i.e. a soil is comprised of mineral matter, organic matter, water and air in various proportions. A more wholesome definition includes soil as a natural body comprised of minerals, organic compounds, living organisms, water and air (Gerrard, 2014).

### 2.1 Soil formation

How a soil develops depends on five factors; parent material (mineralogy of the rocks), time, climate, topography and organisms (vegetation, fauna and soil biota) (Dokuchaev, 1898; Jenny, 1980; Brady & Weil, 2016). Brady and Weil (2016) describe the sequence of factors as "*dynamic natural bodies having properties derived from the combined effect of climate and biotic activities (organisms), as modified by topography, acting on parent material over periods of time*". Subsequent processes of hydrology and human influence have been added later. To study the effect of one factor, a soil forming factor (state factor) approach is used, where all others remain constant e.g. chronosequence (soil age changes), climosequence (climate changes), and toposequence (elevation changes). This

approach, however, assumes that these factors are independent and each has an equal influence on soil formation. Shaw (1930) modified the original Dokuchaev formula for principal factors of soil formation:

$$S = M (C + V)^t + D$$

*Equation 1: Dokuchaev formula for principle factors of soil formation, where soil (S) is formed from parent material (M), by the operation of climatic factors (C) and vegetation (V) over time (t), modified by erosion or deposition on the soil surface (D).*

**PARENT MATERIAL (M):** The characteristics of different soils tend to reflect their parent material (particularly in young soils); igneous or metamorphic rocks tend to produce acidic and sandy (siliceous granite and gneiss) or non-acid and clayey (basalt and diorite) soils. Sedimentary rocks e.g. limestone and sandstone produce sandy or clayey soil, and shale produces clayey soil due to the presence of clay minerals. Parent material can come directly from bedrock in situ such as colluvium or scree at the base of hillslopes and mountains moved via the force of gravity, but much parent material is derived from other sources and deposited. Material can be transported by ice in terminal or lateral moraines, where the deposit is called glacial till and has a large particle size distribution. Matter deposited by water (rivers and overland flow, lacustrine (lakes) or marine (oceans)) provides greater sorting of particles as greater forces are required to move larger pieces. Large pieces of mineral matter are found upstream at water sources, and smaller fractions are found downstream due to lower water energy and attrition forces acting during particle movement. Loess or aeolian (wind) transport moves the finest particles over large areas.

**CLIMATE (C):** This determines temperature and rainfall regimes, which are typically considered independently despite simultaneous operation (Mackey & Burnham, 1964), and is the most important factor in a soil's development. It governs the rate and type of soil formation due to its effect on weathering (physical & chemical), vegetation and microbes (decomposition and humification), precipitation and evaporation (percolation, capillary action and leaching), and temperature (rate of chemical and biological reactions).

**ORGANISMS & BIOTIC FACTORS (V)** Different species of organism contribute to soil formation in varying degrees, but are vital in the degradation of plant material

and subsequent humus formation (total organic compounds, soil organic matter excluding un-decayed plant and animal matter). They are also significant in physical and chemical weathering. Temperature is particularly important as it affects the range of microorganisms that can be active.

**TIME (t):** The speed of soil production is based on many factors. The type of parent material is significant as generally rocks with a lower silica content are broken up more quickly by physical or chemical weathering. The topography affects the speed of mineral build up, e.g. steep slopes are eroded, thereby constantly impeding the build-up of parent material, whereas river plains constantly have sediment deposition. Soils on alluvial planes and slopes tend to be younger than plateaux as the age of a soil is closely related to relief. Warm climates speed up the production of soil due to increased rates of biological processes. Depending on the climate, a soil can take decades to millennia to form. Soils cannot be characterised once as they do not remain a static, stable object, therefore time is a natural dimension in the formation and consideration of soil through its development and change through pedogenic processes.

**TOPOGRAPHY (D):** This influences the climate due to differences in altitude (colder and more precipitation at higher altitudes). Steeper slopes at higher altitude have thinner, less developed soils due to erosion, lower water infiltration, reduced vegetation and lower temperatures, all of which stunts the development of soil and organic matter. Topography also influences movement and accumulation of water and as discussed in subsequent sections, the water content of soil dictates many of its properties.

### **2.1.1 Natural processes of soil formation**

Natural processes of soil formation and change are split into physical, chemical and biological categories, although of course these are all interlinked as no processes can occur independently. Initially, to start the process of soil formation, rocks are physically weathered (Boeker & Grondelle, 1995). To produce the mineral matter at a sufficient scale to create soil, physical weathering breaks down rock into smaller parts either by thermal or mechanical means. Minerals expand to different degrees due to water or temperature, causing stress within the rock and provide areas of weakness because of changes in temperature either across a surface or between inner and outer layers of the rock. An example of this is onionskin

weathering where the outer layers of a rock are heated and cooled repeatedly, fracturing the outer layers. Mechanical weathering (frost shattering) occurs due to the expansive nature of water when it freezes. When water penetrates small cracks in the rocks surface and freezes, the volume change of the water in combination with mineral swelling and shrinking is sufficient to initiate stresses that fracture the rock. Additionally, plant growth can cause mechanical breakdown due to root growth.

Chemical weathering (hydrolysis, carbonation, hydration, acid dissolution and redox) changes the chemical and physical composition of rock minerals (McBridge, 1994; Sparks, 2003). Simply speaking, it is the transformation of minerals to solutes (dissolved substances) and solid residues. The majority of igneous and metamorphic rocks consist of silicate and metal ions (silicates such as  $\text{Si}_2\text{O}_5^{2-}$ ) with free silica ( $\text{SiO}_2$ ) forms e.g. quartz (Breemen & Buurman, 2002). These primary minerals weather to iron (Fe) and aluminium (Al) oxides, clay minerals and secondary minerals, also called amorphous silicates (Breemen & Buurman, 2002). The formation of clay minerals and iron compounds is extremely important as these secondary minerals have large, charged surfaces, which strongly influence soil characteristics. Primary minerals that resist chemical change e.g. quartz (part of granite) are simply physically broken down to form sand. Minerals such as feldspars or micas are vulnerable to decomposition by organic and inorganic acids and are broken down into secondary (soil) minerals (e.g. clay).

'Soil minerals' is a term used to describe the combination of secondary minerals with highly resistant primary minerals. The formation of secondary minerals and 'soil minerals' is a process that may take thousands of years, depending on climate. Weathering agents for chemical weathering are water, organic and inorganic acids, complexing agents and oxygen. Weathering without leaving a solid residue is called congruent dissolution (Breemen & Buurman, 2002); an example of this is complete dissolution of olivine to  $\text{Mg}^{2+}$  (magnesium cation) and  $\text{H}_4\text{SiO}_4$  (silicic acid, the dominant form of dissolved silica in waters). Where the concentration of these dissolved materials is high, some of this can be precipitated ( $\text{MgCO}_3$ , magnesium carbonate) and is called incongruent dissolution of a mineral by decomposition or reaction in the presence of liquid, converting one solid to another. This is common

due to the presence of iron and aluminium in more minerals. The breakdown of primary minerals into secondary minerals occurs through a variety of mechanisms:

- *Hydrolysis* is the most common of these processes, where water molecules ( $\text{H}_2\text{O}$ ) dissociate into charged particles (hydrogen ions ( $\text{H}^+$ ) and hydroxyl ions ( $\text{OH}^-$ )) that break the bonds holding minerals together.
- *Carbonation* is essentially hydrolysis sped up due to biological activity, where carbon dioxide ( $\text{CO}_2$ ) respired from soil organisms forms carbonic acid ( $\text{H}_2\text{CO}_3$ ) when in contact with water. The carbonic acid dissociates to provide hydrogen ions, enhancing the processes of hydrolysis. Plant roots provide two-fold mechanisms for chemical weathering, due to the production of carbonic acid at the root surface as it respire, and the excretion of sugars that are converted to acids when used by microorganisms.
- *Hydration* is the weakening of minerals after the absorption of water, making them vulnerable. Some minerals such as sodium chloride or potassium chloride may be dissolved by water (dissolution) and removed in solution.
- *The redox process* (oxidation and reduction) weakens minerals due to chemical changes and loss of electrons (oxidation) or gaining electrons (reduction). Oxygen ( $\text{O}_2$ ) is an important weathering agent for minerals containing elements in a lower oxidation state. Iron, sulphur and manganese are examples of elements that often occur in more than one oxidation state, where iron exists as native iron, ferrous iron ( $\text{Fe}^{2+}$ ) and ferric iron ( $\text{Fe}^{3+}$ ). The redox of iron (Fe), manganese (Mn), sulfur (S) and organic compounds influence a number of soil properties. Most soils are oxic, i.e. compounds are in an oxidated state and hydrous oxides of  $\text{Fe}^{3+}$ ,  $\text{Mn}^{3+}$ ,  $\text{Mn}^{4+}$  are stable, whereas in anoxic soils that lack  $\text{O}_2$ , reduced  $\text{Fe}^{2+}$  and  $\text{Mn}^{2+}$  minerals are stable. Organic matter is very influential in the state of soils as photosynthesis by plants produce localised strongly reduced conditions. Well-drained soils remain oxic due to diffusion from the atmosphere. When a wet soil is sealed (perhaps during flooding) and gas diffusion is very slow, if decomposable organic matter is present then the soil will become anoxic very quickly. Once  $\text{O}_2$  surplus is used by respiration,

OM is oxidised by Mn (IV), Mn (III), Fe(III) and S(VI). The presence of Fe (III) usually is predominant and therefore it is the typical oxidant for organic matter in waterlogged conditions; iron plays a critical role in the chemistry of flooded soils.

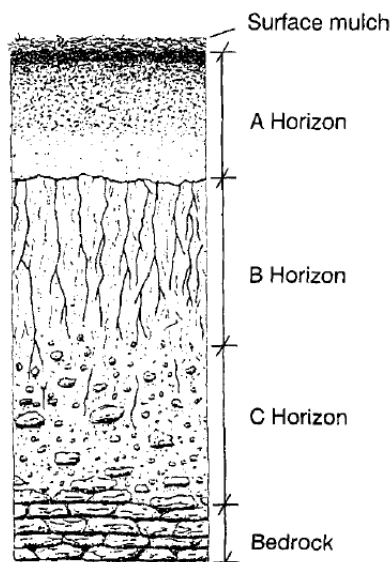
Although biological processes can be covered under both physical and chemical brackets, they are of significant importance due to the complex nature of soil. Biological processes are driven by plant growth and subsequent microbial action on dead plant material, which forms the organic 'parent material' for soils (see section 2.2.1) The influence of soil organisms is vital for the production of a healthy soil, although the biological aspects of soil are not covered in the scope of this thesis. Briefly, biota such as earthworms, nematodes, beetles, ants and millipedes aerate the soil by increasing porosity, adding organic acids and CO<sub>2</sub>. Biota have a significant role in the formation of soil aggregates (see section 2.2.2), whereas organic materials provide physical or chemical binding agents for soil mineral matter in the form of roots and fungal hyphae and humic acids. Soil air is an important constituent that governs the activity of the biological processes in soil. It is typically carbon dioxide enriched and has a high oxygen demand especially at the surface horizons, which diffuses from the large air pool in the atmosphere. The availability of O<sub>2</sub> controls the rate of respiration (along with the temperature, water content, nutrient supply and organic matter content). The diffusion of gases is 10,000 times faster in air than through water, therefore the water content of a soil is particularly important for processes that require oxygen. During wet periods, soils are often anaerobic, inducing redox reactions. Waterlogging encourages the production of gleying conditions (causes production of wetland soil), changes in pH, accumulation of organic matter due to anaerobic decomposition, production of toxic by-products (Ross, 1989), and finally soil ripening.

The pedosphere (soil geosphere) has many important relationships with the surrounding geospheres (atmosphere, biosphere, hydrosphere and lithosphere). The interaction between these spheres is evident in a number of global cycles, namely hydrological, carbon and nitrogen cycles. There are continual exchanges of water between the hydrosphere and pedosphere through various means, such as

precipitation, transpiration and evaporation, as water is transferred in different states. Soil stores vast quantities of carbon, readily available to organisms as CO<sub>2</sub>, and as locked in carbohydrate molecules in soil organic matter and hydrocarbon compounds in rock. Nitrogen moves through the global systems in a variety of forms; plants, species of soil bacteria and cyanobacteria assimilate nitrogen from the atmosphere. Oades (1993) provides a thorough discussion of the biological influence on soil processes, particularly the relationship between biota and aggregation or disaggregation processes. In addition, readers are directed to Paul (2014) and Gerrard (2014) for complete reviews of the interaction between microbes and soil, ecology and biochemistry, as there is not scope for a thorough discussion of the biological elements of soil processes here.

### 2.1.2 Pedogenesis (soil change and characteristics)

As the component parts of a soil are being formed, the soil matter also undergoes a number of complex processes that change the characteristics of the soil, which are dependent on the environment in which soil is formed. Soil formation across the globe occurs differently, and subsequently there are thousands of soils recognised around the world, each of which has a unique set of characteristics based on their pedological development. These are classified into 10 orders that are based on the properties of 'horizons' within the soil. For example, across in the UK, histosols (which frequently form in water saturated areas and are derived from organic matter e.g. peat) account for 3.3% of the world's soils (Bohm, 1976). Alfisols (clay enriched and fertile soil formed under hardwood forests) are most common, covering 13% of the world's land area, (Hillel, 2008).



*Figure 1: Schematic representation of a hypothetical soil profile. The A horizon is shown with an aggregated crumb like structure, the B horizon with columnar structure and the C horizon with incompletely weathered rock fragments (Hillel, 1998)*

According to Breemen & Buurman (2002), the movement of water, dissolved substances (solutes) and suspended particles, temperature gradients or fluctuations, shrinkage and swelling are the main soil physical processes that influence specific soil formation. The interaction of these processes produces soil horizons within a soil profile (Figure 1). Soil is not uniform in nature and consists of a number of layers or horizons that have variable characteristics based on depth. The soil surface conditions affect the important processes such as radiant and thermal energy exchange and the movement of water and gas. The upper horizon i.e. the top layer of the soil is where the majority of biological activity occurs and therefore where proportionally the most organic matter is held within the soil. The next layer, the B-horizon, contains the accumulation of small particles such as clay or carbonates that have been transported by water movement. The parent material forms horizon C; for residual soil formed in situ from bedrock this horizon would be weathered material, or may be alluvial, Aeolian or glacial sediments (Hillel, 1998)

A critical factor in the pedogenesis of soils is the movement of water into, through and out of a soil. Only 0.03% of the world's water is mobile in the hydrological cycle, of which 0.005% is in the soil (Strahler & Strahler, 1976). The movement of water through a soil either by gravity or through capillary action changes the soil properties over time. Capillary action and leaching determine where water and soluble minerals are within a soil profile. Under drying conditions, moisture moves towards the surface against gravity, however where precipitation exceeds evaporation from the soil surface, minerals are leached downward through the soil. Eluviation is the general term for the movement of soil material in solution, which includes leaching, cheluviation and illuviation. Leaching involves the movement of soluble soil components (organic and inorganic) in percolating water, commonly decalcification/calcification and podzolisation on siliceous soils. Cheluviation is the translation of metal cations (iron and aluminium) by soluble organic complexes, which include polyphenols (from plant foliage and litter) and condensed humic and fulvic acids. Lessivage is the movement of clay in suspension (only once detached from colloids), typically deposited when soils dry or water movement becomes negligible. Illuviation is the introduction of salts or colloids (of clay, iron and aluminium oxides and hydroxides, and organic matter) into a soil

horizon by percolating water. The mechanisms of this process are still debated, but thought to happen through changing mobility of solutes.

## ***2.2 Geo-environmental aspects of soil***

Krull *et al.* (2004) provide a vastly expansive discussion of the functions of organic matter and the effect it has on soil properties (particularly on aggregate stability, cation exchange capacity, buffer capacity and water holding capacity); readers are directed to this publication for a thorough review. The following sections summarise the important functions of organic matter and aggregation in soils.

### **2.2.1 Soil organic matter**

Soil organic matter (SOM) is all dead organic material within a soil, although in reality there are many parts of living matter that are incumbent with mineral matter and cannot be readily separated. The largest fraction of SOM is humus, which is heavily decomposed plant material that is dark in colour, acidic and hydrophilic. The decomposition of plant material is the inverse of photosynthesis, and in oxic conditions, SOM is unstable and oxidises to CO<sub>2</sub> and H<sub>2</sub>O when broken down. Nutrients are released in their ionic forms during decomposition (inorganic solutes and gases) and this process, called mineralisation, is completed predominantly by fungi and bacteria. The vast majority of dead biomass is mineralised with a small fraction converted into humus. Decomposition rates are controlled by the climate and the material being broken down, e.g. fine roots and deciduous leaves in moderate temperatures and sufficient supply of oxygen and nutrients are typically broken down within a year but branches take decades and trees up to centuries to break down (Breemen & Buurman, 2002).

To form a soil, organic matter must be incorporated to the mineral matter produced from weathering of a parent material, occurring due to bioturbation (the movement of roots and soil animals through the soil profile altering the structure of sediments and weathered material on the surface of a weathering horizon by homogenising the soil constituents). Soil organic matter binds the mineral particles together into aggregates, which are an essential part of a soil's structure. The majority of soil organic matter is present in the upper layers of the soil (top soil), and ranges in composition from plant matter and animal tissue to humus

(decomposed matter). Humus is dark decomposed material that forms the majority of SOM, produced by humification. Humus does not have a specific form and humic substances are characterised by their behaviour, such as interaction with metals. The rate at which humification occurs is based on the temperature (optimum temperatures are in the range of 25-35° C), water content, nutrient availability and microbial biomass. The processes of decomposition and humification by enzymes, earthworms and other organisms, determine the quantity of typically plant-derived material that is broken down into the basic constituents; cellulose, hemicellulose, lignin, protein & amino acids and waxes. In an environment where there is no organic matter present, and only mineral matter from the weathering of rock is available, organisms that are able to survive in water and nutrient limited environments are able to colonise in order to produce a soil. They are able to obtain nitrogen, an essential element plant growth using photosynthesis and N-fixation. Lichens are typically a primary coloniser as the symbiotic relationship between algae and fungi, where algae obtain carbon and nitrogen through photosynthesis and N-fixation and pass the surplus to fungi, which chemically weather the rock with acid to release minerals for algae. Once the lichens die, mineral matter weathered from the rock surface combines with the organic material to produce soil, a process that allows further colonisation by plants requiring soil to grow. Organic matter accumulates at the surface of the soil and is mixed with mineral matter to form aggregates by microbial activity. It is typical of the uppermost horizons of the soil to be darker and higher in organic matter than lower down in the profile as soil organisms are only active near the surface.

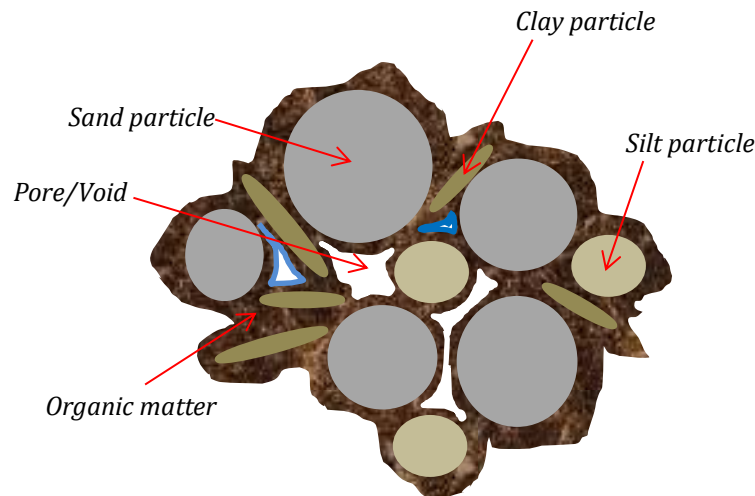
It is well known that organic matter is a critical substance in the formation and function of soil due to its influence on soil stability, aggregation, water holding capacity, carbon sequestration, infiltration, permeability, microbial function, crop yield and has implications for the engineering properties of a soil (Adejumo, 2012; Arias *et al.*, 1999; Ekwue 1990; Hayes & Swift, 1990; Hudson, 1994; Hollis *et al.*, 1977; Jastrow & Miller, 1997; Lal, 1993; Le Bissonais 1996; Majumder *et al.*, 2008; Puppala *et al.*, 2007;). As such the quantity of organic matter in a soil is often used as an indicator of soil health due to its vital role in the improvement of soil properties and processes (Lal, 1993; Obalum *et al.*, 2017). It is commonly stated

that organic matter has the ability to hold up to 10 times its own weight in water, although this may vary in magnitude where Hudson (1994) showed that a silt loam with 4% organic matter holds more than twice that of the same soil with only 1% organic matter. As well as being able to hold water in the material itself, organic matter also aids the aggregation of materials in the soil, which creates a network of voids pores within the soil, through which water, air and microbes move through the soil profile. In this thesis, there is a focus on the influence of organic matter on the water holding capacity and water retention properties of a soil as well as the effect on soil structural stability. These prominent beneficial effects of organic matter are seen as a result of the relationship between organic matter and soil aggregation, as described below.

### **2.2.2 Soil aggregation**

Soil aggregates are fundamentally grouped soil particles that are bound strongly by physical and chemical bonds, providing soil structure. A range of biotic and abiotic processes create aggregates, which are characterised by their higher internal strength the surrounding soil matrix (Bronick & Lal, 2005; Kemper & Rosenau, 1986). A number of comprehensive reviews on the detailed processes of aggregation are available (Harris *et al.*, 1966; Swift, 1991; Tisdall, 1994), so these are summarised briefly in order to provide understanding in their role in soil health and erodibility. Aggregates are formed when the colloid of particles is able to resist the breakdown forces of rehydration, and typically its resistance to breakdown during wetting tests the “stability” of an aggregate. As shown in Figure 2, the formation of aggregates creates voids between the larger colloids called pores. Pores are classified by their equivalent diameter, and although these values are variable in older literature, the Soil Science Glossary (2008) defines a macropore as  $>75 \mu\text{m}$ , mesopores  $30 - 75 \mu\text{m}$ , micropores  $5-30 \mu\text{m}$ , ultramicropores  $0.1 - 5 \mu\text{m}$  and cryptopores as  $<0.1 \mu\text{m}$ . Generally, macropores only contain water if the soil is saturated and found between aggregates, caused by root penetration and movement of biota within the soil. Mesopores typically hold water under suction (capillary water, see section 2.4.1) and are critical sources of water for plants. Micropores contain water that is only extractable by plants (but otherwise immobile) and the only movement of solutes into and out of the pore are by diffusion. Ultramicropores provide habitats for microorganisms, however

anything contained within cryptopores is protected from microorganisms due to their size.



*Figure 2: Simplified schematic of a soil aggregate, where sand silt and clay particles are bound together by organic matter. Pore spaces are created within the aggregate which may be filled with air, water or a combination of the two.*

Aggregate stability affects a number of key soil parameters including shear strength and structural stability carbon stabilization, soil porosity, water infiltration, aeration, compactibility, water retention, hydraulic conductivity, resistance to erosion by water and overland flow (An *et al.*, 2010). As such aggregate stability is widely recognised as a key indicator of soil as it determines the productivity and resistance to degradation (An *et al.*, 2010; Barthes & Roose, 2002). It is closely related to organic matter quantity, water content, cropping history (Beare *et al.*, 1994), clay particles, humic substances, oxides of iron and aluminium, free  $\text{CaCO}_3$ , silica and polyvalent cations (Almajmaie *et al.*, 2017; Blackburn & Pierson, 1994; Cruse & Larson, 1977; Herrick *et al.*, 2001; Karlen & Stott, 1994; Nearing & Bradford, 1985; Pierson *et al.*, 1994; Tisdall, 1996; Wander *et al.*, 1994). In general, good structure for plant growth relies on the presence of aggregates 1-10 mm in diameter that remain stable when wetted (Tisdall & Oades, 1982). Aggregates that are not water stable are liable to breakdown by runoff and rainfall and release individual soil particles that cause the surface of the soil to seal and crust (Fattet *et al.*, 2011; Legout *et al.*, 2005; Loch & Foley, 1994; Martinez-Mena *et al.*, 1999; Ramos *et al.*, 2003).

Six *et al.* (2004) provide a thorough review of aggregate formation, and readers are directed to their publication for an in-depth review of the plethora of processes important for this process. Four substances are stressed to be critical in the formation of peds (aggregates); organic binding agents (microbial gum, an organic polysaccharide in the form of ropes and nets), organic matter, iron and aluminium oxides, and clay (Abiven *et al.*, 2009; Arias *et al.*, 1996; Edwards & Bremner, 1967; Mortland, 1970; Tisdall & Oades, 1982). Microaggregates are assumed to be stabilised by persisting binding agents (humic substances), whereas macro aggregates by transient or temporary organic materials (Six *et al.*, 2004).

Organic binding agents can be split down into transient (polysaccharides), temporary (physical binding by roots and fungal hyphae) and persistent (resistant aromatic components, polyvalent metal cations) (Tisdall & Oades, 1982). Polysaccharides originate from microbial cells and plant roots and contain both hydroxyl and carboxyl groups that bind through Van der Waal bonds and H<sup>+</sup> bonds between clay surfaces and micro-aggregates in order to form larger aggregates. The effect of organic matter is not to hold primary materials together; rather it modifies the forces by which particles are attracted to each other (Chesworth, 2008). Tisdall & Oades (1982) considered microbial gum and organic matter separately and found that the relationship between organic matter and soil aggregation is only marginal, and it is the microbial gum within organic matter that means its effect on aggregation is significant.

Most soil scientists acknowledge that soil organic matter conservation has a positive effect on the soil properties (Chirinda *et al.*, 2010; Hargreaves *et al.*, 2008; Papini *et al.*, 2011), where aggregate stability is strongly linked to organic matter (Arthur *et al.*, 2011; Le Bissonnais & Arrouyays, 1997; Leroy *et al.*, 2008; Six *et al.*, 2004), especially when the clay content of a soil is low (Hartmann & De Boodt, 1974). Tejade *et al.* (2006) found that organic matter acted as a cementing factor necessary for flocculating soil particles and forming stable aggregates. Soil organic carbon (SOC) input in the soil by roots corresponds to temporary binding agents which bind micro-aggregates into macro-aggregates, and SOC and root length density (roots equivalent <0.5 mm dia) were the variables best explaining

variations in aggregate stability (Fattet *et al.*, 2011; Gale *et al.*, 2000; Wander & Yang, 2000).

Grieve (1980), however, found that the decline in aggregate stability is not always proportional to reduction in organic matter. The effects of organic matter on aggregate stability are two fold as it can act as both aggregating and disaggregating depending on its composition (Mbagwu & Bazzoffi, 1996). On one hand organic matter reduces aggregate stability as it allows water to penetrate the soil more quickly due to improved soil structure and pore size, which encouraging erosion by slaking. On the other hand, it can reduce slaking as it reduces infiltration rate and causes runoff (Wallis & Horne, 1992) due to increased water repellency and cohesion once the organic matter has been dried, and improves long-term stability when erosional processes such as rainfall and runoff are dominant (Chenu *et al.*, 2000; Sullivan 1990). To resolve this issue of simultaneous increase and decrease in soil erodibility from the addition of organic matter due to changing speed of movement into and through the soil, cohesion must be increased, i.e. improve the shear strength. As cohesion increases, the properties of individual particles become less important and removal of particles is resisted by the shear strength of a cohesive soil fabric (Bryan, 2000).

There is general agreement that Fe and Al oxides are proficient in the stabilisation of aggregates (De Ploey & Poesen, 1981) as they interact with organic matter in macro-aggregate stability through their flocculation capacity. Harris *et al.* (1966) suggests that the stability of aggregates is based on cementation of finer soil particles by CaO, CaCO<sub>3</sub> and iron & aluminium oxides. Oades (1990) observed that in soils with >10% Fe and Al oxides, the contribution of organic matter to aggregate stability is diminished. Arias *et al.* (1996) found that iron oxides are a major flocculating and binding agent in the formation of micro-aggregates and appears to be the main inorganic binding agent of aggregates >200 µm. This is due either to (1) electrostatic binding between positively charged oxides and negatively charged clay minerals, neutralizing the surface charge and allowing colloids to form, (2) the formation of a bridge between particles by oxide coating or (3) organic materials being bound and adsorbed by oxides on mineral surfaces

(Whalen & Sampedro, 2010). Oxisols are very stable due to the presence of iron oxide, and this bonding prevalent in soils with less organic matter.

Clay particles tend to flocculate in an orderly fashion and have a large surface area producing surface tension of the curved menisci (Chesworth, 2008), and when dried with organic molecules the proximity means that hydroxyls of the polymers form hydrogen bonds with exposed oxygen atoms on the clay surfaces, and this leads to aggregate formation. The binding effect of clay particles to organic molecules is discussed by Zhang & Horn (2001) and possible precipitation as gels on clay surfaces is discussed by Amezketa (1999).

The complex relationship between the factors affecting aggregate stability (AS) are reviewed in detail by Amezketa (1999), Emerson & Greenland (1990), Harris *et al.*, (1966), Lynch & Bragg (1985), Mbagwu & Bazzoffi (1998), Oadies (1984), and Saygin *et al.*, (2012). Briefly, the factors affecting AS are grouped into two categories; firstly intrinsic (invariant) primary characteristics or external factors (climate, biological factors, agricultural management) and secondly dynamic internal factors (electrolyte concentration, types of exchangeable cations, exchangeable sodium percent, clay mineralogy, contents of CaCO<sub>3</sub>, organic matter, Fe and Al oxides). As they are critical in the formation of aggregates, in general soils with higher amounts of iron oxide, organic matter, calcium ions in association with clay, and microorganisms have a greater aggregate stability (USDA, 1996). Saygin *et al.* (2012) concluded that three main soil properties play a major role in aggregate stability, these are: organic matter (Chenu, 1989; Emerson, 1967; Haynes & Swift, 1990; Kazman *et al.*, 1983), the presence of iron and aluminium oxides and oxyhydrides (Le Bissonnais & Singer 1993; Römken *et al.*, 1977) and exchangeable sodium percent (in sodic soils). An assessment of how to quantify soil erodibility and soil aggregate stability can be found in section 2.3.4

## **2.3 Geotechnics of soil**

The previous section highlighted the important chemical and biological processes in soils, which are important for the soil ecosystem and its function as a producer, its job in the carbon/nitrogen cycles etc. The following section looks at soil in a geotechnical aspect, where soils are characterised based on their physical structure and mechanical characteristics, which are important in civil engineering.

### **2.3.1 Soil structure**

A soil's structure influences its ability to perform critical ecosystem functions such as the cycling of nutrients, water and carbon. Structure is the arrangement of primary particles into aggregates, which are separated by planes of weakness. The structure enables a dynamic relationship between solid, water and air where the discrete nature of aggregates results in the creation of pores (voids) that are larger than possible between the primary particles. The soil structure affects root development and penetration, movement and retention of water in the soil (permeability, infiltration and percolation rates), soil erodibility and shear strength of a soil (Gerrard, 2014). The term structure refers to the way in which individual particles that comprise soil are bound together and can be described in reference to the size, shape and arrangement of the aggregates, in reference to the voids or a combination of the two. The soil structure or matrix can be determined as incoherent or coherent (Paton *et al.*, 1995), where incoherent soils behave as if they were single grained and coherent soils have a stable relationship between particle and voids. Coherent soils are further classified by their predominant grain size, closeness of packing and degree of inheritance (of characteristics from the parent material). Soil can be described by its consistency when manipulated, where laboratory tests (Atterberg Limits) are used to classify the soil as brittle, plastic, friable, compact, loose, soapy, firm, sticky, tenacious or thixotropic. These properties are based on the physical make up of a soil and the water content.

In the field, the description of soils is based on the shape, arrangement, and size of peds that separate them into classes (spheroidal, plate-like, block-like and prism-like). There is a large volume of literature on the intricate details of soil structure, and a number of comprehensive reviews have been produced, with far more information than can be included in the following thesis (Harris *et al.*, 1966, Horn

*et al.*, 1994, Kay, 1998). The most important elements of a soil's development and related processes detailed above enable subsequent discussions on soil structure and the specific relationship between organic matter and water. The classification of soils, which can be divided into form, stability, resilience and vulnerability, is dependent on the properties of the soil (Lal *et al.*, 1997). The geometric characteristics are based on the organisation of the solid components within the soil matrix, a heterogeneous arrangement of void and solid space. The spatial arrangement of particles and interstitial spaces forms a structure that spans 9 scales (nm-m).

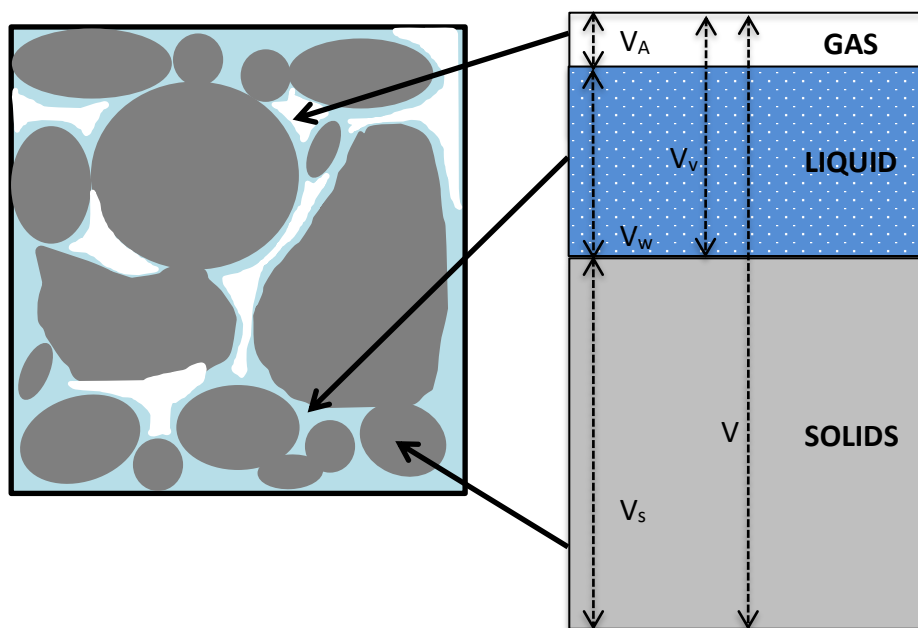


Figure 3: A simplified diagram of a partially saturated volume of soil and the associated three-phase soil diagram where  $V_A$  = volume of air (gas),  $V_w$  = volume of water (liquid),  $V_v$  = volume of voids,  $V_s$  = volume of solids (soil) and  $V$  = total volume.

The relative proportions of the three phases - solids (mineral matter and organic matter), liquid and gas - are dynamic, where soil water and air are more readily changeable, and the solid phase (mineral and organic matter) is less readily changed in short periods of time. These are mostly determined by their development environment, and an example of this ratio is presented in Figure 3. Where structure refers to the arrangement of particles within a soil, texture refers to the relative proportions of sand silt and clay in a soil, which includes gravel and stones. Mineral particles of a soil that are <2 mm are split into three fractions of material which determine their classification; sand, silt and clay. Sands are the

largest fraction (quartz grains) at 0.06-2 mm in diameter; silt the interim (0.002 - 0.06 mm), clay the smallest fraction  $<2 \mu\text{m}$  (0.002 mm) and any particles  $>2 \text{ mm}$  are stones or gravel. Sand and silt particles are created as a function of physical weathering and are chemically similar or unchanged from their parent rock (typically silicate minerals). Clay particles, conversely, have undergone chemical weathering and are therefore different physically and chemically in composition to the parent material, and are smaller than silt or sand particles. Chemical weathering, as described above, disintegrates minerals into their constituents, after which they are able to crystallise to form a variety of secondary clay minerals. Interlocking silica sheets and sheets of aluminium oxide form the structure of clay minerals, with the ratio of silica to aluminium being the dividing factor when classifying them (common clays are kaolinite, illite and montmorillonite).

It is important to note that although fractions of clay are denoted by their size, the physical breakdown of rock can produce any size particle so that clay size particles may in fact be quartz (Rautereau *et al.*, 2017). Clays and clay size particles hold water because they are more compactible, have a high surface to volume ratio, and they reduce pore size. Readers are directed to Mitchell & Soga (2005) and Powrie (2004) for a detailed description of a soil's mineralogy, structural and compositional characteristics and the formation of different clay species. Soils are split into types or classes based on the ratio of sand, silt and clay as shown below in Figure 4. There are a variety of laboratory and manual tests that can be carried out to determine classification; these will be discussed in Chapter 4. There are various different global soil classification systems such as the North American Unified Soil Classification System (USCS), but in the UK the BS ISO 11277:2009 is used as a standard (and shall be in the following thesis). An in-depth discussion of the many different soil types and intricacies of their formation and make up is not needed here, readers are directed to Avery (1980) for a complete soil system summary, which describes the characteristics of different soil types/groups and sub groups.

Particle size distribution (PDS) is a necessary index for soils, as it is crucial to make this characterisation to determine which fraction may control the engineering properties; texture is an important characteristic for critical soil parameters such

as water holding capacity and hydraulic conductivity due to the effects of texture on the voids in the soil matrix (Gupta & Larson, 1979; Van Genuchten, 1980), where soils with a high sand content have a high hydraulic conductivity and low water retention at 'field capacity' and the inverse relationship is apparent for soils with a high clay content (Rawls *et al.*, 1982, Saxton & Rawls, 2006). This is as a result of a higher surface area in clayey soils due to the small particles, which provides the soil with a high number of small pores that retain water. Conversely sandy soils have a lower surface area and fewer, larger pores. The proportion of each of these fractions determines the classification of soil according to the textural triangle (Figure 4). Although there are large areas that, for example determine a soil as clay, these soils are very rarely only clay and will contain silt-sized particles exhibiting the properties of clay.

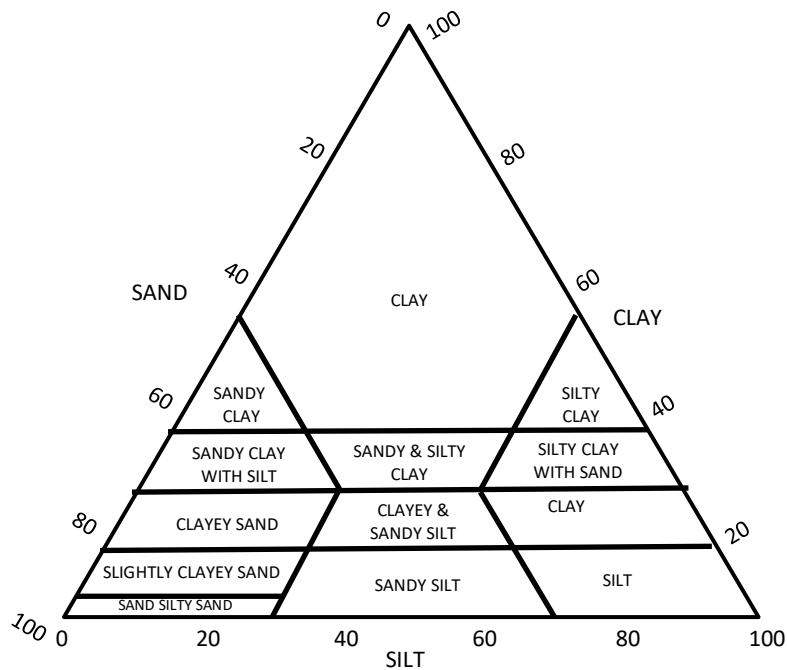


Figure 4: Triangular classification chart of soil based on texture (Head & Epps, 1980)

### 2.3.2 Definitions of soil structure

The terms associated with the structure of soil are variable, with no standalone or universal definitions that are applied to the literature despite common standards in place for their measurement (Koolen, 1987). Table 1 provides a summary of the discrepancy between common terms used to describe soils such as bulk density. This term is routinely used but often refers to the mass and volume of soil in one of two states, wet (field moist) or dry (oven dried). The term bulk refers to the

collective mass of an object, and density is the mass divided by volume, therefore the bulk density of a soil can simultaneously refer to the dry or wet mass, e.g. bulk density is described by Leege & Thompson (1997) as the volume occupied by soil in both wet and dry states depending on the application. Furthermore, issues arise in converting between dry density and wet bulk density as the dry density of a wet sample (i.e. the oven dried mass divided by the volume of the wet soil) is not equal to the wet bulk density of the same sample when dry, due to shrinkage and one cannot simply convert from one to the other. As shown in Table 1 below, the terms 'bulk' and 'density' when used together mean 'total mass over total volume'.

<b>Term</b>	<b>Definition</b>
Bulk	Collective mass of any object
Density	The quantity of matter in a unit of bulk (mass/volume)
<b>Term &amp; Definition given to describe BD</b>	
<b>Source</b>	
Bulk Density	
<i>Referring BD as total wet mass/volume</i>	
Ratio between total weight (mass) and total volume of soil	ISSMFE (1981)
Mass per unit volume of the soil including any water it contains	BS1377 (Part 2, 1990)
The weight (mass) of a material (including solid particles and any contained water) per unit volume including voids	DSIR, 1952 p 524
Total weight (mass) including contained water divided by the volume	Capper & Cassie (1949)
Mass of bulk soil, including solid particles, water and air, contained in a unit volume	Head (1980, Vol1)
Mass per unit volume which includes mass of air or water in the voids	Chudley (2006)
Total wet bulk over volume	Schaub-Szabo & Leonard (1999)
Bulk density	
<i>Referring to BD as total dry mass/volume – soil is measured in wet state and then</i>	

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*dried to determine moisture content*

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The mass of dry soil per unit bulk volume	Soil Science Society of America (1997)
Ratio of the mass of dry solids to the bulk volume of the substrate	Blake & Hartge (1986)
Dry mass per unit volume (in a moist state)	Wallach (2008)
The mass of soil solids per unit volume	Van den Akker & Soane (2005)
Mass of unit volume of dry soil	Buckman and Brady (1960)
Weight of solids divided by total volume	Brewer (1964)
Unit dry weight	Haug (2018)
Volume weight	Levanon <i>et al.</i> (1988)
Apparent bulk density	Wilson (1983)
Bulk density/specific volume	Foth 1991)

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*Table 1: Summary of varying definitions of the terms bulk density and dry density*

The definitions deemed “correct” and therefore used in the remainder of this thesis are:

**BULK DENSITY (BD)** = total mass of bulk divided by total volume of bulk

**DRY DENSITY (D<sub>d</sub>)** = mass of dry solids divided by total volume of a wet sample.

Typical bulk densities for soils in the UK are between 0.2 g/cm<sup>3</sup> for highly organic soils and 1.95 g/cm<sup>3</sup> for very compacted soil (Emmett *et al.*, 2010). For organic matter (compost) dry density ranges from 0.1 to 0.4 g/cm<sup>3</sup> and bulk density ranges from 0.5 to 0.9 g/cm<sup>3</sup> (Agnes & Leonard, 2003). The differences in bulk density between soil and organic matter are due to the particle densities of each material, where soil mineral matter is typically cited as 2.65 g/cm<sup>3</sup> and this is the particle density of the main component, quartz. There are a number of other important terms used when describing the structure of a soil that are pertinent to this thesis and require outlining as a point of reference for further discussion in the chapter, relating to the internal properties of the soil (Table 2).

Term (and synonyms)	Definition
Porosity	A measure of the volume of voids, $V_v$ (which is occupied by air and/or water) within a volume of soil, $V$ .
Void ratio	Ratio between volume of voids, $V_v$ , to the volume of soil, $V$ . ( $V_v/V_s$ ) <i>Can be calculated using the dry bulk density (BD) and particle density (Pd)</i> $100\% - (BD/Pd * 100) = \% \text{ pore space}$ <i>e.g. <math>1.56/2.65 * 100 = 60\% \text{ solid matter} = 40\% \text{ pore space}</math></i> <i>Can be calculated by the difference between the mass at saturation of a sample and the oven dried mass, as mass = volume for water due to density of <math>1\text{g/cm}^3</math> e.g. for a 400 g sample of soil at saturation, with the mass of 200 g being water, the porosity is 50%.</i>
Particle density (Specific density Absolute density True density)	Mass of a particle per unit volume (Flint & Flint, 2002)
Specific gravity	Specific gravity is a ratio of the mass of a material to the mass of an equal volume of water at 4 °C. $G_s = (P_s/P_w)$ where $P_s$ = density of solid and $P_w$ = density of water <i>Because specific gravity is a ratio, it is a unit-less quantity. For example, the specific gravity of water at 4 °C is 1.0 while its density is <math>1.0\text{ g/cm}^3</math>.</i>
Saturated density	Bulk density at full saturation
Submerged density	When a soil mass is submerged, buoyancy reduces the mass. Upward force is equal to the volume x density of water
Specific volume	The volume containing unit mass of solid material (i.e. the volume of 1 kg of soil)

*Table 2: Summary of applicable geotechnical definitions on soil*

The bulk density and water content of compost can be readily measured (mass per volume technique, Agnew & Leonard, 2003) but particle density is more difficult as it needs an air volume measurement (Agnew *et al.*, 2003). Particle densities of compost (organic matter) of a biosolids origin are between 1.3 and 1.4  $\text{g/cm}^3$  (Das & Keener, 1997), Agnew *et al.* (2003) suggest a typical range of 1.5 to 1.8  $\text{g/cm}^3$ , and values of up to 2.31  $\text{g/cm}^3$  have been reported for dairy manure (Weindorf & Wittle, 2016). The bulk density values are generally much higher for soils due to structural differences between the materials in addition to their

particle differences, soil minerals have no voids and regular packing, whereas organic matter has a vast number of voids and air space contained within its structure (Villar *et al.*, 1993).

### **2.3.3 Factors affecting soil structure**

The distribution of the solid phase is based on the ratio of sand silt and clay (soil texture) and presence of organic matter often governs the relationship between solid matter and air or water phases. Moisture content is recognised as being one of the most important factors in soil structure (Soane & Kershaw, 1987). However, the relationship between solid and liquid is co-dependant, i.e. the structural properties of a soil both determine and are determined by the water content of the soil. The amount of water and organic matter in a soil determines the extent to which a soil becomes compacted under pressure, which in turn determines the bulk density, pore space/porosity and void ratio of the soil. The bulk density and porosity of a soil then subsequently determine the infiltration rate (movement of water into the soil surface, under unsaturated conditions) and permeability (rate of movement through a soil, also called hydraulic conductivity) and therefore affect the water content of the soil. This section aims to explore these co-dependant relationships.

Soil compactibility (i.e. the inverse of a soil's ability to resist compaction) is directly related to water content, texture, and organic matter content during the application of force (Mosaddeghi *et al.*, 2000). Compression in soils causes a reduction in total pore space and void ratio, by reducing macro-pores and increasing micro-pores (Richard *et al.*, 2001), and thus changes hydraulic conductivity as compaction reduces total pore space and macro-pore space while increasing micro-pore space (Foth, 1991). There isn't a standalone or universal definition of compactibility, rather a number of ways of measuring it (Koolen, 1987), which includes uniaxial compression or tests measuring bulk density at a given level of impact loading (typically Proctor, 1933). Compression testing uses a range of pressure (up to 1000 kPa) to calculate a compression index. Proctor (1933) showed that soil compaction under a given effort is changeable dependant on the water content of a soil. It is clear from Figure 5 that the water content at compaction controls the density of a soil, resulting from the change in suction (the

energy required to extract a unit volume of water from soil) and physical properties that are changeable with water content.

Soils have large suctions when they are relatively dry (section 2.4.1) meaning that large aggregates are difficult to deform and the compactibility of the soil is low. Soil increases in susceptibility to compaction with increasing water content to the point of maximum density (optimum water content), after which the water within the voids prevents compaction, and additional water reduces the bulk density due to an increased proportion of lower density material in relation to the soil mass (i.e. water at 1 g/cm<sup>3</sup> instead of soil minerals of 2.65 g/cm<sup>3</sup>). The optimum water content for maximum (dry) density is reached at a point where aggregates are packed most efficiently (Agnew & Leonard, 2003; Ahn *et al.*, 2008; Madejon *et al.*, 2002; Malinska & Richard, 2006; Mitchell & Soga, 2005; Tarantino & Tombolato, 2005).

Soils compacted dry of optimum water content have a flocculated structure i.e. random soil particle orientation, and when wetted will take up much more water and swell to a greater extent. They will exhibit better multidirectional permeability and be less compressible than wet compacted soils (although unsaturated soils may collapse or compress upon wetting). Soils compacted wet of optimum have a dispersed structure and are orientated perpendicular to the application of stress due to intra-particle lubrication as a result of the film of water surrounding each. These soils shrink more when dried due to better packing of particles (as charge deficiencies are satisfied, resulting in particle orientation) and are only permeable along particle orientation. Although bulk density increases with moisture content when the soil is compacted dry of optimum, the particle size does not have a significant effect on bulk density (Druilhe *et al.*, 2008). With more small particle sizes (<20 mm), the effect of higher bulk density with high water content is greater (Huet *et al.*, 2012). Second to water content, organic matter content is one of the largest influences on the compactibility and bulk density of a soil (although of course the amount of organic matter also influences how much water is in the soil before it undergoes compaction).

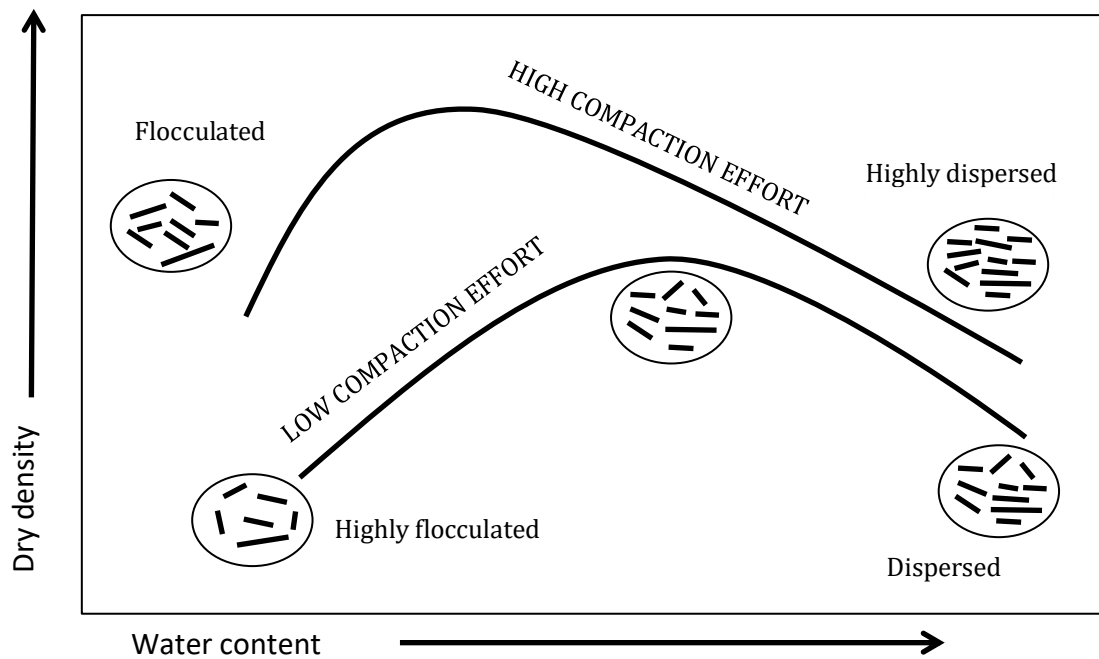


Figure 5: The effect of compaction on soil structure (adapted from Lamb, 1958)

Organic matter affects the compactibility, bulk density and water content of a soil in the following ways, according to Soane (1990). Firstly, it provides binding forces within aggregates or between particles, where long chain molecules bind soil mineral particles together, helping aggregation and resisting compactive efforts. Secondly, it gives the soil elasticity, which is often determined by the relaxation ratio i.e. the bulk density of a test material under specified stress in relation to the bulk density after stress has been removed. Thirdly, organic matter has a dilution effect, as the density of organic matter is significantly lower than soil matter; bulk density of organic matter is between 0.5 and 0.9 g/cm<sup>3</sup> (Gerrard, 2014) with a particle density ranging from 1.2 to 2.3 g/cm<sup>3</sup> (Agnew *et al.*, 2003; Das & Keener, 1997; Weindorf & Wittle, 2016). Therefore, combining soil matter with organic matter will reduce the bulk density regardless of compaction. Fourthly, organic matter changes the electrical charge within a soil where some organic liquids increase the hydraulic conductivity of clays (Brown & Thomas, 1987), which allows water to move through the soil at a greater rate. Penultimately, organic coating increases friction between particles (Beekman, 1987), making the sample less susceptible to compaction, however this is only applicable at low compost rate application (20-30% amendment) and beyond this threshold there is no further improvement or a decline in shear strength (Mitchel

& Soga, 2005; Puppala *et al.*, 2007). Lastly, and perhaps most importantly, organic matter increases the amount of water that a soil can hold, increasing net water availability near saturation (Sullivan & Miller, 2001).

There are differing arguments on the effect of organic matter on geotechnical properties such as shear strength, volume change and compactibility. Should the organic content fall within a range of 6 – 20% then the soil properties are similar to a mineral soil, but beyond 20% the organic content of the soil governs the entire properties of the soil (Edil, 1997). Mitchel & Soga (2005) argue that organic matter in soil decreases the shear strength and increases compressibility characteristics, reduces volume changes and reduces shrinkage. Similarly Adejumo (2012) found that organic matter addition increases plasticity and compressibility, and reduces shear strength. In general the influence of organic matter on compressibility is based on the initial void ratio, i.e. the greater the porosity, the greater the compressibility. Zaffar *et al.* (2017) found that waste water biochar amendment reduced the plastic limit, tensile strength and cohesion, but simultaneously improves the hydraulic conductivity, bulk density, water retention capacity, aggregation and aggregate stability, total porosity and pore size distribution (Aggelides & Londra, 2000). Puppala *et al.* (2007) found that the addition of organic matter increased the optimum moisture content (as more water was required to compact the sample to the maximum density), and increased the free swell of samples. The shear strength increased with an addition of organic matter, although beyond 40% application rate, the shear strength decreased. Similarly, Zong *et al.* (2014) found that the pH, swelling behaviours, shear strength parameters, compaction and Atterberg limits are reported to be improved by the addition of organic matter, in the form of biochar. It appears therefore that the effect of organic matter in soil is two-fold; organic matter increases the volume of water likely to be held in a soil sample, while providing structural stability that reduces the compactibility of the soil.

From a geotechnical perspective, the addition of organic matter has beneficial effects for the water holding capacity (reduced bulk density, increased macroporosity, improved hydraulic conductivity, high infiltration rate, aggregate stability, volume change, primary compactibility etc), but is detrimental to the

shear strength, secondary compactibility, cohesiveness, and vulnerability to particular erosional processes. Therefore there is a trade-off between the positive and “negative” impacts of organic matter inclusion. The based on the application for which soil is being used, where in civil mechanics organic matter is detrimental to applications such as road manufacture, but in softer soil engineering the addition of organic matter may alleviate shortcomings of the soil.

#### **2.3.4 Soil erosion**

As discussed in the introductory chapter, soil erosion is one of the main causes of land deterioration, with 1094 million hectares of land affected globally (Bridges & Oldeman, 1999; Lal, 2003), resulting from aggregate breakdown and detachment (Le Bissonnais, 1996). On a macro-scale, the climatic factors controlling the severity of erosion are rainfall, topography and vegetative cover. However, when these remain constant there is a degree of variability in soil loss, a factor recognised by Bennett (1926), leading to Middleton (1930) coining the term soil erosivity and Cook (1936) soil erodibility.

Erodibility is a term used to describe the inverse ability of a soil to resist the detachment and transportation of particles by erosional forces such as rainfall impact and runoff water (Coote *et al.*, 1988; Saygin *et al.*, 2012). It cannot be measured directly but is inferred, by the combination of a number of given conditions, from simulated rainfall and runoff (Larionov *et al.*, 2017). There is no single, simple and measurable soil property that can wholly represent the response of a soil to erosion factors (Lal, 1990), nor is there a standardised procedure or instrumentation with which is it measured (Almajmaie *et al.*, 2017; Bryan 1968). In fact the use of the term ‘erodibility’ in difference contexts by different research has meant that the definition is exceptionally vague and can be used to summarise all erosional processes on a soil, or in some cases is restricted to particular processes (Bryan *et al.*, 1989). For example, Imerson & Vis (1984) used the term to describe the susceptibility of an aggregate to raindrop impact, however Bryan (1974) used the term to describe breakdown in small flumes under a rainfall simulator.

Initially soil erodibility literature revolved around three assumptions: Firstly, that erodibility can be wholly defined and is valid for all breakdown mechanisms. Secondly, that it can be defined using a small number of physical soil processes and lastly, that erodibility is not affected by short term changes e.g. moisture content (Bryan *et al.*, 1989). However the processes that are now known to largely govern erodibility are dynamic and have changing cycles of varying magnitude (Bryan, 2000; Wischmeier & Mannering, 1969). The multitude of factors contributing to soil loss include the particle size distribution (sand, silt & clay and organic matter), soil pH, soil structure (Wischmeier & Smith, 1978), soil density, gradient of the soil slope, air-filled pore space, aggregation (Cruse & Larson, 1977), parent material, water temperature (Mutchler & Carter, 1983) and soil moisture potential (Bruce-Okine & Lal, 1975).

There are four categories of soil erosion; slaking, breakdown by differential swelling, mechanical breakdown by rainfall and physio-chemical breakdown. When wetted there are a number of forces that breakdown soil; the air within aggregates and in soil pores is rapidly compressed, soil materials undergo differential swelling, and the energy of rain splash and runoff produce shear forces. These forces are most apparent when the soil is dry and produce maximum aggregate rupture by slaking and swelling. However, as the soil begins to wet and higher degrees of saturation are reached, the initial stresses are reduced and rainfall impact and runoff forces become the predominant mechanisms of erosion. This is as a result of the difference in bonding mechanisms between aggregates (strong long-term slowly developing) and the bonds that form coherent, shear resistant soils (short-term weaker bonds that form quickly and dominate fabric coherence) (Bryan, 2000).

Slaking is the breakdown of aggregates when dry soil is rapidly immersed in water, which causes air trapped in pore space and inter-aggregate air to compress and then expand (Truman *et al.*, 1990). Slaking is affected by the rate of water movement into the soil (affected by soil porosity, pore connectivity, antecedent moisture and rate of wetting, Loch, 1994). Soils are eroded by swelling as soils containing high amounts of clay are liable to swell and slake upon wetting due to differential swelling rates as water pushes clay particles apart (Le Bissonnais,

1996; Reichert *et al.*, 2009). Rainfall is a predominant soil erosion mechanism, where the net loss of soil downslope consists of three parts: impact of the raindrop, soil particle detachment and then displacement of the soil particle (Terry *et al.*, 1993). The rate of loss is determined by the soil erodibility and rainfall erosivity i.e. the ability of rain to detach and transport soil (Epema & Riezebos, 1983), which is dependent on the kinetic energy of the rain (Morgan, 2009). Lastly mechanical breakdown, which includes rainfall impact, is erosion from runoff due to the shear forces of rainfall and water running over the surface of the soil. These forces cause aggregates to shatter into fine soil particles, the degree to which is proportional to the raindrop size and energy (Furbish *et al.*, 2007). These fine particles block pores throats as they are moved downward by capillary flow (Legout *et al.*, 2005) and significantly reduces infiltration causing surface ponding and further slaking (Gholami *et al.*, 2013).

### **2.3.5 Soil erodibility indices**

To understand the risk of erosion, the identification of erosion indicators is necessary as the direct measurement erosion in the field is expensive and time-consuming (Barthes & Roose, 2002). There is a wealth of research that has suggested a number of indicators for erodibility, as it cannot be directly measured using a single index. The erodibility of a soil can be inferred directly using aggregate stability or indirectly from easily measurable parameters such as organic matter content, shear strength and bulk density. There are numerous methodologies for the determination of erodibility based on aggregate stability (Kemper & Rosenay, 1986; Le Bissonais, 1996; Marquez *et al.*, 2004; Yoder, 1936) and readers are directed to Lal (1988) for a thorough list of soil erodibility indexes based on aggregate stability parameters that can be measured in the laboratory. More recently Nimmo & Perkins (2002) discuss the variations on widely used standardised methods. The following section briefly outlines aggregate stability as an index for soil erodibility and the use relationship of indirect proxies (organic matter and shear strength) with soil erodibility.

Aggregate stability and the related breakdown processes are very closely linked to soil erodibility (Andre & Anderson, 1961; Barthes & Roose, 2002; Hairsine & Hook, 1994; Le Bissonais *et al.*, 2007), and although the use of aggregation as an

erodibility index is very complex, it is a well-used parameter. Aggregate stability can be inferred by evaluating the percentage water stable aggregates, the degree of soil detachment from rainfall/runoff or linked with the organic matter content, clay content and shear strength of a soil (Amezketta *et al.*, 1996; Beare & Bruce, 1993; Bruce-Okine & Lal, 1975; Le Bissonnais, 1990; Kemper & Koch, 1966; Kemper and Rosenau, 1986; Lock & Foley, 1994; Loch & Smith, 1986; Pierson & Mulla, 1989; Ramos *et al.*, 2003; Young, 1984). However these indicators of aggregate stability are often considered independently, meaning that soil aggregates shown to be water stable by testing methods, may not be so when subjected to rainfall testing methods and vice versa, showing that a single index is not appropriate to determine aggregate stability as breakdown mechanisms work in different ways (Ramos *et al.*, 2003). Another caveat to using aggregate stability as an indicator for erosion through comparisons of water stable aggregates, runoff and rainfall simulations is that these methods are often conducted on rehandled or sieved samples, which may not be at all representative of field phenomena.

At present there is no standard choice for one test of aggregate stability over another, and the selection is based on the researcher or the type of erosion that is predominately important (An *et al.*, 2010). A number of publications have attempted to fine the 'best' method for testing aggregate stability but at present that is no single equation that could be utilised to related soil detachment of nine soils to the ratio identified (Al-Durrah & Bradford, 1982). Larionov *et al.*, (2017) suggest that rupture rate of inter-aggregate bonds (through various mechanisms) can be used for the determination of erodibility as it correlates well with easily determined soil parameters e.g. density, infiltration rate, bulk density and organic matter. The most common direct indicators of aggregate stability are the percentage of water stable aggregates (after slaking), or the degree of soil detachment from rainfall/runoff. Although these methods are considered independently, there is often a direct relationship between them. Some work also infers the aggregate stability from readily measurable parameters that are known to influence aggregate stability (organic matter, Fe, clay etc).

Each researcher suggests a particular method to best characterise aggregate stability. For example Amezketta *et al.* (1996) found that tests that involved slaking

were the closest correlated with rainfall simulations and suggest that either test suffices. However, Loch & Foley (1994) recommended that simulated rainfall is preferable because the results are relevant to soils in the field. Others discuss the merits of different sieving methods for water stable aggregates, for example Le Bissonais (1996) who compares the results of fast wetting, slow wetting and stirring after pre-wetting to cover all breakdown mechanisms. Barthes & Roose (2002) suggest fast wetting as the simplistic way to test aggregate stability.

The percentage of water stable aggregates (WSA%) remaining after a period of testing (e.g. aggregates tested by cracking (slow wetting), slaking (fast wetting) and mechanical breakdown) is one of the most efficient indicators of erodibility (Haynes & Swift, 1990, Le Bissonais, 1996; Luk, 1979). The test involves taking air-dried soil sieved to a particular fraction (typically 2 mm), and wetting it in various ways before sieving it for a given period of time, after which the proportion of aggregates remaining on the sieve is taken along, enabling the calculation of dispersion ratios (Lal & Elliot 1994). There are strong relationships between water stable aggregates and the detachment of soil from rainfall and runoff, where Luk (1979) found that for simulated runoff and raindrop impact, soil detachment was strongly negatively correlated with % of water stable aggregates >0.5 mm, and suggested that erodibility was better indicated by aggregate stability than the component parts of a soil (i.e. organic carbon, sand or clay content). In addition WSA% aggregates have a strong negative relationship with shear strength and gravimetric water content (Coote *et al*, 1988). However the results of sieving to determine WSA% are very dependent on the preparation and specific test conditions. For example Haynes & Swift (1990) compared Yoder's (1936) method of wetting against different periods of wetting and field moist vs air dried aggregates. They found that the duration of sieving was a key factor and found that prolonged sieving reduced the WSA% to a near constant value for all samples tested. They also concluded that unstable aggregates have low organic content, air-drying of aggregates before testing has variable effects and that WSA% can be inferred by soil properties such as clay ratio and particle size distribution, although direct testing is preferable.

Rainfall erosion occurs when an aggregate is destroyed or broken down by raindrops, should the detaching force of the raindrop overcome the intrinsic resisting force of the aggregate (Mbagwu & Bazzoffi, 1998; Kinnell, 2005). Aggregate stability can be inferred by the amount of soil lost as a result of rainfall impact. Once soil particles are detached they are moved by the processes of drop splash, raindrop-induced flow transport, or transport by flow without raindrop. Soils that are exposed to rainfall are susceptible to the formation of a seal, which reduces infiltration and increases subsequent erosion by runoff, but protects the underlying soil from further rainfall erosion (Le Bissonais & Arrouays, 1997; Kinnell, 2005; Legout *et al.*, 2005; Loch & Foley, 1994; Römken *et al.*, 1977; Moore & Singer, 1990). Degradation of the soil surface by rainfall has been identified as a key factor in the degree of erosion by runoff and flow (Léonard & Richard, 2004). Cruse & Larson (1977) and Al-Durrah & Bradford (1982), Nearing & Bradford (1985) and Luk *et al.*, (1989) have observed significant relationships between soil strength and splash detachment in the lab. In addition soil strength is the only proxy parameter that consistently correlates with rainfall detachment (Agassi & Bradford, 1999).

Varying developments on the raindrop techniques used by McCalla (1944), Low (1954), Bruce-Okine & Lal (1975), De Vleeschauwer *et al.* (1978), Mbagwu (1986) are used to test the vulnerability of soil to raindrop erosion, however they are not without their limitations as the majority cannot duplicate rainfall intensity and energy (Agassi & Bradford, 1999). There is a large range of drop size, drop heights and drop rates used for aggregate stability determination (An *et al.*, 2012, Norton, 1987). The power of the raindrop is critical in the breakdown of an aggregate on impact, where the most important factors affecting the total shear stress of a raindrop are; impact velocity, angle of impact, raindrop diameter, shape, surface tension, number and duration of impacts (Nearing *et al.*, 1986; Truman *et al.*, 1990). These conditions are difficult to replicate in a laboratory setting (using rainulators), therefore the kinetic energy (KE index) required to breakdown or disrupt an aggregate to a given degree has been used as an indication of the susceptibility of a soil to erosion by rainfall.

Rudimentary testing of aggregate stability by rainfall conducted by Cruse & Larson (1977) investigated the weight of detached particles through water drop impact as an estimate for aggregate stability. Similarly the water drop test procedure described by Low (1967) used large mass water drops to compensate for low fall height, as with increasing height the aim of drops is reduced. The research suggested that large drops at a rapid rate of delivery allow extreme rainfall conditions to be replicated. However if the aggregate fails to respond after 40-50 drops then subsequent drops will have no further impact effect and other mechanisms of breakdown will take over. Imeson & Vis (1984) used a 1 cm<sup>3</sup> piece of soil and dropped water by a pipette from a height of 10 cm. The number of water drops required to breakdown the soil structure was recorded (raises human error and bias for end point). Soils were kept field moist to avoid irreversible formation of stable aggregates by drying and the test specimens were passed through a 4.8 mm sieve, after which water drops were added until all aggregates passed a 2.8 mm sieve. Imeson & Vis (1984) suggest this as the most suitable method for evaluating highly erodible soils (20-30 impacts).

More recently Loch *et al.* (2001) used an oscillating rainfall simulator for rainfall experiments and found that higher aggregate stability was associated with lower bulk densities (as a function of land management where compacted soils were subjected to poor farming practises, destroying aggregate stability and macroporosity ratio). Many studies are limited as they only consider the single drop effects of rainfall (Ekwue & Seepersad, 2015), neglecting the soil wetting that occurs in continuous rainfall (Stuttart, 1984), which reduces soil shear penetration resistance (Cruse & Larson, 1977) and increases infiltration rates, aggregate breakdown and seal formation (Bryan & Poesen, 1989). Barthès *et al.*, (1999) compared the values of soil erodibility tested by rainfall simulation to values derived from wet sieving for WSA% and found that at the start of rainfall simulation, there was a close relationship between soil loss from runoff and WSA%.

There are two primary proxy indicators of aggregate stability, the organic matter content and the shear strength of the soil. These two parameters go hand in hand as organic matter influences the shear strength of a soil, however testing methods

are such that one either characterises one or the other. As discussed previously in section 2.2.2 soil organic matter plays a chief part in aggregate stability due to its influence on cohesion and wettability (Chenu *et al.*, 2000; Oades, 1984; Sullivan, 1990; Tisdall & Oades, 1982) and can be therefore be used as a proxy to determine aggregate stability. Haynes & Swift (1990) found that soil organic matter content and water content were the best indicators of aggregate stability, and similarly Wischmeier & Mannering (1969) consider organic matter to be the second most influential property affecting soil erodibility after soil texture and in general, the aggregate stability of soil is positively correlated with the organic carbon content and therefore it can be used as a predominant indicator of aggregate stability due to the protection provided against slaking (Le Bissonnais, 2006). Chenu *et al.*, (2000) showed that organic matter in association with clay minerals increased the hydrophobicity, assessed by measuring drop penetration times on 3-5 mm aggregates. Water repellency is a phenomenon where the wetting of a soil is delayed from being immediately absorbed (Scott, 2000). During extensive drying periods, the organic fraction becomes water repellent (Doerr *et al.*, 2000) causing inhibited infiltration (Imeson *et al.*, 1992) and increases overland flow (McGhie & Posner, 1980, Witter *et al.*, 1991). Soils that are water repellent are more susceptible to erosion by overland flow, caused by the reduction in infiltration capacity of the soil (Shakesby *et al.*, 2003, Scott & Van Wyk, 1990).

Lastly, the shear strength of a soil represents a simple but physically significant parameter that integrates physical, chemical and mineralogical soil properties into one readily measureable parameter, which can then be associated with erodibility (Agassi & Bradford, 1999). Soil erosion mechanics are strictly linked to indices of soil strength (Mouzai & Bouhadef, 2001; Nearing & Bradford, 1985). A major factor governing substrate mass movement is the shear strength of soil (Terzaghi, 1942) and there are numerous studies that have linked a soil's shear strength with the erodibility of a soil, by using aggregate stability as an erodibility indicator (Fattet *et al.* 2011; Frei *et al.* 2003). If a direct link could be firmly established between aggregate and soil shear strength through further research, it would allow a better understanding of the mechanisms involved (Frei *et al.*, 2003).

A full discussion of shear strength and stress characteristics pertinent to soils is discussed in the subsequent section (2.3.6), although a brief description is needed here in relation to soil erosion mechanics. Soil shear strength is one of the best predictors of critical shear stress, although few studies have explored the relationship between the two (Franti *et al.*, 1999, Torri *et al.*, 1987, Rauws & Govers, 1988). The critical shear stress is an important soil parameter that governs detachment of soil particles by runoff, giving the threshold at which soil aggregates will break down due to shearing forces. There are a number of factors upon which critical shear stress depends; firstly cohesion of the small aggregates which is dependent on the chemistry of the colloid, secondly the size, form and spatial organisation of aggregates and lastly the presence of roots and hyphae that directly impact structural stability and indirectly improve aggregation (Léonard & Richard, 2004).

The relationship between shear strength of soils and their erodibility has been well established. Cruse & Larson (1977) showed that detachment of soil by raindrops was negatively correlated to shear strength (for wet soils), measured by triaxial testing. Léonard & Richard (2004) used a shear vane device to measure shear strength of the soil immediately after a flow experiment measuring soil loss and found a direct correlation between erosion and shear strength. Similarly Nearing & West (1988) found a relationship between shear strength (from fall cone and torvane methods) and mean weight diameter of aggregates.

### **2.3.6 Soil shear strength and stress**

Stress (the intensity of force) in soil causes deformation in three ways, elastic deformation, change in volume due to water expulsion (consolidation), and slippage of soil particles relative to one another (shear failure). Soil strength can be expressed in various forms, such as compressive strength, shear strength, and tensile strength. In a soil subject to tillage, compressive strength is a measure of the soil surface's ability to resist penetration, shear strength is a measure of how well a soil resists varying directional forces before it fails and tensile strength is a measure of how much a soil can resist being pulled apart. The stress/strain relationship reaches a point at which the soil cannot longer deform through expansion or contraction and the bond between particles is broken. The term

shear strength is defined as the maximum shear resistance that a soil can offer under defined conditions of effective pressure and drainage against shear force (Head, 1980) as the result of resistance to movement at interparticle contacts due to particle interlocking, physical bonds formed across the contact areas (resulting from surface atoms sharing electrons at the interparticle contacts) and chemical bonds or cementation (Craig, 2004). At any given point on any plane in the soil mass, if the shear stress is equal to or greater than the shear strength of the soil, then failure will occur (Craig, 2004). It is not a fundamental property of soil and is related to the conditions of the soil; effective stress, drainage conditions, density of particles, and rate and direction of strain (change in unit length/deformation due to stress). The shearing behaviour of a saturated soil is related to the effective stress ( $\sigma'$ ), which is total stress ( $\sigma$ ) minus the pore water pressure ( $u_w$ ). The shear strength of a soil was originally expressed by Coulomb as a linear function of the normal stress at failure on the plane;

$$\tau_f = c + \sigma_f \tan \phi$$

*Equation 2: Coulomb failure criterion, where  $\tau_f$  is normal stress at failure  $c$  and  $\phi$  are the cohesion (intercept) and the angle of friction (angle of shearing resistance) respectively.*

However, equation 2 does not hold for saturated soil as under load, the saturated soil shares the normal stress between soil particles and the water and the resistance to shear therefore depends on the effective stress ( $\sigma'$ ). Shear stress in a soil can only be resisted by the soil matrix, i.e. the solid particles, therefore shear stress must be expressed as a function of effective normal stress, denoted with ' $\sigma'$ ' shown in Equation 3:

$$\tau_f = c' + \sigma'_f \tan \phi$$

*Equation 3: Critical shear stress as a function of effective cohesion and effective friction angle, where  $\tau_f$  is effective normal stress at failure  $c'$  and  $\phi'$  are the effective cohesion (intercept) and the angle of friction (angle of shearing resistance) respectively.*

By conducting a series of compression tests on an set of identical specimens of soil, the states of stress can be presented using Mohr circles which plot shear stress ( $\tau$ ) against effective normal stress ( $\sigma'$ ), acting across two planes within a body of soil. Here we are interested in the ability of saturated samples to resist shearing forces, as amendments aim to improve the ability of saturated soils to remain intact against erosional forces.

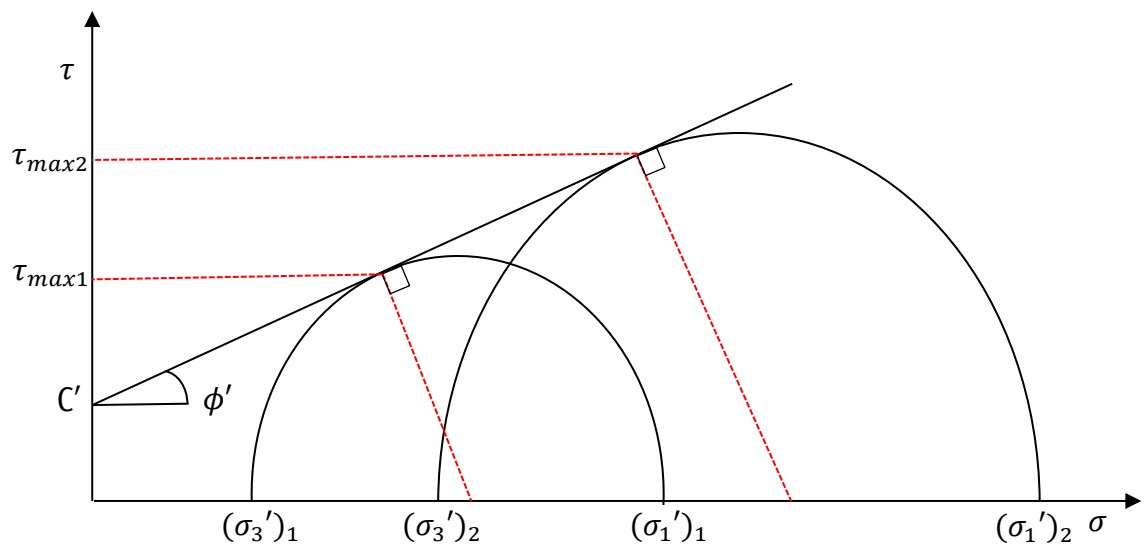


Figure 6: Mohr-Coulomb failure envelope where  $\tau$  is shear stress and  $\sigma'$  is effective stress.

By drawing Mohr circles, calculated from data produced typically in a triaxial cell, one can establish the critical shear stress (failure envelope) above which the sample experiences failure. Figure 6 provides an example of Mohr circles with a failure envelope (joining the maximum stress of each circle, each of which correspond to difference cell pressures during testing,) which plots the shear stress against the normal effective stress.  $c'$  (cohesion) is taken at the intersect of the failure envelope with the Y axis, and the angle of friction ( $\phi'$ ) is measured from the angle of the failure envelope line against the x-axis. Granular soils with little cohesion will have a steep failure envelope gradient that crosses the Y-axis near 0 with a high angle of friction, and crumble easily when dry. Conversely cohesive soils, typically fine grained or highly clayey have a low gradient failure envelope. and low angle of friction (Figure 7).

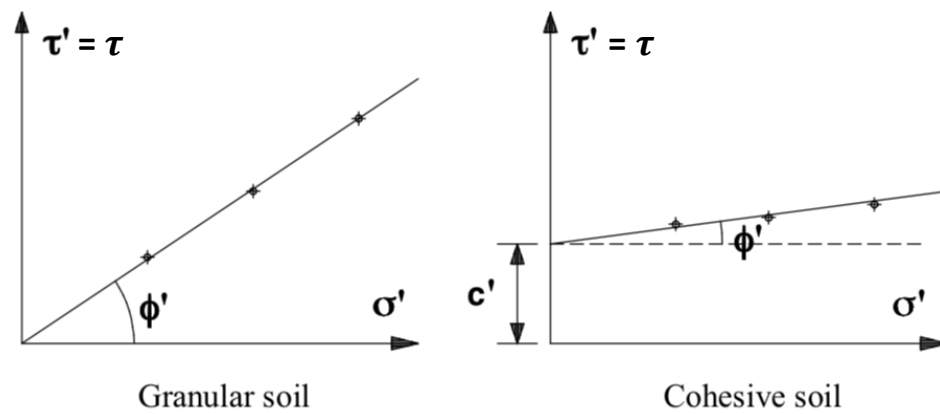


Figure 7: Examples of failure envelope gradients, cohesion and angle of friction values for granular and cohesive soils.

Acquiring the value for the shear strength of a soil is obligatory for many applications, particularly in civil engineering where the stability of slopes, embankments and foundations are critical, as well as with agricultural engineers and soil scientists. The shear strength of a soil is strongly related to the water content of the soil, and as such it can be inferred from the soil water retention curves (Vanapalli *et al.*, 1996). As discussed subsequently in section 2.4, in a saturated state the soil suction is either positive or zero, however unsaturated soils have a negative pore water pressure and experience matric suction (difference between pore air pressure,  $\mu_a$  and pore water pressure  $\mu_w$ ). Where saturated soils are dependent on one stress state variable (effective stress), unsaturated soils are dependent on two stress state variables; net normal stress ( $\sigma - \mu_a$ ) and matric suction ( $\mu_a - \mu_w$ ).

Few testing methods can be applied in the field due to the multidirectional nature of shearing forces in soil, therefore most measurements and quantification are taken from reformed or largely whole soil blocks in the laboratory. Fredlund & Vanapalli (2002) provide a good summary of the variety of guidelines and methods of measuring shear strength, however this thesis focuses on the fall cone test and triaxial testing, the merits and limitations of which are discussed in Chapter 4. Briefly, the determination of soil strength can be obtained using direct shear test, the vane test, fall cone penetrometer, unconfined compression test, and the triaxial compression test (Head, 1980 vol 2). The Casagrande direct shear test (Olson, 1989) is the simplest and most straightforward and measures soil in terms of total

stress, during which the sample is slowly horizontally sheared until a point of failure. The in-situ vane test, covered extensively by Chandler (1988), is a quick and simple test that may be applied in the field to determine the undrained shear strength of soil using an instrument with four blades. Penetrometers measure the force required to push or drive a device into the soil and readers are directed to Bengough *et al.*, (2001) and Lowery & Morrison (2002) for extensive reviews on soil penetrometers and penetrability.

Triaxial testing, covered in detail in Chapter 4, can be further divided into unconsolidation-undrained test (UU), consolidated undrained test (CU), and the consolidated drained test (CD). The UU test is the fastest where failure occurs within 25 minutes and the drainage valves are closed, the CU test is sufficiently slow to allow equalisation of pore pressure during consolidation, and the CD test is slow enough to allow negligible pore pressure variation. This apparatus allows the total control of parameters such as pore water pressure, consolidation and shearing, which allows a thorough analysis of the characteristics of a soil under shearing conditions.

## ***2.4 Soil water***

The following section covers the two most important soil water relationships, the relationship between water content and suction and the hydraulic conductivity of a soil. One of the greatest points of contention and a key argument of this thesis is the lack of multidisciplinary terms for water in soil and in particular the lack of wholesome parameters with which to determine how a soil responds to water, where single and sometimes vague terms such as water holding capacity are insufficient to describe a variety of soils to wetting and flooding (Kerr *et al.*, 2016).

### **2.4.1 Important soil water relationships**

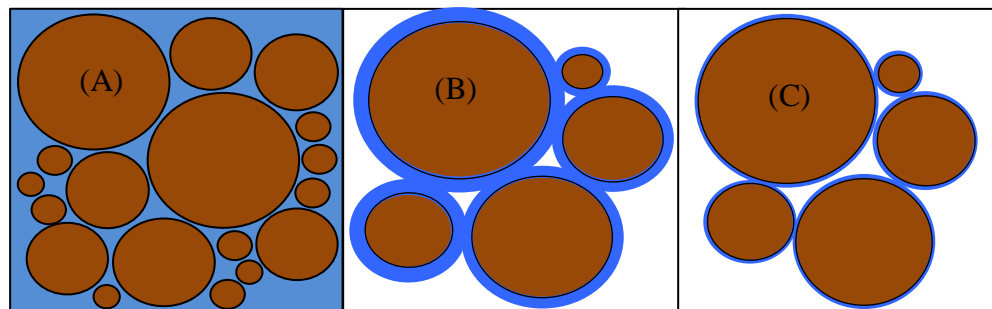
The water content of a soil plays a crucial role in the physical and biological functions of soil including water infiltration, redistribution and movement of water through the soil, shear properties, germination of seeds, plant growth and microbial functions. The interaction of the three soil phases (described in section 2.3.1) creates negative pore water pressure known as soil suction, which is a

critical component for the geotechnical properties of a soil. Suction is simply defined as the potential energy of water within voids of a soil (unsaturated) in comparison with 'free water' (Lu & Likos, 2004; Beckett, 2011), where the relationship is briefly; the drier a soil, the greater the suction and the lower the potential. Values for suction are expressed using negative kPa or pF (capillary potential, which represent the logarithm of the suction expressed in cm of water, Schofield, 1935), as water in voids has less potential than free water and can be expressed using equation 4;

$$\psi_m = C(\kappa) + A(t)$$

*Equation 4: Matric suction, where C is the capillary component described as a function of the liquid-gas curvature  $\kappa$ , and A is the adsorptive component as a function of film thickness (t) Gens (2010).*

Osmotic suction occurs when a semi-permeable membrane separates two solutions, one with a higher concentration of solute. This 'membrane' can be created by clay particles that form very small voids (Beckett, 2011), and the chemical potential is reduced. Matric suction, also known as capillary pressure, occurs due to pressure from dry soil on its surrounding soil to equalise the moisture content of the entire unit. The combination of matric and osmotic suction is referred to as the total suction. There are three types of forces attributed to the presence of water: adhesion (attraction of soil water to soil particles), cohesion (attraction of water to water molecules), and capillary (through adhesion and cohesion water can move through small tubes against the forces of gravity).



*Figure 8: Stages of water retention in the soil matrix. (A) Gravitational water at complete saturation, no air present ( $\theta_{SAT}$ ) (B) Capillary/meniscus water in unsaturated soil, both air and water present between soil particles ( $\theta_{FC}$ ) (C) Hygroscopic water at permanent wilting point, where only water adsorbed to soil surface remains ( $\theta_{PW}$ ) from Kerr et al. (2016)*

Wheeler & Karube (1996) suggest that water is held in four layers around soil particles, and excludes water that is chemically combined (hydration) in the mineral structure. The key properties of these different categories of water in soil are shown in Table 3 and these four layers are;

- Hygroscopic adsorbed water held to the solid particle by electrical attraction and unable to be removed by oven drying (Figure 8C)
- Hygroscopic water that cannot be removed by air drying but can be by oven drying
- Capillary water, held by surface tension and removable by air drying (meniscus water, Figure 8B)
- Gravitational water, removable by drainage (bulk water, Figure 8A)

WATER TYPE		
Gravitational water (A)	Capillary Water (B)	Hygroscopic water (C)
Moves under the influence of gravity	Mostly available for plant growth	Not available to plants
Found in macropores	Held by cohesion and adhesion in capillaries (micropores)	Held on the particle surface very tightly by adhesion (high in clays due to high surface area) caused by forces of Van der Waals or chemisorption
Moves rapidly out of well drained soils	Quantity of water is a function of pore space (total volume) and pore size	Force of gravity insufficient to break the force between soil and water molecule
Not available to plants	As soil dries, water tension (kPa) increases	Only removed with heating (oven drying)
Occupies air space, therefore can drown plants	Removable by air drying	
Leaves soil within 2-3 days		

*Table 3: Key properties of three soil water types; gravitational, capillary and hygroscopic*

### **2.4.2 Soil water definitions**

Before the chapter progresses further, a list of soil water definitions is required as many terms are used synonymously or incorrectly according to their original definition, which is confusing to researchers and does not allow for confidence in comparing values presented in literature. This section also discusses how some of these terms are not adequate to cover the response to soil under conditions where the soil changes over time physically (volume change) and at saturation. Table 4 gives definitions of water terms as used in this thesis and where applicable a description and synonyms commonly associated with the term. These are derived from as many sources as possible, and the given description is the best fit or a standardised definition (e.g. as given by British Standards).

There are many different ways in which the mass or volume and distribution of water in soil can be quantified, both directly and indirectly. It is important to know the water content of a soil for a variety of applications such as the calibration of climate models and use in agricultural/horticultural systems as it affects parameters such as soil organic matter decomposition, soil respiration and carbon sequestration (Bittelli, 2011). The measurement of soil water through direct or indirect methods is standardised and simple (such as British Standards and ASTM (American Society for Testing and Materials)) and will be discussed in Chapter 4. However the definition or interpretations of 'water holding capacity' (WHC), or maximum water holding capacity is exceptionally variable within soil science literature and across other disciplines. WHC is used synonymously with other descriptors such as; the water held at field capacity i.e. maximum amount of water held in a soil against the forces of gravity OR the water available to plants (i.e. the amount of water held between field capacity and wilting point). It is the use of interchangeable terms that suggests the 'maximum' amount of water a soil can hold is only the water held against gravity, meaning research on the end point of the saturation scale i.e. flooding is limited by definition.

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**SOIL WATER TERMS (terms and *description or alternative term*)**

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Saturation	All pores are filled with water. Air is no longer present in the pore space; therefore soil matrix is a two-phase material with zero suction. <i>This is not always equal to the pore space due to air trapped within the soil (usually 0-10%) and saturation is usually 0.95 x porosity (effective saturation).</i>
Effective saturation	If air is present, saturation occurs when water fills all the pores that it can reach as air will still remain in the smallest pores. This is more likely in the field than saturation as defined above. <i>Usually equal to porosity.</i>
Degree of saturation	The volume of water divided by the volume of voids, generally expressed as %. It is 100% when the soil is fully saturated, and requires a knowledge of the total volume of the specimen.
Field Capacity <i>Drained upper limit</i>	Moisture content of the soil after all gravitational water has drained and usually occurs 2-3 days after rainfall. <i>This is the maximum amount of water a soil can hold against gravity. It can be measured using a value of 0.1 mm/day flux or the water held at a suction of -0.33 bars.</i>
Maximum/Water holding capacity (WHC)	(Maximum) water holding capacity is the amount of water held at full saturation without drainage (undrained). <i>Various additional definitions, where maximum WHC was originally defined as the most amount of water that can be held at field capacity.</i>
Wilting Point <i>Lower limit of extraction</i>	Moisture content of a soil at which plants can no longer reach water within the soil. This is typically defined as -1500 kPa.
Oven dry	Soil that has been dried at 105°C for 24 hours. <i>Removes all types of water from the soil except adsorbed water (held to the soil particle by electrical attraction)</i>

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Plant available water	Water that is held in the soil between field capacity and wilting point (-0.33 and -15 bar) and is able to be utilised by a plant. <i>Generally considered to be approximately 50% of the field capacity.</i>
Porosity	The volume of voids within a bulk of soil.
Pore Space	<i>Calculated as the 1- (bulk density/particle density x 100). ‘</i>
Pore size (distribution)	The cumulative size of each pore within a soil.
Permeability	These terms all describe the rate at which water is able to move through a body of soil through the pore network, with values in mm or inches of water/time.
Hydraulic conductivity	
Infiltration rate	The rate of movement of water into a soil from the surface
Percolation	Downward movement of water within a soil.
Gravimetric water content (GWC)	The mass of water to the mass of dry solids (dry basis GWC) The mass of water to the total mass of the wet sample (wet basis GWC)
Volumetric water content (VWC)	$\theta_v = \theta \text{ (GWC)} * B_d \text{ (OR)} = V_w/V_s$ Volumetric water content as originally defined. $\theta_v = \theta \text{ (GWC)} * B_{di} \text{ (OR)} = V_{wi}/V_{si}$ Volumetric water content using the instantaneous volume of a soil (at the point of measurement). <i>where <math>V_w</math> = volume of water and <math>V_s</math> = volume of solids, <math>D_d</math> = dry density.</i>
Suction	The energy required for extracting a unit volume of water from soil, measured in kPa (Fredlund & Rahardjo, 1993).

Soil water retention curve	The relationship between (matric) suction in a soil and the water content of a soil. <i>Also called suction-water content relationship, retention curves, moisture retention curves and numerous variations thereupon.</i>
Matric suction	The difference between the pore-air and the pore water pressure. The relationship between matric suction and degree of saturation is presented in a soil water retention curve

*Table 4: Important definitions for soil and soil-water relationships*

The difficulty with defining soils in saturated or near saturated state is that the meaning of 'holding' water is too broad; there is a distinct difference between retain and hold. Water may retain a given volume of water under gravity when free draining, but will hold or contain a given volume of water when there is static water i.e. flood conditions. The concept of field capacity is used in most literature as a key soil water indicator and is typically quantified using suction values to determine the point at which all water has drained due to the force of gravity from the largest pores in the soil and only water held against the force of gravity remains. The typical definition is *'moisture content of the soil after all gravitational water has drained and usually occurs 2-3 days after rainfall'*. It can also be quantified using the flux values where field capacity is reached when the flux is negligible (ml/hour), or in the lab the moisture content can be determined as -0.33 kPa suction by calculating a soil water retention curve (SWRC) (see section 2.4.3). However field capacity is largely an academic value and not particularly easy to apply to field conditions as strict laboratory processes control its measurement. In reality, some soils take far longer than 2-3 days to drain freely under gravity. Actual field capacity ranges from -10 to -20 kPa (Rose, 2004), for soils with <20% clay and >20% clay respectively. It is not uncommon for soils to be wetter than field capacity under free draining conditions should rainfall exceed the drainage rate. We are interested in how much water a mass of soil can hold at saturation and how it is retained once drying begins to occur, therefore the concept of field capacity may only be applied during the drying phase.

As mentioned previously, the water holding capacity of a soil, here defined as the maximum amount of water a soil sample can hold when undrained, can be measured gravimetrically and volumetrically. These provide an index of the mass or volume of water that a soil can hold, or give an indication of the degree of saturation. Gravimetric water content (GWC) is referenced to a mass of solids, whereas volumetric water content (VWC) and degree of saturation are based on ratios of the original volume of a soil. However VWC doesn't account for the volume change of a soil under wetting as it swells, as it references the value back to the original volume (based on mass and density), and not the instantaneous volume and assumes that no or negligible volume changes have occurred (Fredlund, 2002). Should the volume of a soil remain stable as the water content

increases, i.e. the soil is non deformable, the gravimetric, volumetric and degree of saturation values can be referenced to the constant start value. However should the volume increase as the water content increases, only gravimetric water content can be referred back to the original constant. Commonly used methods to present the water retention characteristics of a soil, such as a soil water retention curve (SWRC, section 2.4.3) do not take into account the volume change of a sample, and indeed one cannot compare the volumetric or gravimetric values without knowledge of both. This is a major drawback of using single measurements to determine soil change as used in an SWRC. Figure 9 shows the differences in how values are presented, adapted from Kerr *et al.* (2016).

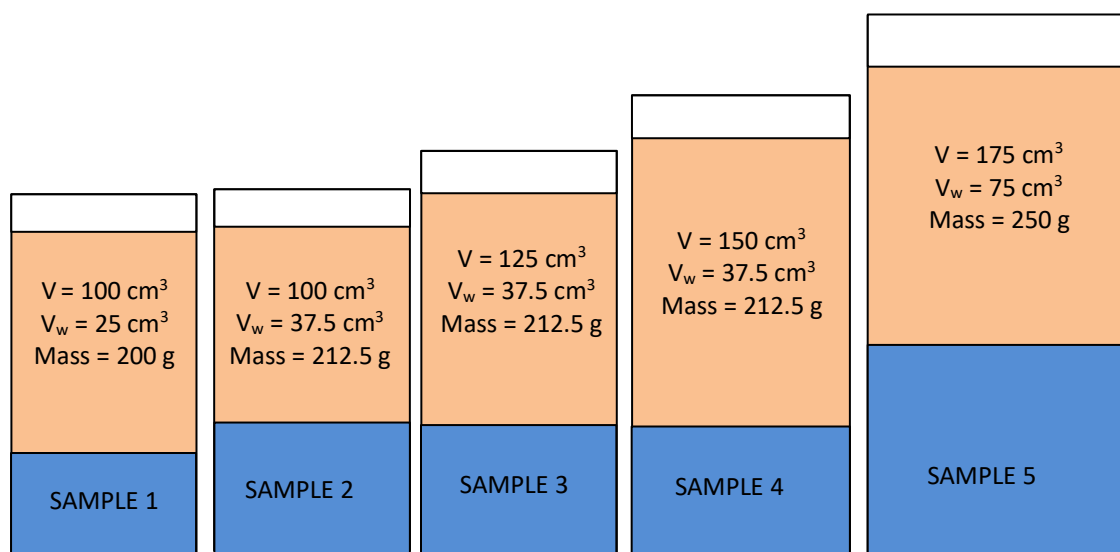


Figure 9: GWC, VWC and density of five samples to represent change during wetting. Sample 1 represents the original sample, where samples 2-5 present theoretical different changes in water content and volume during wetting (adapted from Kerr *et al.*, 2016).

In Figure 9, sample 1 is the original soil mass, volume and water mass, where samples 2-5 represent various changes in water mass, volume, and density. Sample 2 has taken up 12.5 g of water but has not changed in volume as a result of swelling. Sample 3 and 4 have taken up the same mass (and volume) of water as Sample 2, however the bulk volume has changed. Sample 5 has taken up more water than samples 2, 3 and 4 and has increased in volume. These changes result in different gravimetric and volumetric water content values, due to the change in density and volume of samples. It is clear therefore that the terms GWC and VWC are not capable of describing the proportional physical changes in the soil, and

only reference values to the original state. Soils that swell to a greater degree than other soils under the same wetting conditions appear to perform more poorly when assessing their volumetric water content (sample 3 vs sample 4). Therefore it is imperative to record the instantaneous volume and mass of soil samples during measurements over time in order to thoroughly assess the change (as seen in Table 5), presented as VWC<sub>i</sub> (GWC \* dry density of sample at the point of measurement).

Sample	Mass (g)	Mass of solids (g)	Mass of water (g)	Volume (cm <sup>3</sup> )	Bulk Density (g/cm <sup>3</sup> )	Dry Density (g/cm <sup>3</sup> )	GWC(%) dry basis	VWC	VWC (corrected)
1	200	175	25	100	2	1.75	14.3	25	25
2	212.5	175	37.5	100	2	1.75	21.4	37.5	37.5
3	212.5	175	37.5	125	1.7	1.4	21.4	37.5	30
4	212.5	175	37.5	150	1.42	1.17	21.4	37.5	25
5	250	175	50	175	1.43	1.0	28.6	50	28.6

*Table 5: Summary of key changes to samples in Figure 9 (above) where VWC (corrected) is the volumetric water content relative to the instantaneous volume of the sample, not the original volume.*

Samples 3 and 4 have the same water mass change, shown by the gravimetric water content of change of 14.4 - 21.4 %, it is only an index of water content change and is not affected by the physical parameters of a soil, it is a ratio of the water to the dry mass of the soil solids. Volumetric water content, however, is subject to structural changes of the soil mass as it is a ratio of volume of water (which is directionally related to the mass) to the volume of the bulk (wet) soil. Sample 2 and 3 have the same volumetric water content, despite Sample 2 having a proportionally greater volume of water to total soil volume (37.5 cm<sup>3</sup> water to 125 cm<sup>3</sup> of soil in comparison to 37.5 cm<sup>3</sup> to 150 cm<sup>3</sup>, respectively). Original VWC gives an indication of the volumetric change in comparison to the original volume. The instantaneous volume of the soil must be taken and used for the secondary volumetric water content equation (using either the volume of solids or the dry density (D<sub>d</sub>) in addition to using the typical VWC equation in order to provide

accurate volumetric water content of the soil at the time of sampling. Fredlund (2002) suggests that should a volume change occur during wetting, converting the volumetric water content to gravimetric by assuming a specific gravity (definition in section 2.2.2.) for the soil solids gives an accurate representation of real data when the volume change is unknown.

### **2.4.3 Measuring soil water**

The moisture content of a soil determines its behaviour, and measuring this value under defined test conditions can provide classification in order to assess soil's engineering properties (Head, 1980). It can be expressed as the gravimetric water content, volumetric water content, or degree of saturation. A simple method of water quantification is to oven dry samples and obtain a dry mass, where samples are oven dried at 105°C for 24 hours (Evetts *et al.*, 2008) and gravimetric water content is expressed as a ratio of the mass of water to the mass of either the dry solids (dry basis GWC) or to the total mass of wet solids (wet basis GWC) as defined in table 4. However this thermo-gravimetric method is destructive and allows a singular characterisation of the soil to be taken. Indirect methods allow in situ measurements of soil water content by quantifying a proxy variable and using empirical or physical relationships to calibrate the variable against water content. Examples of indirect measurement include; dielectric methods (uses the differences in electric permittivity values between solid, liquid and gas), resistivity methods, neutron scattering, measurements of soil thermal properties. These applications are useful in the field where the transport of samples is not viable (such as a field study over time).

Although the quantification of gravimetric or volumetric water content of a soil is essential, it does not provide information on the relationship between capillary potential/pressure (suction) as a function of the degree of saturation, which is critical for the understanding of soil processes such as infiltration, redistribution, solute transport and compaction (Bachmann & R. van der Ploeg, 2002; Garcia *et al.*, 2014; Klute, 1986; Sorrenti *et al.*, 2016). Knowledge of the water retention characteristics of a soil is also important for understanding the behaviour of soil materials in an unsaturated state (Fredlund, 2002; Fredlund & Xing, 1994; Toll *et al.*, 2015). Capillary pressure (suction) is directly affected by soil texture, particle

size distribution, pore space geometrics, interfacial tension, temperature, and organic matter content (which directly affects water retention due to hydrophilic nature and indirectly affects water retention due to soil structure modification).

The water retention of soils, i.e. the relationship between water content and suction, can be described using a soil water retention curve (abbreviated henceforth as SWRC). An SWRC provides important information on unsaturated soils (Fredlund, 2000; Parvin *et al.*, 2017) and can be used to define important parameters such as plant available water, in the estimation of permeability (van Genuchten, 1980), hydraulic conductivity (Mualem, 1976) and for the interpretation of shear strength (Vanapalli *et al.*, 1996), therefore its usefulness as a soil indicator is widespread. Despite a wealth of research and knowledge on pore space, the SWRC typically uses the volumetric water content and a simplified representation of the pore system. As mentioned previously the SWRC therefore ignores important physical effects such as volumetric change of soil, which limits the accurate identification of the soil's relative water content. There are numerous methods for determining the SWRC, which uses standardised procedures such as the evaporation method (Gardner & Miklich, 1962) to produce a wetting and a drying curve between 0 kPa suction (saturation) and 1500 kPa (permanent wilting point), however each method has its own limitations (Tarantino *et al.*, 2008).

The term hysteresis or hysteric refers to the phenomenon whereby water content at a given pressure for a wetting soil is less than that of a drying soil (Klute, 1986). This gives the 'characteristic' curves as shown below in Figure 10. Should a sample begin in a saturated or close to saturated state and is subject to drying, it will follow the primary drying curve (red, Figure 10). As water content decreases and suction increases, the largest pores begin to desaturate (air entry value), followed by drainage of finer and finer pores until residual suction is reached where there are negligible water content changes with further increases in suction. Beyond the point of residual suction, water is only held within the soil matrix adsorbed onto clay particles (McQueen & Miller, 1974).

Should a sample start from an oven-dried state and then subjected to wetting, the soil will follow the primary wetting curve. The water entry value is the suction at

which water enters pores, at which point the moisture content increases significantly and suction decreases as the water content increases until suction reaches 0, and the soil is saturated. The end point value of gravimetric water content/volumetric water content is lower at the end of an absorption curve (wetting curve is followed) in comparison to the start point value of an adsorption curve (drying curve) due to irrecoverable shrinkage of the sample during drying or air bubbles trapped within the soil pores that prevent full saturation (rendering the sample effectively saturated, see section 2.4.2 for definition). If the wetting or drying of a sample is interrupted, the soil will follow a scanning curve that returns the soil to the alternative curve wetting or drying curve (Figure 10).

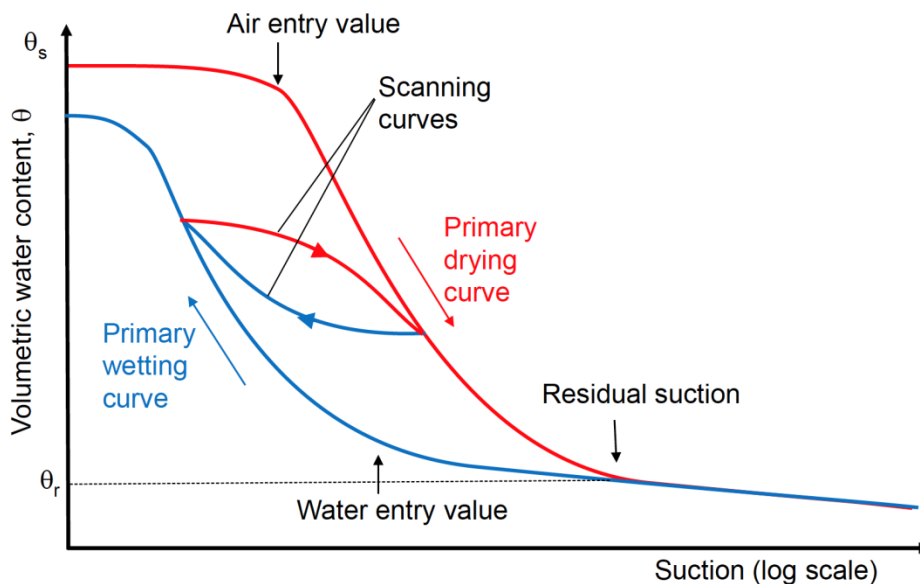


Figure 10: Typical soil water retention curve (after Toll et al., 2012).

#### 2.4.4 Hydraulic conductivity

The speed of water movement through a soil is of significant importance, for functions such as the supply of water to plants and aquifers and entry of water into the soil (Klute & Dirksen, 1986). The hydraulic properties of the soil are a measure of the conductivity and the water retention characteristics i.e. the ability to transmit and store water, respectively. Permeability is the most variable engineering soil property and can change by 10 orders of magnitude considering the size range of particles (gravel – clay), as seen in Table 6.

<i>Classes</i>	<i>Saturated hydraulic conductivity</i>	
	<i>Micrometres per second</i>	<i>Centimetres per hour</i>
Very high	Over 100	36
High	10- 100	3.6 - 36
Moderate	1- 10	0.36 – 3.6
Moderately low	0.1 – 1	0.036 – 0.36
Low	0.01 – 0.1	0.0036 – 0.036
Very low	Less than 0.01	Less than 0.0036

*Table 6: Hydraulic conductivity classes based on speed on water movement (Foth, 1991)*

$$k = \frac{qL}{Ah}$$

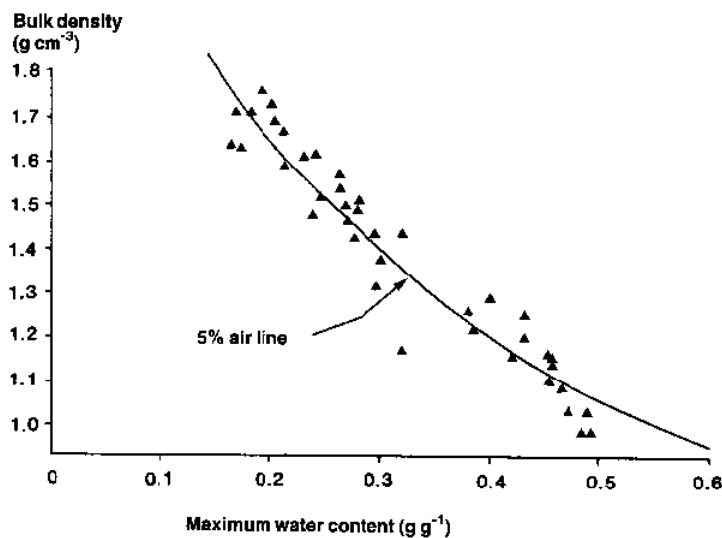
*Equation 5: Hydraulic conductivity (k), where q is the permeability coefficient (flow in m<sup>3</sup>/second), L is the length of the sample in m, A is the cross-sectional area of the soil (m<sup>2</sup>) and h is the pressure head (in m). Craig (2004).*

The relationship between the rate of permeant flow and hydraulic gradient was discovered by Darcy (1856). The coefficient of permeability (used synonymously with hydraulic conductivity) states that the discharge velocity of flow through a porous medium is proportional to the hydrostatic pressure causing flow (hydraulic gradient) and inversely proportional to the permeant viscosity. The formula in Equation 5 applies Darcy’s Law and is used to derive the hydraulic conductivity obtained from a constant head test. There are a number of factors that affect how quickly water moves through a body of soil; particle size distribution, void ratio (porosity), soil structure, state of stress or stress history (i.e. compaction), degree of saturation, thixotrophy (term discussed below), and gradient (Reid, 1988: Head 1980 vol 2).

Firstly, the particle size distribution of a soil governs the porosity to a large extent (Chan & Govindaraju, 2004) and therefore conductivity, as smaller particles create smaller voids that increase the resistance the flow of water. It is common for the conductivity of a soil to be related to the void ratio of a soil, where the higher the void ratio (volume of pores to the volume of soil), the greater the conductivity due to a higher number of flow paths. The caveat, however, is that two identically prepared samples may have different conductivities due to tiny differences in soil

structure, despite having the same void ratio. Therefore although important, the void ratio only governs the conductivity to a limited extent and it is rather the distribution of voids (tortuosity) through the soil matrix that is important.

Secondly, as discussed previously (2.3.1), the structure of soil is dependent on its forming factors, compaction, water content and organic matter content. Soils that are compacted dry of optimum (see 2.3.3) take up much more water than soils compacted when wet of optimum as they have multidirectional permeability due to random soil particle orientation. Once a soil has been compacted, the resultant density of the soil influences the infiltration rate (speed at which water may enter the soil) is a direct function of the density of a soil, and ultimately determines the maximum water content of the soil (Figure 11).



*Figure 11: Maximum water content as a function of bulk density (from Taylor & Gardner, 1963)*

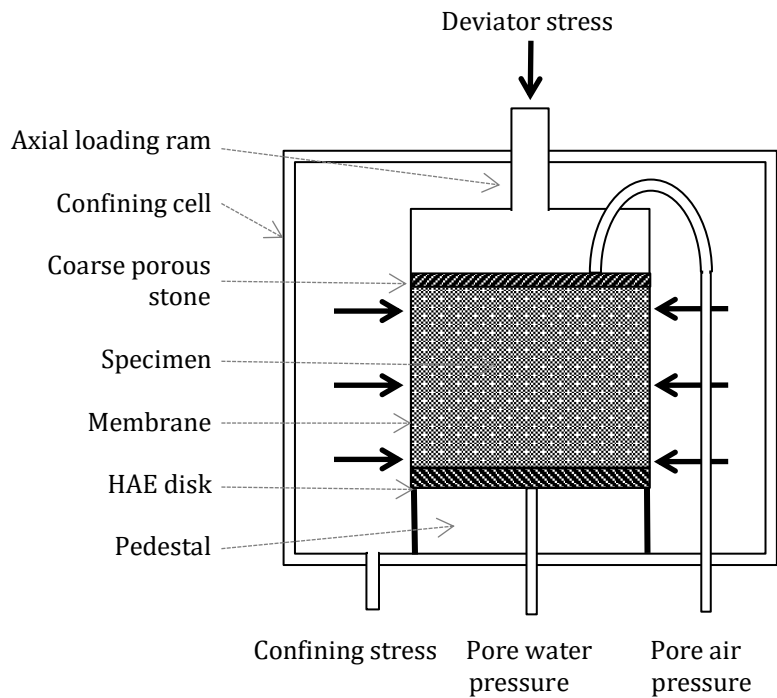
Thirdly, the degree of saturation is also crucially important in permeability measurements. Darcy's Law assumes saturation, but if water does not fill all voids within a soil, air bubbles can block the flow of fluid, which therefore invalidates the assumption (Head, 1980). Soils with a high number of air filled voids have lower conductivity (Olson & Daniel 1981) than pores saturated or partially filled with water. Barden & Sides (1970) reported a difference of 60-100% in hydraulic conductivity as a function of the saturation level.

Fourthly, thixotrophy (the ability of a soil mass to gain strength over time) affects the results of laboratory derived hydraulic conductivity, and indeed other tests. Disturbing the soil creates some alignment of particles during compaction, but

over time the soil will return to a more flocculated state. Mitchell *et al.* (1965) compared specimens immediately after preparation and 21 days after preparation and reported greater conductivities in the latter samples. This effect, depending on the water content at preparation, can be in the order of a magnitude difference in conductivity (Dunn, 1983). Lastly, the gradient (differences in head) in laboratory testing is artificially increased to speed up the processes of testing, however this consolidates the sample through axial deformation, which in turn causes particle migration and closing of macropores. Darcy's Law assumes laminar flow; but artificially increasing the gradient is likely to cause turbulent flow (Reid, 1987). In combination, the increased gradient reduces the hydraulic conductivity of a sample.

#### **2.4.5 Measuring hydraulic conductivity**

Hydraulic conductivity for unsaturated soils can be measured in the laboratory, in the field using suction and tensiometers to measure the change, which includes the instantaneous profile method (Daniel, 1982; Munoz *et al.*, 2008), the Gardner (1956) or Corey's method (Green & Corey, 1971) in various types of infiltration column (Duong *et al.*, 2014), and it can be predicted empirically using the SWRC (Fredlund *et al.*, 1994). For the most accurate assessment of soil hydraulic properties, they should be measured directly whenever possible, therefore the use of empirical methods for estimating hydraulic conductivity will not be discussed further. Testing hydraulic conductivity in a saturated soil can be done using the falling head or constant head test, or alternatively in a triaxial cell (as described in Chapter 4). For simple and rapid testing of hydraulic conductivity, the falling head test uses water in a piezometer (tube) to provide a pressure head (water pressure in terms of the height of a column above the datum level, Head 1980) that passes through a saturated sample of fine-grained soil. The constant head test provides a continuous flow of water at the same pressure, used to test clean sands or large grained soils.



*Figure 12: Triaxial cell apparatus set up for testing hydraulic conductivity and shear strength. A high air entry (HAE) porous stone is at the base of the specimen with an HAE value of 100 kPa. Water pressure is measured at the base (pore water pressure  $\mu_w$ ) and air pressure measured at the normal porous stone at the top of the specimen (pore air pressure,  $\mu_a$ ). From Lu & Likos (2004).*

Controlled tests hydraulic conductivity can be also conducted using triaxial cell apparatus where the suction, water content, pore water & air pressure, and rate of flow through the sample can all be controlled and monitored. As per the set up in Figure 12, the sample tested in a triaxial cell has a porous filter stone at each end with tubing containing the length of the sample.

### **2.5 Concluding remarks**

This chapter has outlined the fundamental knowledge base for the research contained within the thesis, including an introduction to the various important mechanisms of soil pedogenesis, and critical interactions between soil minerals, organic matter and external influences that determine the make-up of a soils; its texture, structure, relationship with water, erodibility and shear strength characteristics. The most important findings from a review of literature are as follows:

- Current definitions surrounding soil, soil water and soil parameters such as density are insufficiently determined. There are no universal definitions for terms such as water holding capacity or soil erodibility. As it is unlikely that a universal term will be used, each research piece needs clear definitions of what they determine to be, for example, the water holding capacity;
  - Here, the water holding capacity is stated as the maximum water content of a soil at saturation. Other documents use the field capacity as the water holding capacity.
  - Soil erodibility has no universal method of quantification, rather a number of indicators that may suggest how erodible a soil is to a water input, which may either be the resistance to degradation under wetting through rainfall or submersion. Soil erodibility may also be inferred indirectly through quantification of organic matter, the presence of clay and water content.
- As summarised in Kerr *et al.* (2016), the quantification of water in soil on both a gravimetric and volumetric basis, after a soil has been subjected to wetting is inconsistent.
  - Gravimetric water content must be reported on a dry or wet basis, without this statement the quantification, one cannot compare literature.
  - Volumetric water content is typically reported according to the original volume of soil, however under wetting most soils swell. Therefore the volumetric water content value, should the original volume be used in the calculation, is incorrect. In order to provide values that compare the original volume of soil to the volume of water to a change in the same sample after water uptake and volume change, the instantaneous soil volume must be used in calculations.

### **3. Water Treatment Residual (WTR)**

This chapter explores the production, storage and disposal processes and physiochemical characteristics of water treatment residual (WTR) with specific reference to waste produced in the NE of England by Northumbrian Water Ltd. (NWL). Much of the information on water treatment processing has come from personal communication with Luke Dennis (NWL) conducted by Finlay (2015), supplemented by personal communication with Ed Higgins (NWL).

#### **3.1 An introduction to WTR production, storage and disposal**

There are 27 different companies across the UK that produce the 5.29 trillion litres of clean water used in the UK each year. Eight companies provide more than 75% of the water; Thames Water provides 18.2% of this, followed by Severn Trent Water 12.6%, United Utilities 11.8%, Yorkshire Water 8.5%, Anglian Water 8.1%, Affinity Water 6.1%, Dŵr Cymru Welsh Water 5.5% and Northumbrian Water 4.7%. Although one of the smaller companies, Northumbrian Water alone has 55 treatment works, supplying 1.1 billion litres of water every day to 4.4 million people in the North East of England (Northumbrian Water Ltd [16]). The remaining 19 companies account for less than 25% of the remaining supply.

'Clean water treatment' refers the production of potable water using raw water from groundwater (aquifers and springs) and surface water sources (streams and rivers), as opposed to 'wastewater treatment', referring to the processing of water containing sewage, agricultural waste and industrial sources of pollution. The primary aim of the clean water treatment process is to remove contaminants to produce water that meets particular thresholds for human consumption, as dictated by the governing body responsible for the water being produced. This includes pathogens e.g. bacteria, viruses and eggs of parasitic worms, potentially toxic chemicals from human activity e.g. fertiliser, and natural chemicals e.g. fluorides, arsenic and those influencing smell, colour and taste. The success of using the coagulant-enhanced flocculation and settlement method, which is common practice for 70% of water treatment works, relies on the removal of turbidity and organic matter such that the water falls within the maximum permitted concentration levels of turbidity according to drinking water standards

(Keeley *et al.*, 2014). Crittenden *et al.* (2012) provide a good summary of important constituents commonly found in water sources (Table 7).

Source	Particulate constituents		Ionic and dissolved constituents		Gases and neutral species
	Colloidal	Suspended	Positive ions	Negative ions	
Contact of water with minerals, rocks and soils (e.g. weathering)	Clay Silica (SiO <sub>2</sub> ) Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> ) Aluminium oxide (Al <sub>2</sub> O <sub>3</sub> ) Magnesium dioxide (MnO <sub>3</sub> )	Clay, silt, sand and other inorganic soils	Calcium (Ca <sup>2+</sup> ) Iron (Fe <sup>2+</sup> ) Magnesium (Mg <sup>2+</sup> ) Manganese (Mn <sup>2+</sup> ) Potassium (K <sup>+</sup> ) Sodium (Na <sup>+</sup> ) Zinc (Zn <sup>2+</sup> )	Bicarbonate (HCO <sup>-</sup> ) Borate (H <sub>2</sub> BO <sub>3</sub> <sup>-</sup> ) Carbonate (CO <sub>3</sub> <sup>2-</sup> ) Chloride (Cl <sup>-</sup> ) Fluoride (F <sup>-</sup> ) Hydroxide (OH <sup>-</sup> ) Nitrate (NO <sub>3</sub> <sup>-</sup> ) Phosphate (PO <sub>4</sub> <sup>3-</sup> ) Sulphate (SO <sub>4</sub> <sup>2-</sup> )	Carbon dioxide (CO <sub>2</sub> ) Silicate (H <sub>4</sub> SiO <sub>4</sub> )
Rain in contact with atmosphere			Hydrogen (H <sup>+</sup> )	Bicarbonate (HCO <sup>-</sup> ) Chloride (Cl <sup>-</sup> ) Sulphate (SO <sub>4</sub> <sup>2-</sup> )	Carbon dioxide (CO <sub>2</sub> ) Nitrogen (N <sub>2</sub> ) Oxygen (O <sub>2</sub> ) Sulphur Dioxide (SO <sub>2</sub> )
Decomposition of organic matter in environment	Humic substances	Cell fragments	Ammonium (NH <sub>4</sub> <sup>+</sup> ) Hydrogen (H <sup>+</sup> ) Sodium (Na <sup>+</sup> )	Bicarbonate (HCO <sup>-</sup> ) Chloride (Cl <sup>-</sup> ) Hydroxide (OH <sup>-</sup> ) Nitrate (NO <sub>3</sub> <sup>-</sup> ) Nitrite (NO <sub>2</sub> <sup>-</sup> ) Sulphide (HS <sup>-</sup> ) Sulphate (SO <sub>4</sub> <sup>2-</sup> )	Ammonia (NH <sub>3</sub> ) Carbon dioxide (CO <sub>2</sub> ) Hydrogen sulphide (H <sub>2</sub> S) Hydrogen (H <sub>2</sub> ) Methane (CH <sub>4</sub> ) Nitrogen (N <sub>2</sub> ) Oxygen (O <sub>2</sub> ) Silicate (H <sub>4</sub> SiO <sub>4</sub> )
Living organisms	Bacteria, viruses.	algae, Algae, diatoms, minute animals, fish etc.	-	-	Ammonia (NH <sub>3</sub> ) Carbon dioxide (CO <sub>2</sub> ) Hydrogen sulphide (H <sub>2</sub> S) Hydrogen (H <sub>2</sub> ) Methane (CH <sub>4</sub> ) Nitrogen (N <sub>2</sub> ) Oxygen (O <sub>2</sub> )
Municipal, industrial and agricultural sources and other human activity	Inorganic and organic solids, causing colour, chlorinated organic compounds, bacteria, worms, viruses etc	Clay, silt, grit and other inorganic solids, organic compounds, oil, corrosion products etc.	Inorganic ions, including a variety of anthropogenic compounds and heavy metals, organic molecules, colour etc.		Chlorine (Cl <sub>2</sub> ) Sulphur dioxide (SO <sub>2</sub> )

*Table 7: Summary of important particulate, chemical and biological constituents found in water according to their source (Crittenden et al., 2012, adapted from Tchobanoglous and Schroeder, 1985)*

Typically water in NE England is sourced predominantly from surface water sources such as rivers that contain suspended (>1.0 µm), colloidal (0.001-1 µm) and dissolved particles (<0.45 µm), the remaining water being taken from groundwater sources. Northumbrian Water, for example, takes 85.5% of its water

from surface sources, 4.5% from ground sources and 10% from mixed sources. In comparison with groundwater, there is a significantly greater quantity of natural organic matter (NOM) and inorganic material (from the weathering of rocks) in surface water. The presence of NOM, a complex matrix including components such as zooplankton, bacteria, viruses, clay-humic acid complexes, humic acids, proteins and polysaccharides (Bolto & Gregory, 2007), has several effects on water quality and creates significant issues during the process of water treatment as it reacts with metal ion coagulants. Typically the quantity of NOM is indicated using total organic carbon (TOC) as a proxy measure where NOM is approximately twice the concentration of TOC. Groundwater sources commonly have TOC ranges of 0.1-2 mg/L and surface sources have a TOC range of 1-20 mg/L (Crittenden *et al.*, 2012). The concentration of NOM in the water source and pH often determines the coagulant dose, as at a higher pH NOM is more ionised and the number of essential positive charges on metal coagulants are reduced (O'Melia *et al.*, 1999). Suspended particulates, mainly inorganics such as silica, aluminosilicates, iron oxides, manganese oxides and organics, which range from 10 µm to sub-micron colloidal size (Thurman, 1985), supply an adsorption surface for microbes and humic substances that to some extent protects them from the disinfection process in water treatment. The presence of dissolved organic compounds (materials that pass through a membrane with pores of <0.45 µm) causes discolouration of water, taste and odour in addition to the potential formation of carcinogenic chlorinated hydrocarbons during disinfection with chlorine.

Water treatment is therefore a stringent process to remove the large range of differing sizes of organic and inorganic particulates. The process of extracting these particulates is difficult because the negative charge of each particle enables to them to be held in suspension for many days, meaning that natural sedimentation of water for clarification would be infeasible as a treatment method considering the demand for clean water. Particulates only settle when they lose their negative charge and are then able to coagulate with other particles, but the range of pH at which different particles lose their charge is wide ranging (2-12 pH). This presents difficulty for the water companies and the treatment process requires the careful use of pH regulators. In the majority of treatment plants a pH of between 6 and 8 is maintained to complete the necessary processes whilst

avoiding accelerated corrosion of equipment that occurs at lower pH range (Crittenden *et al.*, 2012).

### **3.1.1 Water processing and production of WTR**

Drinking water production has approximately 11 stages, from abstraction of the raw water until the point at which water exported from the plant is fit for human consumption. Briefly, the steps of water treatment are as follows (Thames Water, 2014 [17]) where WTR is removed from the process at the sedimentation stage (5); subsequent water processing information is only included for informational purpose. Figure 13 on page 72 describes the processing of WTR removed at the sedimentation stage.

1. *Abstract of raw water* – Water is taken and pumped to the treatment plant from surface sources (rivers and streams) and groundwater sources (aquifers and springs).
2. *Reservoir storage* – water is held on a long-term basis before use, here there is natural settling of some contaminants and breakdown of organic matter by UV radiation in sunlight and organism action. This also evens out any temporal changes in water quality.
3. *Screening* – graduated metal grills (5-15 cm spacing) remove large debris such as twigs, leaves and man-made detritus. Fine screens (5-20 mm spacing) trap smaller debris during movement of water from reservoir to treatment plant. Where raw water is of particularly poor quality cascades are primarily used to increase dissolved oxygen content, to reduce carbonic acid content and raise the pH (limiting the corrosiveness of water).
4. *Clarification* – coagulants, flocculent aids and pH regulators are released into the water during rapid mixing to remove fine particulates (or colloids) such as clay, silt, organic materials and metal oxides that cannot be removed by filtering or natural sedimentation alone.
5. *Sedimentation* – Water that has been dosed with coagulants, flocculation aids and pH regulators is retained in sedimentation tanks for the required period for the smallest particles to settle out of suspension with slow water movement (0.1-0.3 m/s, WHO, 1996). This allows coagulated particles to flocculate, creating larger flocs. If the speed of water movement is too fast,

flocs breakdown. The settled particles form a sludge (WTR), at the bottom of the tank, which is then extracted for further processing (see Figure 13).

6. *Filtration* – After the majority of particulate matter has been settled out of suspension, the remaining solids are removed by sieving the material through rapid gravity filters made from gravel, sand and charcoal and then through slow fine sand filters.
7. *Aeration* – removal of compounds and dissolved metals by oxidation in order to make subsequent removal more efficient.
8. *Granular Activated Carbon (GAC)* – water is passed through porous carbon particles to remove organic compounds, chemicals such as pesticides as well as removal of odour and taste.
9. *Ozone dosing* – highly reactive ozone helps to breakdown organic material and pesticides that are not effectively treated in the previous step.
10. *Disinfection* – typically water is dosed with chlorine for a sufficient time period to kill micro-organisms.
11. *Ammoniation* – addition of ammonia encourages long lasting disinfection by combining with chlorine to form chloramines.

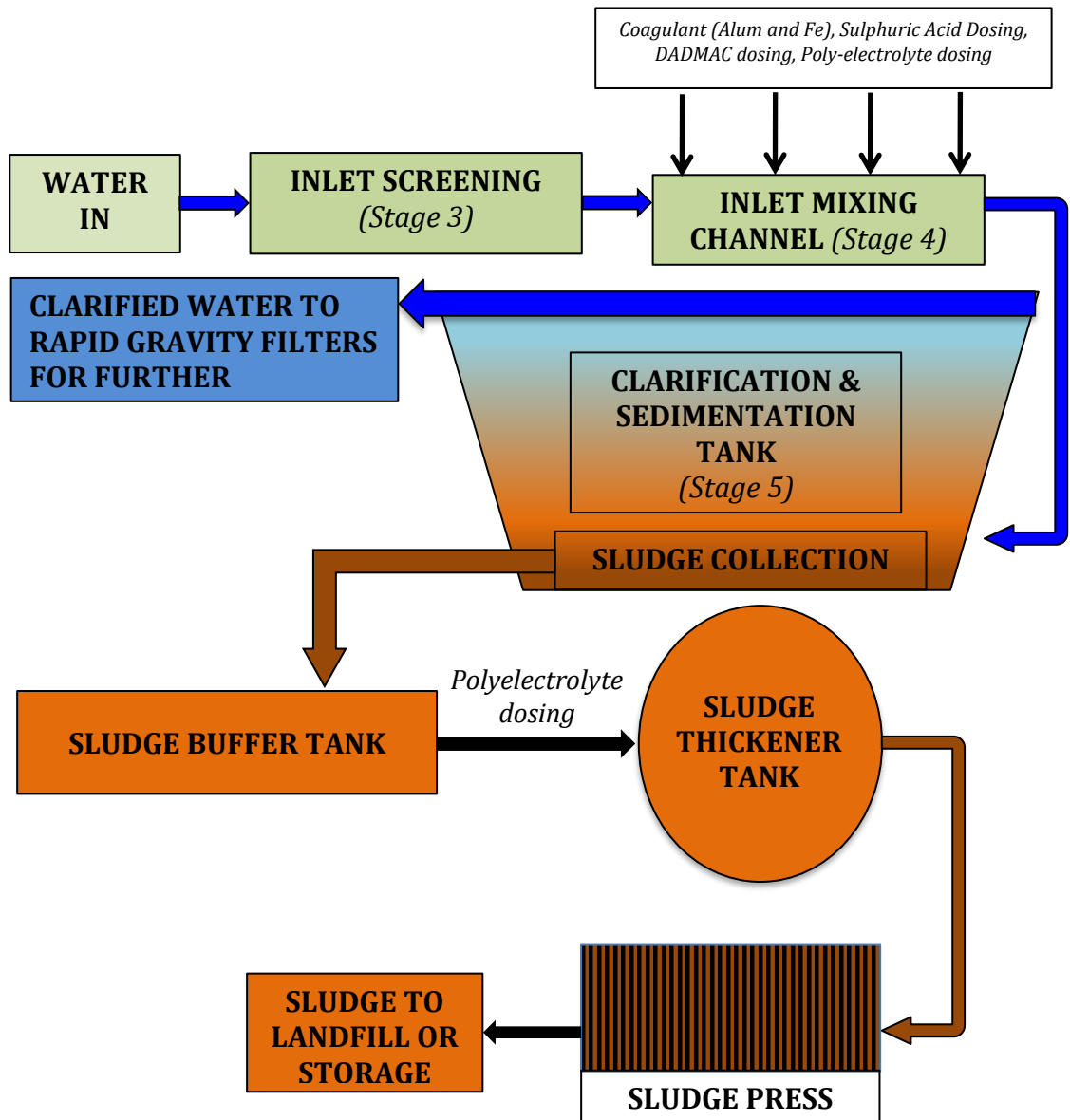


Figure 13: Schematic diagram of clean raw water in a water treatment production plant (Crittenden et al., 2005)

The clarification and sedimentation of materials removed from the raw water are the initial processes in water treatment, as shown in Figure 13. The term water treatment residual (WTR) or water treatment sludge encompasses any liquid, semi-solid, and solid phases of by-product removed during the clean water treatment process. From herewith in we use the term WTR. Although residuals can originate from 22 of the processes in the treatment plant, producing solid and semi solid waste, liquid waste and gaseous waste, here the term WTR refers to the

sludge resulting from the chemical precipitation of incoming waters only. Other waste produced from the plants may include waste from the initial screening process between reservoir and treatment plant or spent sorbents used in the treatment process to sorb constituents such as arsenic, fluoride or remove hardness.

Coagulation is defined by Crittenden *et al.*, (2012) as the addition of a chemical to water with the objective to destabilise particles, so they aggregate or form a precipitate that will sweep particles from solution or absorb dissolved constituents. As outlined above, coagulant is added to take colloidal particles that are present in the incoming water out of suspension by removing the negative charge. This allows particles to coagulate together, flocculate into a larger mass of particles and settle out of suspension. The type and dose of coagulants chosen in the specific water treatment plant depends on the characteristics of source water, such as NOM concentration, temperature, and type of particulates present. Coagulants used in the UK for the water treatment process are hydrolysing metal salts, typically aluminium sulphate ( $\text{Al}_2(\text{SO}_4)_3$ ) or ferric sulphate ( $\text{Fe}_2(\text{SO}_4)_3$ ), however there are also a number of other common coagulants available including prehydrolysed alum and ferric chloride. Typical precipitation reactions for iron and alum-based coagulants can be found in Crittenden *et al.*, (2012) however essentially the end products of the precipitation and dissociation reactions to form metal hydroxides are  $\text{Al}(\text{OH})_3$  and  $\text{Fe}(\text{OH})_3$  depending on the type of WTR. A total of 0.53 kg sludge/kg of ferric sulphate and 0.66 kg sludge/kg of ferric chloride on a dry solids basis is typically produced in these reactions, not including the addition of polymers for increased coagulation. These insoluble hydroxides adsorb onto the negatively charge surface of particulates in the water, meaning the repellent force keeping them in suspension is lost and the coagulation process can begin. Both the zero point of charge of ions and the functionality of the coagulants are dependent on pH, therefore regulators such as calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) or sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) are added to ensure the water is within the operating region of coagulant chemicals, 5.5-7.7 pH for Al and 5-8.5 pH for Fe (Crittenden *et al.*, 2012).

Flocculent aids, such as polyelectrolytes (anionic, cationic or non-ionic), poly-DADMAC (polydiallyldimethyl ammonium chloride), and sodium alginate, are often

added to accelerate the formation of larger and stronger flocs by adsorbing to destabilised particles and creating bridges. This speeds up the sedimentation process as larger flocs settle more quickly according to Stokes Law (Bolto & Greogory, 2007). Typically synthetic organic cationic and anionic polymers are selected as they are cheaper than their natural organic counterparts as effective flocculent aids, but the specific choice depends on the sludge properties and mixing environment (O'Brien & Novak, 1977).

After the clarification stage (4) the water dosed with coagulant, flocculent aid and pH regulators is then piped to sediment tanks, the design of which varies at each treatment plant. During the sedimentation stage (5) the principal in each tank is the same, an inlet channel at the top of the tank delivers water that is slowly mixed in order to increase the contact of coagulated particles and form larger flocs that are quicker to fall out of suspension. This however requires a slow flow in the settling zone of 0.1-0.3 m/s to retain links between the larger flocs. The flocculated particles form sludge (which is now termed WTR) at the bottom of the tank, which is then mechanically extracted. Once the sludge has been removed from the sedimentation tank it is dewatered using centrifuges. In some plants further flocculent aid, e.g. poly-DADMAC, is added to the sludge at this stage essentially to squeeze the water out of the flocs by inward movement of the particles within it. Depending on the process chosen by the plant, WTR is either exported as sludge, if dewatered by centrifuge, or as a 'cake' if a filter press is used to dewater by compacting the sludge. Dry solid concentrations range between 1 and 6% with the use of polymers in the sludge thickener tank, after which centrifuges, filters or belt presses achieve a maximum of around 20% dry solids. Less expensive or less energy intensive options are available at this stage, such as lagoons and drying beds but require large areas of land (Fulton, 1976). Each WTR produced by this process will have different chemical characteristics depending on the raw water source used, but is directly related by the choice of chemicals used during treatment.

### **3.1.2 Water Treatment Residual management and disposal**

The initial processes of removing matter from raw water produces vast quantities of sludge; the UK uses more than 325,000 tonnes of coagulant per year (Henderson

*et al.*, 2009), which costs ~£28 million. Sludge production is increasing because of population growth and temporal changes in raw water quality due to climate fluctuations. In a report published in 2014 (Water UK Standards, 2014), UK production of WTR sludge was approximated to be 131,000 tonnes (of 'dry' solids, approximately 655,000 wet tonnes), 44% of which is Al based, 32% Fe sludge, and the remaining 24% of sludge attributed to 'other formats'. 75,980 tonnes (60%) of produced sludge was disposed of via landfill, 37,990 tonnes (28%) to sewage works for further treatment and the remaining 17,003 tonnes (12%) disposed of via 'minor' disposal routes such as spreading to agricultural land, use as soil conditioners, and use in brick and cement production (Water UK Standards, 2014). The greatest challenge to the water industry, according to the Water UK standards report, is the treatment and disposal of the ~100,000 tonnes of coagulant based sludges produced per year, a figure which has likely increased in the four years since the publication was released. All countries in the EU must comply with the 1998 EU Drinking Water Directive (98/83/EC), under which the Drinking Water Inspectorate (DWI), a UK independent body that was formed in 1990, regulates each regional water company in England, Wales and N. Ireland (the Drinking Water Quality Regulator oversees Scotland) to ensure that companies abide by the rules and regulations stated by the EU directive.

WTR cannot be sent for biological digestion or incineration due to low calorific value, in contrast to sludges produced by wastewater works (sewage) for which this is common practise (Ulmert & Sarner, 2005). The potential high concentration of PTEs and metals in the WTR as well as the high water content further limits disposal methods. To discard the waste from water treatment plants in an environmentally appropriate way, potentially toxic constituents of WTR must be identified before disposal and be compared to the lowest acceptable presence or concentration of a substance, e.g. arsenic as defined by local legal standards.

In the UK prior to 1960 there were very few regulatory constraints on WTR therefore it was typically discharged into local water bodies as a return to source (Elliot *et al.*, 1990), stored in artificial lagoons or spread directly onto land, which could potentially lead to negative environmental and aesthetic impacts such as discoloration of local water sources and turbidity. The physical properties of the

WTR specific to each plant define how the responsible producers must process the waste. Currently there are a number of options currently under research for environmentally sound alternatives for the disposal of WTR via landfill, which include disposal on land as soil amendment (requires a special licence from the Environment Agency) or co-amendment with wastewater biosolids (sewage sludge) and organic matter (discussed extensively in section 3.3), discharge to a wastewater collection system, or reuse in building and fill materials. As they are the easiest forms of disposal, landfill and land spreading are the options typically chosen (L. Dennis, pers comms, 2015).

WTR is tested to determine how it may be disposed of using leachate tests, in which WTR is exposed to a mildly acidic solution similar to what may be found in municipal waste plants and the resulting leachate exposure is tested for the concentration of toxic elements, according to the waste acceptance procedures outlined by The Landfill Directive. The majority of WTRs tested in this way are non-hazardous, therefore landfill is an appropriate (but not sustainable) method for disposal. WTR disposed of via landfill is typically transported from the water treatment plant in haulage trucks; as the transport and landfill fees are charged per mass, WTRs are dewatered to the minimal volume that is economical (Keeley *et al.*, 2014).

Landfill is regulated by the Landfill Directive (Council Directive 1999/31/EC) and has three classifications; hazardous waste, non-hazardous waste and inert waste. Landfill had previously been recognised as the 'best practicable environmental option' for the disposal of particular waste types, including WTR, however the implementation of landfill tax in 1996 as a deliberate policy intervention to find more sustainable reuse of waste has made this an ever decreasingly economical option; since 1996 the tax has increased from a standard rate of £7 per tonne to £82.60 per tonne from April 2015. Lower risk wastes, i.e. 'naturally occurring materials' determined by the Waste Framework Directive (2008/98/EC) and the Landfill Tax Order, have a tax of only £2.60, usually determined by the LOI test where wastes <10% qualify for the lower band of tax. WTRs are considered an inert waste and are charged as per the lower band. Considering that the UK produces approximately 655,000 tonnes of WTR per year, this would raise an

annual disposal cost of £2 million should it all be disposed of via landfill. The limitations for the addition of waste to landfill are as follows according to the Council Decision (2003/31/EC). For waste to be accepted for landfill they must comply with the limits outlined in Table 8.

The increasing levies on landfilling enforced by the government have forced water companies to investigate alternative disposal strategies (Keeley *et al.*, 2014). Since 2008 the Environmental Permitting Regulations have monitored waste management activities, however these are now called Environment Agency standard rules (2016 [18]). The Standard Rules regulate the quantity of waste allowed to be applied during one year, where WTR is classified as 'sludges from water clarification' (List B: waste code 190902), under the general bracket of recover or use of waste on land. Standard rules 2010 No 4 (mobile plant for land-spreading), No 5 (mobile plant for reclamation, restoration or improvement of land), No 11 (treatment to produce aggregate or construction materials) and No 12 (treatment of waste to produce soil, soil substitutes and aggregate) all allow WTR to be applied to land with a number of limitations. Table 8 highlights the typical values for WTRs in the UK, Mosswood specific details and a comparison with soils, and biosolids. These values are assessed against limits for land application, based on two directives, the Sludge directive (and the BSI PAS 100 specification (which sets a minimum compost quality baseline for biowastes) as currently there is no particular directive for water treatment sludge.

Briefly, these detail that the application is not permitted in the following areas: within groundwater source protection zone 1, 10 m of a watercourse, 50 m from any spring, well, or any borehole used to supply water for domestic or food production purposes, nor 50 m from any well, spring or from any borehole used for the supply of water for human consumption. The application is also not permitted within 250 m of the presence of Great Crested Newts, where it is linked to the breeding ponds of the newts by good habitat, nor within 50 m of a site that has relevant species or habitats protected under the Biodiversity Action Plan that the Environment Agency considers at risk to this activity, nor within 50 m from a National Nature Reserve (NNR), Local Nature Reserves (LNR), Local Wildlife Site (LWS), Ancient woodland or Scheduled Ancient Monument.

<b>WTR</b>	<b>Cd</b> (mg/kg)	<b>Cr</b> (mg/kg)	<b>Cu</b> (mg/kg)	<b>Hg</b> (mg/kg)	<b>Ni</b> (mg/kg)	<b>Pb</b> (mg/kg)	<b>Zn</b> (mg/kg)
<b>Mosswood</b>	<u>2.36</u> ± <u>1.42</u>	31.5 ± 7.2	27 ± 8	0.28 ± 0.25	<u>92 ± 35</u>	85 ± 72	<u>665</u> ± <u>405</u>
<b>WTR range</b>	0.2 - 3.6	7.8 - 39	7.9 - 36	0.06 - 1.4	10 - 120	4 - 160	28 - 1100
<b>Soil range</b>	0.06 - 1.1	7 - 221	6 - 80	0.02 - 0.41	4 - 55	10 - 84	17 - 125
<b>Biosolids</b> <b>typical</b>	0.1 - 13.6	28 - 509	25 - 2481	0.1 - 2.0	2.6 - 389	8.1 - 850	32 - 2070
<b>PAS-100</b> <b>regs</b>	1.5	100	200	1	50	200	400
<b>Biosolids</b> <b>regs</b>	20	/	1000	16	300	750	2500

*Table 8: Concentration of PTE in WTRs as derived by Finlay (2015), Elliot (1990), McBride (1994), BSIPAS-100 specification, and Sewage Sludge Directive 86/278/EEC. Underlined = exceedance of PAS-100 regulations*

The main concerns for application on land are due to concerns over aluminium based coagulant sludges becoming mobile at pH <5, and deficiencies in P as a result of either Fe or Al based WTR application which stunts plant growth and causes induced phosphate deficiency in plants (see section 3.2.3). Before the application of waste to land, it must be analysed thoroughly to indicate all substances that may be reasonably expected to be present, i.e. evidence of potential nutrients, and whether the sludge presents any agricultural benefit to where it's being applied, in addition to any potential contaminants that may harm the land. Evidence must also be provided that application of waste will not cause the build up of PTEs (potentially toxic elements) to harmful levels. Table 9 presents the potential benefits and negative impacts of land spreading of WTR according to the Natural Resource Wales (2017 [19]) advice on the Standard Rules. Currently there is a significant body of research on the effect of adding WTR to soil (discussed in section 3.3), however concerns over the long-term effects of land application have meant that this method of disposal is still limited.

<i>Potential benefits</i>	<i>Potential negative impacts</i>
Nitrogen and potassium source from bacterial cell debris	Iron staining on crops
Precipitated phosphate	Elevated aluminium levels in soil
Source of secondary plant nutrient, sulphur	Phosphorous availability limitation at extremes of pH (optimum between pH 6 and 7)
May contain trace elements e.g. Mg	At low pH Al, Mg, Mn, Fe and Zn become more available and may induce plant toxicity.
May contain a significant amount of organic matter (is raw water source is from peaty soil)	Damage to roots through cell division inhibition and reduction in transport of P from roots to shoots.
Sludge may provide sand and fine grit for the improvement of heavy soils, and also adding body to medium and lighter soils	
Some sludges can providing a liming benefit	

*Table 9: Standard rules summary of positive and negative impacts of land spreading of WTR (Natural Resources Wales, 2017).*

### **3.1.3 Northumbrian Water WTR disposal**

The following information has been obtained via personal communication with Luke Dennis and Ed Higgins. Table 10 below presents a summary of WTR produced by Northumbrian Water between 2010 and 2018 (includes Essex and Suffolk production), which highlights that all WTR is disposed of via land spreading with application rates of up to 120 tonnes per hectare (in non-nitrate vulnerable zones). Considering that Northumbrian Water only produces 4.7% of clean water in the UK, total annual figures may total around 1.49 million wet tonnes (around 300,000 total dry solids), which is almost double the 131,000 total dry solids estimated in 2014 by the UK Water Standards (2014). For biosolids the application rate tends to be about 17 tonnes/Ha (and will be lower in Nitrate Vulnerable Zones) whereas WTR, due to its much lower nutrient levels, can be

applied up to 120 tonnes/Ha. This value is up to the discretion of the Environment Agency (EA) and if necessary, the EA can request only 80 or even 60 tonnes/Ha is applied.

<b>WTR type</b>	<b>Total WTR produced (wet tonnes)</b>	<b>Ferric-WTR (%)</b>	<b>Alum-WTR (%)</b>	<b>Alum/Ferric mix-WTR (%)</b>	<b>Disposal route</b>
2010-11	69666	80	13.5	6.5	
2011-12	64871	78.4	13.9	7.7	
2012-13	62871	76.4	16	7.6	Recycled to land
2013-14	55966	72.1	14.7	13.2	
2014-15	61235	67.2	17.8	15	
2014-15	73331		Total dry solids 13973		WTR to
2015-16	78253		Total dry solids 14610		agriculture
2016-17	75835		Total dry solids 13411		under permits
2017-2018	78599		Total dry solids 14488		Waste recovery permit

*Table 10: WTR production figures from Northumbrian Water Ltd, 2010 – 2015 (Finlay, 2015 via pers comm L Dennis, NWL), and WTR production figures from 2014-2018 from Greenhouse Gas Data Returns, which include Essex and Suffolk values (pers comms E. Higgins, NWL)*

Almost all WTR produced by NW is recycled to land under the Environmental Agency Standard Rules, however other companies do not recycle to the same extent and rely on landfill. Some WTR went to different outlets in 2015 (reasons undisclosed), but in general NW avoid landfill due to its vast expense and instead it is taken to sewage treatment plants. For land application, their contractor has a standard rules permit and then applies to the EA to carry out a deployment on a particular farm, where the WTR is incorporated into the soil. The WTR is analysed for an extensive range of values such as PTEs, nutrients, dry and organic matter; a range which is actually a more extensive list than the biosolids one but does not require analysis or microbiological determinants. A qualified expert has to explain to the EA why the WTR is of benefit to the land and compare the sludge to a suitable set of limits. The comparison might be with the Sludge Regulations or with the PAS100 list (see Table 8) but either way, the WTR sludge cannot exceed the

limits. If the application rate is sufficiently low that fewer PTEs are applied to the field and the mean concentration is then within limits, then contractors may discuss this with the EA to allow spreading of WTR exceeding these thresholds. The field soil is also analysed as above but interestingly, the EA very rarely asks for this analysis and concentrates far more on what is in the WTR. The deployment for each farm lasts for one year therefore analysis must be conducted annually (however NWL analyse on a quarterly basis for operational reasons). The EA have it within their discretion to ask NWL to lower the application rate or they may even disallow the deployment.

There are a number of implications for WTR's use as a resource, despite currently being successfully applied to land as a disposal mechanism. Firstly, as the treatment of drinking water is designed to remove PTEs and undesirable substances, these substances may be concentrated in the residual, whereby the concentration fluctuates depending on the composition of raw water used at the time. Some WTRs may therefore exceed the maximum threshold of BSIPAS100 or EU Biosolids regulations, preventing them from use in applications such as land spreading. To be able to use the WTR for soil remediation or improvement, these values will need to be routinely monitored with careful consideration of mean concentration of PTEs (see 3.1.3). Secondly WTR exported from water processing plants has a very high water content, as a result of the cost/benefit trade-off between the costs to dewater the material at the treatment works against the cost of transporting and disposing of the material. It is currently uneconomical to dewater the WTR further to achieve a higher dry solids content to create a lower volume waste stream, despite growing expenses associated with disposal. It is critical therefore to evaluate the effect of water treatment residuals in a wet format, or to determine if further processing is required such as continued dewatering or drying of the WTR.

### 3.2 WTR characterisation

At the most basic level water treatment residual (WTR), is typically comprised of 50-60% FeOOH (iron oxide, where Fe makes up 59% of the mass of FeOOH) and ~40% natural organic matter, which is predominately exported as a sludge with 20% solids from water treatment plants. The typical physical and chemical properties of WTR presented below are were available compared with 'typical' soil values (although soils are exceptionally heterogeneous in their nature).

#### 3.2.1 Physical properties



*Figure 14: Photos of WTR, (a) freshly produced from the water treatment plant at Mosswood water treatment plant (b) oven-dried water treatment residual, clearly showing iron oxide (orange rust colour) precipitates.*

Basim (1999) states that WTRs have significantly different characteristics depending on the water treatment plants, and therefore it is difficult to give 'typical' values. Figure 14 shows the WTR in its raw 'wet' form, resembling a peaty soil/used coffee grinds (A), and oven dried WTR (B), which shows how the

material when dried becomes brittle, angular particles. The greatest influence on the variation in geotechnical characteristics is the water content, as it alters the floc structure, particle size of solids and ion concentration. Table 11 presents a range of ‘typical’ values from Crittenden *et al.*, (2012).

<b>Property</b>	<b>Units</b>	<b>Alum Sludge</b>	<b>Iron Sludge</b>
Total solids	%	0.1-4	0.25-3.5
Dry bulk density	g/cm <sup>3</sup>	1.2-1.5	1.2-1.8
Wet bulk density	g/cm <sup>3</sup>	1-1.1	1.05-1.2
Specific resistance (rate at which WTR can be dewatered)	mins/kg	10-50 x 10 <sup>-11</sup>	40-150 x 10 <sup>-11</sup>
Dynamic viscosity at 20 °C (resistance to tangential or shear stress)	N.s/m <sup>2</sup>	2-4 x 10 <sup>-3</sup>	2-4 x 10 <sup>-3</sup>
Initial settling velocity	m/h	2.2-2.5	1-5
pH		6-8	6-8
Al	%	15-40	-
Fe	%	-	4-21
Silicates and inert materials	%	35-70	35-70
Natural organic matter (NOM)	%	10-25	5-15

*Table 11: Typical physical properties and chemical constituents of alum and iron sludges from chemical precipitation (from Crittenden et al., 2012)*

WTR sludge in its raw form is the combination of fine dissolved and suspended solids found in the raw water source, metal hydroxides used in the coagulation phases and a large quantity of water trapped in the loose structure. The coagulants are attached to the suspended solids by electrostatic bonds that physically trap material and water and allow flocculation to occur. Water is therefore the limiting factor to how coagulated the sludge can become. Typically WTR is characterised by the parameters highlighted in Table 11, as the disposal methods and management of WTR is determined by these values (after Crittenden *et al.*, 2012). The physical characteristics of WTR summarised in Table 11 suggest what one might assume to be free flowing sludge, however as shown in Figure 14, freshly produced WTR ‘cake’ or ‘sludge’ has a texture not unlike soil, with a crumbly texture similar to a sandy loam soil. Without handling of the substance, it is difficult to see that it has ~80% water content and appears like used coffee grinds. Over time, presumably as

a function of the continued work of flocculants, water held within the structure is exuded from the clods, which forms irregularly shaped, denser material surrounded with water containing suspended solids that render it completely black.

O'Kelly (2008) performed geotechnical analysis of WTR and found that it had high plasticity, high compressibility and very low permeability, factors that were attributed to the coagulant bound water, high organic content and charge destabilisation within the flocs. Basim (1999) characterised WTR as plastic but unlike clays, WTRs lose all plasticity when dried and instead behave like granular materials. Proctor compaction testing methods are covered in section 4.3; the compaction characteristics vary depending on whether tests are conducted on WTRs that have gone from 'wet' to 'dry' or from 'dry' to 'wet' (Hseih & Raghu, 1997). Soils typically exhibit a one-peak compaction curve, where the soil becomes denser when compacted at higher water content to a point of 'optimum water content' at which the highest density is reached. In typical dry to wet testing, the WTR exhibits a typical compaction curve shown by soils (Figure 15), however in wet to dry tests, the WTR exhibits no optimal water content for maximum dry density and continues to increase in density as it dries (attributed to the loss of low density water, floc structure collapse and the process of cementation). Hseih & Raghu (1997) conclude that unless the solids content of the WTR is near to the optimum solids content of around 85% (water content at which the compaction test gives the highest density), it is very difficult to compact the residual.

Interestingly, after testing of wet to dry samples, Hseih & Raghu (1997) found that after submerging the samples for one week, the strong interparticle adhesion could not be broken down, similar to thixotropic characteristics shown by some soils that results from the reorientation of soil particles. This characteristic is attributed to the cementation occurring when soluble ions in floc water ( $\text{Ca}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ) are adsorbed in solid particles during drying and interparticle bonding occurs. Although thixotropic soils do not retain strength lost after drying, WTR have an increase in strength because of the reorientation (termed 'thixotropic hardening'), which is an irreversible process. As a result of differences in water

content, the shear strength of WTRs range between 70 kPa and 316.8 kPa, where higher solids content correlates with a higher shear strength.

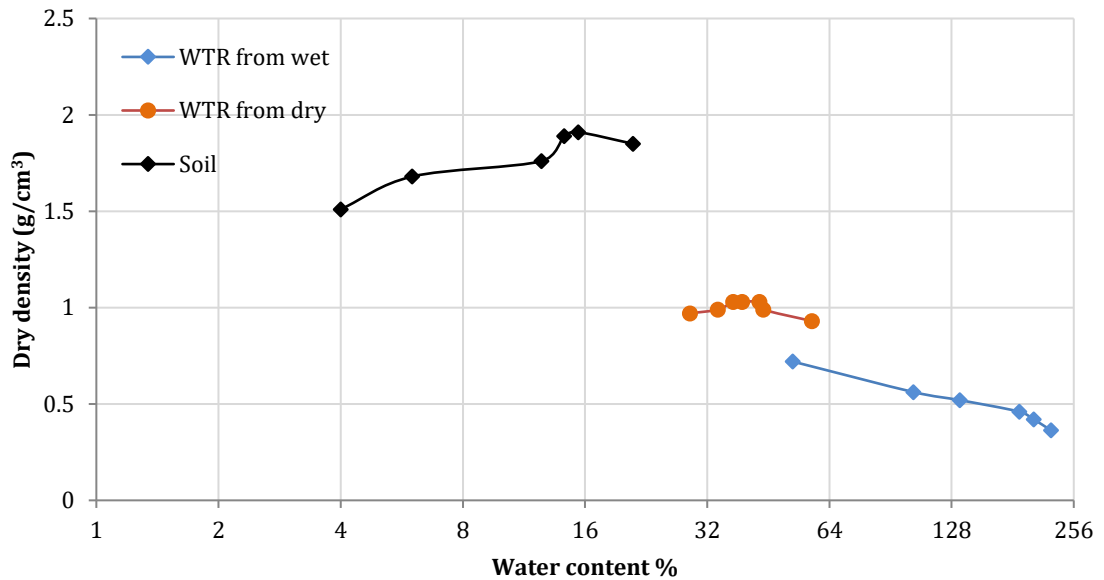


Figure 15: Comparison of WTR compaction curves from dry and wet sides for WTR (Hseih & Raghu, 1997), against the compaction curve of a typical loamy clay soil.

The particle density of WTRs range from 1.87 – 2.71 g/cm<sup>3</sup> (Basim, 1999; Hseih & Raghu, 1997), where the higher values are due to higher proportions of iron and lower values are from higher proportions of comparatively of low-density organic matter. Much like water in soils, water in WTR residuals can be classified into four categories:

1. Free water – surrounding residual flocs, freely moves by gravity.
2. Floc water – water trapped within voids of the flocs, can freely move within the floc but cannot be removed unless the floc structure is destroyed.
3. Capillary water – held by surface tension on the particles.
4. Adsorbed water – held within the colloidal solids and only removable at very high temperatures.

Hseih & Raghu (1997) also found that there was little difference in the determination of water content at room temperature (~24 °C), low-temperature oven (35-40 °C) and conventional oven drying at 105 °C, although there was no concern over the organic matter fraction removal at the high temperature. The solid phase of WTR consists mainly of particles ranging from 1 nm to 1 μm (clay sized fraction), which do not undergo any chemical reactions during the water treatment process (Bohn *et al.*, 1985), and organic materials (colloidal polymers).

Basim (1999) attributes the high plasticity, shrinkage, compressibility and low strength to the presence of organic matter. The two-sided effect of organic matter on the engineering properties of soil is well known (Chapter 2, section 2.2.1), as organic matter may both improve the aggregate stability through water repellency and bonding but may also work to the detriment of a soils shear properties.

There have been numerous studies that have used WTR in its various formats (dried, dewatered or wet) as a soil substitute or for soil amendment. For example Dayton & Basta (2001) tested the potential for 17 WTRs (from the US) that had been air-dried and crushed to 2 mm to be used as a soil substitute for the growth of tomatoes. The WTRs were all considered sufficient for crop growth in terms of nutrient provision, but due to phosphorous immobilisation the yield was poor. Similarly, dewatered WTRs were used by Basta *et al*, (2000) due to their physical and chemical characteristics similar to fine textured soils (DeWolfe, 2006). Rengasamy *et al*. (1980) and Scambilis (1977) found that adding wet sludge altered the mechanical properties of soil by increasing aggregation and cohesion within the samples with application rates of up to 2 tonnes/ha (10,000m<sup>2</sup>). Gharaibeh (2009) has provided a thorough review of the drying process, which provides the characteristics of WTR through a series of drying experiments on ferric residuals, conducted because of the need to optimise or accelerate drying time for WTRs for operational reasons in treatment plants. However, there are no known publications that have directly compared the potential effects of WTR at different moisture contents for various soil parameters.

### **3.2.2 Chemical Properties**

As discussed previously there are two main types of WTR, one produced as a bi-product of water treatment using Al based coagulants and the other Fe based. There are a number of treatment plants in North of the UK that use Al, Fe, or a mixture of the two for their production. Table 12 presents a physiochemical comparison of WTR properties analysed in 2011 from nine plants in NE England (Finlay, 2015). Due to different source water in each location and particular processes in each individual treatment plant, there is a broad range of property values. These would likely change throughout the year, where the concentration of contaminants is dependent to some degree on the volume and movement of

precipitation preceding water collection. Typically WTRs have a very high water content despite their peaty soil like appearance, where solids comprise only 20-30% of the mass. The Fe content varies between 31% and 37% in iron-based residuals, where Al content varies between 15% and 18% for Al based residuals. WTR from Horsley combined Al & Fe treatment has 6.1% Al and 14% Fe content.

WTR	Coag type	WTR form	GWC %	pH	EC ( $\mu\text{S}/\text{cm}$ )	Al %	Fe %	LOI <sub>550</sub> (%)	Total C (%)	Mn (mg/kg)
Moss wood	Fe	Sludge	78 $\pm$ 1.6	4.7 $\pm$ 0.5	239 $\pm$ 168	0.28	31	48 $\pm$ 2.7	21.4 $\pm$ 2.2	1825 $\pm$ 665
WTR	Fe/Al	Sludge or cake	75 - 85.2	4.1 - 7.2	39 - 405	0.21 - 21	0.8 - 41	36 - 70	13 - 26	370 - 5100
Soils	n/a	n/a	2-50	5 - 8	<4000	7.1	4	5	1.5 - 35	80 - 1300

*Table 12: Data on the chemical and physical parameters of WTR obtained across various locations in the NE of England (WTR range) compares with specific Mosswood data and typical soil values. Obtained from research conducted November 2011 on WTR retrieved from 9 different water treatment plants (Finlay, 2015), soil typical from Brady & Weil (2016) in Dayton & Basta (2001).*

Loss on ignition testing (LOI) in this case overestimates the organic matter content present in the WTR, as in addition to the burning of organic material, dehydration of metal oxides and clays also occurs between 105 °C and 550 °C, therefore LOI can only be used as an approximate measure. Total carbon provides a good representation of organic C content in WTRs, and the use of Thermo TOC1200 found <0.1% inorganic C. Mosswood WTR, used in this research, contains roughly 31% FeOOH & water (where FeOOH accounts for 50% and water for 8%) and 40% NOM.

The conductivity of WTR is relatively low, suggesting that despite the high concentration of metal ions (iron, aluminium and manganese) these are not in a soluble form (Dayton & Basta, 2001; Nagar, 2009). All the WTRs tested by Finlay (2015) were acidic, ranging from pH 4.1-7.2 attributed to the coagulants and

organic matter. Aluminium coagulant is required in smaller doses for the same particulate removal than iron coagulant, explaining the discrepancy between quantity in the end product (Crittenden *et al.*, 2005). The Fe and Al present in WTR are mostly amorphous hydrous metal oxides (Elliot, 1990).

Iron oxides comprise the majority of WTR, but importantly are also common constituents of soils (Cornell & Schwertmann, 2003), and play extremely important roles in the chemistry of soils (e.g. sorption, redox, aggregation, plant nutrients, pedogenesis and absorbent properties) due to high specific areas and reactivity (Sparks, 2003). Iron oxides are able to influence soil properties even at low concentrations, where the typical range for the majority of soils varies between <1 and 100's of g/kg. As discussed in Chapter 2, the fundamental role of organic matter and Fe in the formation and stabilisation of aggregates is well known (Arias *et al.*, 1999). Iron oxides and OM are the most important constituents in soil affecting phosphorous reactions and the rate of adsorption/desorption (Fink *et al.*, 2016) affect the reactions and rate. The positive effect of organic carbon on the water retention characteristics of a soil are well studied, where compost is commonly stated as being able to hold 10 times its own weight in water (Hudson 1994; Rawls *et al.*, 2004), although there is debate on the opposing effects of organic matter to either increase aggregate stability (Bartoli *et al.*, 1992) or favouring dispersion (Arduino *et al.*, 1989). Aggregation is key for soil structural properties including porosity (a measure of the volume of pore to the volume of soil material), which has a fundamental role in the water holding capacity of a soil. In addition, aggregation plays a key role in the shear strength and erosional resistance of soils to erosional forces such as overland flow.

### **3.2.3 Nutrient values**

The nutrient values and potentials of WTR have been at the forefront of WTR research to date. The excessive application of organic amendments as method to increase yield on agricultural land has negative environmental impacts. The contamination of ground and surface water with excess nitrogen and phosphorus is a key problem associated with organic rich amendments, leading to eutrophication of rivers and lakes (Sharpley *et al.*, 2001). Phosphorus and heavy metal mobility in soils are also of environmental significance as they pose threats

to both humans and animals. As such, WTR has been used as a low-cost co-amendment with biosolids to control nutrient rich leachates and runoff resulting from the application of organic matter, due to its ability to sorb phosphorous (Agyin-Birikorang *et al.*, 2007; Ahmed *et al.*, 2012; Basta *et al.*, 2003; Bayley *et al.*, 2008; Dayton *et al.*, 2003; Dayton & Basta, 2005; Eaton & Sims, 2003; Elliot *et al.*, 2002; Gallimore *et al.*, 1999; Ippolito & Barbarick, 2006; Jacobs & Teppen, 2001; Makris *et al.*, 2005, Makris *et al.*, 2004, Mahdy *et al.*, 2013; O'Connor *et al.*, 2002; Staats *et al.*, 2004;). There are numerous studies on this application of WTR owing to the economic benefit of recycling the material in addition to biosolids (Athamenh *et al.*, 2015) and success in nullifying issues of surplus P and N in agricultural settings where a range of organic matter has been applied, although some report P deficiencies at higher application rates and concerns around potential elevated Al-phytotoxicity after the application of Al based WTRs (Agyin-Birikorang *et al.*, 2007; Bai *et al.*, 2014; Basta *et al.*, 2003; Bayley *et al.*, 2008; Busalacchi, 2012; Cox *et al.*, 1997; Dayton *et al.*, 2003; Dayton & Basta, 2001; Elliot *et al.*, 2002; Gallimore *et al.*, 1999; Ippolito *et al.*, 2009; Tvergyak *et al.*, 2012; Peters & Basta, 1996; O'Connor & Elliot 2003; Wendling *et al.*, 2013). Much of this literature is revolved around the nutrient behaviours of soils due to co-amendment, but there are no known publications that have compare the effects of WTR and organic amendment with a specific focus on the water retention properties or soil structure, perhaps as a result of the well-known effects of the component parts of WTR and organic matter on soil properties such as aggregation.

Nitrogen, phosphorus, potassium and nitrogen are four of the most important nutrients required by plants as these support essential plant functions such as creation of amino acids for proteins, DNA and cell membranes, enzymes for respiration and photosynthesis and production of chlorophyll (Epstein, 1972; Gurevitch *et al.*, 2002; Hewitt & Smith, 1974; Sahrawat, 2006). Therefore the concentration of these substances in WTR is important if WTR is to be used as a soil amendment or substitute and must be considered when using it as a co-amendment. In Table 13, WTR's total N is slightly higher than found in soil (0.5-1.1% compared to 0.5). The ratio of C:N is important as at a ratio of 25:1, mineralisation and immobilisation of N, i.e. the microbial transformation of organic

N to inorganic N and vice versa, is in balance. At ratios >30(C):1(N), there is a risk of N depletion due to rapid increase in plant biomass as a result of C availability and lack of plant available N (Pierzynski *et al.*, 2005). The range of WTRs is between 15.5 and 39, meaning that it may not always provide a good source of N should the lower end of the range be reached. Overall the concentrations of P, K, and Mg are deficient when compared to soils, therefore added to the soil as a single amendment they are likely to cause P and Mg deficiencies and will need sufficient dilution with organic matter or a very low application rate.

<b>WTR</b>	<b>Total N</b> (%)	<b>C:N</b>	<b>P</b> (mg/kg)	<b>K</b> (mg/kg)	<b>Mg</b> (mg/kg)
<b>Mosswood</b>	0.8 ± 0.1	27 ± 3.0	472 ± 175	833 ± 565	335 ± 183
<b>WTR range</b>	0.51 - 1.1	15.5 - 39	4.0 - 1528	170 - 3900	170 - 2900
<b>Soil typical*</b>	0.2-5	10	1000	640	
<b>Compost typical</b>	1.2	14-20	3000	4000	3000
<b>Biosolids typical</b>	4	10	25000	3000	2000

*Table 13: Concentrations of nutrients in WTR compared to typical soil, compost and biosolids values (Dayton & Basta, 2001; Elliot, 1990; Finlay, 2015; Rowell, 2004)*

### 3.2.4 Potentially toxic elements (PTEs)

The largest component of Fe based WTR is considered to be hydrous ferric hydroxide or amorphous iron oxide (Ippolito *et al.*, 2011), and as such previous studies have shown that due to this chemical composition, WTR can be used as a sorbent of PTEs in contaminated land including arsenic, mercury, cadmium, copper, lead and zinc and the sorption of other undesirable substances in water courses, such as antibiotics (Brown *et al.*, 2012; Fan *et al.*, 2011; Finlay, 2015; Makris *et al.*, 2006; Nagar *et al.*, 2015; Punamiya *et al.*, 2013; Wang *et al.*, 2018; Zhao *et al.*, 2015). An example of the use of WTR to regenerate brownfield sites is the ROBUST project at Durham University (2009-2014), and readers are directed to Finlay (2015) for a discussion of the potential of WTR for this application.

However, as WTR is the by-product of the removal of substances potentially harmful to health, it is important to note that WTR may also contain PTEs if these contaminants are present in the catchment, which may limit their suitability for

land application. Table 8 (from section 3.1.2, displayed again on page 91,) provides concentration values of a number of PTEs present in WTR in comparison to the PAS100 and biosolids (Directive 86/278/EEC) regulations. Substances that are within the PAS100 threshold are allowed to be spread across land without the need of an environmental permit, however those exceeding PAS100 thresholds must be disposed of in a different way. WTRs that did not fall below the threshold concentration for PAS100 of a particular substance are underlined, however it can be seen that the majority of WTRs have low concentrations of PTEs in comparison to typical biosolids and in some cases low in comparison to soil e.g. Cr.

<b>WTR</b>	<b>Cd</b> (mg/kg)	<b>Cr</b> (mg/kg)	<b>Cu</b> (mg/kg)	<b>Hg</b> (mg/kg)	<b>Ni</b> (mg/kg)	<b>Pb</b> (mg/kg)	<b>Zn</b> (mg/kg)
<b>Mosswood</b>	<u>2.36</u> ± <u>1.42</u>	31.5 ± 7.2	27 ± 8	0.28 ± 0.25	<u>92 ± 35</u>	85 ± 72	<u>665</u> ± <u>405</u>
<b>WTR range</b>	0.2 - 3.6	7.8 - 39	7.9 - 36	0.06 - 1.4	10 - 120	4 - 160	28 - 1100
<b>Soil range</b>	0.06 - 1.1	7 - 221	6 - 80	0.02 - 0.41	4 - 55	10 - 84	17 - 125
<b>Biosolids typical</b>	0.1 - 13.6	28 - 509	25 - 2481	0.1 - 2.0	2.6 - 389	8.1 - 850	32 - 2070
<b>PAS-100 regs</b>	1.5	100	200	1	50	200	400
<b>Biosolids regs</b>	20	/	1000	16	300	750	2500

*Table 8: Concentration of PTE in WTRs as derived by Finlay, 2015, Elliot (1990), McBride (1994), BSIPAS-100 specification, and Sewage Sludge Directive 86/278/EEC. Underlined = exceedance of PAS-100 regulations*

The focus of this research is on the WTR produced from the use of Fe based coagulant, specifically from Mosswood water treatment works. The site, located at NZ0650, next to Derwent Reservoir, which collects water from a catchment of 110km<sup>2</sup> and provides a daily yield of 112,320 m<sup>3</sup> per day [NWL, 20]. Water coming into the treatment plant may vary over time due to fluctuations in climate or land use practises that affect the chemistry and physical properties of water, therefore there are changes in the physiochemical properties of WTR over time. This means that any results obtained for experiments using Mosswood derived WTR may not be wholly comparable to WTR used from a different time or place.

### 3.3 WTR as a resource

#### 3.3.1 Using WTR as a single amendment to soil or as a soil substitute

Although much literature has discussed the use of WTR to reduce the excessive addition of P and N to groundwater and surface water sources resulting from the application of biosolids or other organic matter, relatively there is much less literature on the merits of WTR as a single amendment soil conditioner or as a soil substitute. Land based applications have historically centred around using sludge as a substitute for agricultural limestone but increasing attention has been paid to land application as a sustainable disposal mechanism, as already employed by NW (Basta, *et al.*, 2000, Titshall & Hughes, 2005), due to the similarities in geochemical properties of WTR and soil (Elliot & Dempsey, 1991).

Soil conditioning (treatment that modifies a soil's properties to improve crop growth) resulting from the input of organic matter is an advantage of the application of WTR and although Elliot & Dempsey (1991) suggest that the effects are quite small, there is a consensus that the use of WTR as a soil conditioner is generally beneficial to soil, with the exception of potential P depletion causing reducing in crop yields. For a residual to be considered as a substitute for soil it must imitate soil and/ or maintain soil quality defined as "*the capacity of a soil to function, within an ecosystem and land-use boundaries, to sustain biological productivity, maintain environmental quality and promote plant and animal health*" (Doran & Parkin, 1996) and similarly "*to perform its three principal functions (economic productivity, environmental regulation and aesthetic and cultural values)*" by Lal (1993).

There has been a handful of research papers that considers the single application of WTR or use of WTR as a soil replacement, with conflicting results in respect to phosphorus and crop growth; Heil & Barbarick (1989) found that WTRs at >15 g/kg reduced the yield of sorghum-sundangrass and similarly Skene *et al.*, (1995) experienced decreased growth of broad beans at 20 g/kg even with the use of fertilizer. Basta *et al.*, (2000) also evaluated the use of WTR as a soil substitute by measuring the growth of bermudagrass, and showed that mean yields were

dependant on the type of WTR used, where both tissue P concentrations and available P were below adequate for two out of three WTRs tested.

Similarly, Dayton & Basta (2001) characterised WTR for use as a soil substitute and found that high application rates (>10%) have caused P deficiency in crops. However, Oladeji *et al.* (2009) suggest that excessive application of WTR could deplete plant available P and cause Al phytotoxicity when considering Al based residuals, however even with an application rate of 25% single amendment or up to 10% in a 2 year field study, there were no effects on the yield of grass nor the phytotoxicity. Silveira *et al.* (2013) that found that incorporation of WTR caused an 11% yield suppression compared to surface application, but in general there were no adverse effects of up to 70 Mg ha<sup>-1</sup>. Elliot (1990) however found that 20-100 g/kg enhanced tomato growth due to a liming effect (5.3-8.0 pH change). Beneficial use of WTR therefore in a geochemical perspective is heavily dependent on the application rate, where careful monitoring is needed of both the constituents of the WTR to be spread, and the effect on the receiving soil.

Most of the literature on WTR looks at whether it can improve crop yields and this literature is not covered in detail here. The reader is referred to Ippolito *et al.*, (2011) which provide a brief summary on this topic. While some most studies have reported the effects of amendment of soil WTR from a purely geochemical perspective, some research has mentioned positive improvements in soil qualities, such as water retention and pH (Basta *et al.*, 2000; Bugbee & Frink, 1985; Owen, 2002; Pecku *et al.*, 2005; Rengasamy *et al.*, 1980;), although none have these as a primary objective of study. Bugbee & Frink (1985) used WTR amendment up to 670 g/kg, which yielded increases in water holding capacity and aeration (although these values are not quantified). They also found that the increase in productivity as a result of improved soil structure was sufficient to offset P deficiencies. Owen (2002) applied ferric WTR to agricultural land and suggested it was beneficial to grassland and livestock. They found that the most economical way of spreading sludge (accounting for all costs including centrifuging) is using 4% dry solids directly onto land at £4.50/m<sup>3</sup> (which is £107/tonne of dry solids).

Moodley *et al.* (2004) and subsequently Moodley & Hughes (2006) used WTR as a single amendment to soil and evaluated the changes in water retention, hydraulic conductivity and evaporation at the field scale. Moodley *et al.* (2004) found that differences from the control plot were only measurable after 2 years and only at the highest application rate of 1.3 tonnes/ha were water retention and hydraulic conductivity improved, and hence suggest that very high application rates of WTR are required to observe differences in the physical properties of soils). At application rates of up to 1.3 tonnes/ha, they reported that WTR incorporated to a depth of 20 cm increased the saturated hydraulic conductivity of soils linearly with application rate, and increased total porosity due to reduction in bulk density. An increase water holding capacity was attributed to the performance of polymers in the WTR in aggregation of the soil, where WTR amended soil increases the water held at a particular matric potential, although the change in retention properties were suggested to be affected by the WTR properties itself rather than an interaction with the soil. The water loss from WTR amended soil was less than a control soil which echoes findings from Kemper *et al.* (1994), where evaporation was reduced by 10-20%. This finding was attributed to the addition of coarse WTR grains which reduce the capillary action at low matric potentials and therefore lower the unsaturated hydraulic conductivity. Moodley & Hughes (2006) also suggest that aggregates of WTR have high stability and a limited potential for swelling due to the binding effects of polymers.

### **3.3.2 Recycling waste**

Moves to reuse many different types of waste over the past decade have meant that the investigation into the potential benefits of waste is ever growing. This comes as a result of increasing costs for disposal and concerns over sustainability; DEFRA's 2010-2015 policy paper on waste and recycling stated that the UK generates 177 million tonnes of waste annually, which is both a poor use of resources and has detrimental effects on business and household economies. This includes industrial waste products, where the EU directive aims to reduce the dependence on landfill for disposal and increase the recycling of wastes. In a report on UK statistics on waste generation (DEFRA, 2018 [21]), mineral waste (39%, 79 million tonnes) and soils (26.7%, 54.2 million tonnes) were the largest contributors in 2014 (from a total of 202.8 million tonnes in 2014), compared to

just 10% by households. Of the waste that is deposited into landfill, mineral wastes only account for 7%, however soils account for 45% of the total material. Mineral wastes account for 50% of the recycled and other recovery treatment method total, where soils only account for 10%. These figures go some way to showing that there is increasing recycling of natural materials, but that it is not yet sufficiently sustainable.

The cost of processing and disposing of WTR accounts for a significant proportion of the operating costs of water treatment works, the cost of which increases each year because of increasingly stringent disposal regulations (Babatunde & Zhao, 2007) therefore the disposal of residuals is a formidable challenge (Hseih & Raghu, 1997). As outlined in the introductory chapter, the reduction in soil quality worldwide is a major issue and the remediation of soils is key on the agenda (Tvergyak, 2012). Simultaneously there is a need to close the loop between sustainable sludge management and water treatment and as such there are increasing attempts to find beneficial uses for WTR (Babatunde & Zhao, 2007). The potential for using a waste material as a sustainable method with which to improve soil has been identified for the immobilisation of PTEs (McCann *et al.*, 2018) and phosphorous (Ippolito *et al.*, 2011), however there are few studies that have explored the effect of WTR amendment on the water retention and hydraulic conductivity of soil, and none that have explored the effect of WTR amendment on the water retention characteristics and shear strength of soils. The exploration for WTR's use in soil for structural and water holding improvements is, for lack of a better word, exciting. Being able not only to recycle a waste, but also use it to benefit the function of soil may change the value of WTR and indeed Goldbold *et al.*, (2003) and Rensburg & Morgenthal (2003) have advocated that WTR could offer one of the greatest commercial potential for reuse.

### **3.4 Concluding remarks**

The increasing generation of WTR remains an inevitable prospect as our demand for clean water grows globally, and therefore the sustainable disposal of increasing volumes of sludge is an ever-growing concern. While small-scale land application is sufficient for the time being, further research is needed into the whole scale effect of WTR application on land. To date much focus has been ensuring that the

application of WTR isn't detrimental to soil (as a single amendment or with the application of biosolids), or on its capacity to return soils to acceptable concentrations of P and PTEs. A critical factor in the effective reuse of WTR from water treatment works is legislation (Babatunde & Zhao, 2007) and although companies currently follow guidelines for disposal there are no WTR specific legislations.

There is no question that the reuse of WTR provides a unique and sustainable opportunity for water treatment companies from an economic standpoint (Godbold *et al.*, 2003) and to benefit contaminated land. This thesis questions the opportunity for WTR to be used as a beneficial material for the amendment of soil to improve water holding characteristics, including hydraulic conductivity and to improve soil structure and shear strength in order to make soils more resistant to soil erosion and degradation, rather than exploring land application as just a method for disposal. This in turn provides an economical, chemical and physical benefit to the recycling of WTR into soil, in addition to addressing soil erosion and flooding issues that are of critical concern.

#### 4. Measuring Change in Soils

The following chapter discusses the methodology behind a novel approach to quantifying soils with a focus on what parameters are important for soils to function effectively during flooding events, i.e. its 'flood holding capacity'. This chapter will outline the technical aspects of collecting reproducible and reliable data for this thesis. The first part of the chapter introduces the materials used in this thesis, soil from St Anthony Lead (Soil 1) and Nafferton Farm (Soil 2), Water Treatment Residual (WTR) from Honey Hill (WTR1) and Mosswood (WTR2), compost and silica, with an outline of how these materials were prepared, stored and analysed. The subsequent sections outline the development and refinement of methods to make and test 'cores' for flood holding capacity (Trials 1-4). Three trial studies were conducted in order to develop the processes and methods, before a full-scale experiment was completed, ensuring that these were the appropriate tests to produce robust data that was both valid and most importantly reproducible in other laboratory or field settings.

Chapter 2 discussed the relationship of water to various physical and chemical properties of soil, and concluded that there are currently flaws in the measurement of water in soils. The new methodology described in this chapter addresses some of these issues, thereby providing a method to quantifiably measure soils in a way that encompasses the stresses undergone during flooding. The methods used have been developed using British Standards where applicable, with novel additions where these are not available. The remainder of the chapter outlines other testing undertaken on the samples from Trial 4 including soil erosion tests, shear testing, and hydraulic conductivity testing. A further method to assess soil, X-ray computed tomography (XRCT) has also been used in this thesis but owing to its complexity and novelty in the field of science, a full discussion of methods and results are contained within its own Chapter 6. To the author's knowledge there have been no studies that have used the methods outline in Trial 4 to wholly quantify soils in respect to their maximum water holding capacity, whilst including the shear strength and erosional resistance, volume change and hydraulic conductivity as a function to which 'flood holding capacity' is attributed. In order to provide clarity in terms used hereafter in the chapter and remaining

thesis, the following list of definitions describes what is meant by each term or the acronym for each term.

*AMENDED* – An amended soil contains compost, WTR and or silica in varying degrees in addition to an original soil.

*AMENDMENTS* – The addition of compost or WTR or silica to soil is an amendment of the original soil (S1 or S2).

*COMPOST* – Decomposed organic matter from plants, leaf litter or garden waste used to fertilise soil.

*CORE* – Material of a given sample that has been formed into a cylindrical unit 38 mm x 76 mm for testing.

*FLOOD HOLDING CAPACITY (FHC)* *The ability or capacity of a soil to take up and store flood water upon submersion without significant soil erosion or loss of shear strength, and resistance to the detrimental impacts of flooding on soil structure and critical eco-service functions (Kerr et al., 2016).*

*GRAVIMETRIC WATER CONTENT (GWC)* – a ratio of the mass of water to the mass of dry matter.

*REPLICATE* – An additional sample with the same composition as previously used, e.g. Soil 1 had 12 replicate cores, meaning there were 12 cores produced using only Soil1.

*SAMPLE* – A type of soil amendment, where the ‘sample’ refers to AM1 or Soil2.

*SOIL* – Soil derived from either St Anthony’s Lead (S1) works or Nafferton Farm (S2).

*UNAMENDED* - refers to the use of soil without any additional material.

*VOLUMETRIC WATER CONTENT (VWC)* – a ratio of the volume of water in a soil to the volume of dry solids.

*VOLUMETRIC WATER CONTENT  $i$  (VWC $_i$ )* – a ratio of the volume of water in a soil to the volume of dry solids at the point of measurement.

*WATER HOLDING CAPACITY (WHC)* – the maximum amount water a unit of soil can or could hold at saturation based as a function of GWC, volume and VWC/VWC $_i$

*WTR* – Water treatment residual, a waste from water treatment works as discussed in Chapter 3. WTR is discussed in relation to its water content and termed oven dry (WTR $_{od}$ ), air dried 280% solids (WTR $_d$ ) or as received or ‘wet’ 20% solids (WTR $_w$ )

## **4.1 Materials**

Four trials were conducted to develop a methodology to measure a soil's 'flooding holding capacity', which used a number of different materials as described below. Values for chemical/biological characteristics were obtained using Derwentside Environmental Testing Services (DETS), an ISO 17025 accredited analytical service. Physical testing was completed in the department as per British Standards.

### **4.1.1 Soil**

Two soils were used for experimental trials, Soil 1 from St Anthony Lead works and Soil 2 from Nafferton Farm. Soil 1 was obtained from the former St. Anthony lead works in Newcastle upon Tyne (NZ287629), historically a site of anthropogenic heavy metal pollution in operation between 1840 and the 1930's. Characterisation carried out by Finlay (2015) derived by X-ray Florescence (XRF) for Soil 1, determined 6.6 pH and 73 g kg<sup>-1</sup> organic carbon. The site is heterogeneously contaminated with Pb, As and Zn with a mean concentration of 6954, 8820 and 1987 mg kg<sup>-1</sup>, respectively. This soil was chosen for preliminary trials as it has been well characterised by Finlay (2015). Soil 1 posed a potential threat to human health due to high oral bio-accessibility of lead, however this was taken into account with appropriate COSHH regulation guidance for health and safety.

Soil 2 sourced from Nafferton Farm (NZ064657) is an agricultural soil and replaced Soil 1 (StALW) in latter trials to eliminate health & safety hazards posed by the soil and the degradation effect of long-term storage. Specific preparation and storage methods followed for Soil 1 were unknown, therefore sourcing new material enabled control of the storage and preparation of components from collection to final use. 300 kg of soil was obtained in June 2014 (and stored as per section 4.2) from the 300 ha site at Nafferton farm, which is currently the site for a cooperative project called BIONICS that investigates the biological and engineering impacts of climate change on slopes. Soil 2 was characterised by DETS, the results of which are in 4.3.7

#### **4.1.2 Compost**

All compost used in the experiments was obtained from a Newcastle based company called Com-vert, sourced in 2011 for Trials 1 and 2 (Compost 1) and in 2014 for Trials 3 and 4 (Compost 2). The compost is made to the Soil Association Organic Standards and meet PAS100 (2011) quality standards. The PAS100 specification, prepared and published by the British Standards Institution (BSI) provides requirements for the compost processing, input materials and minimum quality to ensure that composted materials are consistently fit for their intended use (PAS100, 2011). As a result of this process compost can be defined as “solid particulate material that is the result of composting, that has been sanitized and stabilized and that confers beneficial effects when added to the soil, used as a component of a growing medium, or is used in another way in conjunction with plants” (PAS100, 2011).

#### **4.1.3 Water Treatment Residual**

Water treatment residual (WTR) was obtained from Honey Hill (WTR1) for Trials 1&2 and from Mosswood (WTR2) for Trials 3&4. WTR1 was characterised by Finlay (2015) and had a gravimetric moisture content of  $81.4 \pm 0.1\%$ , organic content (determined by LOI)  $54.1 \pm 0.1\%$ , 4.1 pH, Fe 35%. WTR 2 was collected from Mosswood water treatment works due to its proximity to Durham University, where the research was conducted. There was a marked difference between the texture of WTR1 and WTR2, where WTR1 had significantly separated into solid materials and water; as discussed in Chapter 3 the flocculants and coagulants used in the water treatment process continue to bind particles together and expel any water within the matrix. WTR2 in contrast had a dry crumbly soil texture, despite having similar water content to WTR1. The difference in appearance between WTR1 and WTR2 were due to the age of the materials, WTR1 was obtained from Honey Hill in 2011 and WTR2 was obtained in 2014 from Mosswood, therefore water was still held in the coagulated material in WTR2.

#### **4.1.4 Silica**

Washed Silica was obtained from Leighton Buzzard supplies. The silica was oven dried to remove any water accumulated in storage. Silica is chemically inert and is

able to impart structural properties in isolation and therefore was used to replace WTR2<sub>a</sub> in Trial 3.

#### **4.2 Material storage, handling and preparation**

The handling and storage of materials can profoundly affect the results of analysis (Sheppard & Addison, 2008), particularly for subtle soil measures such as biotic functions, bioavailable elements or studies of organic matter (Kaiser *et al.*, 2001). The typical sequence observed and followed for this study is as follows:

- Collect samples from the field
- Reduce clump size and mix sample
- Subsample for appropriate testing e.g. moisture content
- Dry remaining sample if appropriate to a moisture content suitable for storage duration

Typically soils are ground so that all material passes through a 2 mm to ensure a composite and homogeneous sample for testing. Although this is acceptable practice for some testing such as chemical analysis, for more sensitive physical measures such as macro-pore properties, sieving to <2 mm affects microbial mineralisation rates, extractable iron and aluminium concentrations, oxygen uptake, rate of decomposition and can increase extractable phosphorous by 165% in some soils (Craswell & Waring, 1972; Hassink, 1992; Neary & Barnes, 1993; Peterson & Klug, 1994; Ross, 1992; Turner & Haygarth, 2003). Although suggested as best practise for the long-term storage of materials, drying a soil at room temperature (which very often results in a moisture content similar to oven dried soil) causes organisms to die or become dormant, dissolved inorganic materials become concentrated in remaining pore water, organic materials will coagulate and deform, and aggregates will stabilise and cause hydrophobicity of the soil (Elmholt *et al.*, 2008; Semmel *et al.*, 1990). For the types of analysis required in this methodology, the suggested procedures for the storage materials were: field moist or workable moisture content, with moderate breakdown of aggregates, and minimal storage time (unless refrigerated). Therefore, as described below, soils and compost were not dried for storage. The water content of the WTR was used in both wet (as received) and air-dried forms. All materials were sieved to 6.3 mm to reduce sieving artefacts as mentioned above.

#### 4.2.1 Soil

For particular soil tests, preparation and storage can profoundly affect the properties of soil (Rimmer, 1991; Sheppard & Addison, 2008). Air-drying is the accepted procedure to preserve samples without degradation, which may be supplemented with mixing and physical breakdown of aggregates (Tan, 2005), and this method was believed to be the procedure for storage of Soil 1. However air drying as a preservation technique is only accepted in geotechnics and not preferable for geochemical/geoenvironmental use (Dowding *et al.*, 2005), therefore the Soil 2 was kept at field moisture content and was used immediately. Samples were kept indoors to regulate their temperature, as high temperatures, i.e. those exceeding 35°C are detrimental to physical and biological functions of the soil (Bartlett & James, 1980). This includes, but is not limited to, oxidation of sulphur in some soils that produces sulphuric acid when wetting and reduces the soil pH, cementation of soil aggregates (Bullock *et al.*, 1988), oxidation and loss of organic matter, oxidation of Fe<sup>2+</sup> to Fe<sup>3+</sup>, changes in P fixation related to Al and Fe chemistry and an increase of exchangeable Mn (Sherman & Harmer, 1943).

At the site, there was heavy plant growth on the topsoil deposit with numerous large stones and non-organic material. The reason for the growth and origin of contaminants is unknown however the majority of large fragments (>7 mm) present were removed before storage, including roots, biota, pebbles and all non-organic matter such as brick fragments, glass and plastic. For this initial screening, large soil aggregates were broken down through a 20 mm sieve. In total 81 kg (27% of total soil mass) were removed from the excavated soil. The remaining 219 kg was sealed in plastic bags to avoid the loss of soil moisture and stored inside at a constant temperature. Soil2 was then passed through a second screening; Datta *et al.* (2014) found that sieve mesh size was a significant factor in soil disturbance, affecting soil pore structure and organic matter fractions. For physical and chemical analysis soils are typically sieved through apertures ranging from 0.5-10 mm (usually 2 mm) in order to remove unwanted fragments such as litter, roots and stones and then mechanically ground to homogenize the material. However sieving at this scale accelerates C mineralization, N immobilization and denitrification (Situala *et al.*, 2000), with finer sieves exacerbating this effect and

resulting in a flush of C and N due to break down of aggregates and increased surface area.

To maintain the soil as close to field conditions as possible whilst enabling the material to be used at a size appropriate to the scale of research being undertaken, all soil matter was passed through a 6.3 mm sieve (Datta *et al.*, 2014). This accommodated some small aggregate particles that had formed naturally and avoided sieving the soil too finely. Soil cores compacted for testing were 38 mm in diameter; therefore the largest particles in each sample would comprise a maximum of 10% of the total core surface area should one take a slice of the core. A further 14.2 kg of stones and other fragments were removed from the soil during this second screening. Each sample bag contained soil of different areas through the soil profile due to the use of a mattock and shovel to retrieve the soil; the upper most samples contained high quantities of root matter and biota and had low water content with a sandy texture. Samples obtained from lower in the soil profile were much denser and contained larger stones, had a higher water content and appeared to have a more clayey texture. All sieved soil was mixed for a period of 10 minutes to reduce heterogeneity. The fundamental rationale for compositing the soil is that following the mixing process, a sample taken from the whole then yields a valid mean point for chemical or physical testing (Tan, 2005).

The 204 kg of soil passing the 6.3 mm sieve was then divided using the riffing method into 2 kg subsamples and returned to polythene storage bags. Briefly, the riffle method (Head, 1980) uses a large riffle box to divide the sample into two parts. In this process soil is separated through a number of slotted paths in the box with the aim of randomly dividing the sample. Once the sample is passed through the box, one half of the sample is put aside. The remaining half is then passed through the box, again putting one half aside. This process is repeated until the division of the sub-sample firstly gave 500 g for particle size analysis and (8 repetitions) then gave 2 kg sub-samples (approximately 60 repetitions in total).

#### **4.2.2 Water Treatment Residual (WTR)**

WTR was stored in two forms, firstly it was unprocessed and sealed 'as received' to be used for research in the format in which it is produced by the water company (WTR<sub>w</sub>), and secondly was air dried (WTR<sub>a</sub>) to provide a granular, coffee grind like material that was more easily handled and did not completely saturate samples and render them to a slurry. WTR1<sub>a</sub> was air dried at room temperature and broken down by hand periodically. Once dried, this was sieved to 2 mm and stored in sealed plastic bags until use in Trials 1 & 2 and WTR1<sub>w</sub> was unprocessed. Similarly, approximately half of WTR2 was air-dried over a number of days, with periodic breakdown of the larger aggregates to ensure that these did not harden and become one large solid mass. Once dried, WTR2<sub>a</sub> was passed through a 6.3 mm sieve to approximate the maximum aggregate size of the soil and sealed for storage before use in Trial 4 to ensure that it did not take up moisture from the surrounding air. The remaining mass of WTR2 was kept sealed and unprocessed (WTR2<sub>w</sub>).

#### **4.2.3 Compost and silica**

Fifteen 40-litre bags of compost were delivered in October 2014 and stored outside until their use. Compost was not processed before use, with the exception of the removal of large twigs and other large particles during mixing with soil mineral matter and/or WTR. Silica was oven dried to remove any moisture accumulated during storage, but otherwise unprocessed.

### 4.3 Material characterisation

British standards were followed to characterise all materials as described above, where brief descriptions of these methods are subsequently outlined.

#### 4.3.1 Particle size distribution

For Soil 2, a particle size analysis (particle size distribution) was conducted according to BS1377 (Part 2:1990). Three fractions, all of which can be subdivided into finer size fractions, are present in soil, sand (0.06-2 mm), silt (0.002-0.06 mm) and clay (<0.002 mm). Simple dry sieving was not appropriate as the method is only applicable to clean granular materials with negligible quantities of silt or clay. In a dry state, clay and silt are able to adhere to the surface of sand grains. Therefore, the wet sieving procedure was followed to calculate particle size. According to BS1377, the minimum mass of 500 g was obtained using the riffing method (as described previously). Coarse material removed during the secondary screening was re-added to the sieved soil to obtain the coarser fraction particle size curve for the material. The soil sample was weighed ( $W1$ ) using a mass balance and then oven dried overnight at 105°C and left to cool in a desiccator. The dried sample was then weighed on a balance to ensure the minimum mass threshold was met (to an accuracy of  $\pm 0.1\%$  of the total mass) ( $W2$ ). A standard set of sieve apertures was used for the classification; 37.5 mm, 20 mm, 10 mm, 6.3 mm, 3.35 mm, 2 mm, 1.18 mm 600  $\mu\text{m}$ , 300  $\mu\text{m}$ , 150  $\mu\text{m}$  and 63  $\mu\text{m}$ . Soil was crushed and then washed with a water jet to remove the clay and silt particles adhered to the surface of sand grains, as these cannot be removed sieving alone. To avoid overloading of sieves, three sieves were nested together (2 mm, 212  $\mu\text{m}$  and 63  $\mu\text{m}$ ). Material passing through the 63  $\mu\text{m}$  sieve was collected for a pipette hydrometer test and oven dried overnight at 105°C. The dried and cooled material was weighed to  $\pm 0.01\%$  of mass ( $W3$ ). The silt & clay proportion of the total sample was calculated using equation 6:

$$< 63 \mu\text{m} \% = \frac{W2 - W3}{W2}$$

*Equation 6: Calculation of the silt and clay fraction of a soil based on the wet sieving procedure BS1377*

The cooled oven-dried soil was placed into the standard sieves in a stack shaker for 10 minutes. Once the shaking cycle was complete, the proportion retained on

each sieve was weighed, giving the proportional value of each fraction. The pipette method of sedimentation of the fine fraction (also BS1377: 2, 1990) was used to calculate the silt and clay proportion of material passing the <63  $\mu\text{m}$  sieve. The procedure is based on the relationship between settling velocity and particle diameter (developed by Stokes, 1851). Although Stokes' Law makes a number of assumptions, including the assumption that particles are spherical (unlike clays and clay sized grains that are more plate like), the use of sedimentation through pipette analysis is standard practice in geotechnics. Briefly, the test requires a sample to be taken at a depth (h) at a given time (t) at which all particles coarser than X mm have been eliminated (Gee & Or, 2002), where the settling times for clay fractions can be calculated at a given time and temperature. 12.5 g of dry soil that has passed a 63  $\mu\text{m}$  sieve are dispersed with 25 ml of sodium hexametaphosphate ( $\text{NaPO}_4$ , called Calgon) in a conical flask containing 500 ml of water. The conical flask is shaken for 3-4 hours at 25  $^{\circ}\text{C}$  to disperse and disaggregate all individual particles. The contents of the flask are added to a testing bath, after which three 9.67 g aliquots are taken by pipette at 4m30s, 46 minutes and 6h54m to quantify the concentration of particles of 0.02 mm, 0.006 mm and 0.002 mm equivalent diameter, respectively. This assumes a particle density of 2.65  $\text{g}/\text{cm}^3$ .

As discussed in Chapter 3 (section 3.2.1), particle size distribution (PSD) for WTRs has been performed by hydrometer analysis in the wet form (produced by water treatment plants) where 95% of residual solids passed the 74  $\mu\text{m}$  sieve, but when air-dried the PSD included larger grain sizes, even when the air-dried material was pulverised. Issues with settling of both WTR2<sub>w</sub> and WTR2<sub>a</sub> in hydrometer measurements were experienced during testing by Hsieh & Raghu (1997) and Basim (1999), therefore analysis by hydrometer of wet PSD was not attempted for WTR2. For WTR2<sub>a</sub> the process of obtaining a 'particle size distribution' was completed using simple dry sieving (BS1377: 1990 Test 7 B) as per section 4.3.1 and a sample placed into a stack of 10 sieves for a period of 10 minutes. Obtaining PSD from dry sieving does not give an individual particle size of WTR2<sub>a</sub> but was conducted to obtain information on the sizes of WTR2<sub>a</sub> 'grains' achieved by manual breakdown during air-drying in this particular research.

Given the effect that surface area has on the water holding capacity of soil and the shear properties (Chapter 2, section 2.3.6) it is important to know if the predominant size fraction of WTR<sub>2d</sub> is fine or coarse. The caveat, however, is that the test may not have been wholly accurate as, due to the brittle nature of dry WTR, larger pieces may have been broken down due to the sieving process, which may have given an erroneous reading for the contribution of the finer fraction. In general Basim (1999) found that the materials become more granular as they dry due to cementation from organic matter and oxides present, therefore the test wholly reflects the duration of drying and breakdown methods used during the drying process.

#### **4.3.2 Classification and description of soils**

The standard light Proctor compaction test (BS1377, 1990: Test 12) derives the optimum water content at which the maximum density can be achieved with a given compaction effort. This test was completed using Soil 2 and briefly, three layers of Soil 2 were added to the 1000 cm<sup>3</sup> Proctor mould, using 27 compaction strikes of a 2.5 kg hammer from 300 mm to compact each layer. The soil water content is incrementally increased to test compactibility across a range of water contents. The standard light Proctor tests initially uses a dry soil that is tested as described above, then broken down and wetted to increase the water content before completing another test. The density of the sample increases with the increase of water content until maximum density is achieved, after which the density decreases due to the addition of less dense water (1 g/cm<sup>3</sup>) to soil matter (2.65 g/cm<sup>3</sup>); results are typically reported in Figure 16. Atterberg tests were also conducted on Soil2, where Atterberg limits are a measure of the critical water content that separate the soil into different states of consistency, defined at the shrinkage limit (uncommonly tested), plastic limit and liquid limit. A fall cone test was used to determine the liquid limits and a thread (roll) test was completed to determine the plastic limit (BS1377: 1990 Part 2). Soil2 was determined as inorganic clay of medium plasticity (the soil exhibits plastic properties at 7-17% water content).

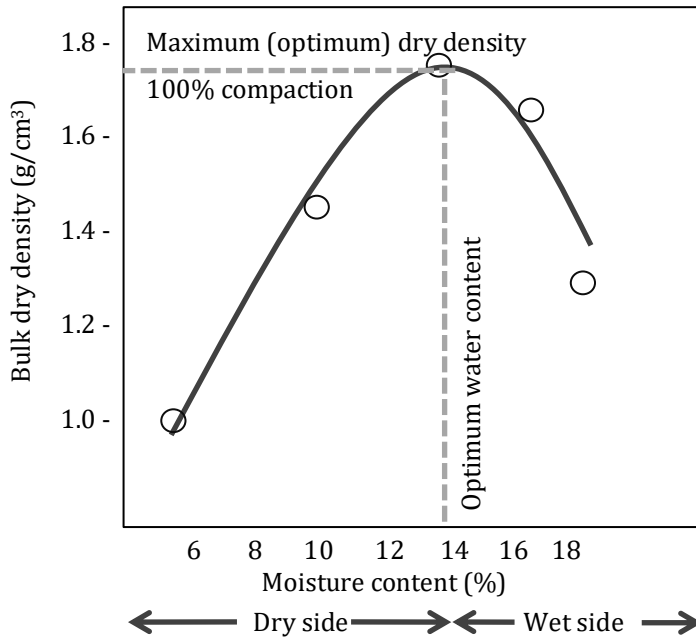


Figure 16: Example of the Proctor standard test for compaction of soil at different water contents, annotated with optimum moisture content, maximum bulk dry density, wet and dry sides of optimum.

#### 4.3.3 Volume and density (bulk, dry & particle)

Bulk density and dry density were calculated for each core using the volume of a sample and mass of dry solids (Figure 17). The volume of individual components also allows empirical calculations of dry density and the void ratio.

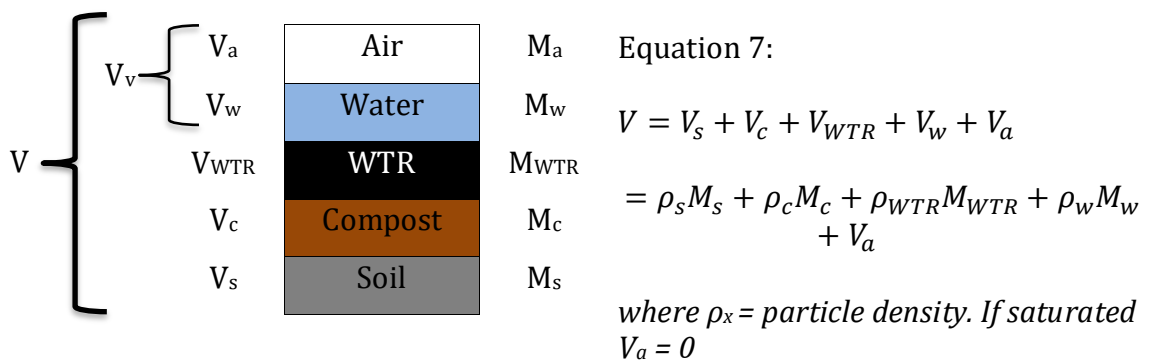


Figure 17: Schematic of volume and mass proportions in a soil with mass and volume equation 7.

Bulk density is ratio of the mass to the bulk volume of the soil, which includes the volume of the solids and pore space, air and water. Dry density is therefore a ratio

of the mass of oven-dried solids to the volume of the sample and 'equivalent particle density' can be calculated using the ratio of components.

$$BD = \frac{M}{V}$$

Equation 8: Bulk density calculation for soil where  $M$ = mass of moist sample and  $V$  is total volume of the sample.

$$R_C = \frac{M_C}{M_S}$$

$$R_{WTR} = \frac{M_{WTR}}{M_S}$$

Equation 9: Ratio of components in amendments where  $R_c$  = ratio of compost,  $M_c$  is mass of compost,  $M_s$  is mass of soil solids,  $R_{WTR}$  is ratio of WTR and  $M_{WTR}$  is mass of WTR

$$D_d = \frac{M_S + M_C + M_{WTR}}{V}$$

$$= \frac{M_S + M_C + M_{WTR}}{\rho_S M_S + \rho_C M_C + \rho_{WTR} M_{WTR} + \rho_C M_W}$$

Equation 10: Dry density ( $D_d$ ) calculation where  $\rho_s$  is particle density of soil,  $\rho_c$  is particle density of compost,  $\rho_{WTR}$  is particle density of WTR<sub>d</sub> (where WTR<sub>2</sub> is assumed to have the same value)

$$\rho_{Eq} = \frac{\rho_s + \rho_c R_C + \rho_{WTR} R_{WTR}}{1 + R_C + R_{WTR}}$$

$$= \frac{1 + R_C + R_{WTR}}{\rho_{Eq}(1 + R_C + R_{WTR}) + \rho_w w(1 + R_C + R_{WTR})}$$

$$\rho_d = \frac{1}{\rho_{Eq} + \rho_w w}$$

Equation 11: Equivalent particle density which links the gravimetric water content ( $w$ ) and particle density ( $\rho_d$ ) for any combination of components

The particle density of WTR<sub>2d</sub> and compost were derived using pycnometry, a method that uses a vessel with a precisely known volume to allow density determination. The particle density (as defined in Chapter 2) was assumed for Soil<sub>2</sub> to be 2.65 g/cm<sup>3</sup>. WTR<sub>2d</sub> was tested for particle density using BS1377: 1990

Part 2 (small pycnometer method). 2 mm sieved WTR2<sub>a</sub> that had been air was oven dried at 30° C for 72 hours immediately before testing to remove remaining water. Air removal is a critical factor in the particle density testing, where testing standards suggest the removal by either gently heating the contents of the pycnometer or applying a vacuum. As discussed in Chapter 3 – heating of WTR has considerable effects on the geophysical properties (loss of volatile solids and organic matter) and therefore the latter was chosen. Each test subject was de-aired for 3 days and produced densities of  $2.11 \pm 0.81 \text{ g/cm}^3$  (where minimum = 2.1099, maximum = 2.118). In comparison, two tests in which subjects were only de-aired for 30 minutes produced densities of 2.127 and 2.103. It must be noted that Basim (1999) found that the higher the temperature used to dry the WTR, the higher the particle density, which is attributed to the loss of the organic phase at temperatures. Therefore should the test be conducted using 105 °C, a higher particle density is expected (approximately 0.2 g/cm<sup>3</sup> increase between samples dried at 60 °C and 105 °C). As the samples are exposed to higher temperatures, in addition to organic matter reduction, calcium oxide, iron oxide and aluminium come out of solution and act as a cement between the solid material, and hence increasing the particle density.

A method described by Weindorf & Wittie (2016), adapted from Blake & Hartge (1986) was followed for measuring the particle density of compost, using hexane in place of traditional methods using water in the pycnometer. The use of hexane facilitates low density particle submersion as it has a lower density than water (0.655 g/cm<sup>3</sup>). The method briefly involved oven-drying compost at 70 °C for 24 hours, before passing it through a 2 mm sieve. Next, 6-8 g of dried compost was added to a 100 ml volumetric flask of known mass and weighed on a mass balance to an accuracy of  $\pm 0.0001 \text{ g}$ . Approximately 40 ml of de-aired hexane was added to the volumetric flask to completely immerse the compost, however following the gentle swirling technique as used by Weindorf & Wittie (2016), many particles remained buoyant and air bubbles continued to be trapped even after 24 hours of immersion. Therefore, the flask containing compost and 40 ml of hexane was de-aired for a period of 3 minutes. De-aired hexane was then added to bring the sample to volume (100 ml) and left to settle for a number of hours. Any additional hexane required was then added and the mass was recorded. Particle density was

then calculated using Equation 12. The compost particle density ( $\rho_d$ ) was found to be  $1.675 \pm 0.33 \text{ g/cm}^3$  where the number of repeats ( $n$ ) = 12.

$$\rho_d = \rho_h(W_c) / [W_c - (W_{cb} - W_h)]$$

*Equation 12: Compost particle density calculation where  $\rho_h$  = density of hexane ( $\text{g/cm}^3$ ),  $W_c$  = weight of oven dried compost (g),  $W_{ch}$  = weight of compost and hexane (g), and  $W_h$  = weight of 100 ml of pure hexane (g)*

#### **4.3.4 Moisture content**

As per discussion in Chapter 2, the moisture content of a soil (or any material) can be expressed gravimetrically or volumetrically. Conventional temperatures for testing (105 -110 °C) remove both gravitational and capillary water present in the pores, however this temperature may also remove significant amounts of organic matter in highly organic soils. According to BS1377 (1990) to determine gravimetric water content (GWC) a moist sample of material of known mass is put into a metal container and placed into an oven at 105°C for 24 hours.

GWC can be expressed using a on a wet mass basis or on a dry mass basis. For example, for a soil that weighs 100 g in a field moist condition, containing 80 g of dry solids, with a dry density of 1.5  $\text{g/cm}^3$  (bulk density = 1.89  $\text{g/cm}^3$ ) and a volume of 53  $\text{cm}^3$  (as shown in Figure 17) the equations for GWC dry basis and GWC wet, give different values. It is therefore very important to note which equation is used in literature. Typically, Equation 13 (dry basis) is used, giving the mass of water in the moist bulk of soil to the mass of dry solids as a ratio (0.2) or can be given as a percentage, i.e. 20%. Equation 14 expresses GWC on a wet weight basis and relates the mass of water to the total mass of the moist bulk of soil (Gardner, 1986). The difference between the two is not negligible, therefore is it critical to note if the GWC is calculated on a dry or wet basis. For soils the difference between wet and dry is not too dissimilar, but for materials such as WTR that may have 80 g of water to every 20 g of dry solids there is a large difference in values for dry basis and wet basis; 4 (400%) and 0.8 (80%) respectively. For the purposes of this thesis, Equation 8 will be used to express the GWC as it is used in the majority of literature.

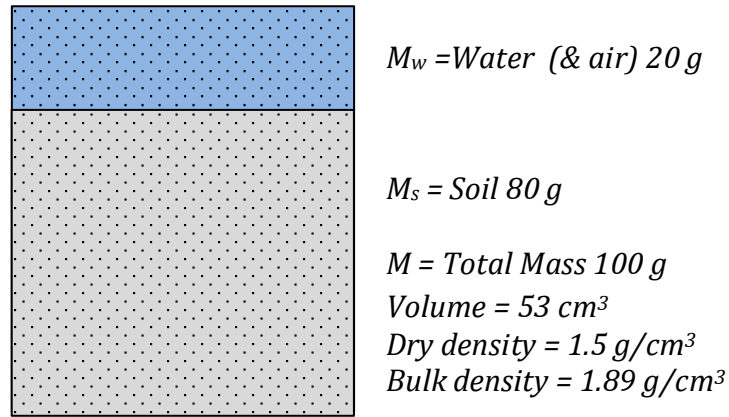


Figure 18: Example of a field moist soil, with the values of properties noted on the diagram.

$$\begin{aligned}
 \theta &= \frac{M_w}{M_s} \\
 \theta &= \frac{M_w}{M_s + M_c + M_{WTR}} \\
 &= \frac{M_w}{M_s(1 + R_c + R_{WTR})} \\
 &= \mathbf{0.25 \text{ or } 25\% \text{ GWC}}
 \end{aligned}$$

Equation 13: Dry basis gravimetric water content calculation for the soil sample on a dry basis in Figure 17 where  $M_w$  = mass of water,  $M_s$  = mass of dry solids

$$\begin{aligned}
 \theta &= \frac{M_w}{M} \\
 &= \mathbf{0.2 \text{ or } 20\% \text{ GWC}}
 \end{aligned}$$

Equation 14: Wet basis gravimetric water content calculation for the soil sample in Figure 17 where  $M_w$  = mass of water and  $M$  = mass of moist soil

Volumetric water content is a measure of the volume of water relative to the volume of the soil bulk, however this value is only calculated relative to the original volume (Equation 15). To account for swelling in the soil, the volume or dry density ( $D_d$ ) of soil at the point of measurement (instantaneous,  $i$ ) is used rather than using the original values (Equation 16). Both volumetric measures will be used in the presentation of results to enable comparison to the majority of data produced, but also to provide an insight into the absolute volumetric water behaviour using  $VWC_i$ , which gives a ratio between the volume of water to the volume of the sample.

$$\begin{aligned}\theta_v &= \frac{V_w}{V_s} \\ &= GWC * D_d \\ &= \mathbf{0.37 \text{ or } 37.7\% \text{ VWC}}\end{aligned}$$

*Equation 15: Volumetric water content ( $\theta_v$ ) calculation for the sample in Figure 17 using the original volume of soil as a constant; where  $V_w$  = volume of water,  $V_s$  = original volume of solids*

$$\begin{aligned}\theta_{vi} &= \frac{V_w}{V_{si}} \\ &= GWC * D_{di} \\ &= \mathbf{0.37 \text{ or } 37.5\% \text{ VWC}}\end{aligned}$$

*Equation 16: Volumetric water content ( $\theta_{vi}$ ) calculation using an instantaneous volume of soil of Figure 17; where  $V_w$  = volume of water and  $V_{si}$  = volume of instantaneous soil*

The determination of the GWC of soil at 105 °C is standard practice, however table 14 provides evidence that values obtained for the GWC of WTR and compost are dependent on the temperature at which the test is conducted. At higher temperatures, the difference in GWC is attributed to the loss organic matter as drying samples at the conventional 105 °C may induce the removal of volatile solids. This gives inaccurately high-water contents for WTR and compost. The GWC of WTR was determined from material air dried at 20 °C (WTR2<sub>d</sub>), 30 °C and 60 °C for 48 hours and at 105 °C for 24 hours. Similarly the water content of compost was derived at both 60 and 105 °C to determine a difference in the water content value. As discussed in Chapter 3 (section 3.2.1), Hseih & Raghu (1997) found that there was little difference in water lost from WTR at temperatures of 24 °C, 30-40 °C and 105 °C. Basim (1999) conversely found that for WTR there was 3.44-10.25 % of weight lost in the test between 60 and 105 °C, due to the loss of organic matter. Table 14 supports Hseih & Raghu's findings, showing that there is little difference between the derived water contents at either 60 °C or 105 °C for WTR2<sub>w</sub> suggesting that organic matter may even be removed at the lower temperature, or not at all in this case. In tests conducted on Mosswood WTR2<sub>w</sub>, there was less than 1% difference in the GWC between 60 and 105 °C.

Sample (n = 3)	Air dried at 20 °C	WC at 30 °C	WC at 60 °C	WC at 105°C	WC at 105 °C – 60 °C
	48 hours			24 hours	
	(wet basis GWC) *values derived by Basim (1999)				
WTR2 <sub>w</sub> <i>From treatment plant</i>	1.14 (0.53) <i>*1.8-5.12</i>	2.18 (0.69)	4.51 (0.82) <i>*1.97-5.7</i>	4.94 (0.83) <i>*2.4-6.0</i>	0.43 (0.001) <i>*0.13-0.45</i>
WTR2 <sub>d</sub> <i>Air dried @ 20C</i>	n/a	0.21 (0.30)	0.53 (0.35)	0.78 (0.44)	0.25 (0.009)
Compost2	n/a	n/a	0.61 ± 1.4	0.71 ± 1.3	0.04 ± 0.005

*Table 14: Water contents of WTR2 dried at different temperatures, where the value is derived using Equation 13 (mass of water/ mass of solids). 'n =' denotes the number of repeat tests. Values in brackets describe the water content by wet basis GWC. These values are compared to a small range of values derived by Basim (1999), denoted by \**

However, there is a 32% difference in values between GWC of WTR2<sub>d</sub> dried at 60 and 105 °C. This is presumably due to the water becoming more difficult to remove as it becomes trapped in the 'dried' WTR (see Chapter 6, section 6.4.3), therefore at 60 °C there is much less water removed than at 105 °C. The alternative is that the water removal is the same for WTR2<sub>w</sub> and WTR2<sub>d</sub> and the difference is due to the more effective removal of organic material once the WTR has been air-dried because of a higher surface area of exposed organic matter. Although literature suggests the use of 70 °C to dry compost to avoid significant loss of organic matter (Agnew & Leonard, 2003; Pansu & Gautheyrou, 2007), results from testing compost showed that there was only a very small difference in the water content of compost dried at 60 °C and 105 °C, therefore calculations were taken using the conventional 105 °C.

### 4.3.5 Material characterisation results

Material	Sourced	Sieving	Moisture content % (dry basis)	pH	Total Carbon %	TOC %	LOI <sub>550</sub> %	EC (µs/cm)	Total N %	Fe %	Al %
Soil 1 40% sand 30% silt 30% clay	St Anthony lead works, 2011	6.3 mm (air dried)	11.7	6.6	4.7	n/a	n/a	220	0.21	4.1	8.1
Soil 2	Nafferton Farm, 2014	6.3 mm	16	7.5	n/a	2.3	3.96	n/a	n/a	25000 mg/kg	n/a
Compost 1	Comvert, 2011	n/a Large twigs and non-organic material removed	33	7.4	15	n/a	n/a	1700	1.13	2.8	4.1
Compost 2	Comvert, 2014		55	8.1	n/a	14	13.9	n/a	n/a	16000 mg/kg	n/a
WTR1 <sub>w</sub>	Honey Hill, 2011	n/a	83 ±2	5 ±0.6	23.4 ±0.2	12.6	51 ±2.4	224 ±119	0.7	29.3 ± 4.2	0.5 ± 0.2
WTR1 <sub>d</sub>	Honey Hill, 2011	2-4 mm	n/a					n/a			
WTR2 <sub>w</sub>	Mosswood, 2014	n/a	80 ± 2	4.7 ±0.5	21.4	27.9	48 ±2.7	239 ±168	0.8	28.8 ±1.7	0.4 ±0.3
WTR2 <sub>d</sub>	Mosswood, 2014	6.3 mm (air dried)	18 ± 2					n/a			
Silica	Leighton buzzard	n/a	0	n/a	0	0	0	n/a	n/a	n/a	n/a

Table 15: Results of material characterisation and analysis including physical and chemical attributes, where values are obtained from analysis undertaken by Derwentside Environmental Testing Services (DETS) using methods DETSC 2301# and 2008# and from Finlay (2015).

Material	Mn mg/kg	Pb mg/kg	K mg/kg	Mg mg/kg	P mg/kg	As mg/kg	Zn mg/kg	B mg/kg	Cd mg/kg	Cr mg/kg	Cu mg/kg	Hg mg/kg	Ni mg/kg	Si %	Ca %	Se mg/kg
Soil 1	350	<b>6954*</b>	15240	4500	407	8820	<b>1987</b>	n/a	n/a	n/a	n/a	n/a	n/a	20.6	0.78	n/a
Soil 2	730	80 ±0.3	n/a	n/a	n/a	6.9 ±0.2	150 ±1	1.2	0.2 ±0.1	17 ±0.15	17 ±0.2	<0.05	15 ±1	n/a	n/a	<0.5
Compost 1	525	136	19010	6560	2942	10.4	250	n/a	n/a	n/a	n/a	n/a	n/a	13.5	3.24	n/a
Compost 2	460	46 ±0.3	n/a	n/a	n/a	9.9 ±0.2	150 ±01	6.3	0.4 ±0.1	19 ±0.15	46 ±0.2	0.10 ±0.05	12 ±1	n/a	n/a	<0.5
WTR1 <sub>w</sub>	1350 ±289	31.6	425	245	352	n/a	210	n/a	0.78	29.5	17.8	0.62	49.3	n/a	n/a	n/a
WTR1 <sub>d</sub>																
WTR2 <sub>w</sub>	1825 ±665	85	833	335	472	n/a	<b>665</b>	n/a	<b>2.36</b>	31.5	26.8	0.28	91.5	n/a	n/a	n/a
WTR2 <sub>d</sub>																

Table 16: Chemical analysis of materials undertaken by DETS using methods DETSC 2301# and 2008#, including information from Finlay (2015). **Bold** typeface signifies that the concentration exceeds BSI PAS100 regulations, and **bold** typeface with \* signifies that the concentration exceeds EU Biosolids regulations (Sewage sludge directive 86/278/EC).

<i>Material</i>	<i>Moisture content %</i>	<i>pH</i>	<i>TOC %</i>	<i>LOI<sub>550</sub> %</i>	<i>Total carbon %</i>	<i>EC (us/cm)</i>	<i>Total N %</i>	<i>Fe %</i>	<i>Al %</i>	<i>Mn mg/kg</i>	<i>Pb mg/kg</i>	<i>K mg/kg</i>	<i>Mg mg/kg</i>	<i>P mg/kg</i>	<i>Zn mg/kg</i>	<i>Cd mg/kg</i>	<i>Cr mg/kg</i>	<i>Cu mg/kg</i>	<i>Hg mg/kg</i>	<i>Ni mg/kg</i>
<i>Soil</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>5</i>	<i>3</i>	<i>n/a</i>	<i>0.02-0.5%</i>	<i>4</i>	<i>7.1</i>	<i>80-1300</i>	<i>10-84</i>	<i>640</i>	<i>n/a</i>	<i>1000</i>	<i>17-125</i>	<i>0.06-1.1</i>	<i>7-221*</i>	<i>6-80</i>	<i>0.02-0.41</i>	<i>4-55</i>
<i>WTR</i>	<i>72-85</i>	<i>4.1-7.2</i>	<i>n/a</i>	<i>36-70</i>	<i>13-26</i>	<i>39-405</i>	<i>0.051-1.1</i>	<i>0.8-41</i>	<i>0.21-21</i>	<i>370-5100</i>	<i>5-160</i>	<i>170-3900</i>	<i>170-2900</i>	<i>4-1528</i>	<i>28-1100</i>	<i>0.2-36*</i>	<i>7.8-38</i>	<i>7.9-36</i>	<i>0.05-1.4</i>	<i>10-120</i>
<i>Compost</i>	<i>30-60%</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>1.2</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>4000</i>	<i>3000</i>	<i>3000</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>	<i>n/a</i>

Table 17: Typical ranges for soil, WTR and compost, sourced from Finlay (2015) in addition to other literature. **Bold** typeface signifies that the concentration exceeds BSI PAS100 regulations, and **bold** typeface with \* signifies that the concentration exceeds EU Biosolids regulations (Sewage sludge directive 86/278/EC).

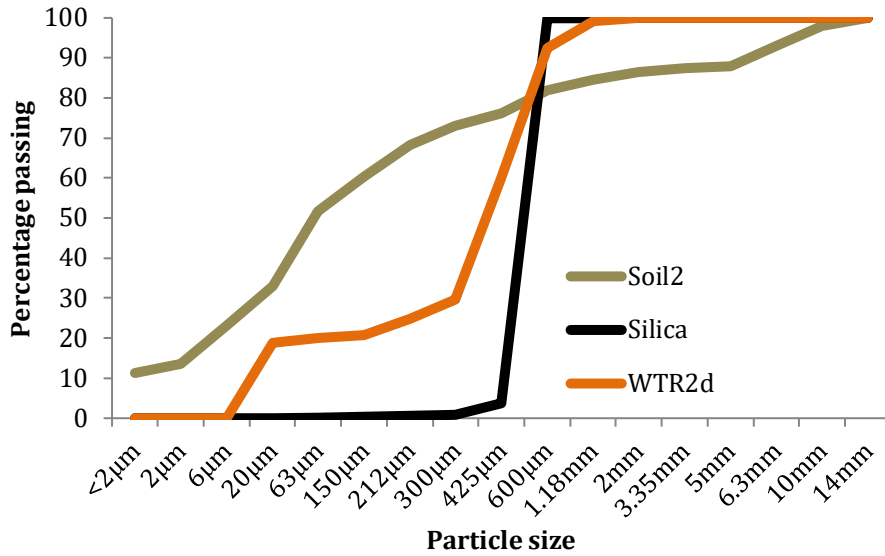


Figure 19: Particle size distribution for Soil2, silica and WTR2<sub>d</sub> performed as per BS1377 (Part 2: 1990)

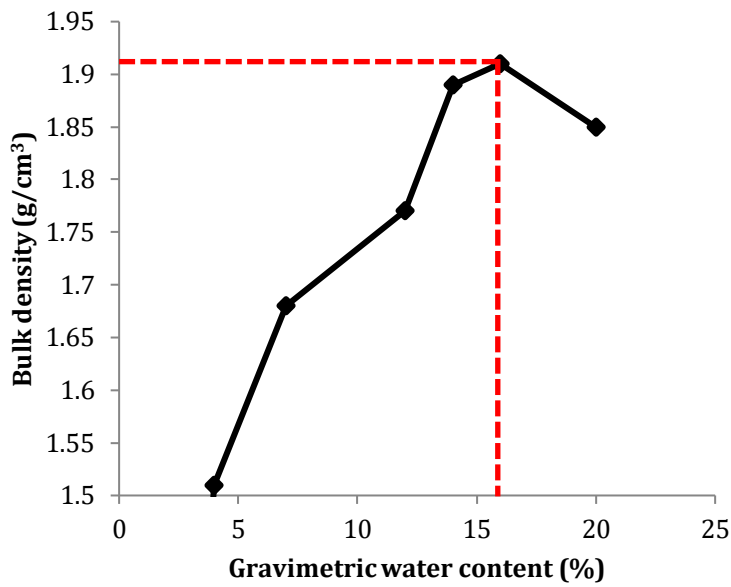


Figure 20: Proctor compaction curve for Soil2 determining the maximum (optimum) bulk density as 1.91 g/cm<sup>3</sup> at gravimetric water content of 16% (0.16), shown by the red dashed lines.

Figure 19 presents the particle size distribution of the three materials used in Trials 3 & 4, retrieved following wet sieving and pipette sedimentation BS1377 (Part 2, 1990). Finlay (2015) conducted PSD testing on Soil 1 and found that it comprised 40% sand, 30% silt, 30% clay and is therefore a clay loam (Rowell, 1994), which tends to have high water holding capacity, poor aeration, is

susceptible to compaction and has a high resistance to pH change (Brady & Weil, 2016). Soil 2 comprised 61.8% sand, 25.1% silt and 13.1% clay, and is therefore classified as a sandy loam. The characteristics of sandy loam mean that this soil type is naturally well draining due to the high proportion of sand, with typical hydraulic conductivity of 14-42.34  $\mu\text{m}/\text{sec}$ , and bulk densities of 1.55- 1.75  $\text{g}/\text{cm}^3$  (USDA, [23]). Figure 20 displays the relationship between water content and bulk density according to the Proctor compaction method for Soil2, where the maximum density of 1.91  $\text{g}/\text{cm}^3$  was achieved at 16% water content.

#### 4.4 Experimental trial methods

As highlighted in Chapter 2 there is a need to quantify the parameters of soil in relation to their 'flood holding capacity'. Traditional testing methods to determine the water holding capacity of a soil use a variety of apparatus, where samples usually start at complete saturation and are then placed into closed chambers on a porous plate, after which negative pressure is applied. Water is forced from the sample under pressure, typically to -0.33 kPa to determine the field capacity, and up to -1500 kPa to determine the water content or degree of saturation at wilting point. To the author's knowledge, there are few if any, known processes to test water holding capacity that are conducted without external pressure in order to determine maximal WHC (saturation) and through the drying phase. To simulate the process of wetting and drying in the field due to the processes of capillary action, infiltration and gravitational drainage, a new method (Kerr *et al.*, 2016) was developed.

Four water holding capacity trials were conducted during the research for this thesis, where Trials 1 and 2 were used for the development of a core making methodology, and Trials 3 and 4 were more expansive robust trials (greater repetitions) that produced the data on which this thesis is based. The terms 'sample', 'core' etc have already been defined at the start of the chapter. Each amendment (sample) henceforth is given a single label (e.g. A1), as shown in Figure 21.

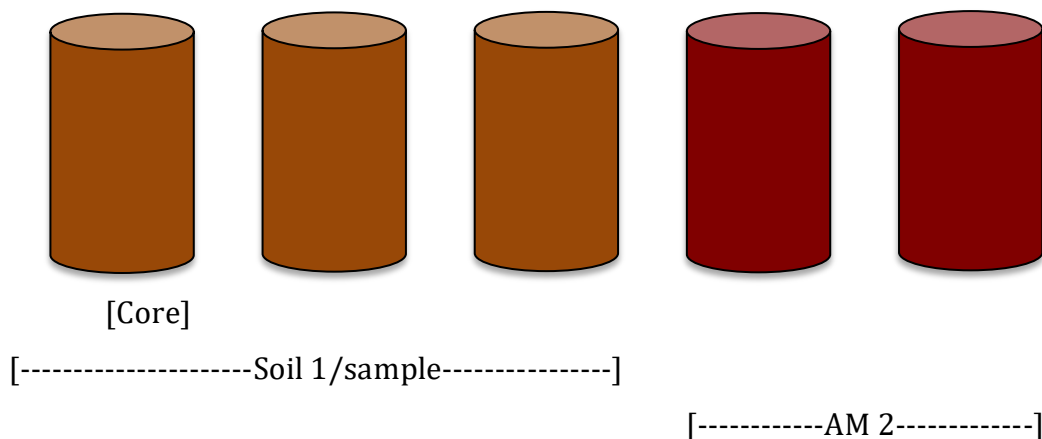
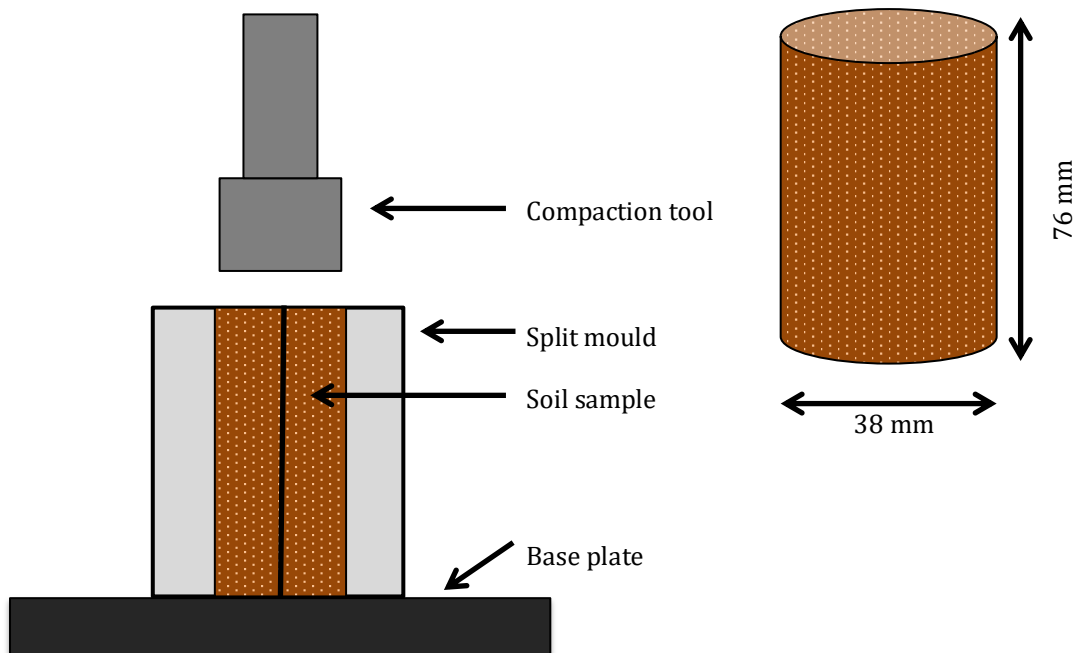


Figure 21: Sample annotations based on individual units (core/sample), groups of cores of the same composition (S1 or AM8), and samples of different composition (Soil 1, AM2 etc)

#### 4.4.1 Core production methodology

Cores were created using either a split mould (Figure 22: Trials 1 and 4) or using the Proctor compaction mould (Figure 23: Trials 2 and 3). The 76 mm x 38 mm cylinder was chosen as this size of split mould allowed many samples or 'cores' to be produced using the same procedure and apparatus found in many soil laboratories, with the additional benefit of being a manageable size of sample when considering the number of samples required to be made. Soil is very heterogeneous in its nature; therefore many replicates are required in order to produce statistically significant results.



*Figure 22: Schematic of sample (core) production using a split mould and compaction pin. The core removed from the mould is 76 mm x 38 mm.*

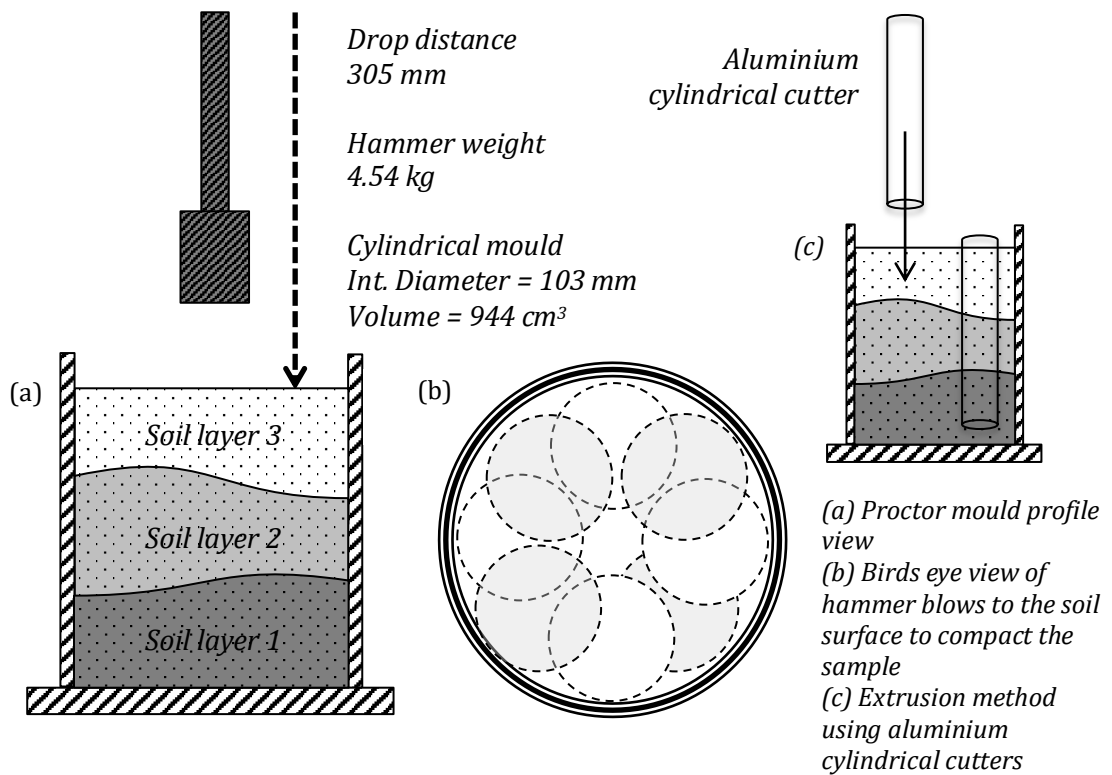


Figure 23: Proctor compaction mould method for soil core production

#### 4.4.2 Trial 1

Trial 1 was a preliminary trial conducted during the first year of research in February 2014 and aimed to establish a fundamental basis for developing effective and statistically viable experimental techniques. The initial research was very much exploratory in its concept and was used to help design the final methodological strategy. Trial 1 used Soil1, Compost1, WTR1<sub>w</sub> and WTR1<sub>d</sub> in proportions outlined in Table 18, where n = 1. Samples were mixed at the original water content in Table 15 (materials summary), according to the dry mass of individual materials. For example, for an amendment requiring dry component masses of soil (50 g), compost (25 g) and WTR1<sub>w</sub> of (25 g) would require 56 g of soil, 38 g of compost and 45 g of WTR, assuming soil has a water content of 12%, compost a water content of 52% and WTR a water content of 80%.

<b>Sample</b>	<b>Soil %</b>	<b>Compost %</b>	<b>WTR%</b>	<b>GWC</b>	<b>Bulk density (BD) &amp; dry</b>
<b><i>n</i> = 3</b>	<b>SOIL 1</b>	<b>COMPOST1</b>	<b>WTR1</b>	<b>%</b>	<b>density (<math>D_d</math>) g/cm<sup>3</sup></b>
Soil 1	100	0	0	12	1.71 (BD) 1.55 ( $D_d$ )
T1A	50	50	0	33	1.67 – 1.87 (BD) 1.12 – 1.25 ( $D_d$ )
T1B	50	25	25 (WTR1 <sub>w</sub> )	44	1.53 – 1.71(BD) 0.86 – 0.93 ( $D_d$ )
T1C	50	25	25 (WTR1 <sub>d</sub> )	22	1.63 – 1.76 (BD) 1.27 – 1.38 ( $D_d$ )
T1D	50	40	10 (WTR1 <sub>w</sub> )	38	1.54 – 1.60 (BD) 0.96 – 0.99 ( $D_d$ )
T1E	50	40	10 (WTR1 <sub>d</sub> )	29	1.59 – 1.69 (BD) 1.13 – 1.20 ( $D_d$ )

*Table 18: Amendment proportions for samples used in Trial 1, using three materials. 'n = ' denotes the number of repeat tests.*

Each core was made using a split mould (Figure 22, section 4.4.1) and a plastic graduated compaction plunger, where three equal layers of sample were compacted into the mould using 25 strikes, which approximates the Proctor light compaction method, although the compaction effort was unregulated as a hand-held hammer was used directly on the compaction plunger. The water content of samples ranged from 12% to 44% for the different amendments (Table 18). As discussed in Chapter 2 (section 2.3.3), samples with a higher water content were compacted to a greater extent and produced very dense samples, however drier amendments were more difficult to compact and required much higher compactive effort to yield a sample with sufficient aggregate stability for handling and extraction from the mould. The mass of samples ranged from 112 g to 146 g as mass was added to mould until the extracted core would hold together, giving a broad range of densities.

Once produced, each core was weighed and measured with digital callipers before being inserted into a latex membrane (typically used for tri-axial testing) and stoppered at each end with a 38 mm porous disk to minimise soil loss when submerged. Samples were completely submerged for a period of 24 hours and weighed and measured again once the membrane had been carefully removed. This method was not sufficiently precise and yielded poor results, with the

following action points leading into Trial 2: control of water content at initial production, control of compaction effort to ensure a similar sample density, greater homogenization of component parts to reduce error, change of saturation method and sample casing and increase of sample size to account for unavoidable heterogeneity.

#### **4.4.3 Trial 2**

In this second trial, Soil 1 and WTR1<sub>d</sub> were air-dried and WTR1<sub>w</sub> was excluded so that water content could be controlled, as soil only retains hygroscopic water once air dried (Figure 8C). Compost was kept as received water content (33%) as air-drying (discussed in section 4.2) has a number of degenerative effects such as increased water repellence, mineral surface acidity and microbe mortality (Kaiser *et al.*, 2015). Soil 1 and WTR1<sub>d</sub> were gently crushed to pass a 2 mm sieve to increase homogeneity of samples. As in Trial 1, samples were mixed proportionally according to the dry mass of components and water was added to make the samples to 13.5% water content (Table 19). The value of 13.5% water content was chosen as it was the driest state in which the samples could be produced and still retain their form. Following the standard light proctor test protocol (27 compaction blows and three layers of sample), one large cylindrical sample was created, from which four cores were extruded using aluminium cylindrical cutters and a machine press (Figure 23). Cores were then painted with three coats of liquid latex to provide a thin, impermeable membrane designed to replicate the external pressure of surrounding soil and to reduce the soil loss from the cores during wetting. To initiate testing, samples were put onto wet sand to allow water to enter the sample gradually over 48 hours before complete submersion; this was to avoid trapping air in the centre of the sample and reduce erosion of the end faces by slaking. At 48 hours samples were completely submersed and a 5 cm head was maintained. Every 24 hours the samples were weighed and measured over a period of 72 hours.

The drawback to the method used in Trial 2 was that the structure and natural heterogeneity of soil was partially destroyed by air drying and breaking down to <2 mm, which may have reduced the water uptake rate and total water holding

capacity (as a result of pore size reduction and potential hydrophobicity as discussed in relation to aggregate size and air drying in section 2.2)

<i>Sample</i>	<i>Soil 1 %</i>	<i>Compost 1 %</i>	<i>WTR1<sub>a</sub> %</i>	<i>Bulk density (BD)</i> <i>dry density (D<sub>d</sub>) g/cm<sup>3</sup></i>
Soil 1	100%	0	0	1.86 – 1.93 (BD) 1.64 – 1.70 (D <sub>d</sub> )
T2A	50%	50%	0	1.61 – 1.66 (BD) 1.42 – 1.46 (D <sub>d</sub> )
T2B	50%	0	50%	1.51 – 1.58 (BD) 1.33 – 1.39 (D <sub>d</sub> )
T2C	50%	25%	25%	1.55 – 1.61 (BD) 1.37 – 1.42 (D <sub>d</sub> )
T2D	50%	40%	10%	1.54 – 1.61 (BD) 1.35 – 1.41 (D <sub>d</sub> )
T2E	50%	30%	20%	1.56 – 1.63 (BD) 1.37 – 1.44 (D <sub>d</sub> )

*Table 19: Soil amendment ratios used in Trial 2 according to the dry mass of each component. 'n =' denotes the number of repeat tests.*

#### **4.4.4 Trial 3**

Trial 3 included a greater number of amendments and repetitions (Table 20), which included the use of silica that aimed to replicate the structural effect of WTR (due to the inert nature of silica), rather than the geochemical effect. Soil<sub>2</sub>, WTR<sub>2a</sub>, Compost 2 were used as the materials for this trial and were processed as outlined in section 4.2 (sieved to 6.3 mm, Soil<sub>2</sub> field moist and WTR<sub>2a</sub> air dried). Samples were mixed according to their dry mass and then slowly air-dried to 14% water content. For those samples that were <14% GWC when mixed, water was added to make up the deficit. Once at 14% water content the samples were sealed until use to avoid moisture loss.

The proctor mould method (Figure 23) used in Trial 2 was also used for Trial 3. During the production of samples, amendments containing compost (with the exception of A6) would not adhere sufficiently to enable them to be removed from the mould at 14% water content and required a further addition of water (increasing the samples to 25% water content). Once produced, the cores were

painted with liquid latex as discussed previously. For Trial 3, the wetting method was altered from Trial 2 to allow samples to take up water from the saturated sand for a longer period of time before submersion. Cores were measured and weighed at 24 hour intervals for a period of 120 hours before being fully submerged for a further 120 hours.

<b>Sample</b> <b>N= 8</b>	<b>Soil</b> <b>2%</b>	<b>Compost</b> <b>2%</b>	<b>WTR2<sub>d</sub></b> <b>%</b>	<b>Silica</b> <b>%</b>	<b>Bulk density (BD)/</b> <b>dry density (D<sub>d</sub>)</b> <b>g/cm<sup>3</sup> average</b>	<b>Water</b> <b>content</b>
Soil 2	100				1.85/1.60	14
F1	50	50			n/a	n/a
A1	50		50		1.55/1.37	14
A2	50			50	1.89/1.66	14
A3	50	25	25		1.49/1.19	25
A4	50	25		25	1.56/1.25	25
A5	60	40			1.42/1.25	14
A6	60		40		1.75/1.54	14
A7	60			40	1.96/1.73	14
A8	60	20	20		1.49/1.19	25
A9	60	20		20	1.50/1.20	25
A10	70	30			1.51/1.20	25
A11	70		30		1.62/1.43	14
A12	70			30	1.78/1.57	14
A13	70	15	15		1.52/1.22	14
F2	70	15		15	n/a	14

*Table 20: Soil amendments used in Trial 3. Bulk & dry density and water content values are for individual cores at production. F1 and F2 were omitted from the trial as the composition F1 would not yield a stable core before testing began and F2 fell apart considerably during wetting, invalidating the data. 'n =' denotes the number of repeat tests.*

F1 and F2 were omitted from the trial; during the production of F1 it was clear that this amendment would not yield sufficiently stable cores to extrude and paint with latex. During the wetting of F2 there was significant soil loss from the ends of the cores, giving a false indication of the mass; therefore, this data could not be used.

Although the water content and compaction effort remained consistent across the board, due to the variation of specific density of component parts and the different extents to which samples compacted under the same effort, the dry and bulk density of samples were significantly different. In some cases, the degeneration of a number of cores meant a reduction in the total number of cores for different amendments; some cores had significant loss of soil from the open ends during the wetting process, giving a false reading for the mass and indicating what would appear as a loss in water content.

#### 4.4.5 Trial 4 (Final Trial)

Trial 4 included the finalised water holding capacity method and four other tests (outlined in section 4.5). In this trial, the mass and volume change during both the wetting and drying was observed over time, with two wetting and two drying periods. Silica was removed from the testing materials and replaced by WTR<sub>2w</sub> as realistically, WTR would be added to soil in the form in which it is produced by water treatment companies, i.e. unprocessed and with a GWC of ~80%. Once the unprocessed WTR (WTR<sub>2w</sub> in this case), is incorporated into the soil, it would air dry naturally over time, this is the reason why all component materials were mixed at their stored water contents (Table 15) relative to their dry mass and then air dried to 17.5% GWC (Table 21).

<i>Sample</i>	<i>Soil2 %</i>	<i>Compost2 %</i>	<i>WTR2</i>
Soil2	100	0	0
AM1	90	10	0
AM2	90	0	10 WTR <sub>2d</sub>
AM3	90	0	10 WTR <sub>2w</sub>
AM4	90	5	5 WTR <sub>2d</sub>
AM5	90	5	5 WTR <sub>2w</sub>
AM6	80	20	0
AM7	80	0	20 WTR <sub>2d</sub>
AM8	80	0	20 WTR <sub>2w</sub>
AM9	80	10	10 WTR <sub>2d</sub>
AM10	80	10	10 WTR <sub>2w</sub>
AM11	70	30	0
AM12	70	0	30 WTR <sub>2d</sub>

AM13	70	0	30 WTR2 <sub>w</sub>
AM14	70	15	15 WTR2 <sub>d</sub>
AM15	70	15	15 WTR2 <sub>w</sub>

*Table 21: Soil amendment ratios used for Trial 4.*

Sixteen cores were made for each amendment, twelve of which were used for WHC testing, four of which were stored for use in triaxial testing (section 4.6) and XRCT (Chapter 6). These parameters were chosen so that all amendments would hold together during both the preparation stages and through wetting and drying cycles. By controlling the mass of each core, the resulting data would give an indication of the water holding capacity per unit mass of material. Previous trials had cores of varying density and mass, which meant there were many independent factors to consider during the analysis of results. The density of cores in Trial 3 were representative of what would occur in field conditions, where soil or amended soil at GWC of 14 or 25% had been subjected to a given compactive effort (e.g. by machinery or cattle). Trial 4 in comparison relates the water holding capacity to a given volume and mass of soil or amended soil.

The split mould method (Figures 22 and 24) was used to create 175 g cores, BD of 2 g/cm<sup>3</sup> and D<sub>d</sub> 1.75 g/cm<sup>3</sup>, using Soil 2, WTR2<sub>d</sub>, WTR<sub>w</sub> and compost 2. Each 25 g layer was scored on the surface after compaction to ensure that subsequent layers adhered to the previous layer. For samples that contained compost, each core was left in the split mould on the static press until the displacement needle remained stable (Figure 24A) to ensure that compost did not rebound and deform the sample before it had been painted with latex as in Figure 24C. Although the water content of different amendments was the same, it is likely for amendments with compost that the majority of water retained by the sample was contained within the compost as it acts as a sponge, rather than in the surrounding soil material. Samples without compost in contrast are likely to have homogeneous dispersion of water within them. By leaving the core in the mould with a static pressure applied, water held preferentially by compost is able to disperse evenly through the sample.

Once cores were removed from the split mould, they were immediately painted with liquid latex and geotextile (both highly permeable and elastic) was tied around the base to ensure no loss of soil. Cores were stored in airtight containers in the fridge until all cores were made. Samples, as with Trials 2 & 3, were placed onto saturated sand to begin the wetting cycle, during which the cores were weighed and measured every 24 hours. The initial wetting took up to 216 hours (the point at which the mass plateaued), after which they were flooded for a period of 48 hours (total of 264 hours). Cores were then placed on oven dry sand to begin the drying cycle, and once again weighed and measured every 24 hours until their mass reached 175 g. This process was completed twice to give two wetting and two drying cycles.



*Figure 24: Split mould and static compaction press method. (a) Top left - Pressure gauge on static compaction press. (b) Top right - Split mould and static compaction press. (c) Bottom left - Samples with high compost additions (where left has been removed from mould immediately after compaction and right after 1 hour of pressure equilibration). (d) Bottom right - Finished sample with latex coating and geotextile wrap*

#### 4.4.6 Density and water content considerations

Below is a summary of methods, method development and parameters used during Trials 1-4 (Table 22). As discussed in Chapter 2 (section 2.3.3) the following relationships between water content, compactive effort and density are known;

*Water content:* the water content of a soil controls the resultant degree of compaction for a given compactive effort, where the addition of water increases susceptibility to compaction and the bulk density increases up to the 'optimum' point where maximum (dry) density is reached (Figure 20).

*Compaction effort:* compacting different soils at a constant water content and compaction force will result in samples of different densities as some materials deform more easily than others (e.g. soil becomes much denser than compost at the same compactive force).

*Density:* different soil composition requires variable compactive effort to make a sample to a given dry density when using a constant mass. Amendments with high proportions of compost will require a greater compactive effort to reach the required dry density.

Water content was chosen as the control parameter for Trials 2-4, as it is a critical factor in the structure of soils during compaction and it is important to have a constant starting moisture content for trials considering the water content change over time. As Head (1980) states, the criterion for compaction must be ascertained: either compaction to bring the soil to a specified dry density (or void ratio) or to apply the soil for a known compactive effort. Compaction was controlled in Trials 2, 3 and 4 using either the Proctor method or a static press, giving variable resultant densities of samples, despite constant water content, due to the properties of the amendments.

As discussed in previous chapters the dry density and state of compaction of a soil sample largely governs how water moves into and through a soil (see section 2.4.4), where dry density and total porosity are commonly used parameters to characterize this attribute (Håkansson & Lipiec, 2000). Efforts to find a parameter that eliminated the differences in optimum compaction values have mainly revolved around relating the bulk density to a reference point, i.e. a bulk density obtained by a standardised compaction effort e.g. 200 kPa at a standard water

content. The issue is that much of this testing is for different soils, not amendment soils that contain high levels of organic matter.

For Trial 4 the water content and density were chosen control factors. Low dry density samples of the sample soil composition have a greater total pore volume and therefore have more space for water than denser samples, the exception being clays that can hold large amounts of water due to their high surface area and high micro-porosity despite their density. The bulk density and mass of cores were therefore chosen as constant factors to remove the effect of density variability, in addition to the control of water content. The known drawbacks to the use of this method firstly that compost is compacted to a significant degree to achieve the required density, removing one of the positive effects on structure that compost imparts, and secondly each amendment will be at different degrees of compaction. Unfortunately, one can only choose two options from the three parameters of density, water content and compactive effort.

<i>Trial #</i>	<i># of samples</i>	<i># of amendments (inc. soil alone)</i>	<i>Period of wetting</i>	<i>Period of drying</i>	<i>Bulk density (BD) dry density (D<sub>a</sub>) g/cm<sup>3</sup></i>	<i>Water content %</i>	<i>Exterior coating of cores</i>
1	3	6	24 hours	n/a	1.53 – 1.87 (BD) 0.86 – 1.55 (D <sub>a</sub> )	11.7-44	Rubber membrane
2	4	6	48 h sand bed, 72 hours flood	n/a	1.51 – 1.93 (BD) 1.33 – 1.70 (D <sub>a</sub> )	13.5	Latex
3	8	16	144 h sand bed Up to 336 hours flooding	n/a	1.42 – 1.96 (BD) 1.19 – 1.66 (D <sub>a</sub> )	14 & 25	Latex
4	12	16	216 h sand bed 48 h flood	216 h	2.0 (BD) 1.73 (D <sub>a</sub> )	17.5	Latex & geotextile

*Table 22: Summary of method parameters for Trials 1 -4*

#### 4.4.7 Statistical testing

The use of statistics is widely used in science to test whether the central tendencies, i.e. mean or median, of two or more groups are significantly different from one another (Ruxton, 2006). In statistical testing a null  $H_0$  and an alternative hypothesis  $H_a$  are required, where the former is the default position that there is no relationship (dependent) or no difference (independent samples), and the alternative hypothesis states that there is a relationship or difference between two data sets. The significance level ( $\alpha$ ) is the probability of rejecting the null hypothesis, where typically this value is 0.05 (95%) or 0.01 (99%), and the  $p$  value is the probability of a result being of the same value should  $H_0$  be true. Statistical significance is achieved when  $p < \alpha$ , and therefore one can accept the alternative hypothesis by rejecting the null hypothesis. The smaller the  $p$  value, the greater the significance, therefore one can have degrees of significance. Data can be split into two groups based on their theoretical distributions (parameters), which determine the type of statistical analysis that can be used. Parametric statistical tests make a number of assumptions about the data including the normality population distribution, and variance, however non-parametric tests make fewer assumptions and do not require 'normally distributed data' (Altman & Bland, 2009).

Parametric methods include  $t$ -tests and analysis of variance (ANOVA) for testing differences and least squared regression and correlation for testing the relationship of dependent variables. Non-parametric methods include the Wilcoxon Mann-Whitney U or rank sum test (MWU), Kolmogorov-Smirnov and sign test, which work using the rank of data rather than making assumptions about the distribution or variance of the data. The issue with checking normality in small data sets is that it is difficult as formal testing has low power, so violations of the assumptions may not be detected, but with a large number of samples ( $n$ ) you can depend on the central limit theorem. This means that with enough observations with a finite level of variance, the mean of the samples will equal the mean of the population as a whole, the asymptomatic normality of the test statistic and  $t$  distribution. With no way to accurately check this, one must assume no normality and use non-parametric methods. Rank tests are reasonable defaults if one expects non-normality, due to small sample size and apparent presence of outliers that

skew plotted data (which is likely to be the case with data based on soil variability).

In an attempt to statistically model data produced, a report conducted by Perksy (2017) developed a mathematical model that allowed important qualitative statements about the physical properties of the tested soils with mathematical rigour, focusing on the drying phase of data from Trial 4. Bayesian Inference with Markov Monte Carlo was used to perform analysis and deduce the properties of a population as a whole by incorporating prior knowledge of soil characteristics and data from the experiment. However, the use of this method to determine statistical difference requires an extended knowledge of mathematics, beyond the current knowledge base of the author.

The Mann Whitney test (MWU, Mann & Whitney, 1947) is commonly used to compare the efficacy of two treatments in medicine, and can be applied to other disciplines that have a similar research aim. It is commonly used as an alternative to t-tests when data are not normally distributed or other assumptions of parametric data are not met, therefore the data are non-parametric (Ruxton, 2006; Fagerland & Sandvik, 2009). As the MWU test compares the sum of ranks, it is less likely to spuriously indicate significance because of the presence of outliers (therefore it is more robust). The MWU test is the default test for comparing ordinal measurements (such as mass) with similar distributions. It does however assume that two independent samples have the same shape (distribution) and spread (variance), but with difference medians and location (Skovlund & Fenstad, 2001).

The MWU detects if 2 or more samples come from the same distribution, or whether the medians of the group are different. The null hypothesis is therefore that it is equally likely for one randomly selected value from one sample to be less or greater than a randomly selected value from the other sample. For this research therefore, the null hypothesis is that a core of material is equally likely to have a higher or lower water holding capacity than a core of material from a different amendment. The test therefore determines if the samples were taken from the same population (i.e. there is no difference in WHC) or different populations. The

alternative hypothesis states that one distribution is greater than the other. The test makes the following assumptions:

1. The data are ordinal i.e. one observation can be shown to be greater than another e.g. by use of decimals.
2. The dependent variable (WHC as defined by the GWC) is not normally distributed.
3. The data of the two groups have equal variances.
4. The shapes of the distribution are the same, although the location can be different.
5. The sample drawn from the population is random.

The data from WHC testing were assumed to be non-parametric due to the small sample size ( $n = 12$ ) that effectively invalidates normality testing and in addition typical parametric analysis requires groups of data greater than 15. The MWU is commonly known as a test for equality of medians, however it actually is able to detect differences in shape and spread rather than just a difference in means (Hart, 2001). The Mann Whitney statistic is highly informative as it gives the probability that one group individual will score higher than the other, particularly in the analysis of controlled treatment trials. For the purposes of this test, only the statistical significance is needed, as the distribution of data is of lesser importance and the hypothesis are as follows:

*$H_0$  = there is no difference between the control sample (Soil 1 or Soil 2) and the amended sample (AM1-AM16)*

*$H_{a1}$  = there is a difference between the control sample (Soil 1 or Soil 2) and the amended sample (AM1-AM16), where the amendment is expected to increase the water holding capacity of the soil, as indicated by the gravimetric water content.*

*$H_{a2}$  = there is a difference between samples with different amendments (e.g. AM1 vs AM2), however the direction of change is not known*

The primary alternative hypothesis ( $H_{a1}$ ) was tested assuming the amended sample would be greater than the control, which suggests the use of a one-tailed

test however a two tailed test was used for both the primary and secondary alternative hypothesis to avoid missing a detrimental (decrease) impact of amendment on the WHC. A significance level of 95% was chosen, therefore 'a' must be less than 0.05 to be given a significant result. Due to the differences in initial parameters (water content and density) and very small data sets where  $n = 5-8$  statistical testing between sample populations, testing was not appropriate on data from Trials 2-3.

For the statistical analysis of Trial 4, significance tests were not performed on the time series as a whole, but at important points through the wetting and drying cycles; 24 hours, 288 hours (maximum GWC), 312 hours (24 hours of drying), 600 hours (24 hours of rewetting), 912 hours (second maximum GWC), 936 (second 24 hours of drying), 1068 hours (end point).

#### **4.5 Erosional resistance testing**

According to Lal (2003) the choice of an appropriate index for testing erodibility of soils is governed by the relevance to the processes that control soil erosion in the natural field environment. It is for this reason that, as stated in Chapter 2 there is no standard index by which erodibility is measured. Two methods were used to measure the erodibility and cohesiveness of amended soils; the Veitch method and fall cone penetrometer. The former is a novel method using water drop erosion to a determined point of failure (KE index, measuring the kinetic energy required to disrupt an aggregate), and the latter is a standardised method that tests the shear properties of a saturated soil. These tests were chosen to test the effect of amendment on the cohesion and stability of samples under rainfall simulation. As stated in literature (Chapter 2, section 2.3.5), rainfall simulation is suggested as the most suitable method to approximate the erodibility of a soil. For this reason, the Veitch method was developed to test the stability of a small soil sample under a controlled supply of drops.

##### **4.5.1 The Veitch method**

Discs of 10 mm height x 38 mm diameter were formed as per amendments in Trial 4 (Table 21), using the static press and split mould method with a single 25 g layer of material compacted at 17.5% GWC to a density of 2 g/cm<sup>3</sup> (BD), 1.73 g/cm<sup>3</sup> (D<sub>d</sub>).

The test was designed to induce rapid wetting under rainfall simulation to test the ability of the sample to resist slaking and aggregate breakdown. The subjectivity of other tests, which use single drops to break a sample down to a given end point, is somewhat negated by designating an easily identifiable end point of the test (when the surface of the sample or the whole of sample first experiences failure, through cracking as in Figure 25, or slumping) and using a controlled rate of water delivery. Drops fell onto the centre of the sample from a height of 310 mm, with a resultant drop velocity of 2.46 m/s and a kinetic energy of 0.00036 Joules. Although the test had significantly lower velocity than natural rainfall, which ranges from 2 – 7 m/s depending on drop size 0.5- 2.6 mm and rainfall intensity 1- 6 mm/hour respectively (Pruppacher, 1981), this was the greatest height achievable in lab to ensure that drops reliably fell centrally on the sample. Samples were mounted onto a curved plastic standing with a radius of 37 mm. 24 replicates of each amendment were tested, where half were tested immediately after production, and the remaining 12 samples were completely saturated and dried back to a mass of 25 g. This was to replicate the effects of a wetting and drying cycle on the materials. The time from initial drop to the point of failure was recorded, from which the number of drops and mass of water required for failure could be calculated. The sample was immediately removed from the plastic mount, weighed and placed into an oven for 24 hours at 105° C to calculate the water content.

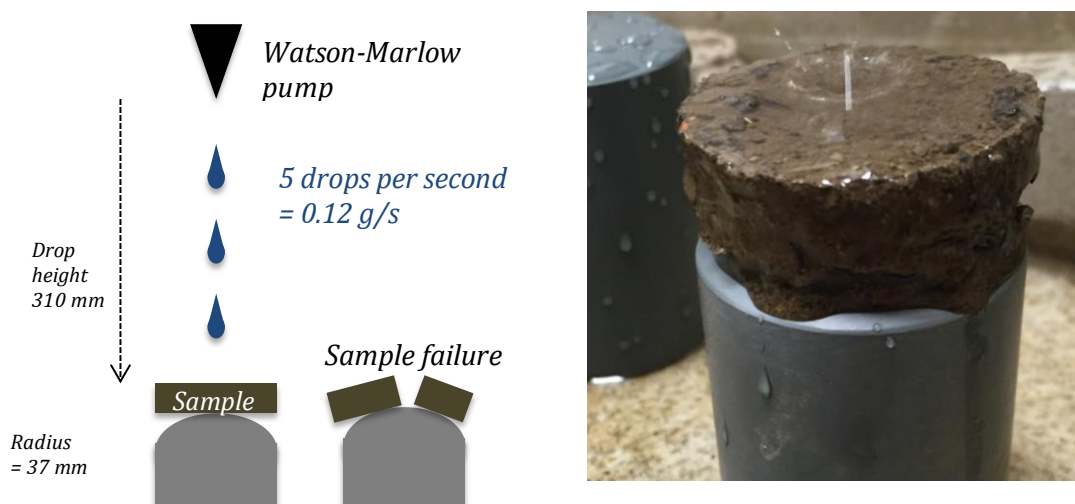


Figure 25: Schematic diagram (left) of the Veitch method used to test aggregate stability of disk shaped samples and a photo of a sample during testing (right)

#### 4.5.2 Fall cone test

The fall cone test is commonly used as a quick test of undrained shear strength following Hansbo's equation (Equation 17) and testing using this method followed the procedures described in BS1377: 1990. Samples were made following the procedures used in Trial 4, but to dimensions of 38 mm x 38 mm (four layers rather than seven) to match the fall cone test apparatus requirements. Briefly, the test uses a stainless steel cone to penetrate a saturated soil sample under the influence of gravity where the apex is flush with the surface of the sample, after which the depth of penetration is measured using a dial gauge after 5 seconds have passed from release of the cone (Tanaka *et al.*, 2012). 24 replicates were completed for each amendment, 12 of which were tested 24 hours after production and 12 of which were tested having been fully saturated and air-dried back to the initial water content. The undrained shear strength in kPa can be calculated using Equation 12 from the data obtained in the testing.

$$C_u = k \propto \left( \frac{mg}{d^2} \right)$$

*Equation 17: Hansbo formula, where  $C_u$  = undrained shear strength,  $m$  is mass of the cone,  $g$  is gravity,  $d$  is the penetration depth and  $k \propto$  is the cone factor based on cone angle (ranges between 0.1 and 1). Hansbo (1957).*

#### 4.6 Triaxial testing

Triaxial testing has several advantages over other shear testing methods as complete control of testing conditions, including the measurement of the drainage conditions, pore pressure, volumetric changes, compressibility, permeability, stress distribution can be obtained with ease. The drawback of this method is that it is very time intensive (and therefore expensive) and may not truly reflect the effects of loading that may occur in the field. However the test provides accurate, adaptable data that provides a reliable test for the shear characteristics of a specimen. The triaxial test is performed on cylindrical specimens that are either trimmed from field samples, or as in this research, synthesised in the lab to best represent field conditions. The sample is contained within an impermeable latex membrane before being mounted for testing in the triaxial cell (Figure 26).

Some cores may have been subject to thixotropic effects due to the duration over which testing was completed. The samples were tested according to their amendment number (i.e. Soil 2 and AM1 were tested first and AM13, AM14 last); therefore this must be factored into the results. The procedure for testing the shear strength of the soil was as follows and includes a brief description of the steps taken. For a thorough review of the process readers are directed to Lade (2016) for information on initial set ups and test procedures. All samples from Trial 4 were tested for their permeability at pressures of 25 kPa, 50 kPa and 100 kPa. Three cell pressures of 25 kPa, 50 kPa and 100 kPa were used for Soil 2, and AM11-AM15 in order to produce a set of Mohr's circles, stress strain curves and stress paths. This meant three samples were used for the shear strength testing as the process destroys each core by testing it to failure. For permeability measurements (AM1-AM10), only one core was used where permeability testing at 25 kPa was completed before increasing the cell pressure to test at 50 kPa and so forth.

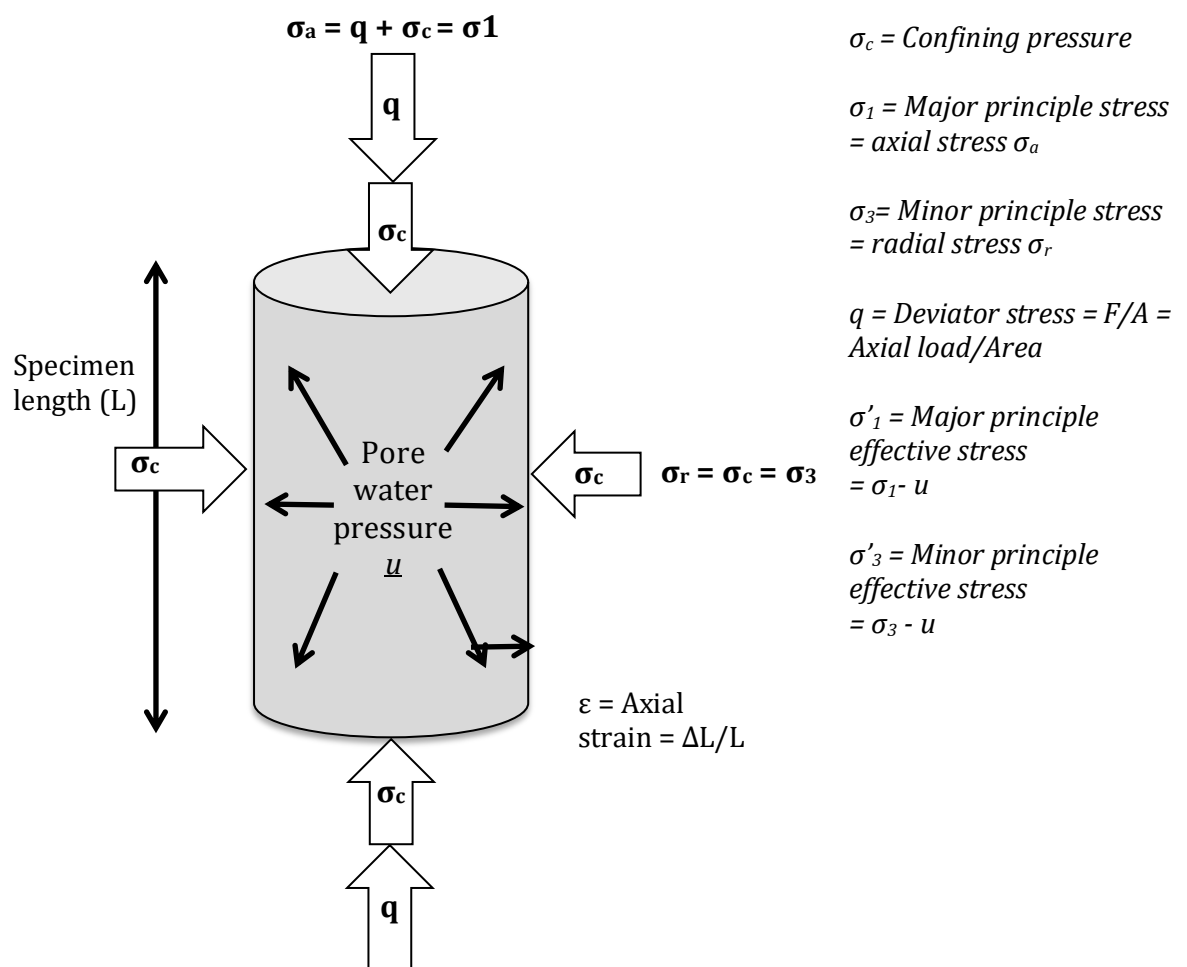


Figure 26: Schematic of the strain ( $\epsilon$ ) and stress ( $\sigma$ ) states undergone by a sample during triaxial testing

An experienced lab technician completed the consolidated undrained triaxial testing according to BS 1377-8:1990 Part 8, which comprises briefly: firstly, test specimens were measured (average radius and height), weighed and inserted into a latex tube, into which air free porous disks are placed at the end face of each cylindrical specimen. The sample was then placed into the triaxial cell and secured using rubber O-rings. Once the cell was sealed, it was filled with water and the saturation of the sample was started, where cell pressure (not exceeding 305 kPa) and back pressure (aiming to reach 300 kPa) was applied. The saturation process aims to fill all voids with water and properly de-air the pore pressure transducer and drainage lines. Pore water pressure readings were taken until the change was negligible, as an increase in pore water pressure suggests air presence. Skempton's B value (a determination of how saturated the sample is) was calculated once cell pressure was increased by 100 kPa. For some materials, the B value will reach 1 (100%) at full saturation, however for very dense samples this value may only reach 0.91. The level of saturation, calculated using Equation 18 must have been < 95% or 0.95 before consolidation could begin, which indicated a saturation level exceeding 99%.

$$B = \frac{\Delta u}{\Delta \sigma_3}$$

*Equation 18: Skempton's B value calculation where  $\Delta u$  is the difference between initial and maximum pore pressure and  $\Delta \sigma_3$  is the difference between initial and maximum cell pressure.*

At this point the sample was fully saturated and all voids were filled with water. In the subsequent phase, called consolidation, cell pressure was increased to the specified limit (325, 350 or 400 kPa). Readings were taken until the volume change of the specimen was negligible and 95% of the excess pore pressure, created as a result of cell pressure change, had dissipated. This process brought the specimen to a given effective stress (total pressure/stress – back pressure) required for shearing and permeability tests. The permeability of the sample was tested prior to shearing by applying a 15 kPa difference in pore pressure, and the volume change in water above and below the sample measured to be able to calculate the permeability using Equation 5 (section 2.4.4.)

$$k = \frac{qL}{Ah}$$

*Equation 5: Hydraulic conductivity ( $k$ ), where  $q$  is the permeability coefficient (flow in  $m^3/second$ ),  $L$  is the length of the sample in  $m$ ,  $A$  is the cross-sectional area of the soil ( $m^2$ ) and  $h$  is the pressure head (in  $m$ ). Craig (2004).*

After testing the permeability of the sample, it was sheared; during the shearing process the vertical load, pore water pressure and vertical displacement were monitored until there was a change in displacement (height of the sample) of 20% of the original height of the sample. This entire process was completed three times, to test the hydraulic properties and shear strength at three pressures, 25, 50 and 100 kPa.

From this data, hydraulic gradients, Mohr circles/stress strain graphs and stress paths may be determined. The shear strength is the maximum shear stress, determined from the radius of the Mohr's circles (example in Figure 27) or from the stress strain graph (Figure 28). From Mohr's circles we may obtain the angle of friction and the cohesion values. However cohesion is a vague concept, as only soils with physio-chemical bonding have a true cohesion, but many soils will show an apparent cohesion in triaxial testing as the result of volume change, which gives a higher peak state than critical state (apparent cohesion, as discussed in Chapter 5, section 5.3.2). As the failure envelope drawn in Figure 27 is at the discretion of the person obtaining the value, the values of cohesion and angle of friction are exceptionally sensitive to the gradient of the failure line, which may be drawn far from the 'true' value. In addition the Mohr circle only shows the condition at one axial stress point. As we are able to obtain the maximum shear strength from a stress strain curve (by dividing the deviator stress by two, Figure 28), and from stress paths (Figure 29) one can obtain the angle of friction, critical state line and peak line, we may use these two representations rather than Mohr's circles to analyse the continuous variation in behaviour rather than just a snapshot of the point of failure.

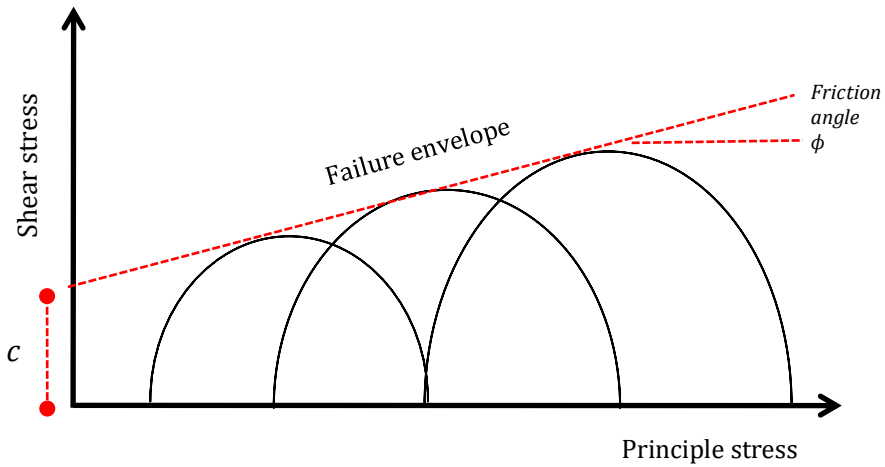


Figure 27: Example of Mohr Circles, plotted with a failure envelope (which is idealistic and unlikely to be linear), angle of friction, and cohesion ( $c$ ).

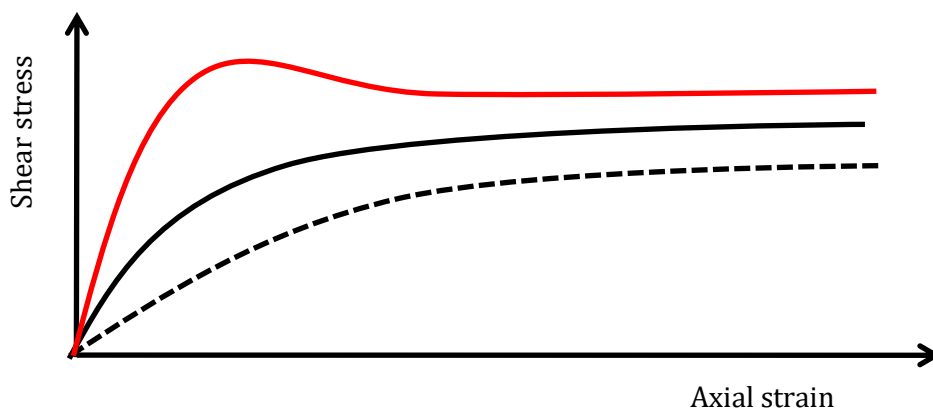


Figure 28: Example of stress strain curves for a soft material (black dashed), a typical stress strain curve of a soil for which the maximum strength is coincidental with the critical state (black), and an overconsolidated sample that reaches a peak strength and subsequently reaches an ultimate state with increasing axial strain (red).

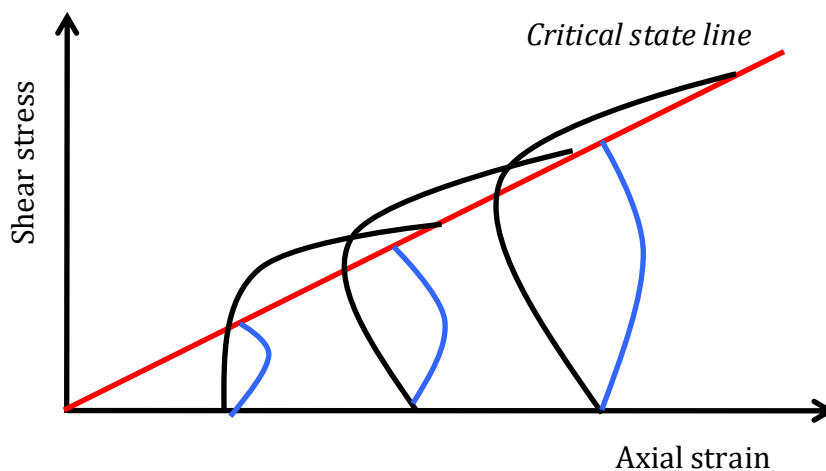


Figure 29: Example of stress paths for dilatant (black) and compressive (blue) samples to the critical state line (red)

#### 4.7 Concluding remarks

This chapter has outlined specific details regarding the source, preparation and storage of materials used in this research. These materials have been characterised using British Standards. The chapter clarifies the methods of testing gravimetric water and volumetric water content values, and subsequently outlines the development of methods to determine the water holding capacity through Trials 1-4 which briefly comprise;

- Trial 1: Exploratory trial to test core production methods with materials in 'field' condition.
- Trial 2: Exploratory trial to test core production methods using texturally homogenised and air-dried materials, wetted to a consistent water content.
- Trial 3: Large scale trial using two controls (water content and compaction effort) for core production using the Proctor mould method, testing 13 different amendments, up to 50% amendment, with up to 12 repetitions ( $n = 12$ ). Materials used in this trial were Soil 2, compost, silica and WTR2<sub>d</sub>
- Trial 4: Final large-scale trial using two controls (water content and bulk density) for core production using the split mould method. Trial 4 tested 15 different amendments, up to 30% amendment where  $n = 12$ . Materials used in this trial were Soil 2, compost, WTR2<sub>d</sub> and WTR2<sub>w</sub>.

In addition to water holding capacity trials, the chapter outlined triaxial apparatus methods by which the hydraulic conductivity and shear strength of soils were tested, a British Standard test to determine the undrained shear strength (fall cone test), and a novel test (the Veitch method) to determine the erodibility of a soil via raindrop detachment.

## 5. Results and Discussion

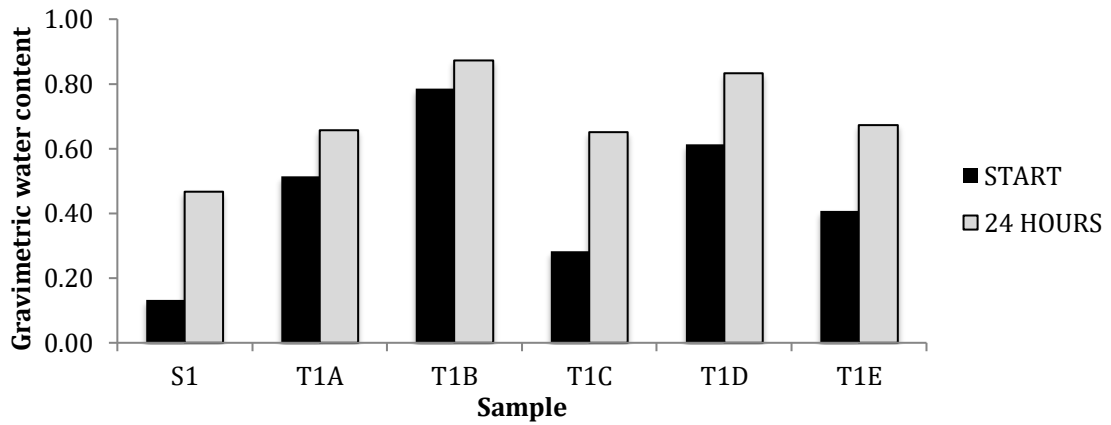
The following chapter presents data on four soil functions, WHC, erosional resistance, shear strength and hydraulic conductivity. This includes experiments completed for this thesis and supplementary work completed conducted by students under supervision of the author, for the purposes of measuring soils and their amended counterparts, which is pertinent for the discussion on what effect amendment has on soils.

### 5.1 Water holding capacity

The testing of the water holding capacity of soils and amended soils was completed over four trials using a novel methodology. Initial testing used only a small variety of amendments, where Trials 3 and 4 had a much greater range of amendments and replicates (denoted by  $n =$ ).

#### 5.1.1 Initial results: Trials 1 & 2

The outcome of Trials 1 & 2 determined how Trials 3 & 4 were conducted and as such the results from Trials 1 & 2 are presented here in order to briefly discuss the limitations of their design and are not used to inform the discussion of hypotheses. Each trial is discussed to demonstrate how the various methods and initial parameters such as moisture content are enormously important in determining the data produced. Figures 30-34 below show the change in gravimetric water content, volumetric water content and density respectively in Trial 1. As discussed in Chapter 4, all volumetric water contents (VWC) will also be displayed with the instantaneous volumetric water content (VWC<sub>i</sub>), which is a ratio of the volume of water to the volume of bulk soil at the point of measurement rather than VWC, which is the ratio of volume of water to the original volume of the soil. Samples are labelled S1, corresponding to unamended Soil1, and T1A, T1B, T1C, T1D and T1E corresponding to amendments using Soil1 (S), Compost1 (C) and WTR1<sub>d</sub> or WTR1<sub>w</sub>, which are outlined below each figure.



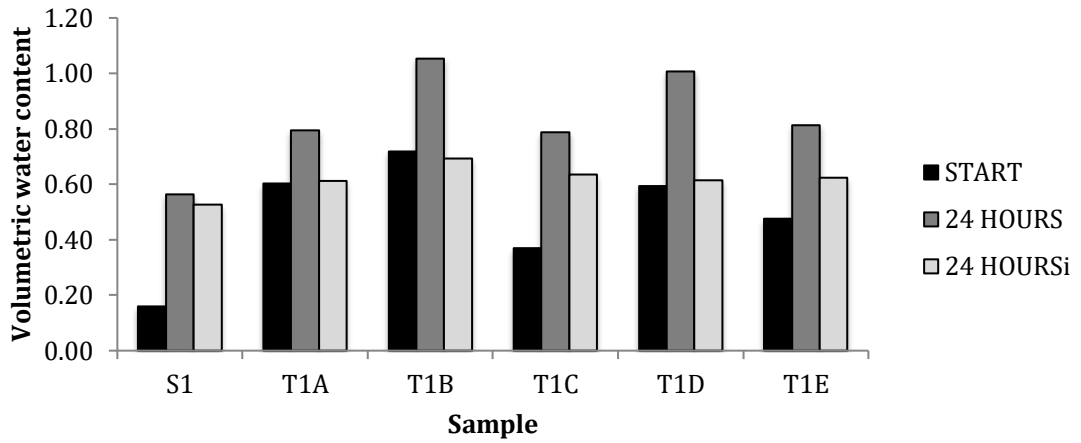
<i>S1</i>	<i>T1A</i>	<i>T1B</i>	<i>T1C</i>	<i>T1D</i>	<i>T1E</i>
100 % soil	50S 50C	50C 25C	50S 25C	50S 40C	50S 40C
		25WTR1 <sub>w</sub>	25WTR1 <sub>d</sub>	10WTR1 <sub>w</sub>	10WTR1 <sub>d</sub>

where S = Soil1 and C = Compost1

Figure 30: Trial 1's average gravimetric water content of Samples S1 and T1A-T1E where  $n = 1$ . 'Start' indicates the initial conditions of the samples, and '24 hours' indicates the GWC after 24 hours of submersion.

The initial GWC for Trial 1 (shown in Figure 30) was considerably different between samples, ranging from 0.13 (S1) to 0.79 (T1B). In general, the samples with the highest change were the ones that began with the lowest GWC, where T1C had the greatest increase (from 0.28 to 0.65) compared with unamended soil (S1: from 0.13 to 0.47). The starting water content is therefore of essential importance as samples with greater initial gravimetric water content will be able to take up less water pro rata and therefore a direct comparison cannot be made.

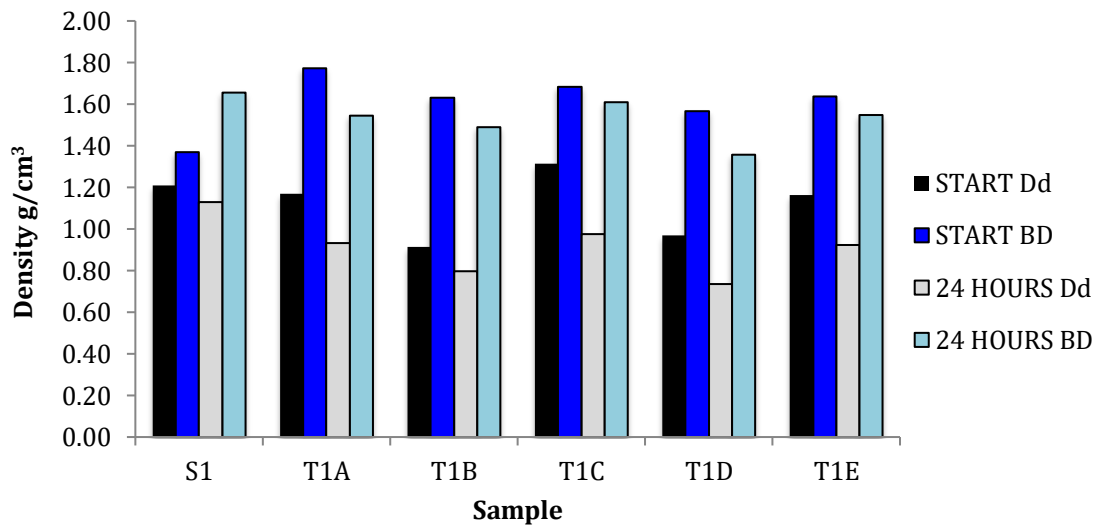
The volumetric responses of samples (Figure 31) are similar to the gravimetric water content change show in Figure 30. In general, the greatest change in VWC was shown for samples with lower initial VWC (S1 and T1C).



S1	T1A	T1B	T1C	T1D	T1E
100 % soil	50S 50C	50C 25C 25WTR1 <sub>w</sub>	50S 25C 25WTR1 <sub>d</sub>	50S 40C 10WTR1 <sub>w</sub>	50S 40C 10WTR1 <sub>d</sub>

where S = Soil1 and C = Compost1

Figure 31: Trial 1's average volumetric water content (VWC) and VWCi, where  $n = 3$  with the exception of S1 where  $n = 1$ , at the start point and 24 hours after submersion.

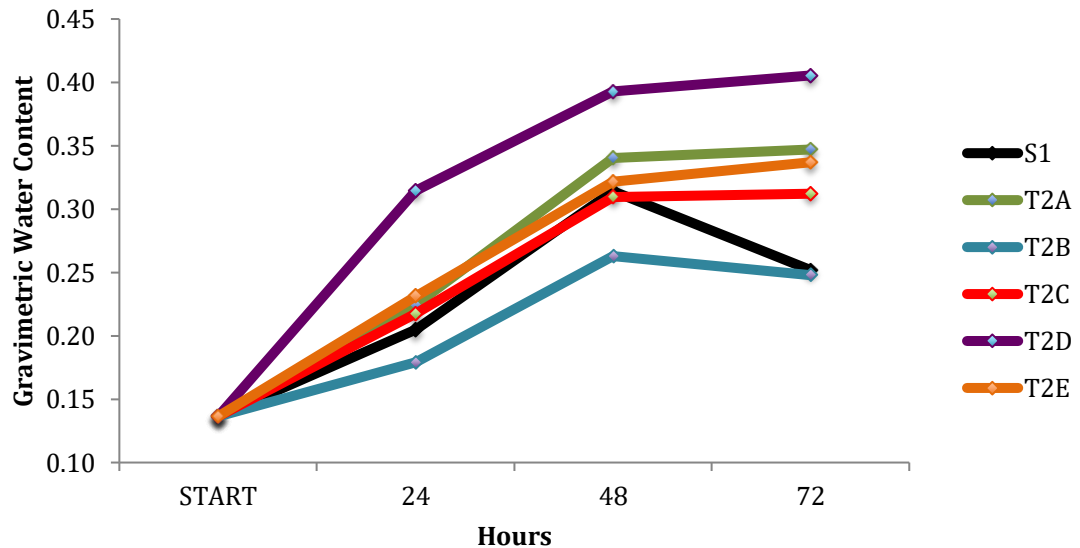


S1	T1A	T1B	T1C	T1D	T1E
100 % soil	50S 50C	50C 25C 25WTR1 <sub>w</sub>	50S 25C 25WTR1 <sub>d</sub>	50S 40C 10WTR1 <sub>w</sub>	50S 40C 10WTR1 <sub>d</sub>

where S = Soil1 and C = Compost1

Figure 32: Trial 1's average dry density ( $D_d$ ) and bulk density (BD) of samples, where  $n = 3$  with the exception of S1 where  $n = 1$ , at the start point and 24 hours after submersion.

As shown in Figure 32, there were small changes in the bulk density and dry density as a result of volume change of the samples as they took up water. The dry density decrease was lowest for S1 (0.08 g/cm<sup>3</sup>), and largest for T1C (0.34 g/cm<sup>3</sup>). The bulk density of all samples increased with the exception of S1, which had a 0.29 decrease, the former of which is due to an increase in water in amended samples without a large change increase in sample volume.



S1	T2A	T2B	T2C	T2D	T2E
100 % soil	50S 50C	50S 50WTR2 <sub>d</sub>	50S 25WTR1 <sub>d</sub>	25C 50S 10WTR1 <sub>d</sub>	40C 50S 30C 20WTR1 <sub>d</sub>

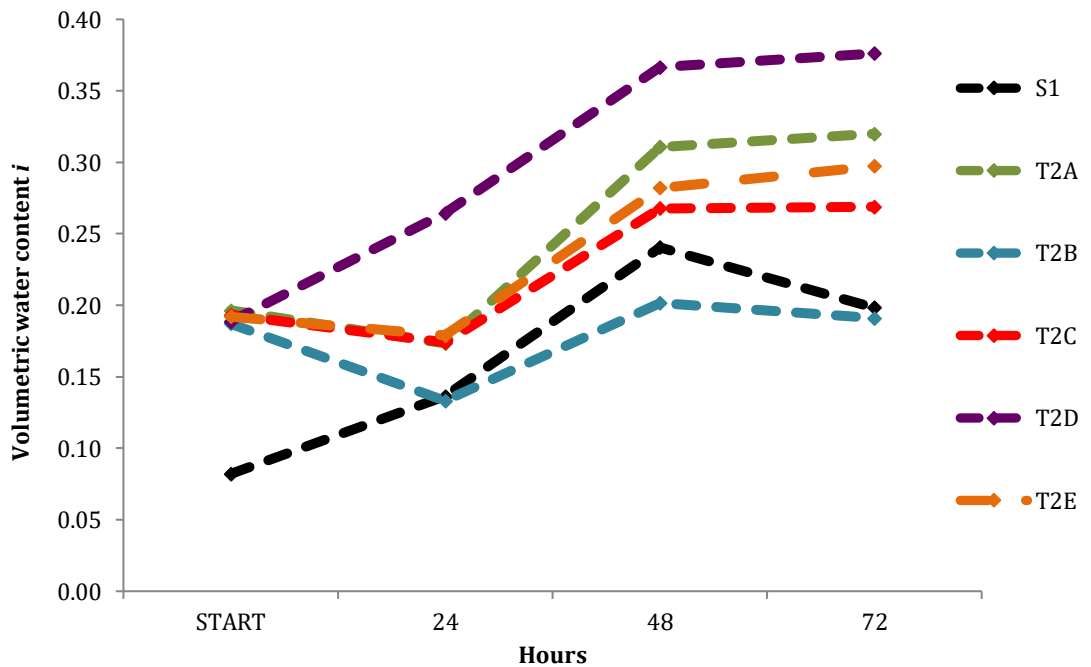
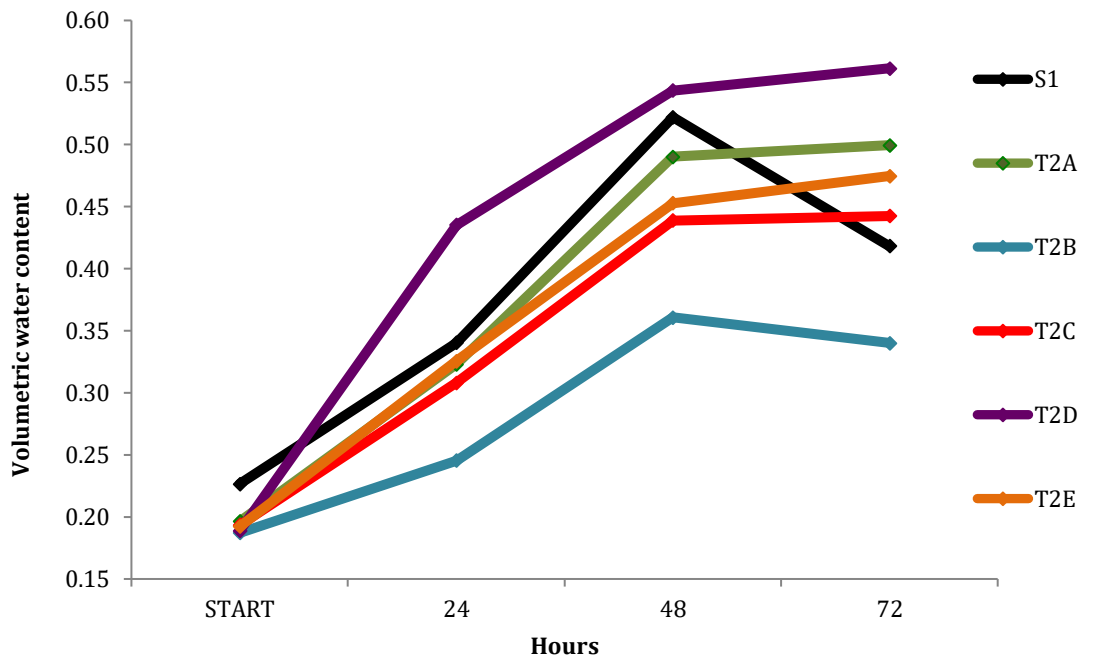
where S = Soil1 and C = Compost1

Figure 33: Trial 2's average GWC (where n = 4) over 72 hours of submersion.

In Trial 2, S1 remains as the unamended soil and 5 amendments are annotated T2A – T2E. All component materials (soil, compost and WTR) were air-dried before mixing and the controlled addition of water after combination meant that the initial gravimetric water content was equal for all samples, improving the methodology from Trial 1. As a result of this change a better comparison can be made between samples than in the previous trial where the initial water content was variable (Figure 31). All values of GWC were greater than unamended soil over time with the exception of T2B (50S 25C 25WTR1<sub>w</sub>). T2D (50% co-amendment) had the highest GWC after 72 hours of submersion, above the single 50% amendment of compost (T1A). However, literature suggests that the soil with the greatest amount of organic matter is likely to hold the most water (up to 10

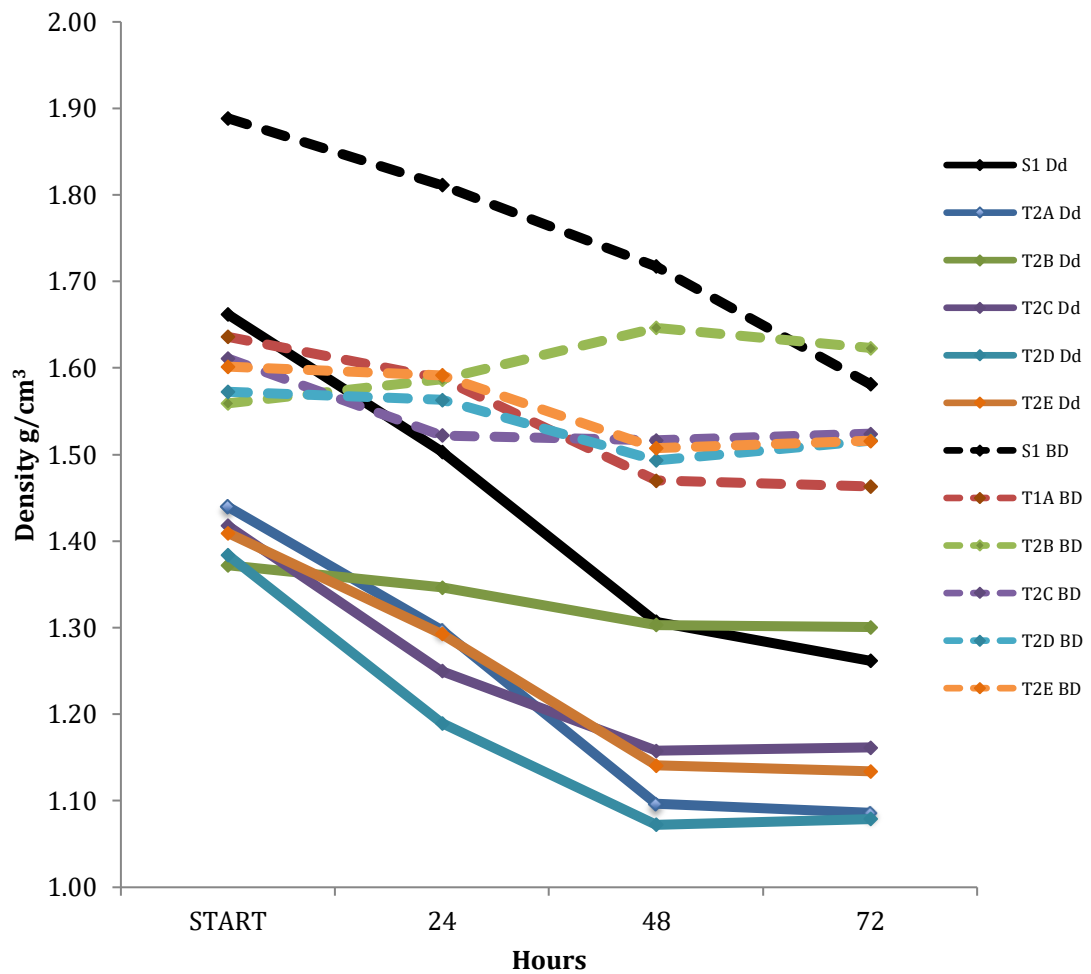
times its own weight, see section 2.2.1). This result comes as a non sequitur and provides an example of why keeping materials such as compost as close to 'field' conditions as possible is crucially important, rather than removing their beneficial properties (water holding capacity and structural improvement) by air drying and grinding. Although all amendments were made up to the same water content, the air-drying of compost and soil would have been detrimental to the water holding capacity. As discussed in the methods chapter, the breakdown and drying of materials is likely to have compromised or altered the benefit of the addition of dry WTR or compost from a structural perspective (Kaiser *et al.*, 2015). Therefore these results do not reflect how an amendment may affect the maximum GWC of a soil.

Figure 34 shows the volumetric response of samples over the wetting period of 72 hours. It clearly displays the importance of having both the VWC and VWCi information. Should one just look at the VWC of S1 vs amended samples, only T2D has a higher VWC, however this is compared to their original volumes. What you cannot see is that for all amended samples (excluding T2B) is that their bulk volume has also increased, meaning that the VWCi (ratio of volume of water to volume of solids) is greater than unamended soil.



<i>S1</i>	<i>T2A</i>	<i>T2B</i>	<i>T2C</i>	<i>T2D</i>	<i>T2E</i>
100 % soil	50S 50C	50S 50WTR <sub>2d</sub>	50S 25C 25WTR <sub>1d</sub>	50S 40C 10WTR <sub>1d</sub>	50S 30C 20WTR <sub>1d</sub>
where <i>S</i> = Soil1 and <i>C</i> = Compost1					

Figure 34: Trial 2's average VWC and VWC<sub>i</sub> (where  $n = 4$ ) over 72 hours of submersion.



<i>S1</i>	<i>T2A</i>	<i>T2B</i>	<i>T2C</i>	<i>T2D</i>	<i>T2E</i>
100 % soil	50S 50C	50S 50WTR2 <sub>d</sub>	50S 25C 25WTR1 <sub>d</sub>	50S 40C 10WTR1 <sub>d</sub>	50S 30C 20WTR1 <sub>d</sub>

where *S* = Soil1 and *C* = Compost1

Figure 35: Trial 2's average dry density (annotated with Dd) and bulk density (annotated with BD) of samples ( $n = 4$ ) over 72 hours of submersion

Figure 35 shows the density change (dry density and bulk density) over the 72-hour wetting period of Trial 2. The values of T2B appear to be erroneous as the values of bulk and dry density must both either increase or decrease together, whereas in fact they increase and decrease respectively. In this case the bulk density of T2B increases at 48 hours but continues to decrease in dry density. This is not a trend that can be observed unless the fluid entering the soil mass is denser than substrate and there is an increase in substrate volume (which in this case it is not). Therefore this must be due to measurement error for this particular sample.

In general, all amendments appear to reduce the respective bulk and dry densities in comparison with unamended soil.

### 5.1.2 WHC Trial 3

Briefly, samples were placed onto a saturated bed of sand to allow uptake from the base of each core before being fully submerged after 96 hours. The test was deemed complete and the measurement of mass and volume of samples ceased when the mass reached a stable value (<0.5 g difference to previous value), indicating no further uptake of water, or a decline in mass that indicated that the soil was beginning fall away from the ends of the core. The number of repeats within each amendment sample (n) is noted in the each figure reference, as these were variable; eight cores were made for each amendment, however some of these cores deteriorated during the wetting process and data is not provided for these cores. Figure 36 provides an overview of the gravimetric water content (GWC) of Soil2 and 13 amendments over a wetting period of 240 hours, following the methods outlined in Chapter 4 (4.4.4), where the amendment proportions are outlined subsequently in Table 23.

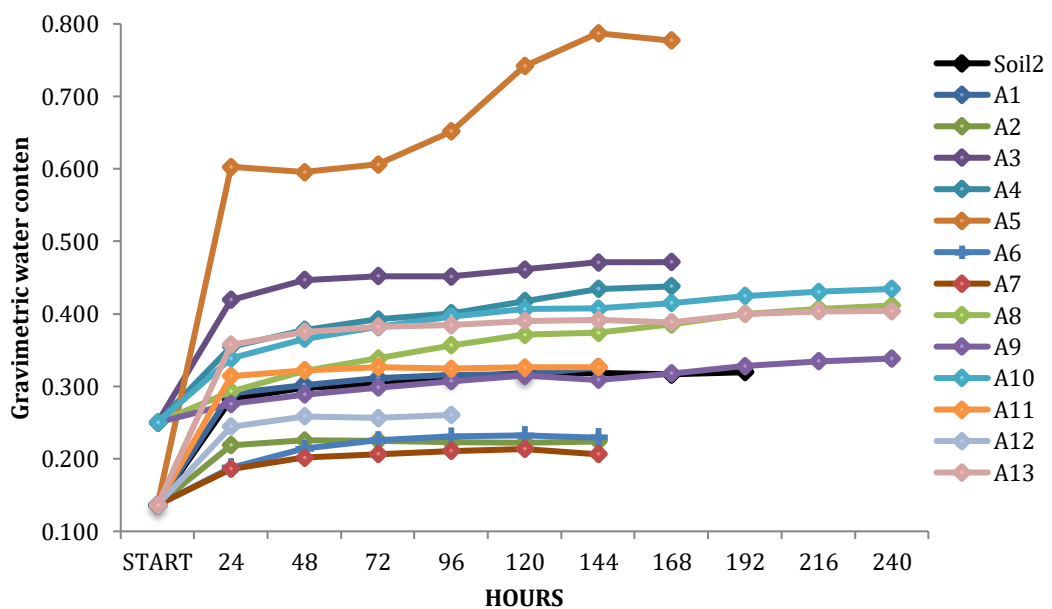


Figure 36: Average GWC of samples up to 240 hours of wetting according to the method outlined in section 4.4.4. n is between 5 and 8. The measurement of some amendments ceased once the mass reached a plateau. Note that samples have two different starting GWC, 0.14 (14%) and 0.25 (25%).

<i>Sample</i> <i>n= 8</i>	<i>Soil</i> <i>2%</i>	<i>Compost</i> <i>2%</i>	<i>WTR2<sub>d</sub></i> <i>%</i>	<i>Silica %</i>	<i>Bulk density (BD)/dry</i> <i>density (D<sub>a</sub>) g/cm<sup>3</sup></i>	<i>Water</i> <i>content</i>
Soil 2	100				1.85/1.60	14
F1	50	50			n/a	n/a
A1	50		50		1.55/1.37	14
A2	50			50	1.89/1.66	14
A3	50	25	25		1.49/1.19	25
A4	50	25		25	1.56/1.25	25
A5	60	40			1.42/1.25	14
A6	60		40		1.75/1.54	14
A7	60			40	1.96/1.73	14
A8*	60	20	20		1.49/1.19	25
A9*	60	20		20	1.50/1.20	25
A10	70	30			1.51/1.20	25
A11	70		30		1.62/1.43	14
A12	70			30	1.78/1.57	14
A13	70	15	15		1.52/1.22	14
F2	70	15		15	n/a	n/a

*Table 23: Soil amendments used in Trial 3. Bulk & dry density and water content values are for individual cores at production. F1 and F2 were omitted from the trial, as the composition of F1 would not yield a stable core before testing began and F2 fell apart considerably during wetting, invalidating the data. A8 and A9 are noted with an asterisk as these samples, across all testing measures, were substantially lower than trends in the remainder of tests would suggest.*

Table 23 provides a summary of the amendment proportions; however, these are also provided on each of the subsequent Figures (37-54) for better interpretation of results. As noted previously, the number and letter annotation at the end of each line represents the relative proportion and amendment respectively; the shorthand used is S (Soil2), C (compost2), WTR2<sub>d</sub>, and Si (silica). Error bars on each graph represent the minimum and maximum values obtained for each amendment. At this stage, the data were not searched for anomalies as soil is exceptionally heterogeneous by nature and therefore for a small sample size (maximum 8) the assumption that a data point is anomalous, is not reasonable. For this reason, the data were not statistically tested for significance of differences. The following sections will review the effect of amendment on the GWC as a result

of different amendments, and the effect of amendment on the density and volume change trends as a result of different amendments. One must note that some samples start the wetting cycle at a GWC of 0.14 and some start at 0.25. As stated in section 4.4.4 of Chapter 4, this was due to the need for more water in amendments containing compost so as to create robust cores (with the exception of A5 = 60S 40C).

### 5.1.2.1 Effect of single amendment at different proportions on GWC: 5050, 6040, and 7030

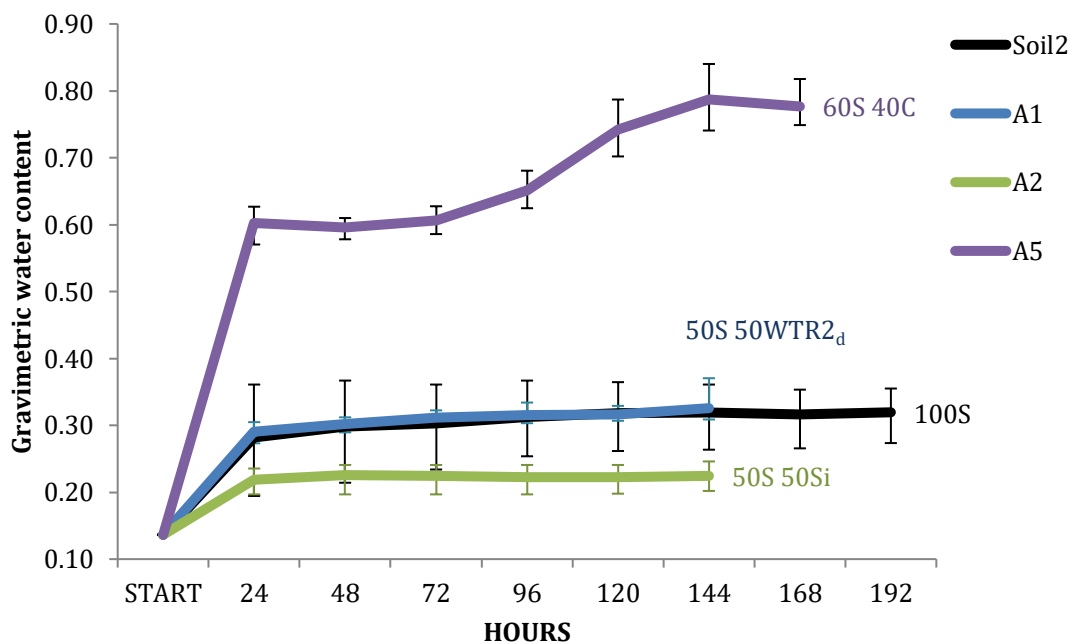


Figure 37: Average effect of single amendment at a 50% rate on gravimetric water content. A1 is 50S 50WTR2<sub>d</sub> and A2 is 50S 50Si. A5 is also included as a single compost amendment as F1 (50S 50C) was not included in the data set. Soil2 is the control. Soil2 n = 5, A1 n = 5, A2 n = 7, A5 n = 7

Figure 37 presents the GWC values for unamended soils and two 50% single treatment amendments (A1 and A2). For comparative purposes, A5 is also included as a single amendment of compost at 60%. As discussed in Chapter 4, the production of cores at 50% compost was not possible (F1). The GWC of A1 (50% soil, 50% WTR2<sub>d</sub>) is almost identical over time to the control sample (unamended soil, Soil2), suggesting that the addition of dried WTR does not improve or decrease the GWC, where the maximum GWC for A1 was 0.326 and the maximum for unamended soil was 0.319. The addition of silica at a 50% rate (A2) however,

appreciably reduces the amount of water the soil can hold, where a maximum GWC of 0.224 was reached. The rate of mass increase was similar for both unamended soils and AM1, nearing maximum GWC after 24 hours of wetting. A single amendment of compost, even at a lower amendment ratio of 40%, performs better than any single amendment at 50%, reaching a maximum of 0.777. There are no overlaps in the maximum/minimum error bars, suggesting this result is significant (although no statistical testing has been performed).

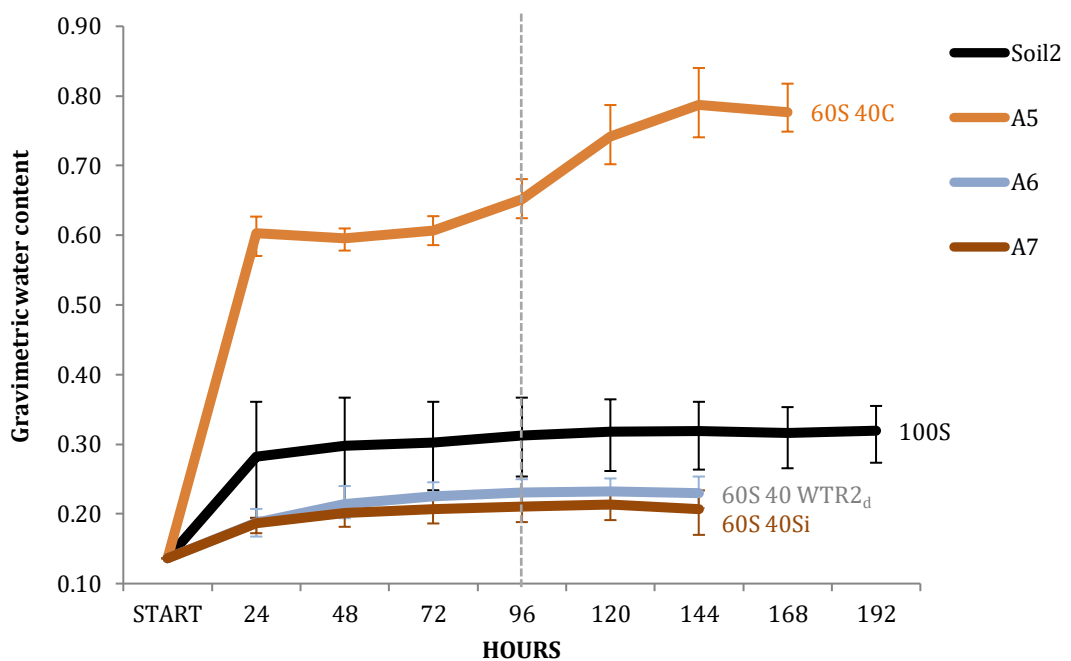


Figure 38: Average effect of single amendment at a 40% rate on gravimetric water content. A5 is 60S 40C, A6 is 60S 40WTR2<sub>d</sub> and A7 is 60S 40Si. Soil2 is the control. Soil2 n = 5, AM5 n = 7, AM6 n = 7, AM7 n = 7. The dashed grey line indicates the point of flooding.

Figure 38 compares the GWC of unamended soil and three amended samples at a 40% amendment rate (A5, A6 and A7). A 40% amendment of WTR2<sub>d</sub> reduces the maximum GWC of the soil compared to the control (0.319) with a maximum of 0.229. The 40% amendment of silica has a similar effect, reaching a maximum GWC of 0.207. Within 24 hours of wetting the majority of GWC change has occurred for unamended soil, A6 and A7, and the GWC plateaus. In contrast, a 40% amendment of compost (A5) gives a maximum GWC value of 0.787; the increase in GWC is rapid in the first 24 hours and continues to increase at 72 hours after a small lull, presumably due to the compost slowly expanding once wet. It is difficult to tell is

this is a measurement error or a real effect. Although no statistical testing has been conducted, there is no overlap of error bars between soil and AM5 in Figure 38, which may suggest that the difference is significant.

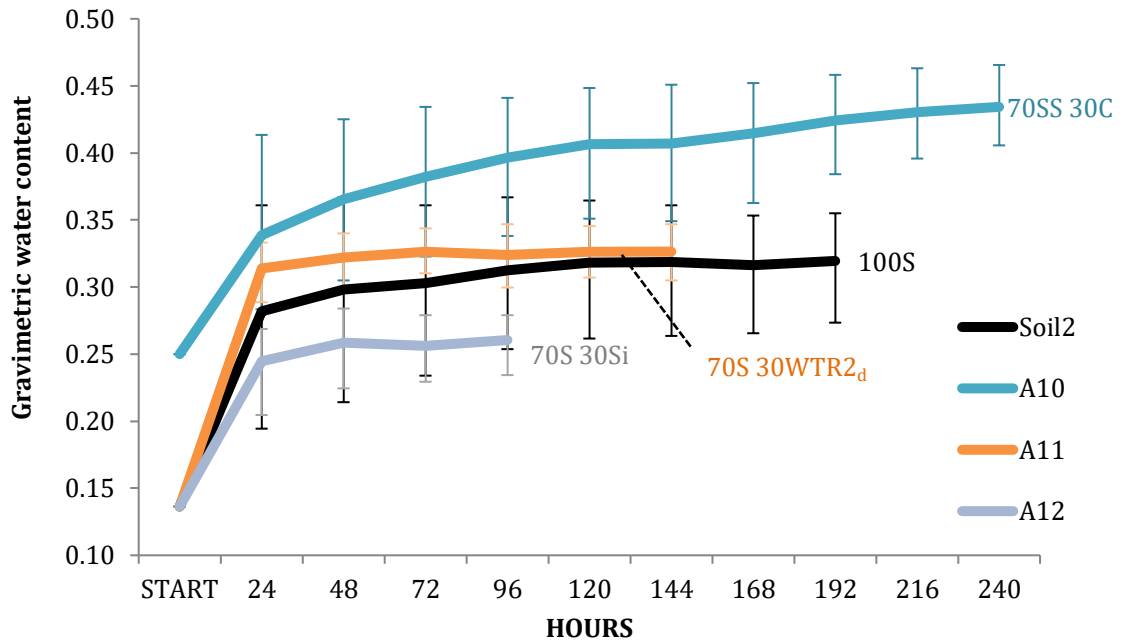


Figure 39: Average effect of single amendment at a 30% rate on gravimetric water content. A10 is 70S 30C, A11 is 70S 30WTR2<sub>d</sub> and A12 is 70S 30Si. Soil2 is the control. Soil2 n = 5, A10 n = 8, A11 n = 8, A12 n = 5.

Figure 39 compares the GWC over time of unamended soil and three other amendments (A10, A11 and A12). The amendment at a 30% rate of silica (A12) reduces the GWC in comparison with unamended soil and reaches the maximum of 0.261 within 48 hours. The addition of WTR2<sub>d</sub> at a 30% rate (A11) marginally increases the maximum GWC compared with unamended soil (0.326 and 0.319 respectively) and increases the rate of GWC change in comparison with unamended soil in the first 24 hours. The amendment of compost at a 30% rate (A10) results in a higher GWC than any other 30% single amendment, reaching a 0.434 maximum. This however may be due to a higher initial GWC, effectively giving this amendment a head start. As discussed in Chapter 2 (section 2.4.4), the hydraulic conductivity of a more saturated sample is higher than that of an unsaturated sample, therefore benefitting the sample with a higher initial water content.

### 5.1.2.2 Effect of single compost amendment on GWC

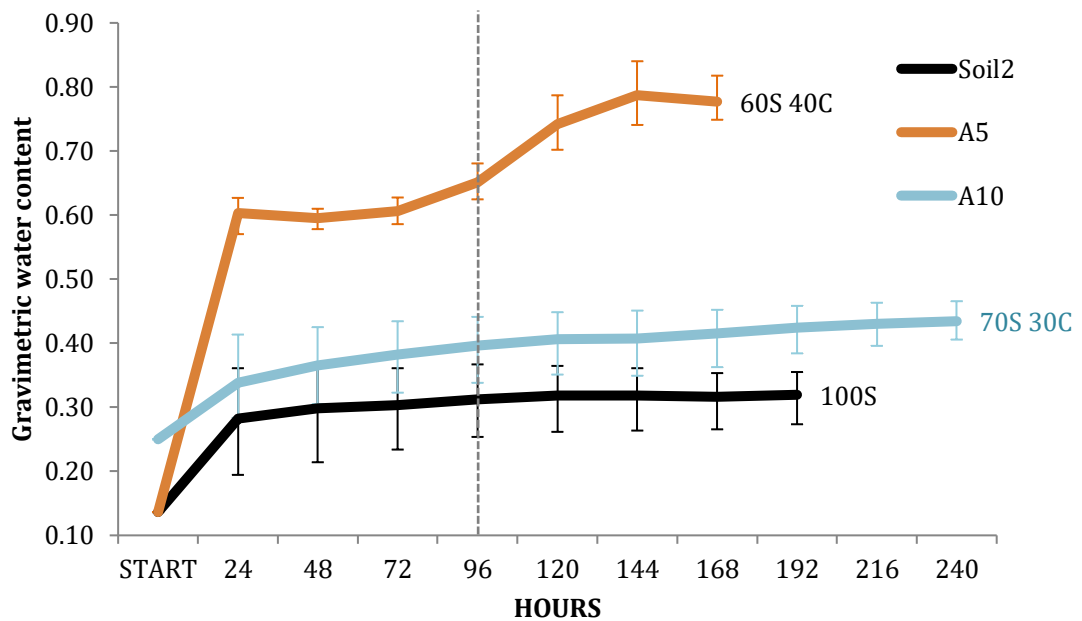


Figure 40: Average effect on GWC of single amendment with compost at 30% and 40% rate against the control, Soil2. A5 is 60S 40C and A10 is 70S 30C. Soil2  $n = 5$ , A5  $n = 7$ , A10  $n = 8$ . A10 started at 0.25 GWC due to the presence of compost. A5 was the only compost amendment to remain at 0.14 GWC. The dashed line shows where the samples were flooded during the wetting process

The effect of a single amendment with compost appears to be related to the proportion of amendment, although not necessarily linearly, where an amendment at a 40% rate (A5) has a much greater maximum GWC than an amendment at 30% (A10) at 0.787 to 0.434 respectively. The trends in the two lines are also different, where A10 appears to reach near maximum GWC within 24 hours but A5 has a sharp increase during the first 24 hours, followed by a lull and then a slow increase until the maximum point. This is likely due to the wetting process and subsequent response of compost; samples were initially only placed onto saturated sand and then flooded after 96 hours. Although unamended soil and 30% compost did not respond to the extra input of water, 40% compost continued to increase in GWC. Considering that 30% compost did not respond in the same way to wetting, suggests that this may be a function of measurement error in some part.

### 5.1.2.3 Effect of single WTR/silica amendment on GWC

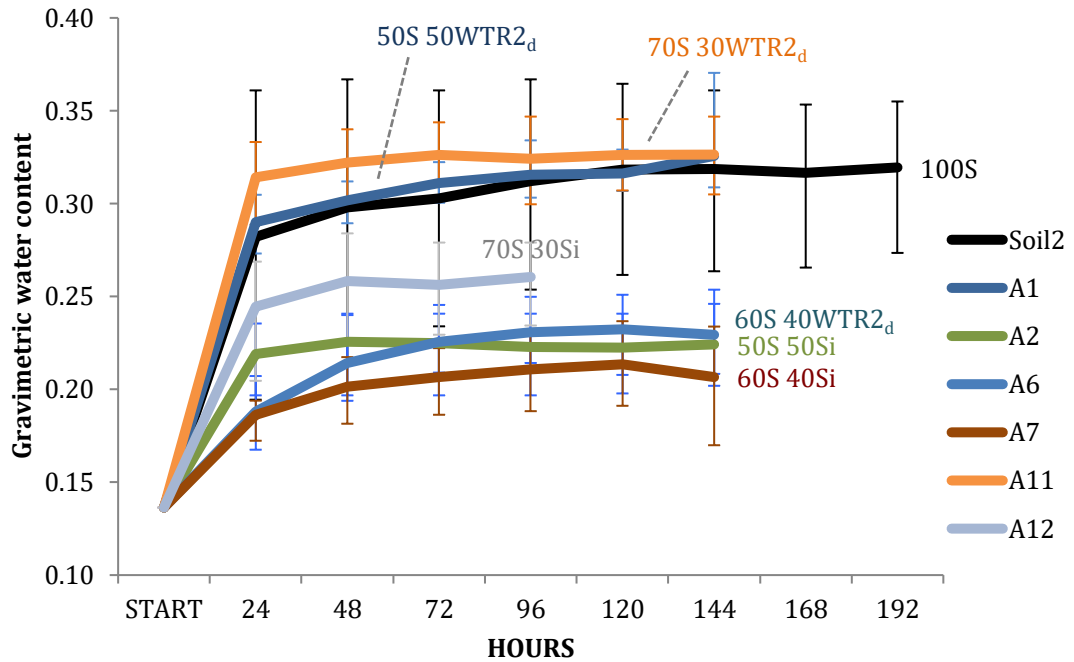


Figure 41: Average effect on gravimetric water content of single amendment with WTR<sub>2d</sub> and silica (Si) at various amendment rates against the control, Soil<sub>2</sub>. A1 is 50S 50WTR<sub>2d</sub>, A2 is 50S 50Si, A6 is 60S 40WTR<sub>2d</sub>, A7 is 60S 40Si, A11 is 70S 30WTR<sub>2d</sub> and A12 is 70S 30Si. Soil<sub>2</sub> n = 5, A1 n = 5, A2 n = 7, A6 n = 7, A7 = 7, A11 n = 8, A12 n = 5.

Figure 41 shows the effect of single amendments of WTR<sub>2d</sub> and single amendments of silica at various proportions. Silica was added to replicate the structural effect of WTR, albeit to a limited extent due to the particle size distribution differences. As shown by Figure 19 the particle size range of silica was quite narrow, where the majority of particles were between 425  $\mu\text{m}$  and 600  $\mu\text{m}$ . In comparison, the WTR<sub>2d</sub> had a large range, where 40% of the WTR<sub>2d</sub> ‘particles’ are smaller than 425  $\mu\text{m}$  and 20% are of a clay size fraction (<63  $\mu\text{m}$ ). The presence of the finer fraction is critical in the water holding capacity of the soil (Rawls *et al.*, 1982; Saxton & Rawls, 2006) and the addition of coarse particles is likely to have caused the reduction in water holding capacity of soils amended with silica. The rate of hydraulic conductivity, based on the same theory, is predicted to increase with the addition of coarser materials; however, this is not evident in Figure 41 as the rate of water uptake is lower for all single silica amended samples than unamended soil.

With the exception of A11 (50S 50WTR2<sub>d</sub>) and A1 (70S 30WTR2<sub>d</sub>), which appear to be within the margin of error shown by the maximum/minimum error bars, the addition of WTR or silica as single amendments have a negative influence on the GWC of a soil compared with unamended soil. The large overlap in error bars for all samples show that there is a significant amount of heterogeneity of GWC within the set of 12 cores in a sample.

#### 5.1.2.4 Effect of co-amendment vs single amendment: GWC

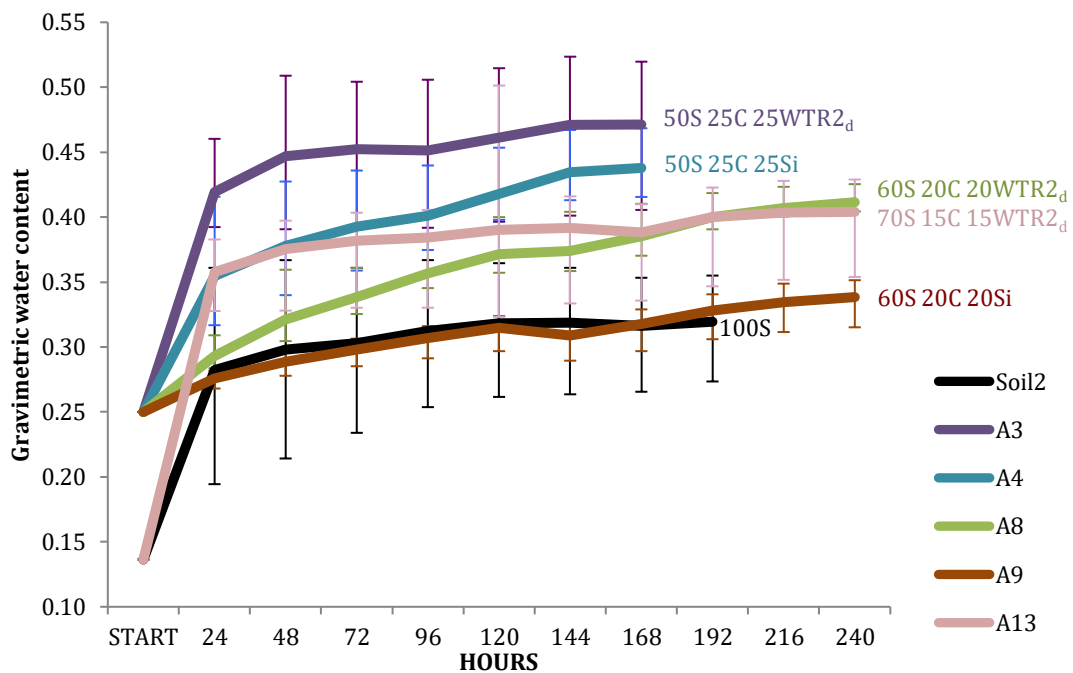


Figure 42: Average effect on gravimetric water content of co-amendment with WTR2<sub>d</sub> and compost at various amendment rates against the control, Soil2. A3 is 50S 25C 25WTR2<sub>d</sub>, A4 is 50S 25C 25Si, A8 is 60S 20C 20WTR2<sub>d</sub>, A9 is 60S 20C 20Si and A13 is 70S 15C 15WTR2<sub>d</sub>. F2 is 70S 15C 15Si but this data is not included. Soil2 n = 5, A3 n = 7, A4 n = 8, A8 n = 8, A9 n = 8, A13 n = 8.

Figure 42 presents a general view of the effect of co-amendment on the GWC of 5 co-amendments against the unamended soil. In general, all co-amendments using WTR2<sub>d</sub> perform better than their silica co-amended equivalent (as discussed previously). With the exception of 40% silica co-amendment, all co-amendments with WTR2<sub>d</sub> increase the GWC in comparison with unamended soil, where greater proportions of amendment increase yield higher GWC despite different initial GWC. Figures 43-45 show the effect of co-amendment against a single amendment at various amendment ratios, where the general trend shows that compost has the

greatest effect on the GWC of a sample, regardless of what other material is added, i.e. co-amendments have higher GWCs than single amendments of WTR or silica, due to the presence of compost.

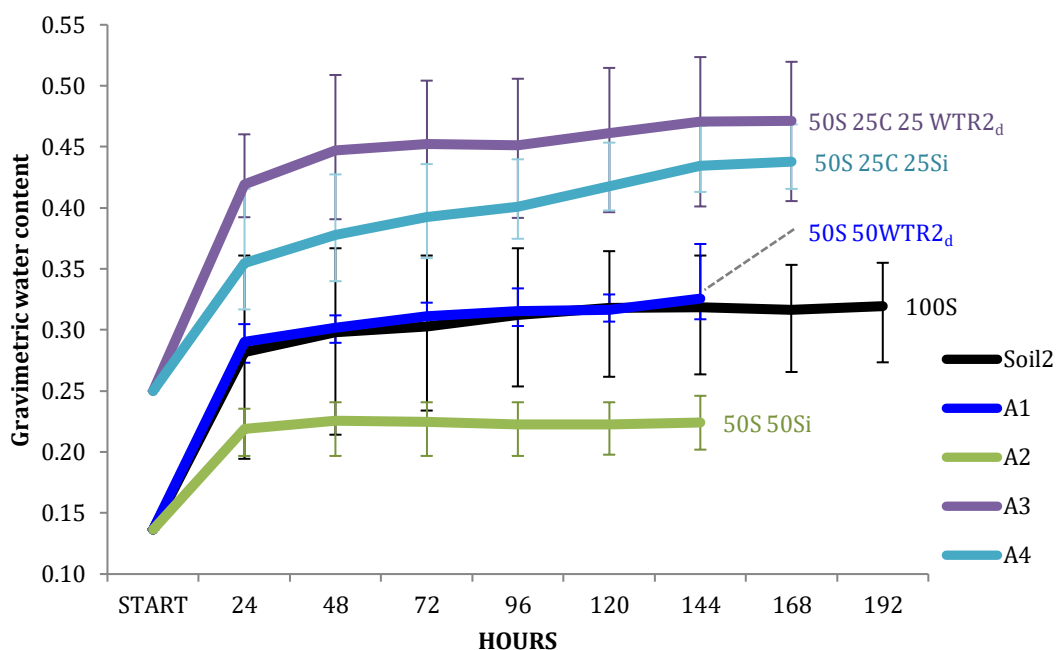


Figure 43: Average effect on gravimetric water content of co-amendment and single amendment at a 50% application rate against the control, Soil2. A1 is 50S 50WTR2<sub>d</sub>, A2 is 50S 50Si, A3 is 50S 25C 25WTR2<sub>d</sub> and A4 is 50S 25C 25Si. Soil2 n = 5, A1 n = 5, A2 n = 7, A3 n = 8, A4 n = 8.

At a 50% amendment rate, as shown in Figure 43, in comparison with single amendment, the co-amendment with WTR2<sub>d</sub> or silica improves the GWC by 31% for WTR2<sub>d</sub> and 49% for silica, although this may be a function of the co-amendments starting at a higher GWC. The co-amendment improves the GWC by 30% (WTR) and 28% (Si). As discussed the single amendment of WTR2<sub>d</sub> (A1) or silica (A2) at a 50% shows either negligible difference or a reduction in the GWC.

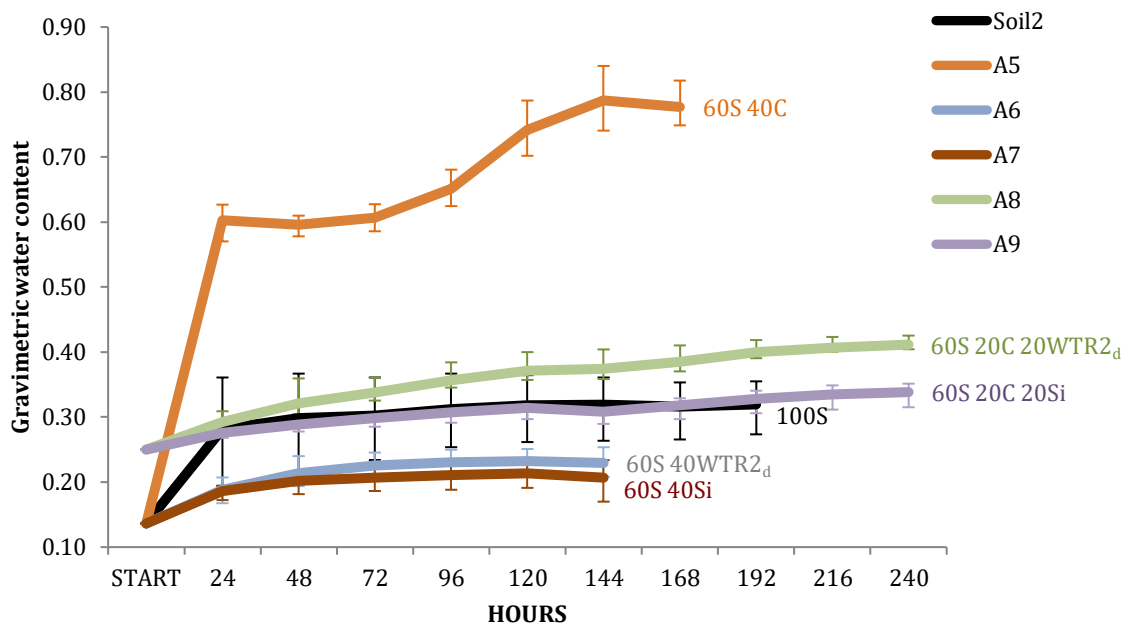


Figure 44: Average effect on gravimetric water content of co-amendment and single amendment at a 40% application rate against the control, Soil2. A5 is 60S 40C, A6 is 60S 40WTR2d, A7 is 60S 40Si, A8 is 60S 20C 20WTR2d and A9 is 60S 20C 20Si. Soil 2  $n = 5$ , A5  $n = 7$ , A6  $n = 7$ , A7  $n = 7$ , A8  $n = 8$ , A9  $n = 8$

At a 40% amendment rate shown in Figure 44 the single amendment of compost (A5) has the highest GWC (0.787) in comparison with any co-amendment at the same proportion. Figure 45 similarly shows that a single amendment of compost at 30% proportion (A10) has a greater GWC than any co-amendment at 0.434 and unamended soil. Importantly however, at a 30% amendment rate, the WTR co-amendment performs almost as well as the single amendment of compost (0.403 vs 0.434). It is also interesting to note that although A13 (30% WTR amendment) had an initially lower GWC than A10 (30% compost), after 24 hours the GWC of the co-amendment was higher than the single amendment. This suggests that although amendments with compost alone hold more total water than amendment with any other material, the co-amendment with WTR2<sub>d</sub> increases the rate of water movement into the material. This suggestion is supported by hydraulic conductivity data later in the chapter (section 5.3.1).

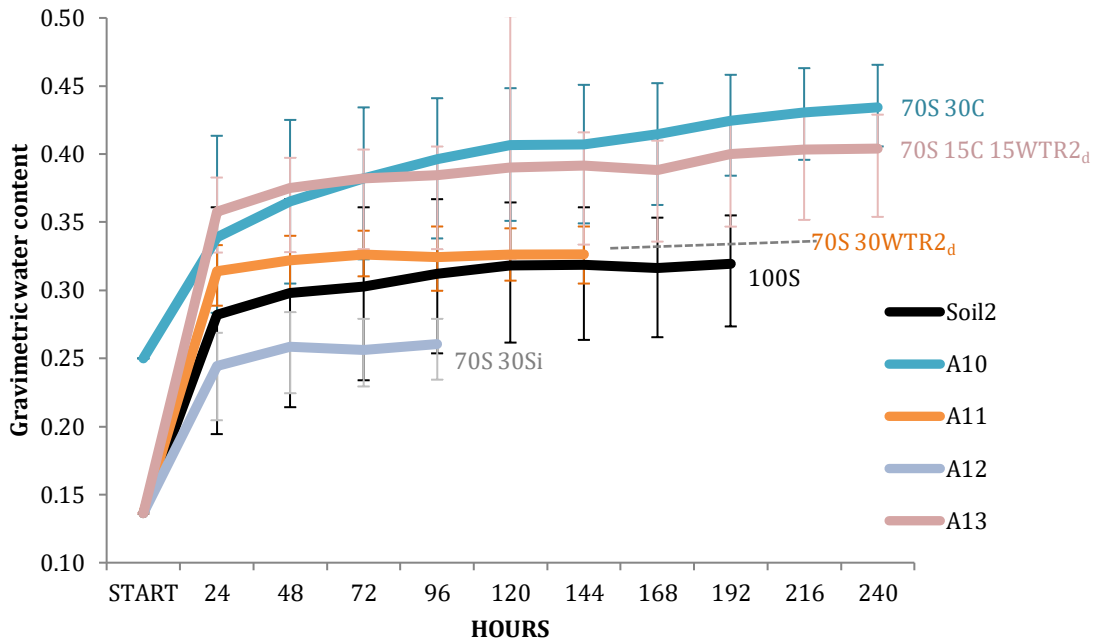


Figure 45: Average effect on gravimetric water content of co-amendment and single amendment at a 30% application rate against the control, Soil2. A10 is 70S 30C, A11 is 70S 30WTR2<sub>d</sub>, A12 is 70S 30Si, and A13 is 70S 15C 15WTR2<sub>d</sub>. Soil2 and A12 n = 5, A10, A11 and A13 n = 8

#### 5.1.2.5 Effect of density and initial water content on GWC

As discussed previously in Chapter 2, the relationship between water content and density is co-dependent (section 2.3) where the water content affects the resultant density of a soil under compressive force, and in turn the density of that soil affects the maximum water content, as a result of porosity differences (where in general lower density soils have higher porosity). In Figure 46, we are able to compare the dry density of amendments based on their initial water contents, where A1, A2, A5, A6, A7, A11, A12, and A13 started at 0.14 GWC and A3, A4, A8, A9 and A10 started at 0.25 GWC (where a reminder of compositions is presented in Table 24). Figure 46 shows that the initial water content at compaction affects the degree to which the sample is compacted, as those samples compacted at 0.25 (orange bars) are less dense than samples compacted at 0.14 (with the exception of A5). However, this difference may be wholly accountable to the variance in materials within each amendment rather than the water content; samples compacted at 0.25 all contained compost, a material of low density with high elasticity parameters, both of which reduce the compactibility of the material. In contrast samples compacted at 0.14 contain denser materials (silica, soil and WTR).

	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10	A11	A12	A13
Soil	50	50	50	50	60	60	60	60	60	70	70	70	70
Compost			25	25	40			20	20	30			15
WTR	50		25			40		20			30		15
Silica		50		25			40		20			30	

Table 24: Summary of amendment proportions for Trial 3

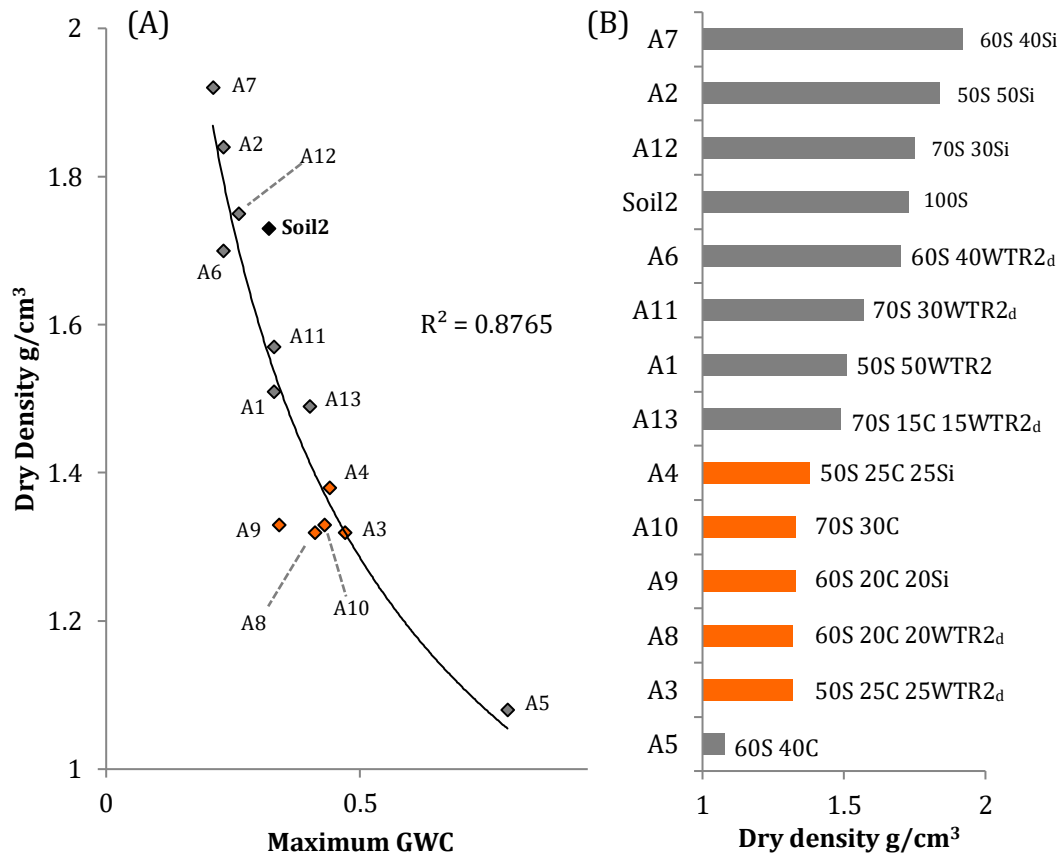


Figure 46: (A) Maximum GWC as a function of the dry density of samples against the control, Soil2. (B) Dry density of samples compared to their initial water content, where samples identified above in grey had an initial GWC of 0.14 and samples identified above in orange had an initial GWC of 0.25.

Samples that have the highest dry densities (A2, A7 and A12) and higher dry densities than unamended soil, all contain silica. Samples that are co-amended or are singly amended with WTR2<sub>d</sub> have an intermediate density and have lower dry densities than unamended soil, which is due to lower bulk density and particle density of the dried WTR2<sub>d</sub> in comparison with soil (2.11 and 2.65 g/cm<sup>3</sup>, respectively) and may be influenced by the packing characteristics of WTR2<sub>d</sub>. The samples that have the lowest dry density (A3, A5, A8, A9 and A10) contain the

highest proportions of compost, which as discussed in Chapter 2 is a function of the low particle density of compost and high resistance to compaction.

The dry density of the sample therefore directly affects the maximum GWC as a result of two factors. Firstly, regardless of the materials within the sample, it is well known that soil with lower dry densities have higher void ratios which allow more water to be contained within the sample. Secondly, the materials that cause a soil sample to have a lower dry density due to either low particle density or due to low bulk density of the material, i.e. compost or WTR2<sub>d</sub>, themselves impart better water holding capacity, giving the result that lower density soils have a higher GWC. In addition, high-density materials such as silica, are unable to incorporate water with their matrix, whereas low density compost is very porous. Therefore, for materials that occupy the same volume, their individual capacity to hold water is very different. Figure 46 shows this, where amendments that have a lower initial dry density (as a factor of initial water content and material make up) have higher maximum GWC than samples with higher dry density.

This effect is supported by the data produced by Laurie (2017, Figure 47 unpublished), which shows that density affects the GWC for unamended soil that have been compacted to different densities at the same water content. Laurie also compared the effect of co-amendment at different densities against unamended soil. Cores were prepared in the same way as those for Trial 4 using a static compaction press (Figure 24), where  $n = 12$  and initial water content was 0.16 (16%) at an amendment rate of 70% soil, 15% WTR2<sub>d</sub> and 15% compost. Cores were compacted to three different bulk densities, 1.4, 1.6 and 1.8 g/cm<sup>3</sup> to test the effect of density on the GWC. For comparison in Trial 3 A13 (also 70S 15C 15WTR2<sub>d</sub>) had an initial bulk density of 1.69 g/cm<sup>3</sup> and unamended soil had an initial bulk density of 1.97 g/cm<sup>3</sup>. Discussed subsequently, this is also comparable to AM14 in Trial 4, which had an initial bulk density of 2 g/cm<sup>3</sup>.

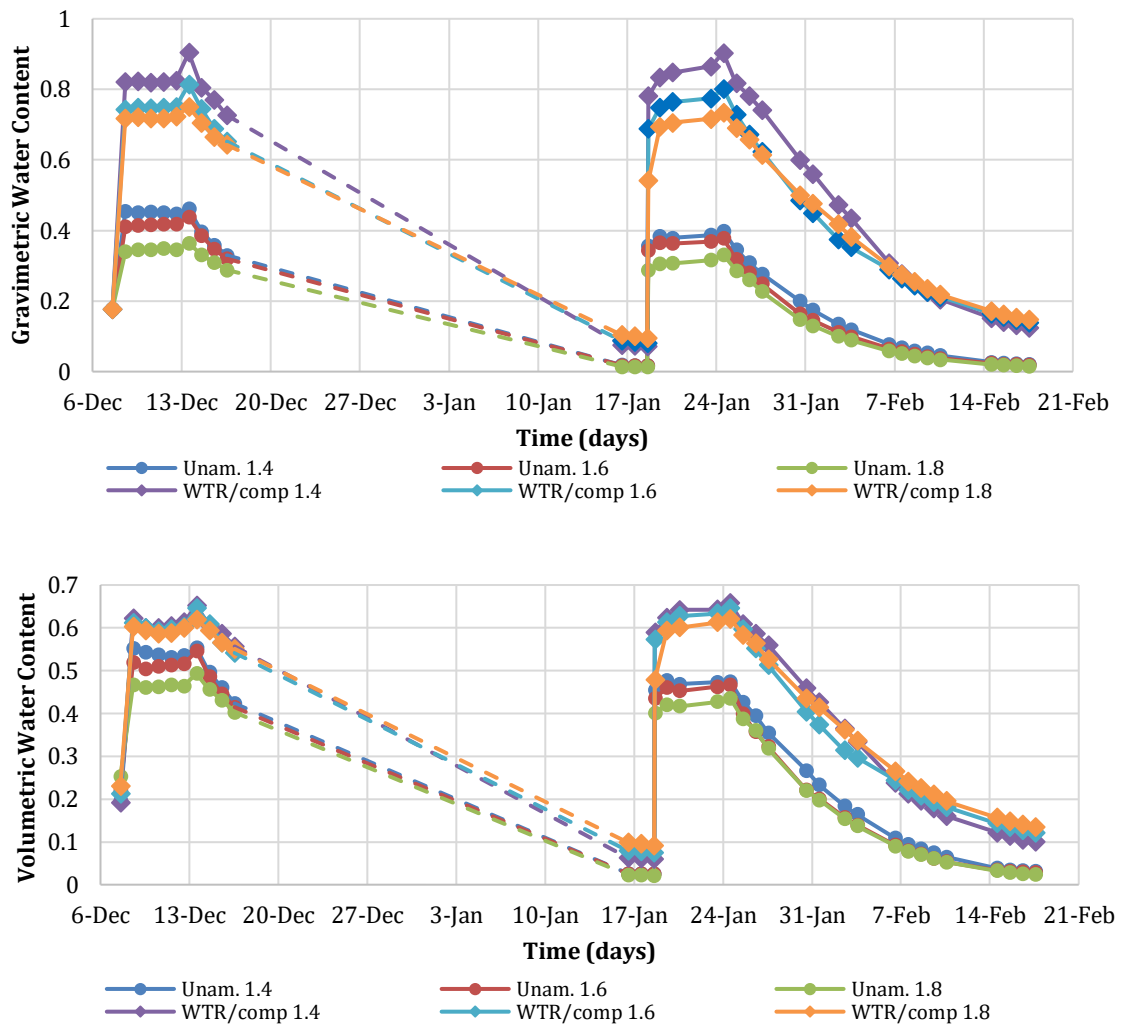


Figure 47: Average gravimetric water content (top) and average volumetric water content (bottom) for unamended soil at three bulk densities (Unam. 1.4, Unam 1.6 and Unam 1.8 g/cm<sup>3</sup>) and (bottom) for a 30% WTR co-amended soil (WTR/comp 1.4, WTR/comp 1.6, and WTR/comp 1.8 g/cm<sup>3</sup>). n = 12. Dashed lines connecting lines between phases indicates an extended drying period during which samples were not measured. Data collected by Laurie (2017).

Figure 47 shows there is a direct relationship between bulk density and GWC across two wetting and drying cycles where the lower the density, the higher the GWC reached. In addition, the maximum GWC of amended soils was higher at all densities than unamended soil e.g. at 1.4 g/cm<sup>3</sup> unamended soil reached a GWC of 0.49 and amended soil reached a GWC of 0.90. The difference between unamended and amended soil was statistically significant (p<0.1). The trends shown in Figure 47 show that during the second wetting cycle, the GWC of co-amended samples was similar to the values obtained in the primary wetting, whereas unamended

samples held less water during the second wetting cycle. This suggests that co-amendment reduced shrinkage (which is unrecoverable in soil) as a result of drying and therefore the soil structure (voids) were maintained, allowing higher GWC (Laurie, 2017).

### 5.1.2.6 Relationships between compost and GWC

The previous figures have gone into detail about the differences in GWC identified between various amendments. The following section attempts to obtain between trends in GWC and the presence of compost with the GWC of samples.

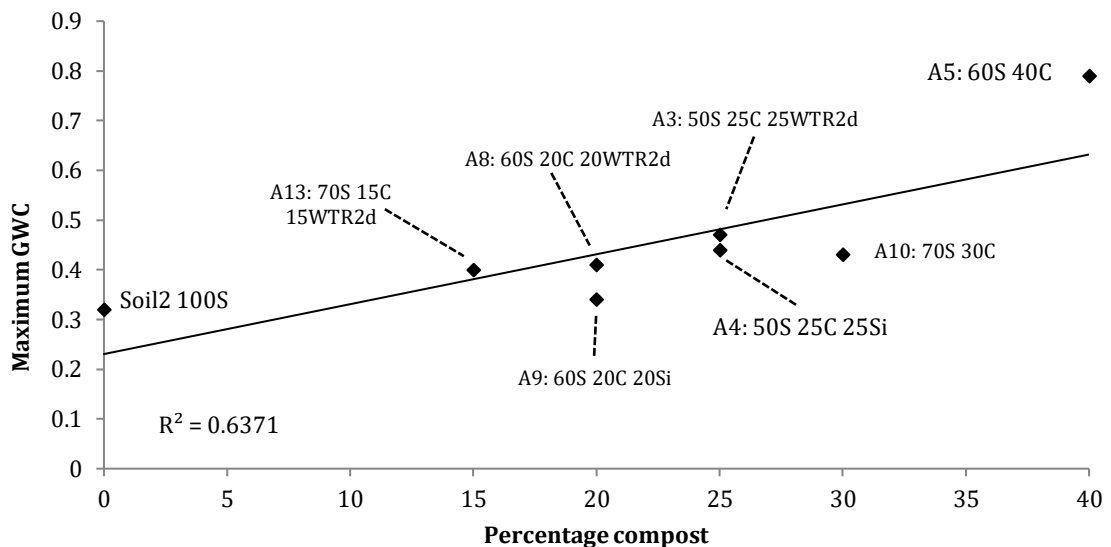


Figure 48: Maximum GWC of samples against the proportion of compost in the amendment as either a single amendment (40 or 30%) or as part of a co-amendment (25, 20 or 15%).

Figure 48 shows that compost amendment has a positive effect on the maximum GWC achieved, which concurs with literature discussed in Chapter 2. There is a significant positive trend denoted by the trend line ( $R^2 = 0.63$ ), indicating that the greater the proportion of compost, the higher the GWC maximum. Only one sample (40% compost) had a higher maximum GWC (A5 at 0.79) than co-amendments. Importantly, at 30% amendment rate, the co-amendment with WTR2<sub>d</sub> (A13, 0.4) reaches a similar maximum to the single amendment of 30% compost (A10, 0.43). This shows that at lower amendment proportions, which are the least unrealistic application rates for land spreading, there is no disadvantage to using a co-amendment with WTR2<sub>d</sub> over compost. The addition of co-amendment improves

the maximum GWC of a soil by between 20 and 32% depending on application rate.

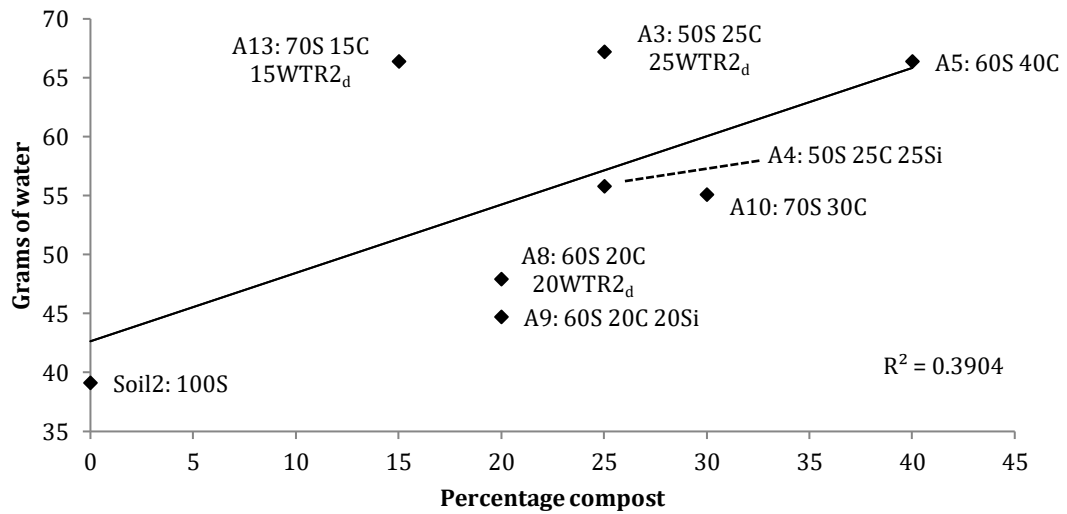


Figure 49: Average increase in grams of water after 24 hours for samples amended with compost as either a single amendment (40 or 30%) or as part of a co-amendment (25, 20 or 15%), suggesting a rate of water uptake.

Figure 49, shows that there is only a very weak relationship between compost proportion and the rate of uptake in the primary 24 hours of wetting, denoted as grams of water taken up in 24 hours. Unamended soil has the lowest uptake of all samples (39.13 g). It is clear that the co-amendment of soil with WTR2<sub>d</sub> yields a better water uptake compared to the single amendment of compost over the first 24 hours of wetting, where 30% co-amendment (A13, 66.38 g) and 50% co-amendment (A3, 67.2 g) have a greater uptake in 24 hours than 40% single amendment of compost (A5, 66.37 g).

Importantly, the co-amendment at 30% (A13) takes up ~17% more water than the 30% single amendment of compost (A10, 55.08 g) in the first 24 hours. There is negligible difference between 50% and 30% co-amendment ratios, despite a greater proportional amendment for the former, however A8 and A9 (20% co-amendment) appear to perform sub-par, where one would expect a co-amendment of 40% (A8) to reach a value between those for 30% (A13) and 50%. The values of A8 and A9 throughout the analysis of Trial 3's data performed poorly, countering the general trend whereby greater proportions of amendment yield greater change against unamended soil. The information in Figure 49 is important as the initial

period of wetting is critical in extreme weather scenarios (where rainfall is heaviest) and the infiltration rate is key to reduce soil erosion through overland flow, as with a good infiltration and percolation rate, water will enter the soil profile rather than immediately forming runoff as with saturated soils. Analysis of data suggests that WTR<sub>2d</sub> increases the rate of uptake whereas compost, although providing a higher maximum GWC, may not have a higher uptake in the first 24 hours due to hydrophobicity initially preventing water from entering the soil body.

### 5.1.2.7 Relationships between WTR/silica and GWC

The important piece of information to take from these figures is that WTR<sub>2d</sub> elicits a higher maximum GWC and greater increase in GWC over 24 hours than the amendments of silica for both co-amendment and single amendment. The addition of silica results in a 3-30% lower maximum GWC than the WTR<sub>2d</sub> proportional equivalent which is likely to be a result of differences particle size distribution discussed in section 4.3.1. Information on the detailed surface and shape characteristics of WTR<sub>2d</sub> are provided in Chapter 6, and this provides evidence as to why the effect of WTR<sub>2d</sub> addition is so effective in improving the WHC in comparison to the amendment of other coarse grain materials.

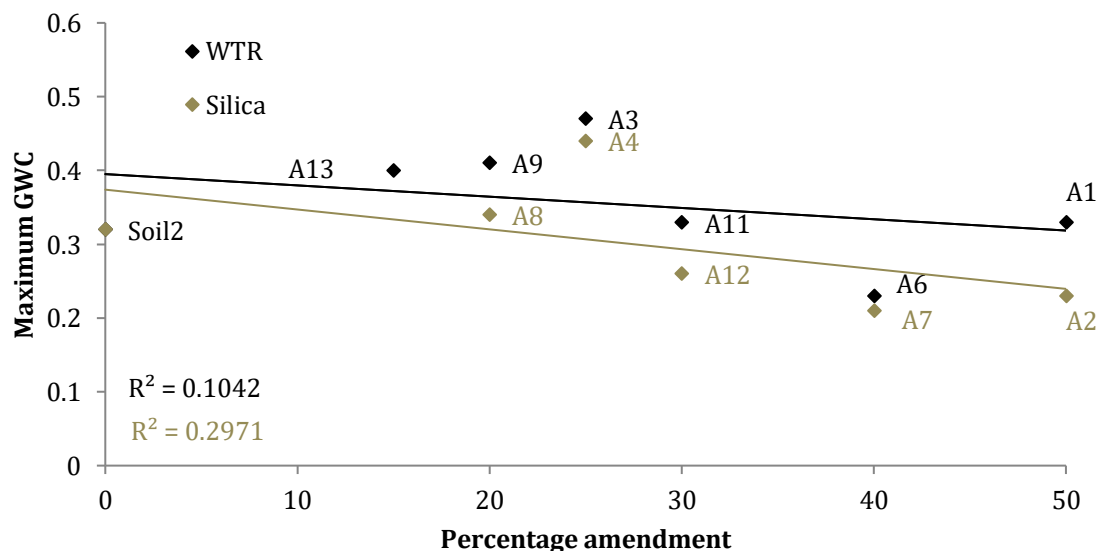


Figure 50: Average maximum GWC of samples amended with WTR or silica either as a single amendment (50, 40 or 30%) or as part of a co-amendment (25, 20 or 15%)

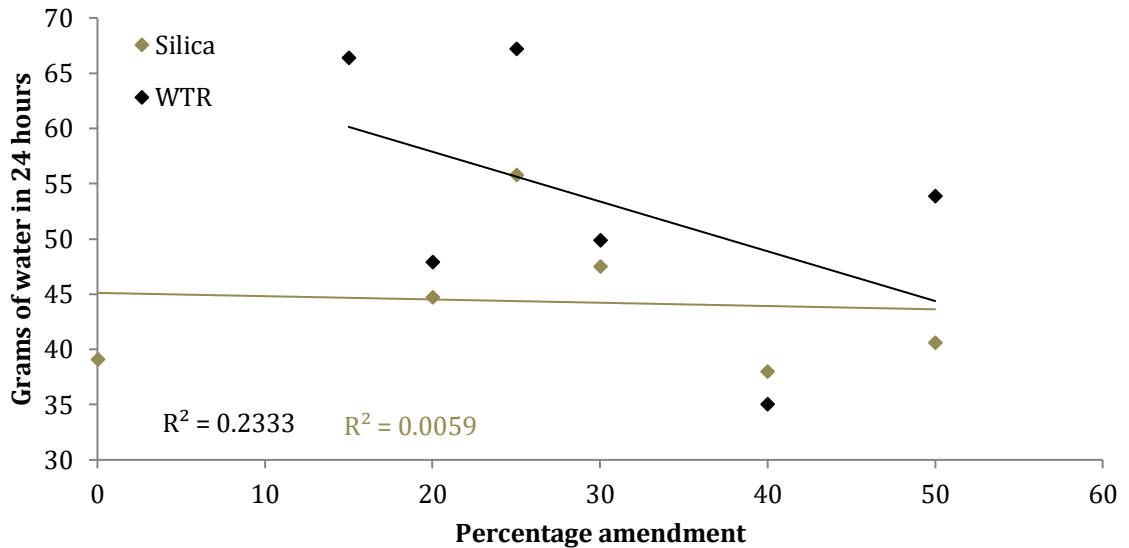


Figure 51: Average increase in grams of water after 24 hours for samples amended with WTR<sub>2a</sub> or silica as either a single amendment (50, 40 or 30%) or as part of a co-amendment (25, 20 or 15%), suggesting a rate of water uptake.

#### 5.1.2.8 Volumetric and density changes

Figures 52 and 53 show the volumetric water content and volumetric water content *i* change over time for 14 samples against the control, Soil2 (unamended). As discussed in section 2.4.2, we must view the volumetric water content with knowledge of either the volume change of samples and/or the gravimetric water content of samples to supplement this measurement so that we may determine relative changes between samples. The volumetric water content change in Figure 52 follows a similar pattern to the trends seen between different amendments for the gravimetric water content where A5 (60S 40C) has the greatest maximum VWC, showing that it had both the greatest change in GWC and volume. In general co-amendments have a greater VWC than single amendments at the same proportion, where silica single amendments had a negligible or detrimental influence, shown by a reduction in VWC.

The VWC<sub>i</sub> in Figure 53 shows the instantaneous volumetric water content, which relates the volume of water in the sample to the volume of the sample at the time of measurement, rather than VWC that relates the volume of water in a sample to the original volume of the sample.

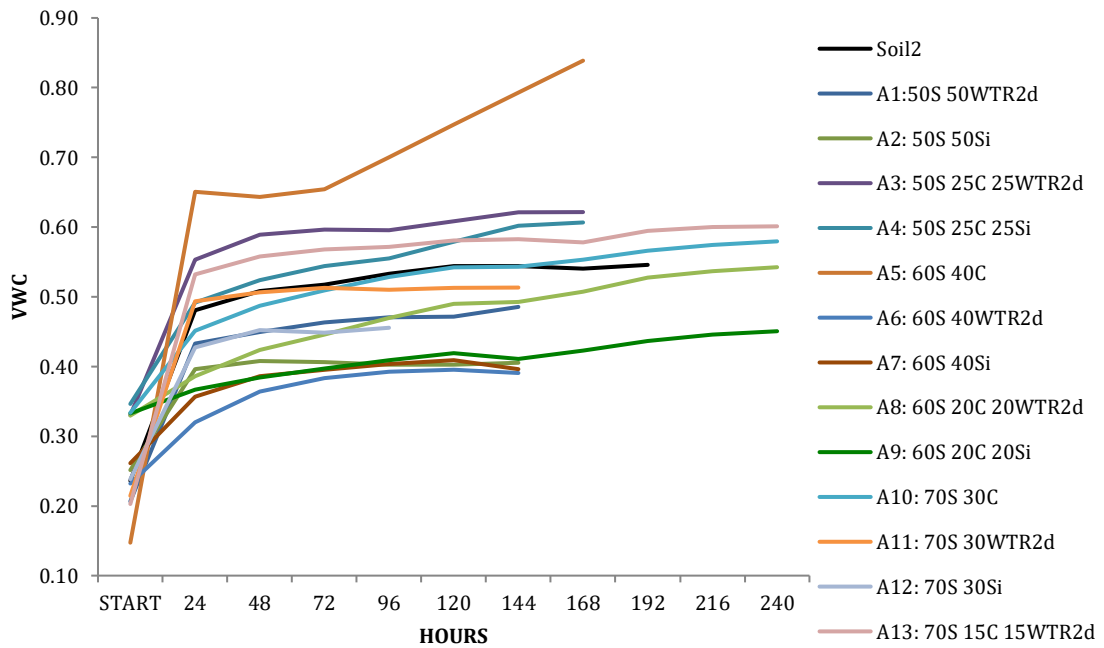


Figure 52: Average volumetric water content change of samples against the control, Soil2, over a 240-hour wetting period. VWC start values range between 0.21 and 0.26 for samples at 0.14 GWC, and between 0.33 and 0.35 for samples at 0.25 GWC due to small differences in sample volume.

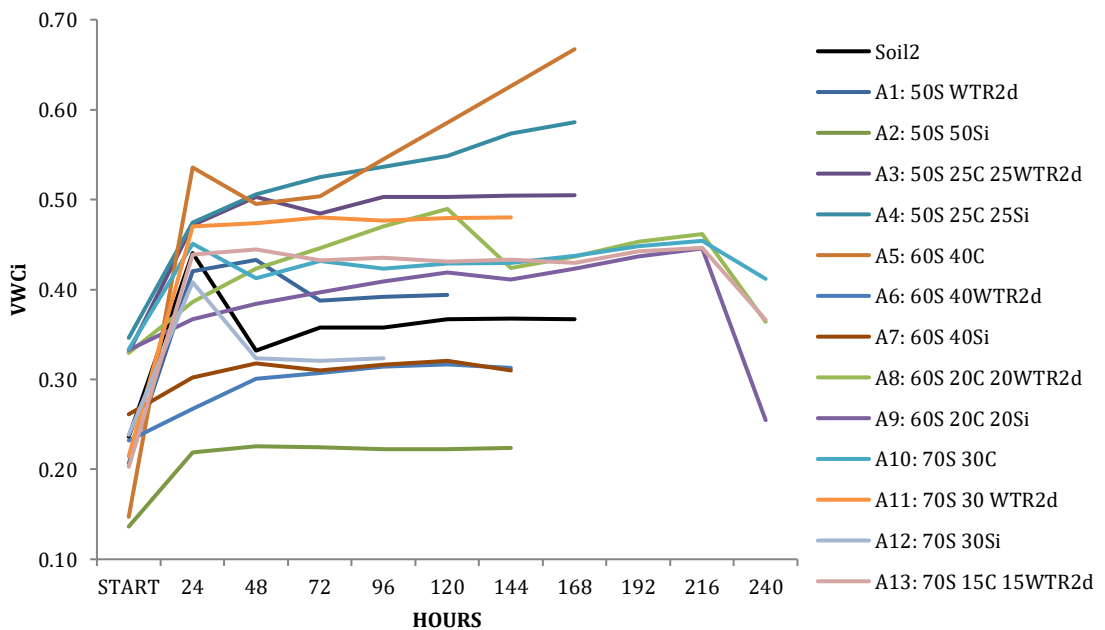


Figure 53: Average volumetric water content (i) change of samples against the control, Soil2, over a 240-hour wetting period. VWC start values range between 0.21 and 0.26 for samples at 0.14 GWC, and between 0.33 and 0.35 for samples at 0.25 GWC due to small differences in sample volume.

As discussed previously, VWC as a measure does not account for volumetric change of the sample. When we view the volumetric changes by VWCi, the decreases shown by unamended soil, A1, A10 and A12 indicate that the samples are increasing in volume at a greater rate than they are taking up water. Samples that have a fairly horizontal trend either experience no change after an initial wetting period or undergo similar increases in volume and gravimetric water content. Samples that have a positive trend increase in GWC faster than they swell. It is therefore vital that the VWC and VWCi are viewed in tandem with the volume and GWC measurements of samples.

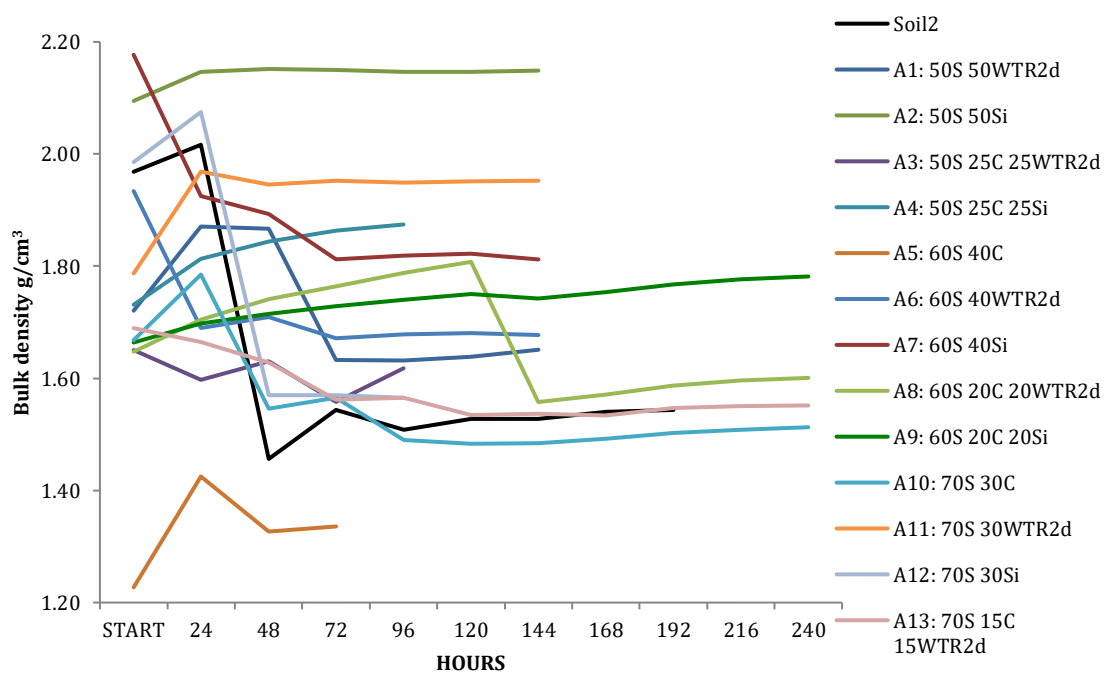


Figure 54: Average bulk density change of samples over 240 hours of wetting.

Figure 54 shows that some amendments are more prone to swelling than others, expanding to hold more water, however it is difficult to compare the relative density changes as the starting density of each amend is different. Amendments that included silica or WTR2<sub>d</sub> tended to have little response to wetting in terms of sample volume, which as Moodley & Hughes (2006) found was due to the binding effects of polymers in the WTR. As samples such as A5 (60S 40C) take up water, the bulk density increases as the water fills the voids present in the compost. Unamended soil experiences the largest drop in bulk density as a result of swelling.

### **5.1.2.9 Concluding remarks: Trial 3**

The results from Trial 3 have shown that the water holding capacity is greatly affected by proportion of compost in the samples, which also determined the initial bulk density of the sample after compaction. Under the same compactive effort, samples with compost required a higher water content to be able to produce samples for testing, and it is unknown to what extent this affected subsequent measurements. The addition of silica, designed to test the physical effect of WTR<sub>2d</sub> addition without a geochemical effect did not provide a large degree of insight, owing to the differences in particle size distribution. By adding silica, the water holding capacity decreased, while the theoretical beneficial effects of a larger grain size in increasing the hydraulic conductivity were not apparent. Should the experiment be repeated, a careful assessment of particle size distribution is needed for the WTR<sub>2d</sub> to ensure that the silica matches the high proportion of fine particles as these are likely the reason for WTR<sub>2d</sub> providing higher water holding capacity. The key summary points from Trial 3 are as follows:

1. Single amendment with WTR does not improve the maximum GWC of the soil but does improve hydraulic conductivity (rate of uptake in 24 hours). Single compost amendments have the highest maximum GWC at all amendment ratios
  - 40% and 50% single amendment of WTR or silica does not improve the GWC compared to unamended soil.
  - Single 40% compost increases the maximum GWC by 60% compared to unamended soil (0.319 to 0.787)
  - 30% single amendment of silica has an 18% lower maximum GWC than unamended soil.
  - 30% single amendment of WTR increases the rate of uptake by 11% after 24 hours, while achieving a similar maximum GWC to soil.
  - Silica doesn't effectively replace WTR due to the difference in size fractions.
  - All single amendments of WTR or silica (excluding both 40% single amendments and 50% silica) increase the rate of uptake in the first 24 hours by up to 42% compared to unamended soil.
2. In general the co-amendments have higher GWC than unamended soil regardless of the addition of WTR or silica, therefore the co-amendment benefit is dependent on the amount of compost you add, not the other half of the co-amendment.

- The addition of co-amendment improves the maximum GWC of a soil by between 20 and 32% depending on application rate.
  - At 50% co-amendment, the GWC is improved by 30% (WTR) and 28% (Si) compared to unamended soil
  - At 50% co-amendment improves GWC by 31% (WTR) and 49% (Si) compared to the single amendment of each material
  - At a 30% amendment rate, WTR co-amendment (0.4) performs equally well to single compost amendment (0.43) and both better than soil (0.32)
3. Co-amendment improves the rate of water uptake compared to single amendment of compost
- Co-amendment improves the rate of uptake by 17% for 30% amendment in the first 24 hours - important in flood scenarios
  - 30 and 50% co-amendment have the same rate of water uptake in the first 24 hours compared to 40% single compost
4. The bulk density of the samples is dependent on the materials used for amendment, not due to the initial water content. Co-amendment stabilises the samples on drying.
- Soil at lower density has a higher GWC as a result of different void ratios, compared to more densely packed soil (Laurie, 2017).
  - Co-amendment reduces the shrinkage during drying and therefore improves the GWC
  - Compost allows the lowest bulk density and greatest volume change (reduction in density during wetting) owing to the material's low density with high elasticity.

### 5.1.3 WHC Trial 4

As discussed in the previous section (5.1.2), there are general trends to suggest that the single amendment of WTR has a negative influence on the maximum GWC compared to soil alone but improves the hydraulic conductivity in the initial 24 hours of wetting. The single amendment of compost increases the maximum GWC to the greatest extent but has much less effect on the rate of uptake. The take home point from Trial 3 is that although compost has the best GWC, the co-amendment improves both the gravimetric water content maximum and the rate of uptake compared to unamended soil, which is important as part of the soil's 'flood holding capacity'. However, a statistically viable comparison of results from Trial 3 are difficult to make due to differences in the density and initial water content of samples before wetting.

In Trial 4 we are able to directly compare the gravimetric and volumetric water content and density responses of different amendments as the initial conditions have been controlled. This data set also provides two wetting and two drying cycles. Table 25 serves as a reminder for the amendment proportions used in Trial 4, Figure 55 provides an overview of the GWC changes and Figure 56-58 show an overview of VWC, VWCi and volume changes. The amendments proportions used in Trial 4 were almost completely different to those used in Trial 3, where the amendment proportions were changed to be much lower and aimed to reflect amendment quantities that may be used in the field.

As discussed in Chapter 4 (section 4.4.7) statistical significance has been conducted using a Mann Whitney test at particular points along the time series. On each figure, green markers indicate that the difference between the amendment and unamended soil is statistically significant ( $p < 0.01$ ), and red indicates no significance. As there are two wetting and two drying phases, the data will be analysed as a time sequence using the terms 1<sup>st</sup> wetting (0-228 hours), 1<sup>st</sup> drying (228-576 hours), 2<sup>nd</sup> wetting (576-816 hours), and 2<sup>nd</sup> drying (816 – 1104 hours). Drying rates in the 2<sup>nd</sup> drying are slower than in the 1<sup>st</sup> drying as container lids were left in place allowing limited airflow and water evaporation, which slowed the rate of drying, and were not a function of the water holding capacity of samples. AM1 and AM2 dry at a much faster rate than any other amendment or

unamended soil due to being unintentionally completely uncovered during the second period of drying. Error bars are not presented on the subsequent graphs as the overlap between them makes them difficult to distinguish.

<i>Sample</i>	<i>Soil2 %</i>	<i>Compost2 %</i>	<i>WTR2</i>
Soil2	100	0	0
AM1	90	10	0
AM2	90	0	10 WTR2 <sub>d</sub>
AM3	90	0	10 WTR2 <sub>w</sub>
AM4	90	5	5 WTR2 <sub>d</sub>
AM5	90	5	5 WTR2 <sub>w</sub>
AM6	80	20	0
AM7	80	0	20 WTR2 <sub>d</sub>
AM8	80	0	20 WTR2 <sub>w</sub>
AM9	80	10	10 WTR2 <sub>d</sub>
AM10	80	10	10 WTR2 <sub>w</sub>
AM11 (T3:A10)	70	30	0
AM12 (T3: A11)	70	0	30 WTR2 <sub>d</sub>
AM13	70	0	30 WTR2 <sub>w</sub>
AM14 (T3:A13)	70	15	15 WTR2 <sub>d</sub>
AM15	70	15	15 WTR2 <sub>w</sub>

*Table 25: Soil amendment ratios used for Trial 4*

Figures 55-58 respectively show the general trends in the GWC, volume change, VWC and VWCi of all samples over two wetting and drying cycles, the second of which (2<sup>nd</sup> wetting and 2<sup>nd</sup> drying) may be more relatable to how the amended samples would perform in the real world. This is because the results from the 1<sup>st</sup> wetting and drying cycle may to some degree reflect the effects of the production process and may have behaved more uniformly because of the control of density, mass, and water content rather than due to the influence of the amendments themselves. In general, during the 1<sup>st</sup> wetting the majority of amendments improve the GWC, volume increase, VWC and VWCi compared to unamended soil.

During the 1<sup>st</sup> drying, AM3, AM9, AM10, AM11, AM12, AM14, AM15 have a slower rate of drying than unamended soil (although this may be a function of starting the drying curve at a higher GWC), and remaining amendments appear to dry faster than unamended soil, regardless of the GWC at the start of the 1<sup>st</sup> drying phase. Through the 2<sup>nd</sup> wetting and drying phases all amendments except AM8 perform better (statistical significance will be discussed subsequently) than unamended soil despite a 13.9% - 16% in reduction GWC (as a result of unrecoverable shrinkage during the first drying). This means that the positive effects of amendment are continued past an initial wetting and drying period.

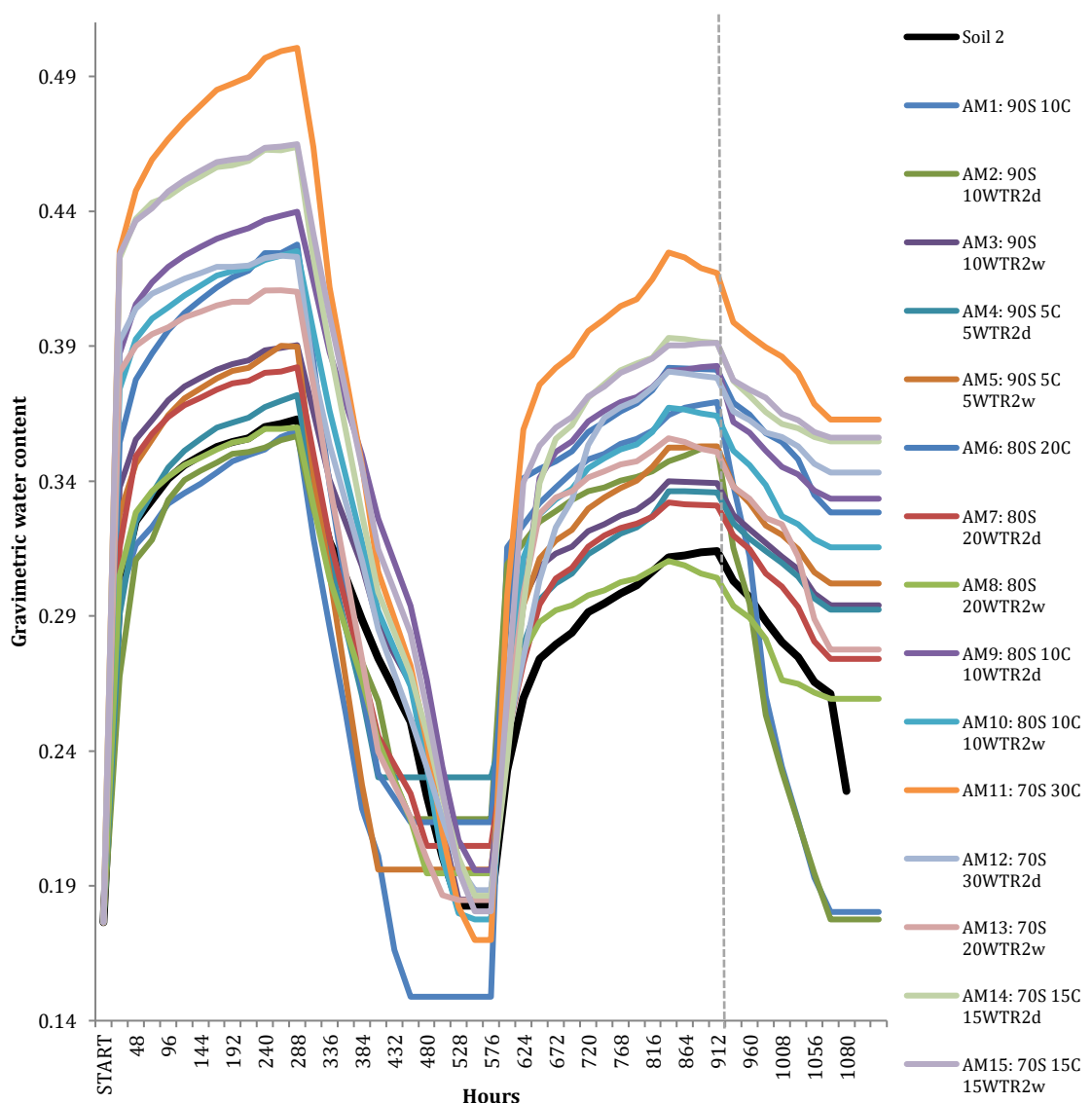


Figure 55: Average GWC change of 15 samples against the control Soil2 (unamended soil), over a 1056 wetting and drying period.  $n = 12$ . Dotted line indicates where the lid was completely removed from samples AM1 and AM2, hence a faster drying rate than other amendments.

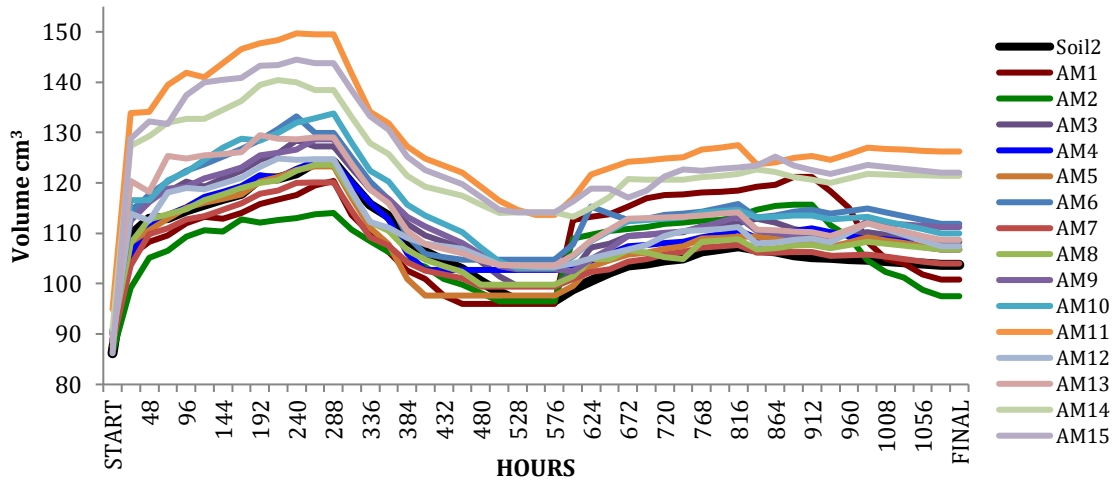


Figure 56: Average change in volume of cores of 15 samples against the control (Soil2) over a 1056 wetting and drying period.  $n = 12$

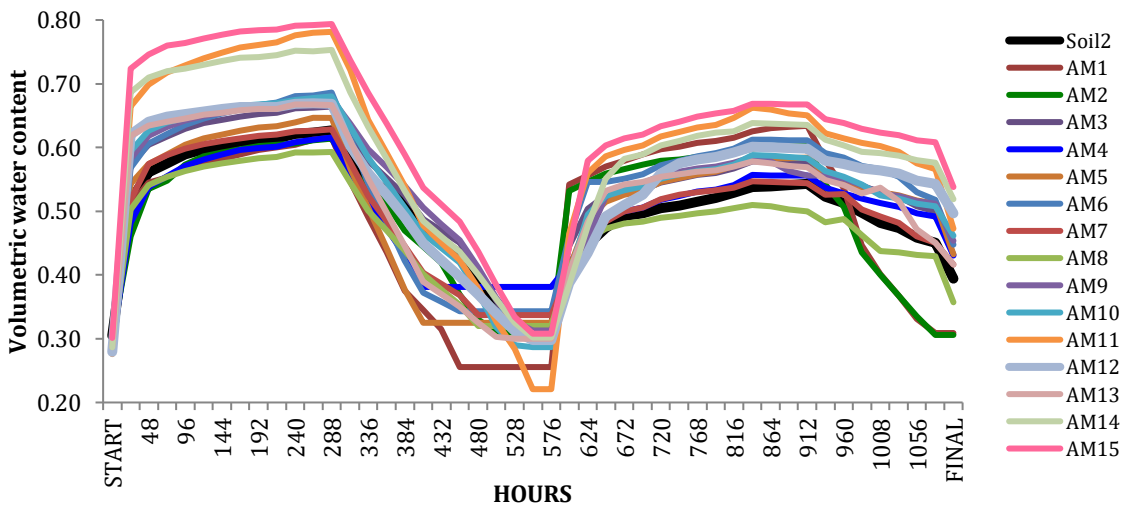


Figure 57: Average change in volumetric water content (VWC) of 15 samples against the control (Soil2) over a 1056 wetting and drying period.  $n = 12$

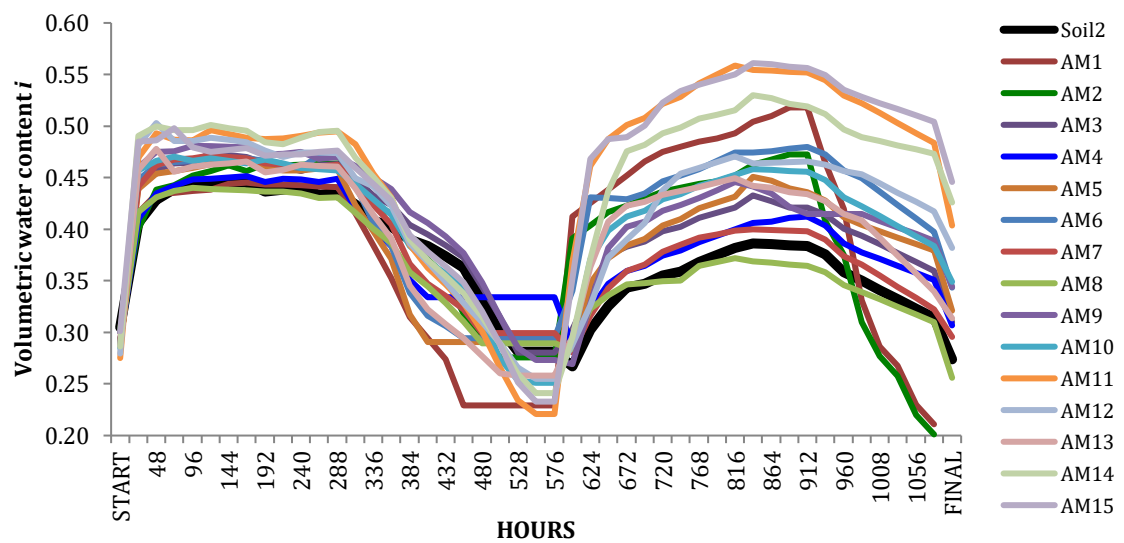


Figure 58: Average change in VWC<sub>i</sub> change of 15 samples against the control (Soil2) over a 1056 wetting and drying period.  $n = 12$

The average volume of soil cores (Figure 56) show considerable fluctuations over time presumably as a result of human error ( $\pm 3.5 \text{ cm}^3$ ) when measuring the samples, as it is illogical for samples to swell and shrink over a wetting cycle unless they undergo collapse (which is not the case here), and values that deviate from what should be a smooth line trend are a function of measurement error only. The values for VWC in Figure 57 are higher than VWC<sub>i</sub> shown in Figure 58 due to difference reference points, the former being the original volume of the sample, and latter being the instantaneous volume of the sample.

Figures 56-58 provide only an overview of results, but a brief analysis of these graphs indicates that, as seen in Trial 3, the volumetric increases are greater in the first wetting than the second wetting, as there is unrecoverable shrinkage during the first drying. A low value for VWC or VWC<sub>i</sub> is not unfavourable provided there is significant volume change of the sample, and hence why the volume data is required as part of the volumetric assessment. An ideal soil, i.e. one that experiences high volume change after wetting (which decreases the bulk density and increases the volume of voids) and has a high value for VWC<sub>i</sub> (not necessarily VWC) will accommodate flood water better than a soil that experiences less volume increase and has a lower VWC<sub>i</sub>. Samples with a higher VWC or VWC<sub>i</sub> in the second wetting, do not necessarily indicate an improvement but instead reflects the volume change of the sample being lower than in the first wetting cycle. This results in a higher value for VWC and VWC<sub>i</sub> as they are relative to the volume of the soil. AM11 (70S 30C) and AM15 (70S 15C 15WTR2<sub>w</sub>) provide an exemplar for improving the WHC as they have the highest sample volumes after the second wetting and the highest proportion of water to solids (VWC<sub>i</sub>).

5.1.3.1 Effect of single amendment at different proportions: 7030, 8020, 9010 on GWC & VWC/VWCi

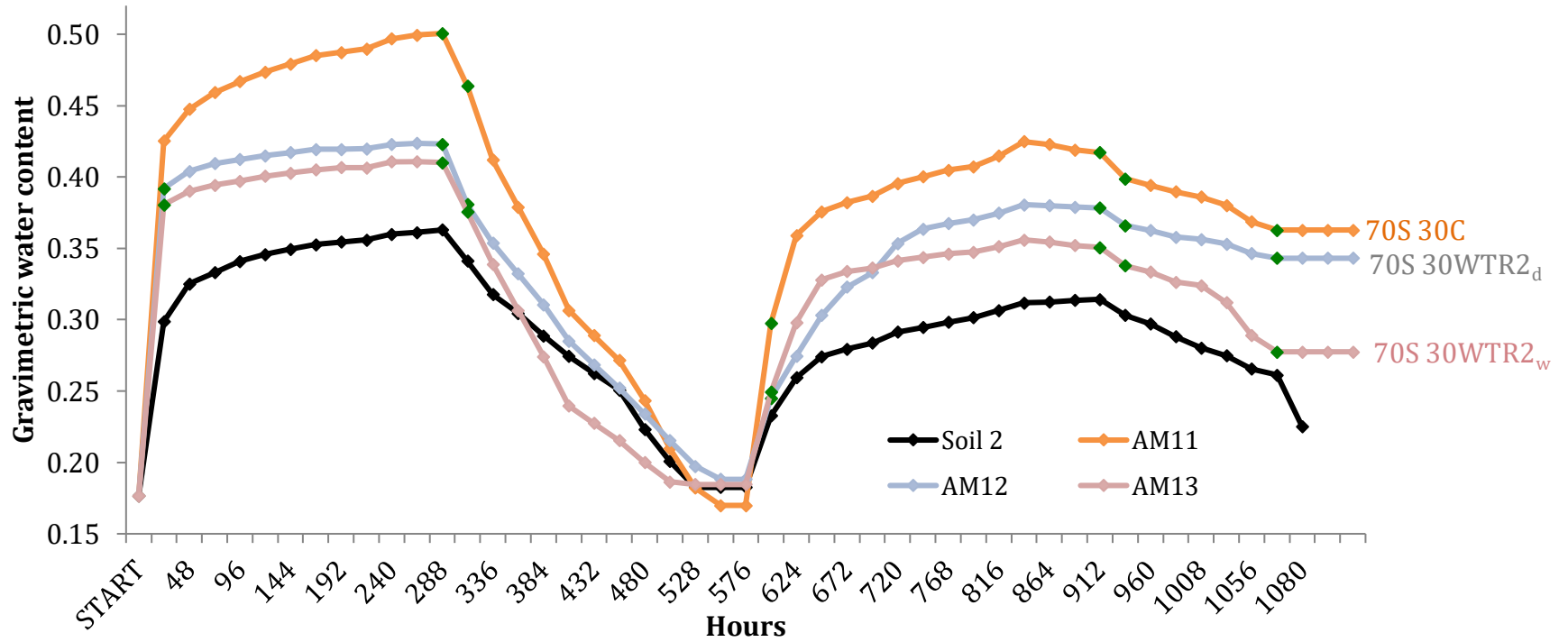


Figure 59: Average GWC for samples with a single amendment at 30%. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

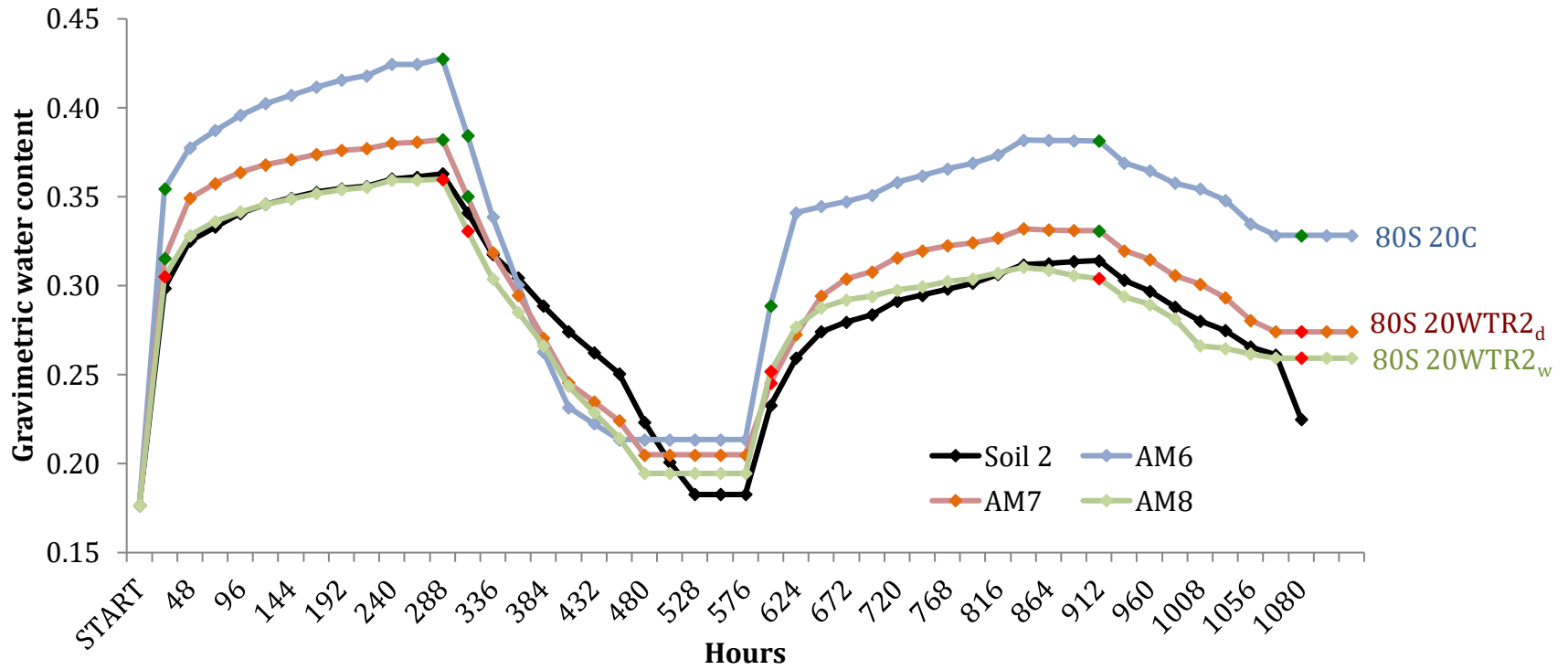


Figure 60: Average GWC for samples with a single amendment at 20%. Green markers indicate statistical significance in the difference between amendments and Soil 2 (unamended soil), red markers indicate there is no significance in the difference.

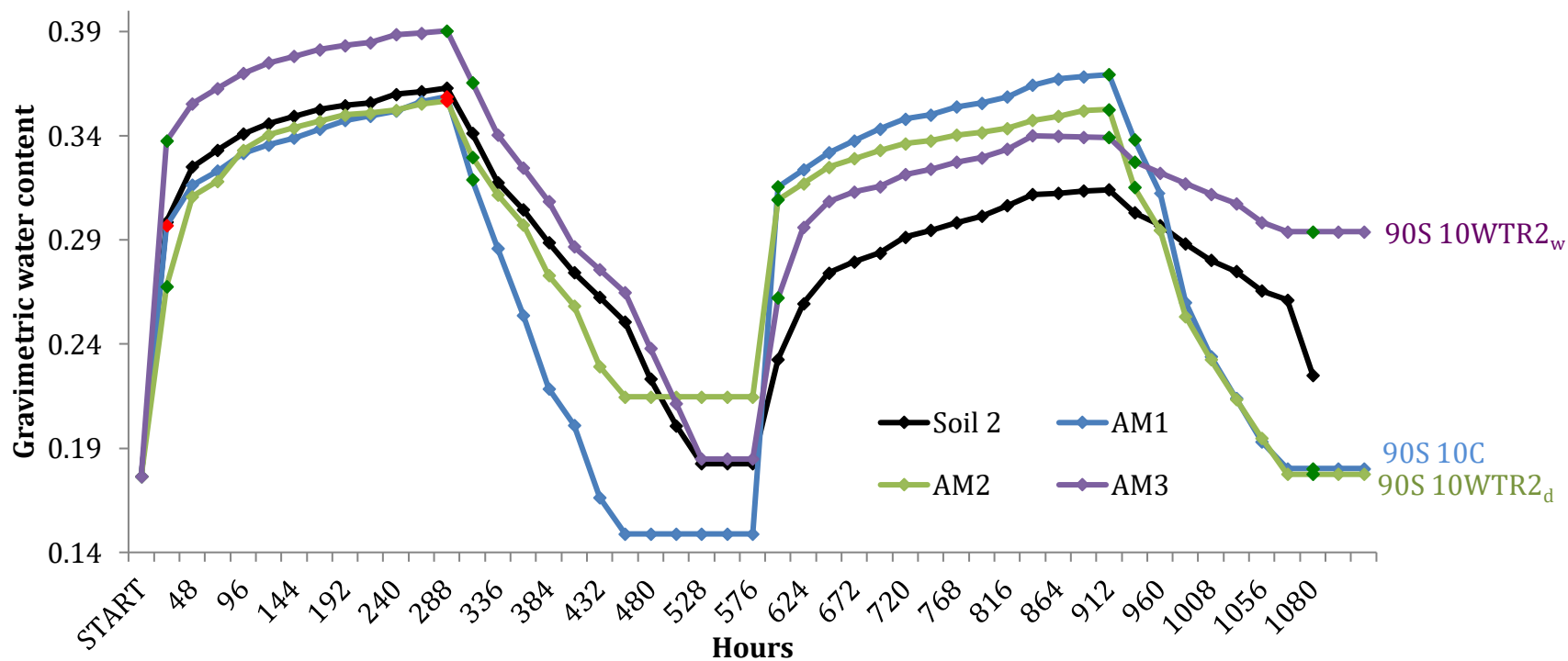


Figure 61: Average GWC for samples with a single amendment at 10%. Green markers indicate statistical significance in the difference between amendments and Soil 2 (unamended soil), red markers indicate there is no significance in the difference

Figure 59 shows that a 30% single amendment of compost results in a higher maximum GWC (0.50) than single amendments of WTR2<sub>d</sub> (0.42) or WTR2<sub>w</sub> (0.41) at 30% or soil alone (0.36). It appears that the rate of uptake into all samples was similar, with the exception of unamended soil, as shown by the gradient of the lines. All samples reached their maximum GWC at the end of the first wetting phase. AM12 (70S 30WTR2<sub>d</sub>) and AM13 (70S 30WTR2<sub>w</sub>) achieved 93% of their water uptake within the first 24-28 hours. AM11 (70S 30C) and Soil2 (100S) in contrast only reached 84% of their maximum GWC within the first 24 hours and continued to take up water at a similar rate until saturation.

During the second wetting phase, in general all amendments held less water and were slower to increase in GWC than the previous wetting cycle (due to expected shrinkage as discussed in Chapter 2). During the second drying phase AM13 (70S 30WTR2<sub>w</sub>) and AM11 (70S 30C) dried faster than AM12 (70S 30WTR2<sub>d</sub>). AM13 (30% WTR2<sub>w</sub>) appeared to take up and release water faster than the other samples, including unamended soil. There is no statistical significance in the difference between AM12 and AM13 until 912 hours is reached, beyond which the difference remains significant ( $p < 0.01$ ). AM11 is statistically significantly different from AM12 and AM13 over the whole time series ( $p < 0.01$ ), as compost is able to take up water within its structure and expands during wetting, therefore it is likely to hold the most water, however WTR2 cannot take water into its structure and is not known to swell during wetting (Moodley & Hughes, 2006). WTR2<sub>d</sub> and WTR2<sub>w</sub> may achieve a higher GWC compared to unamended soil due to an increase in surface area by the addition of fine materials.

The response of soil amendments at a 20% single amendment (Figure 60) are similar to the response at 30%, where compost again has the highest GWC (0.43) and WTR2<sub>d</sub> (AM7) has a maximum of 0.38. WTR2<sub>w</sub> (AM8) and unamended soil both reach 0.36. AM8 (80S 20WTR2<sub>w</sub>), appears to have no effect on the GWC, as shown by the red markers (no statistical difference) at this amendment rate. AM6 (20% compost) and AM7 (20% WTR2<sub>d</sub>) are statistically significantly higher than unamended soil ( $p < 0.01$ ) and the difference between AM6 and AM7 is also statistically significant ( $p < 0.01$ ). Soil2 appears to dry slower than all amended samples during the 1<sup>st</sup> drying phase, however this effect is not apparent during the

2<sup>nd</sup> drying phase. WTR single amendments experience 10% reduction in GWC between 1<sup>st</sup> and 2<sup>nd</sup> wetting peaks, compared to compost amendment that experiences a 12% reduction and unamended soil a 14% reduction. This suggests that the effect of WTR in increasing the GWC is sustained over time (discussed subsequently).

The trend in Figure 61 shows that during at a 10% amendment ratio, during the 1<sup>st</sup> wetting only AM3 (90S 10WTR<sub>2w</sub>) has a statistically significantly higher GWC than unamended soil reaching a peak of 0.39 ( $p < 0.01$ ). AM1 (90S 10C) and AM2 (90S 10WTR<sub>d</sub>) are not significantly different from unamended soil during the 1<sup>st</sup> wetting, both reaching a peak of 0.36. In the first drying phase, 10% compost dried much faster than all other samples (hence it was able to drop to 0.15), as shown by the gradient of the line. Unamended soil and 10% WTR<sub>2w</sub> followed a similar trend to each other whilst drying, however 10% WTR<sub>2d</sub> dried faster than soil alone (conversely to the 30% in which the WTR<sub>2d</sub> dried at a slower rate). This suggests that coarser material added by WTR<sub>2d</sub> increased the rate of drying, possibly due to a higher ratio of large voids which drain faster than smaller pores, present in the unamended soil and WTR<sub>2w</sub> amended soil. 24 hours into the 2<sup>nd</sup> wetting phase, all amendments are significantly higher than soil (as shown by the green markers from 336 hours onwards,  $p < 0.01$ ). Unamended soil, 10% WTR<sub>2w</sub> and 10% WTR<sub>2d</sub> experience a reaction in the maximum GWC (14, 13 and 3% respectively) than in their first wetting phase, however 10% compost had a 3% increase in the GWC. This suggests that at 10% single amendment, the addition of compost or 10% WTR<sub>2d</sub> provide some structural stabilisation such that samples are able to retain porosity after an initial drying period which allows them to hold more water than samples that experience shrinkage.

### ***5.1.3.2 Effect of single amendment at different proportions: 7030, 8020, 9010 on volume & VWC/VWCi***

Figure 62 shows that the single amendment of compost at 30% results in the highest maximum sample volume (149.5 cm<sup>3</sup>) in comparison with unamended soil (124.1 cm<sup>3</sup>) and single amendments at 30% of WTR<sub>2d</sub> and WTR<sub>2w</sub>, reach 124.7 cm<sup>3</sup> and 129 cm<sup>3</sup> respectively. 30% compost has significantly higher sample volume than soil and 30% WTR amendments and 30% WTR<sub>2d</sub> is statistically

better than 30% WTR2<sub>w</sub> ( $p < 0.01$ ), with the exception of the 2<sup>nd</sup> wetting peak (912h) and the end of the 2<sup>nd</sup> drying cycle (1056 hours).

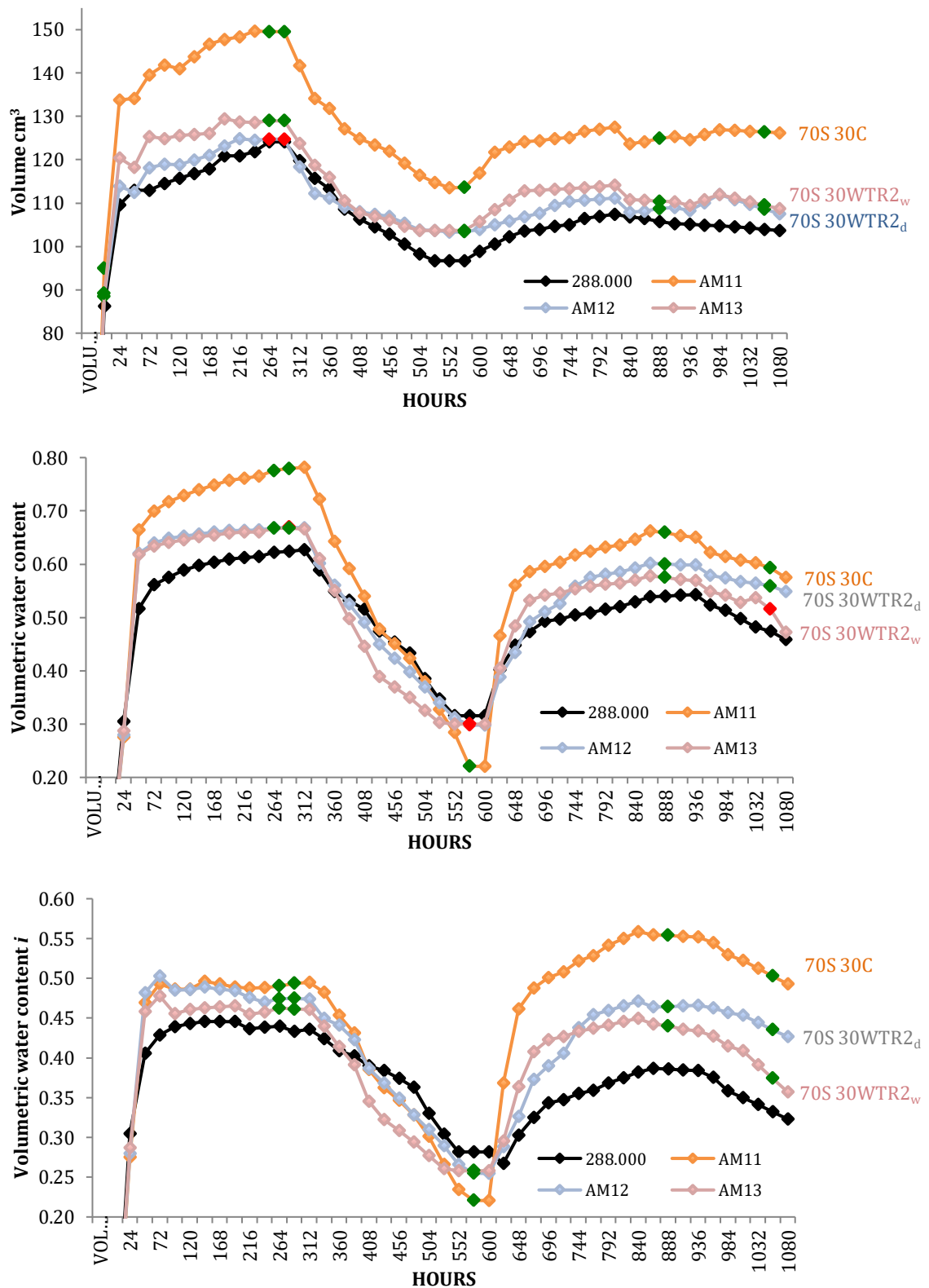


Figure 62: Average effect of single 30% amendments on the volume, VWC and VWC<sub>i</sub> of samples. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

Figure 62 shows that the VWC for all 30% single amendments is statistically higher ( $p < 0.01$ ) than unamended soil (with the exception of 600 hours for both WTR 30% amendments and 1056 hours for 30%). The VWC for 30% WTR single amendments were not statistically different from one another in the first wetting and drying phases however, in the second wetting and drying phases WTR2<sub>d</sub> (AM12) was significantly higher than WTR2<sub>w</sub> (AM13) ( $p < 0.01$ ). Figure 62 shows that the VWC<sub>i</sub>, a ratio of the volume of water to the instantaneous volume of soil, is statistically higher for all amendments at a 30% single amendment than unamended soil. This effect is particularly prevalent during the second wetting and drying phases, where any amendment reduces the shrinkage during the 1<sup>st</sup> drying allowing samples to take up a higher volume of water to the volume of soil in the second period of drying. The higher values for volume and VWC are expected for compost-amended samples due to expansion upon wetting.

Figure 63 below similarly shows that using a single amendment at 20%, compost (AM6) has the greatest volume change, VWC and VWC<sub>i</sub> compared to unamended soil, however at this application rate it is the only single amendment to perform consistently better than soil ( $p < 0.01$ ). AM7 (20% WTR2<sub>d</sub>) has a statistically significant lower volume change than unamended soil ( $p < 0.01$ ) during the first wetting and drying cycle and is no different to unamended soil during the second wetting and drying phases. AM8 (20% WTR2<sub>w</sub>) is either statistically lower ( $p < 0.01$ ) or performs no differently to unamended soil for volume, VWC and VWC<sub>i</sub> (with the exception of the 2<sup>nd</sup> wetting and drying volume changes). This suggests that at a 20% rate, the single amendment of soil with WTR in either form provides either no improvement or a reduction in the sample volume compared to unamended soil.

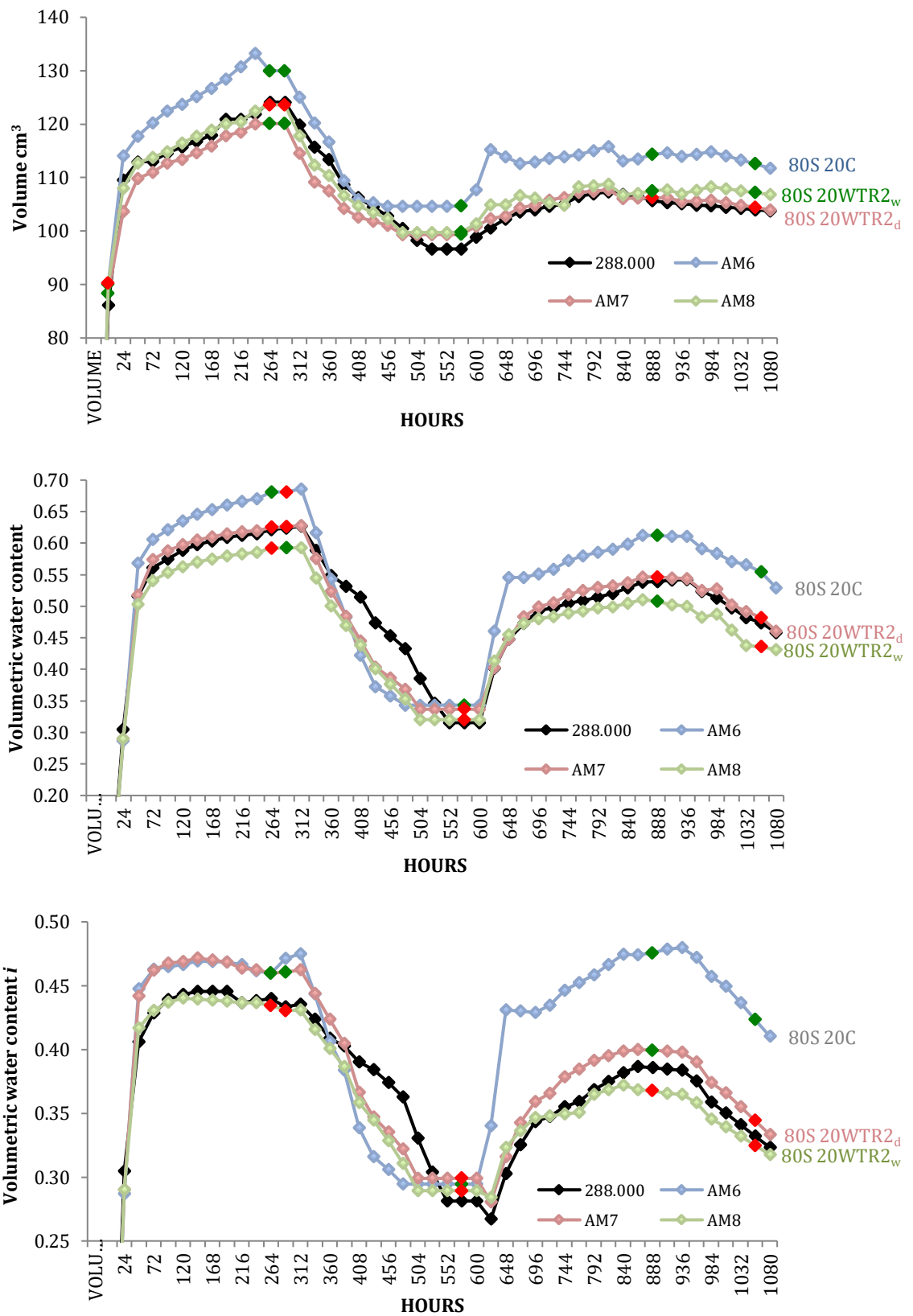


Figure 63: Average effect of single 20% amendments on the volume, VWC and VWCi of samples. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

Figure 64 (below) shows that using a single amendment at a 10% ratio causes little difference in volume, VWC and VWCi during the first wetting and drying cycle compared to unamended soil, a trend also shown in the gravimetric water content change in Figures 59-61. This may reflect the low dosage of amendment where a 10% amendment is not enough to see a difference in response in the first wetting and drying cycles due to limitations on how homogeneously the amendment can be mixed through the soil. However, during the second wetting and drying phases, there is a significant difference between unamended soil and 10% amendments for all volumetric measurements, suggesting that a wetting and drying cycle was needed to initiate to bind potential new aggregates to improve the structure of soil.

A 10% single amendment with compost (AM1) still yields the largest change in volume, VWC and VWCi, in comparison with unamended soil, however the magnitude of the difference is expectedly less than 20% or 30% amendments. The volume change of all 10% amendments during the 1<sup>st</sup> wetting and drying phases are significantly lower or equal to unamended soil, however during the 2<sup>nd</sup> wetting and drying phases, they are significantly higher than unamended soil ( $p < 0.01$ ). This effect is contradictory to the effect of 20%, where the significance in difference remains the same during the 1<sup>st</sup> and 2<sup>nd</sup> wetting and drying sequences. At 10% amendment we see that there is an improvement over time, and in fact the 10% amendments in the second wetting cycle have higher GWC, volume, VWC and VWCi than the 20% amendment. The apparent 'poor' performance of 20% amendments in comparison to the 30% and 10% amendments, where in theory they should fall between the values of the two, may perhaps reflect a fault in manufacture, which would cause these particular set of samples to fall sub-par.

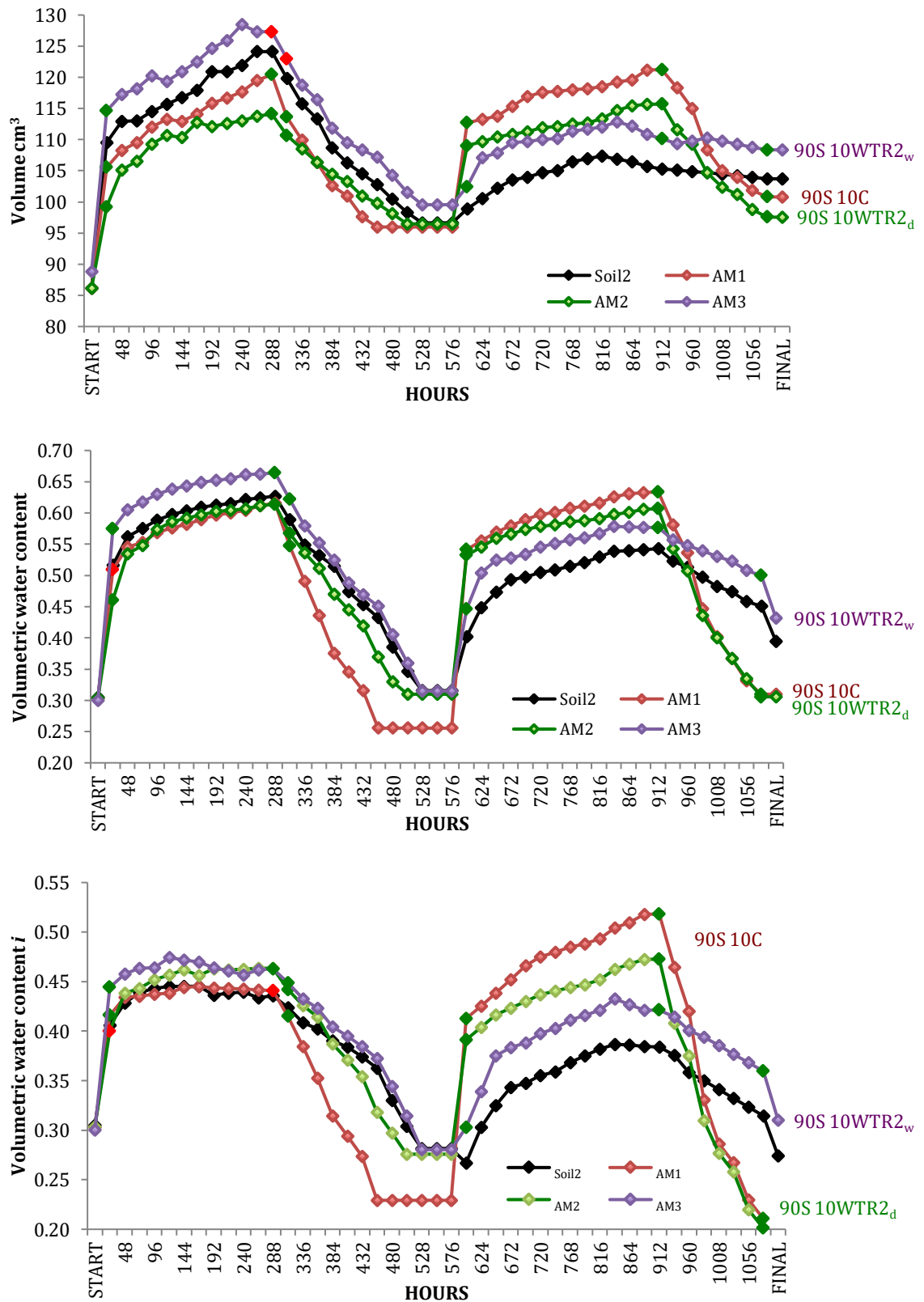


Figure 64: Average effect of single 10% amendment on the volume, VWC and VWC<sub>i</sub> on samples. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

### 5.1.3.3 Effect of single compost amendment on GWC, VWC, VWC and volume

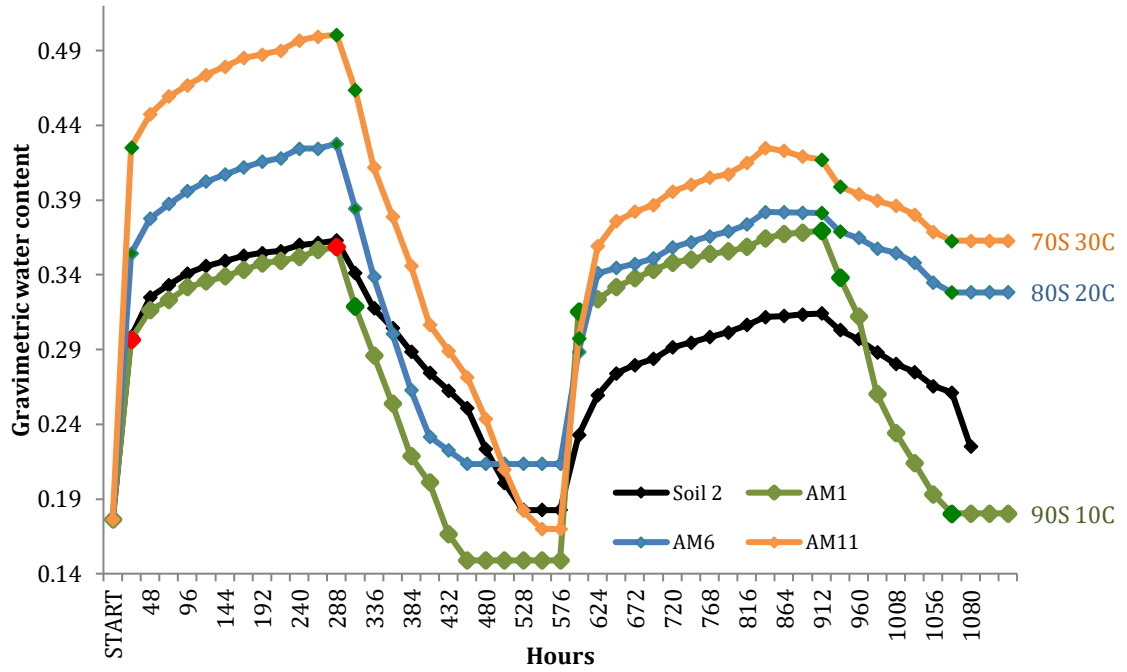


Figure 65: Average effect of single compost amendment at different proportions of amendment, 30%. 20% and 10% on GWC. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

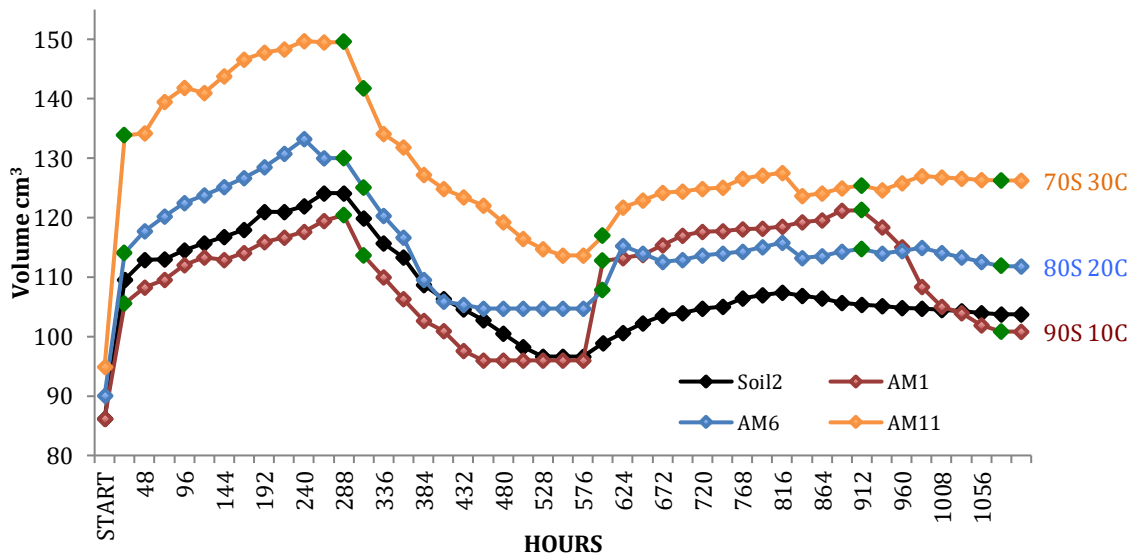


Figure 66: Average effect of single compost amendment at different proportions of amendment, 30%. 20% and 10% on volume. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

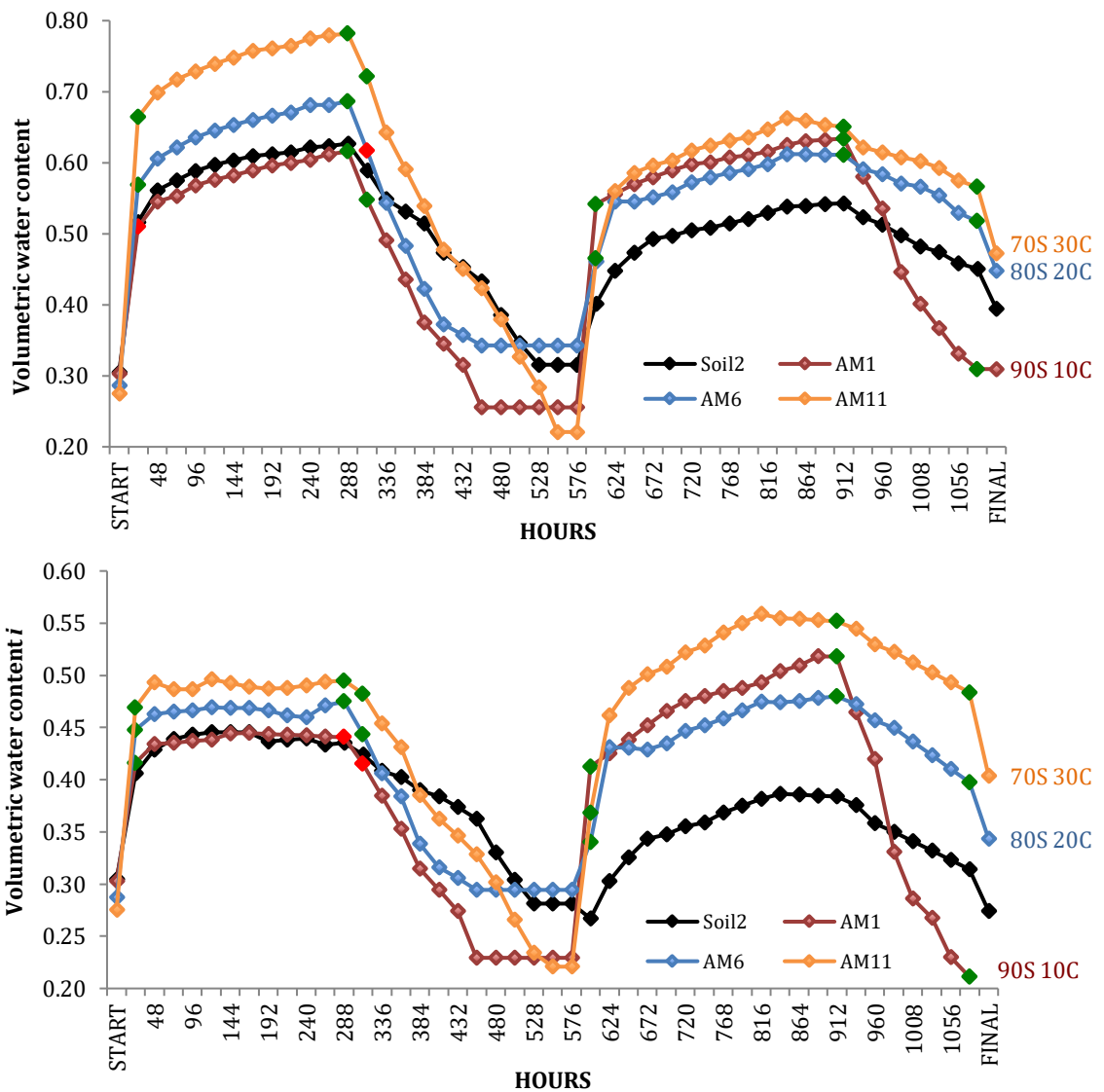


Figure 67: Average effect of single compost amendment at different proportions of amendment, 30%, 20% and 10% on VWC (top) and VWC<sub>i</sub> (bottom). Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference. AM1 was uncovered during the 2<sup>nd</sup> drying, hence a steeper curve than the remaining samples.

Figure 65 indicates that, as seen in Trial 3, the GWC increase is proportional to the ratio of single compost amendment, where a 30% single amendment yields a 34.7% improvement in maximum GWC, a 20% amendment yields a 13.3% improvement compared to unamended soil. 10% compost amendment yields no difference in the first wetting but then shows a 17.5% improvement in the second wetting. Figure 66 shows that the volume change during the 1<sup>st</sup> wetting and drying echoes trends in GWC, where AM11 (30% compost) and AM6 (20% compost) are

significantly higher than unamended soil ( $p < 0.01$ ), but AM1 (10% compost) is significantly lower ( $p < 0.01$ ). Once again, during the 2<sup>nd</sup> wetting, all single compost amendments perform better than unamended soil as they comparatively experience greater volume change during the second wetting cycle. Figure 67 presents the VWC and VWCi of compost single amendments compared with unamended soil. As these values are a function of the GWC and volume change of samples, once again we see that the greater the amendment using compost, the greater the increase in volumetric water content. Interestingly the 9010 (AM1) is higher during the second wetting than the 8020 (AM6) amendment for both VWC and VWCi, however this may reflect the poor performance of 20% amendments in general across the entire Trial.

#### 5.1.3.4 Effect of single $WTR_d/WTR_w$ amendment on GWC, VWC, VWCi and volume

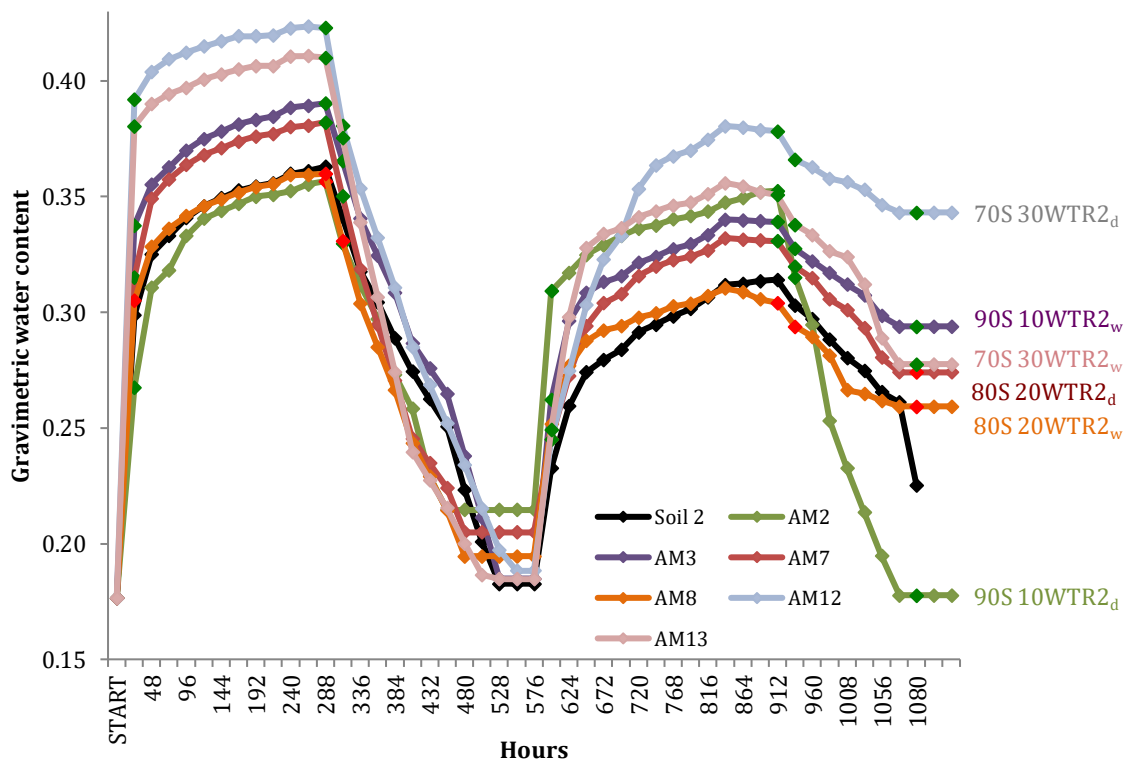


Figure 68: Average effect of single  $WTR2_d$  and  $WTR2_w$  amendment at different proportions of amendment, 30%, 20% and 10% on GWC. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

Figures 68 and 69 show the effect of single amendments of WTR<sub>2d</sub> and WTR<sub>2w</sub> at various proportions on the water holding capacity (GWC, volume, VWC and VWC<sub>i</sub>) compared with unamended soil. In Figure 68, both of the 30% WTR amendments (AM12 and AM13) have the highest maximum GWC (0.42 and 0.41 respectively) in both wetting phases. Although most of the single WTR amendments statistically significantly improve the GWC compared to unamended soil, the effect appears not to be simply a function of the amount or the type of amendment in the soil. Neither 10% WTR<sub>2d</sub> or 20% WTR<sub>2w</sub> are significantly different from unamended soil during the 1<sup>st</sup> wetting and drying cycle, however their counterparts AM3 (10WTR<sub>2w</sub>) and AM7 (20WTR<sub>2d</sub>) both have statistically significantly higher GWC than unamended soil ( $p < 0.01$ ), which suggests that there is not a steadfast relationship between the use of wet or dry WTR with the resultant change in GWC. During the 2<sup>nd</sup> wetting and drying, although the highest amendment proportions still have the highest overall GWC, the 10% amendments of WTR (AM2 and AM3) have higher GWC than the 20% single amendments (AM7 and AM8), which again reflects the general poor performance of 20% amendments. The difference between the use of WTR<sub>2d</sub> and WTR<sub>2w</sub> isn't always significant nor is there a consistent trend with which type of WTR performs best in terms of increasing the GWC of soil.

Figure 69 shows that the samples with the highest GWC also have the highest volume change, however 30% WTR<sub>2w</sub> (AM13) is the only amendment that has a statistically significantly higher volume than unamended soil during the first wetting and drying cycle ( $p < 0.01$ ) reaching a maximum of 129 cm<sup>3</sup> compared to unamended soil achieving maximum of 124.1 cm<sup>3</sup>. All single amendments with the exception of AM7 have a statistically significantly higher sample volume than unamended soil during the second wetting and drying phases, where AM2 (10% WTR<sub>2d</sub>) has the highest secondary max of 115.7 cm<sup>3</sup>. Similarly, to the GWC change shown in Figure 68, there appears to be no trend in volume change that corresponds with either the proportion of amendment or the type of WTR added. We know from informal submersion experiments that dried WTR does not swell upon wetting despite its high organic content (which may be due to the organic matter being 'locked in' by the drying process), however we have not quantified

the behaviour of organic matter present in WTR2<sub>w</sub>. The addition of WTR must therefore either improve soil structure so that the soil is able to swell to a better

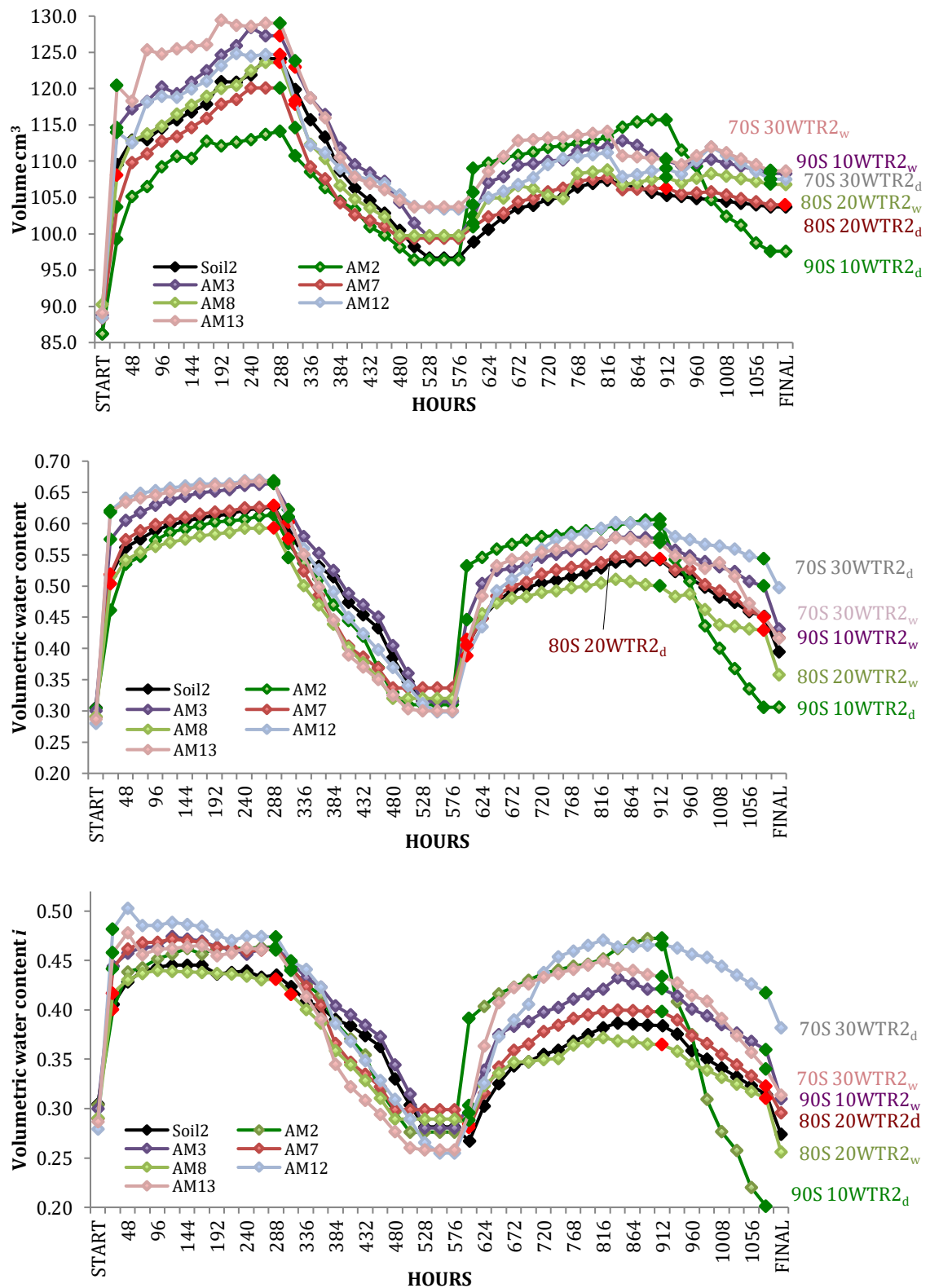


Figure 69: Average effect of single WTR2<sub>d</sub> and WTR2<sub>w</sub> amendment at different proportions of amendment, 30%, 20% and 10% on VWC and VWC<sub>i</sub>. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

extent than soil alone, or the presence of organic matter causes a greater degree of swelling. As the latter is unlikely, as Moodley & Hughes (2006) suggest, due to the presences of binding agents, the inclination is to accept the former conclusion that WTR aids soil structure to improve volume change. The high surface area particularly of wet WTR, due to the high fraction of very fine particles (95% are <75  $\mu\text{m}$ ) may also account for the increase in volume compared to unamended soil, as water can coat each individual particle, whereas in samples amended with compost we see such an increase in volume because the compost swells in isolation within the soil.

Figure 69 shows that AM3 (10 WTR2<sub>w</sub>), AM12 (30 WTR2<sub>d</sub>) and AM13 (30 WTR2<sub>w</sub>) are the only amendments that remain significantly higher in VWC and VWC<sub>i</sub> than unamended soil over all four phases ( $p < 0.01$ ). AM7 and AM8 (20 WTR2<sub>d</sub>/WTR2<sub>w</sub> respectively) are no different to unamended soil, or are significantly lower for VWC and VWC<sub>i</sub>. AM2 (10 WTR2<sub>d</sub>) is only significantly different during the second wetting phase but reaches the highest secondary maximum for both VWC (0.607) and VWC<sub>i</sub> (0.473). Overall, the highest proportion single amendments of WTR2<sub>d</sub> or WTR2<sub>w</sub> (30%) illicit the largest improvements in GWC, volume, VWC and VWC<sub>i</sub>, however the relationship between 10% and 20% amendment proportions is less clear as a 20% amendment does not always perform better than a 10% amendment. Despite the addition of WTR2<sub>w</sub> appearing to outperform WTR2<sub>d</sub> when comparing amendments at the same proportion, there are some exceptions to this which means a preference to the form of WTR is not able to be concluded.

### 5.1.3.5 Effect of co-amendment on GWC & VWC/VWCI and volume

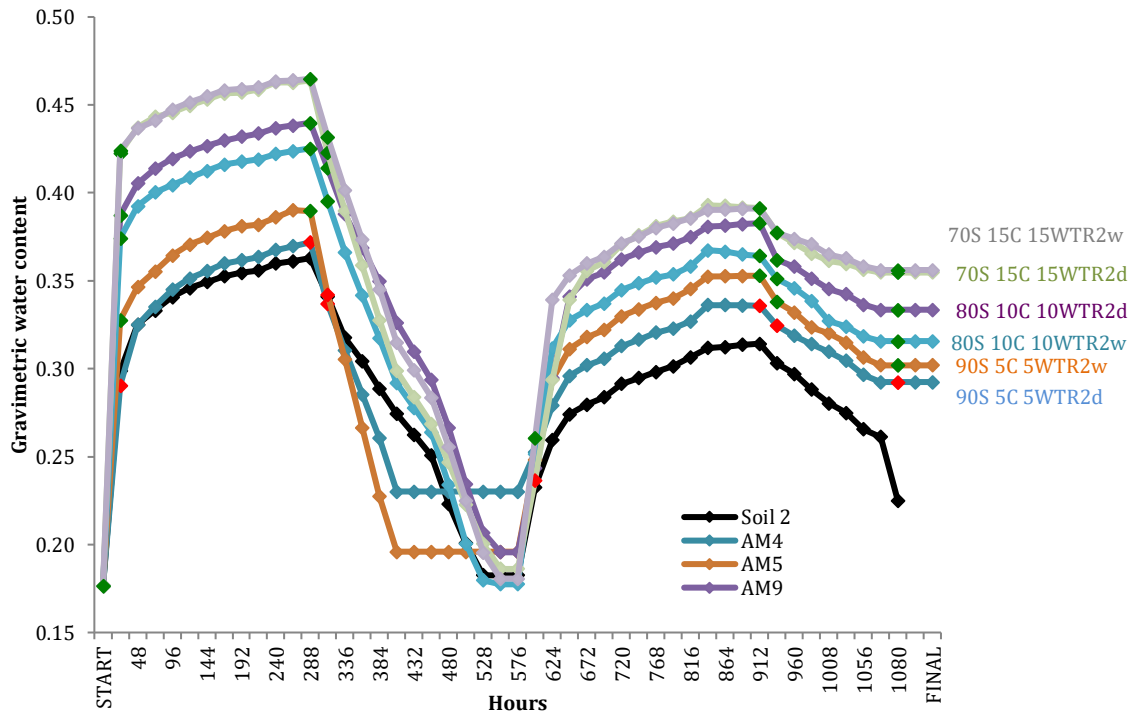


Figure 70: Average effect of co-amendment at different proportions of amendment, 30%, 20% and 10% on GWC. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

Figures 70 & 71 provide an overview of the effect of compost & WTR2 co-amendment on the GWC, volume, VWC and VWCI of samples in comparison to unamended soil. A detailed comparison between single and co-amendments are explored in the next section (5.1.3.6). The maximum observed values for GWC, volume, VWC and VWCI are all reached by the 30% compost/WTR2<sub>w</sub> amendment AM15 (0.46, 143.7 cm<sup>3</sup>, 0.794 and 0.561, respectively). It is clear that measured changes are proportional to the amount of co-amendment, where in Figure 70 it can be seen that a 30% co-amendment (15% compost, 15% WTR2<sub>d</sub> or WTR2<sub>w</sub>) has a significantly greater GWC than both unamended soil and a 20% co-amendment ( $p < 0.01$ ). In addition the difference in GWC between 20% co-amendment and the 10% co-amendment is statistically significant ( $p < 0.01$ ). In general, these results echo those from analysis of the single amendment of WTR shown in Figures 68 & 69, and show that there is no clear pattern that distinguishes WTR2<sub>d</sub> to be better or worse than WTR2<sub>w</sub> in improving the WHC of soil. The volume, VWC and VWCI

changes as shown in Figure 71 below also show that the volumetric change of samples is dependent on the co-amendment rate, where 30% co-amendment using

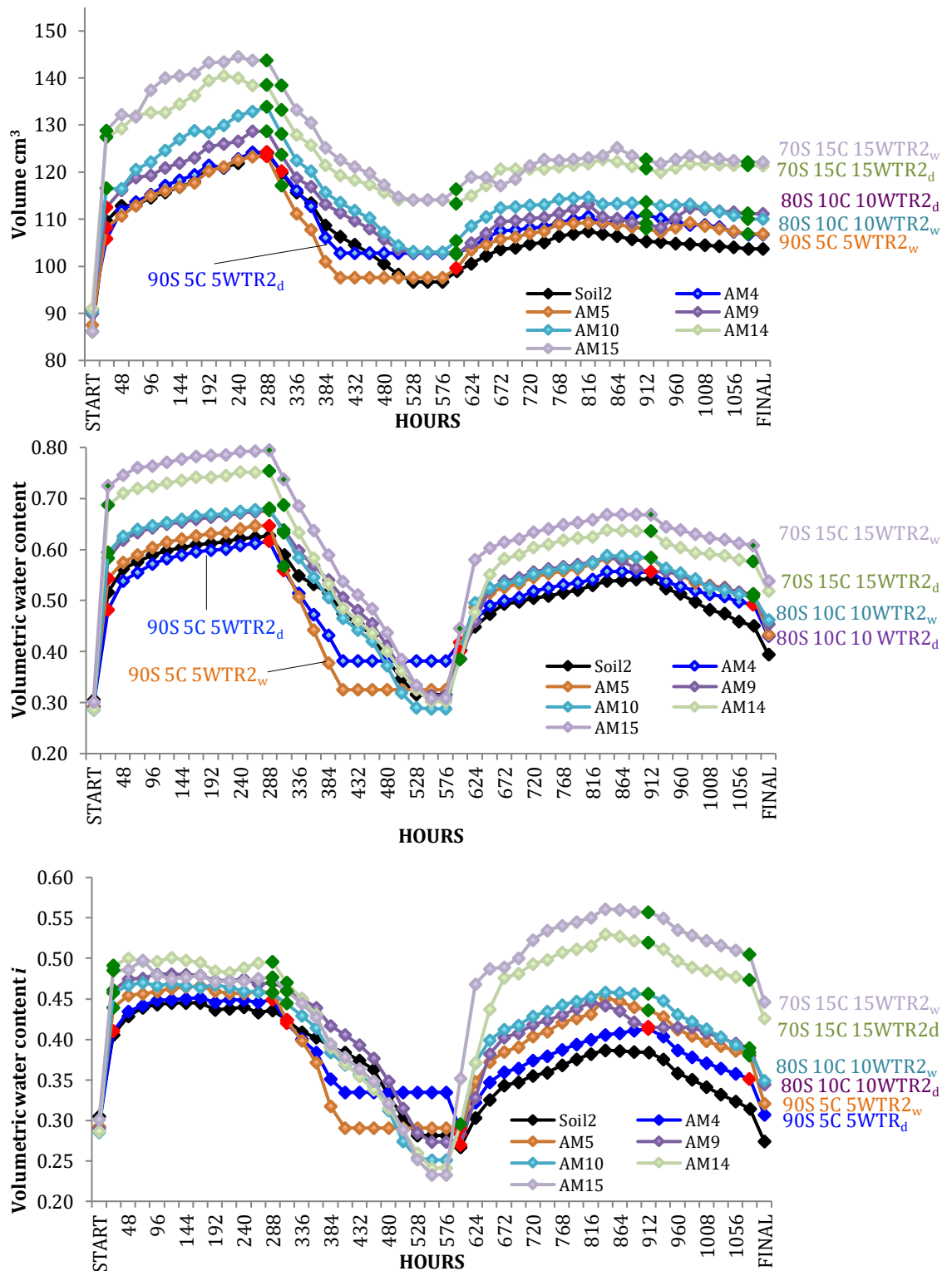


Figure 71: Average effect of co-amendment at different proportions of amendment, 30%, 20% and 10% on volume, VWC and VWCi. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

WTR2<sub>w</sub> (AM15) has the highest volume, VWC and VWC<sub>i</sub> over the entire time series. Wet WTR gives a significantly higher sample volume than dry WTR at the 30% co-amendment rate, however at a 20% amendment rate the volume change is significantly higher for amendments with dried WTR. There is no statistical difference in volume change between wet and dry WTR at 10% co-amendment.

### 5.1.3.6 Effect of co-amendment vs single amendment on GWC & VWC/VWC<sub>i</sub> and volume

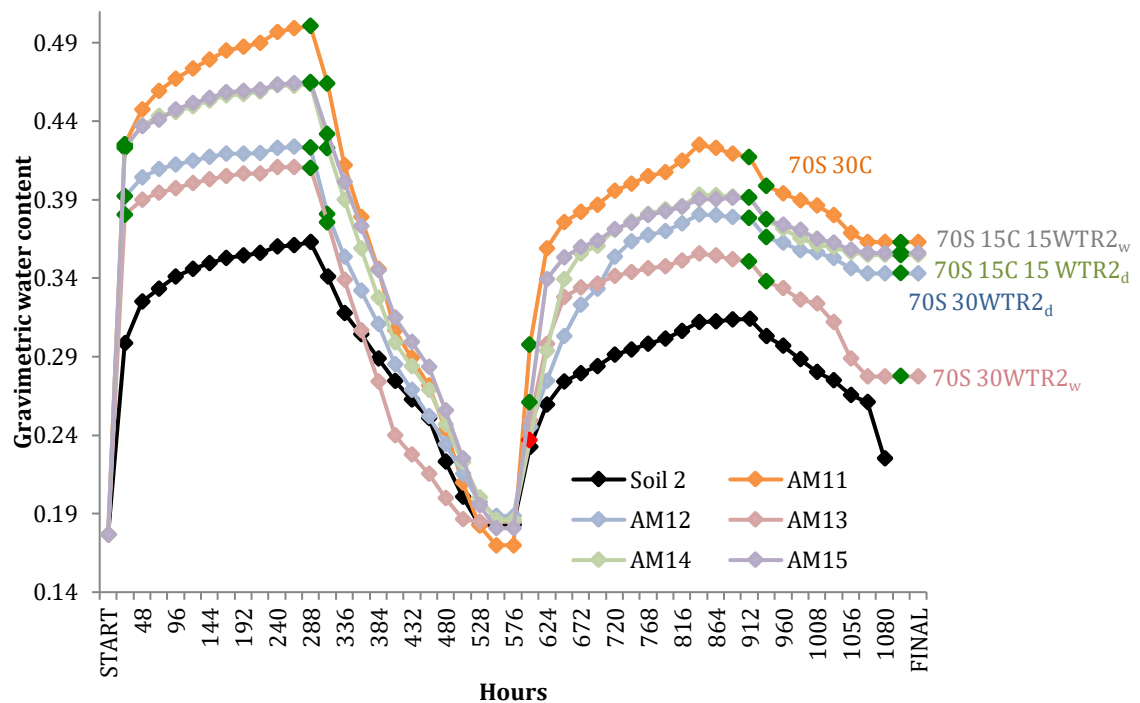


Figure 72: Average effect of single amendment vs co-amendment at 30% amendment on GWC. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

Figure 72 compares the effect of single amendment at 30% to co-amendment at the same rate on GWC through the wetting and drying process. All amendments have a significantly higher GWC than unamended soil, where the single amendment of compost at 30% (AM11) has the highest overall GWC, peaking at 0.50 and is significantly different from the next highest performing amendments ( $p < 0.01$ ). As also seen in Trial 3, AM14 and AM15, the co-amended samples, have higher GWC values than the single amendments of either WTR2<sub>d</sub> (AM12) or WTR2<sub>w</sub> (AM13) across the whole of the time series. Although the single

amendment of  $WTR_d$  performs better in general than  $WTR_w$ , the inverse is apparent when the sample is co-amended, where all co-amendments with  $WTR2_w$  perform better than  $WTR2_d$  counterparts. Importantly, the GWC of all co-amended samples remain higher than unamended soil during the second wetting and drying. In Figure 72, only one amendment dries faster than unamended soil (AM13, 30%  $WTR2_w$ ), which suggests that although this amendment has a high capacity to hold water, this amendment then allows drainage of the soil at a greater rate than unamended soil and does not retain the water.

Figure 73 compares the volume change of samples where the single amendment of compost has the greatest volume change (AM11), reaching a maximum of  $149.5 \text{ cm}^3$  compared to unamended soil at  $124.1 \text{ cm}^3$ , and was significantly better than all other treatments ( $p < 0.01$ ), with the exception of AM15 at 600h and 912h. Although all samples undergo swelling and shrinkage during the 1<sup>st</sup> wetting and drying, the amended samples shrink to a lesser degree than unamended soil (which shrinks back to a volume of  $96 \text{ cm}^3$ ) where in contrast amended samples reach between  $103$  and  $114 \text{ cm}^3$  despite being dried to a similar GWC. There is proportionally much less change during the 2<sup>nd</sup> wetting and drying phases, suggesting that amendment stabilises the soil over time to reduce shrinkage. Figures 72 and 73 suggests that by co-amending the samples, the soil inherits the beneficial water holding capacity of compost while inheriting the beneficial structural changes that WTR impact on the soil.

Figure 73 also compares the VWC and VWCi of single and co-amended samples, which reflect the GWC and volume change of samples over time. Overall, co-amendments have higher values than single amendments at the same amendment proportion, and considering that the co-amendments also have the highest sample volumes this means that they hold more water than unamended soil and single amendments (excluding AM11, 70S 30C). Amendments with the highest VWC, AM11 (70S 30C) and AM15 (70S 15C 15 $WTR2_w$ ), are not statistically different from each other, showing therefore that the co-amendment performs equally well to a single amendment, where AM15 reaches a maximum VWC of 0.79 and AM11 reaches 0.77.

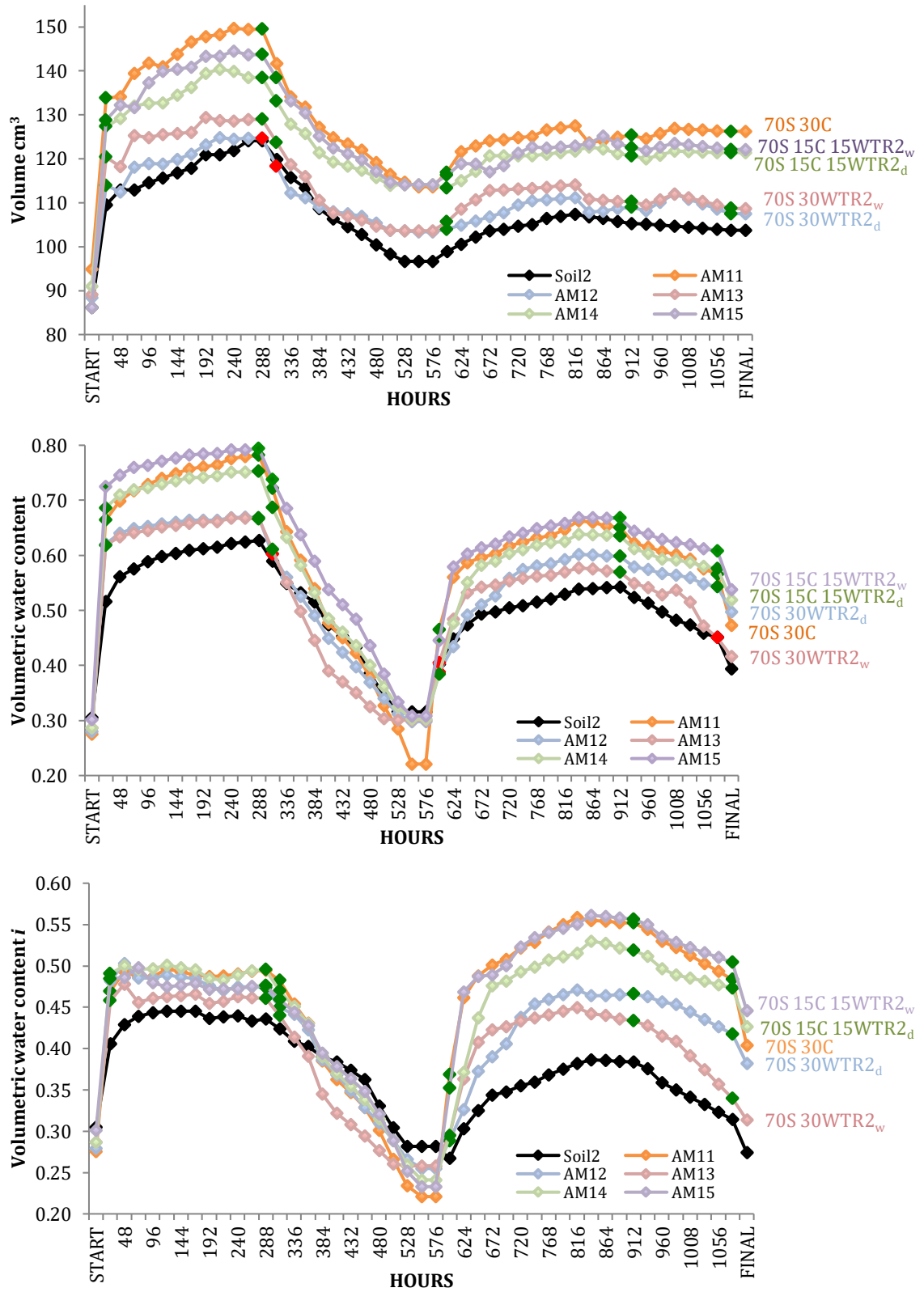


Figure 73: Average effect of single amendment vs co-amendment at 30% amendment on volume, VWC and VWCi. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

In Figure 73, the VWC of single 30% amendments of compost or WTR (AM11, AM12 and AM12) are 16% lower than their co-amended counterparts (AM14 and AM15) throughout the two wetting and drying cycles. VWC<sub>i</sub>, a measure of the instantaneous volume of water to the volume solids provides a good indicator of the volumetric response over time, not just a comparison to the original state of a sample. All single and co-amendments in Figure 73 have significantly higher VWC<sub>i</sub> than unamended soil ( $p < 0.01$ ), however this difference is exacerbated during the 2<sup>nd</sup> wetting and drying phases, where 30% co-amendment has a 31% higher VWC<sub>i</sub> value than unamended soil and importantly performs equally well as a single 30% compost amendment. The higher VWC<sub>i</sub> in the 2<sup>nd</sup> wetting and drying phases indicates that there is a greater volume of water to the volume of solids than the 1<sup>st</sup> wetting and drying phases, however this is due to the reduction of the bulk volume of solids, not an increased GWC. As the volume change is known, it can be concluded that amendment with either 30% compost or a 30% co-amendment of WTR2 yield similar increases in the WHC of the soil, where single using WTR amendment only yields a small increase.

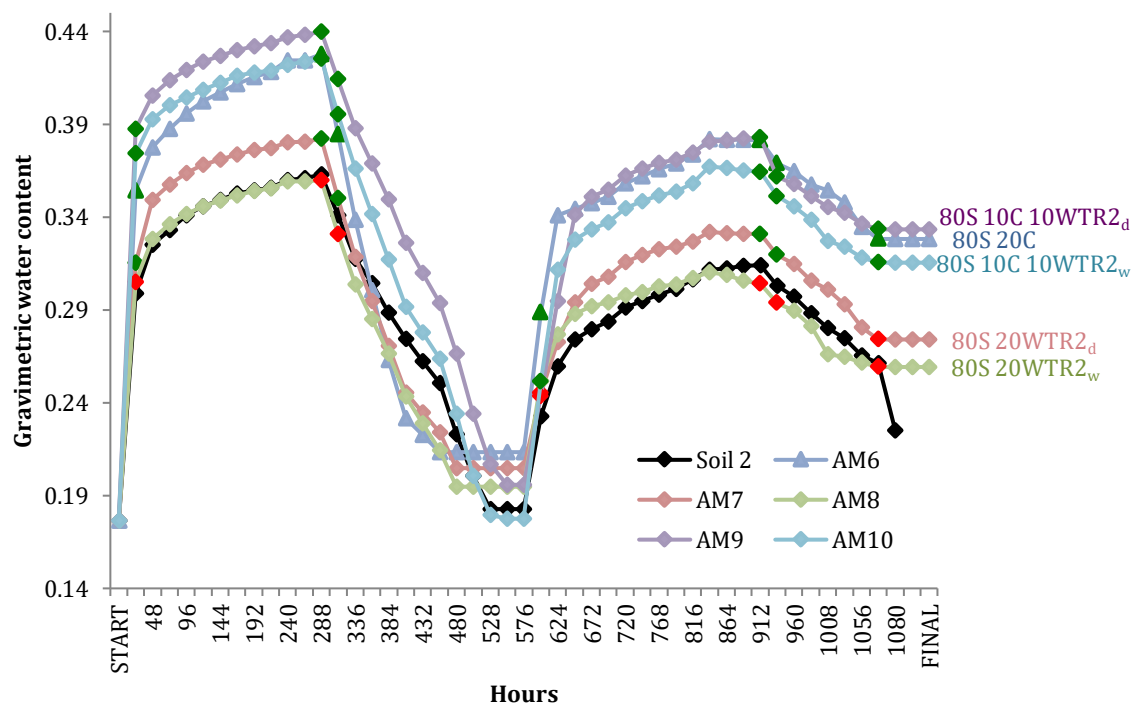


Figure 74: Average effect of single amendment vs co-amendment at 20% amendment on GWC. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

Figures 74 & 75 compare single 20% amendments with co-amendment at the same ratio and have a similar trend to those seen for the 30% amendment. The greatest increase in GWC (up to 0.44) is achieved by both the co-amendments (AM9 or AM10) or single amendment of compost (AM6), where there is no statistical difference between the three. This suggests that at a 20% amendment ratio, the co-amendment performs as well as a single amendment of compost for GWC change. As shown previously, the co-amendments perform better than single amendments, and although 20% single amendment with WTR2<sub>d</sub> (AM7) increases the GWC, it is significantly lower than single amendment of compost (AM6) and co-amendments AM9 and AM10 ( $p < 0.01$ ). The 20% single amendment with WTR2<sub>w</sub> (AM8) has no statistical difference to unamended soil.

In Figure 75 there is no statistical difference between the increase in VWC for the single amendment of compost and the co-amendment at 20%, where co-amendments again perform better than their single amendment. For VWC<sub>i</sub> shown in Figure 75, the trend remains the same. At 20% amendment rate there is no difference in the improvement of GWC, volume, VWC and VWC<sub>i</sub>, between adding compost only and a compost/WTR2 co-amendment.

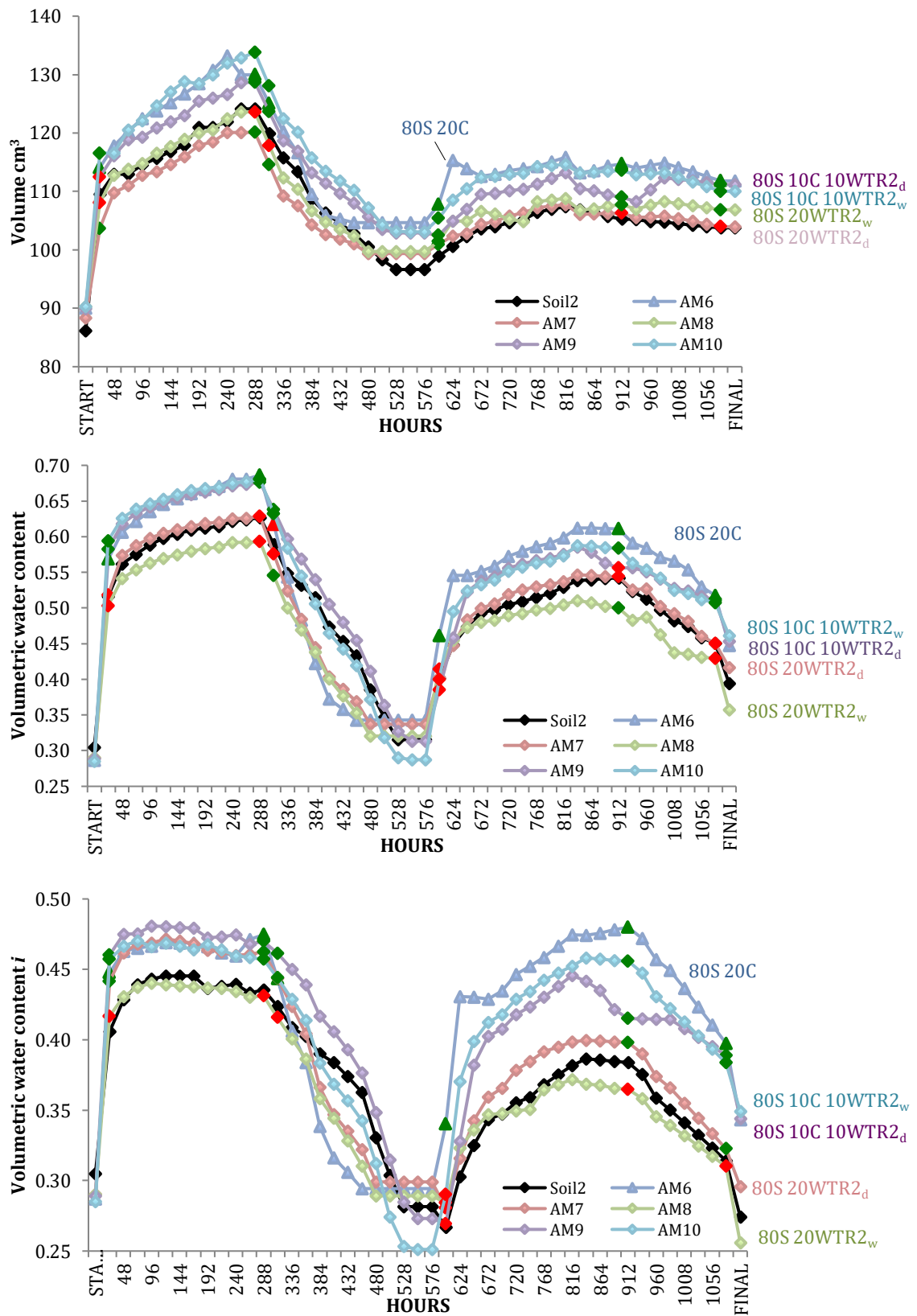


Figure 75: Average effect of single amendment vs co-amendment at 20% amendment on volume, VWC and VWC<sub>i</sub>. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

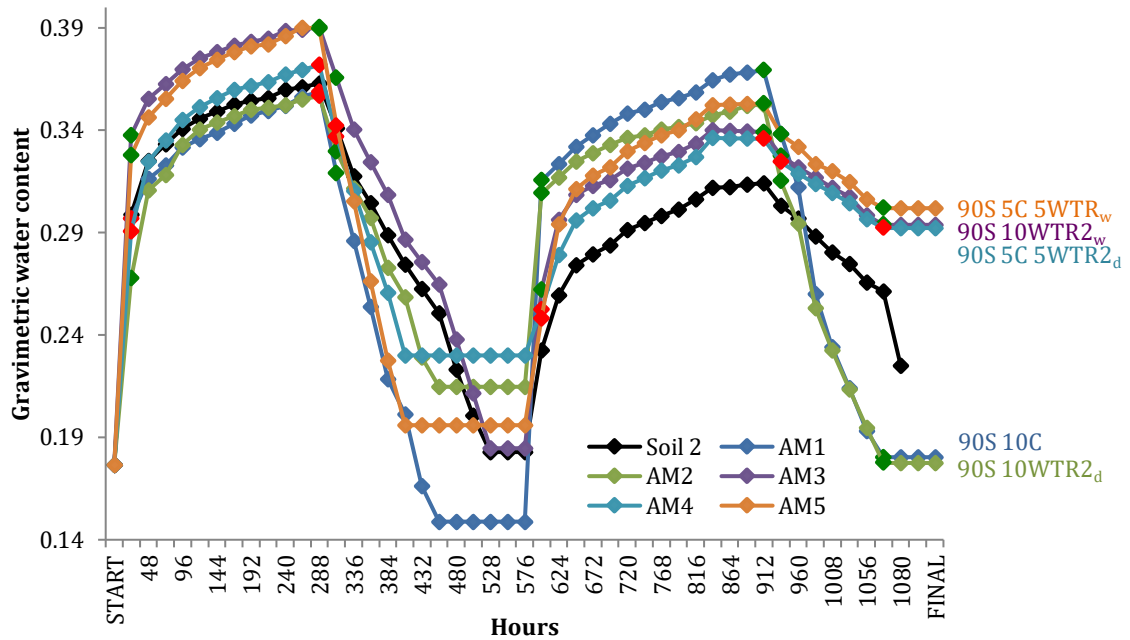


Figure 76: Average effect of single amendment vs co-amendment at 10% amendment on GWC. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

Figures 76 and 77 compare single 10% amendments with co-amendments at the same ratio. The differences between unamended soil and the amended samples are much smaller, as expected, than the larger amendment proportions. At 10% amendment, the single compost amendment no longer has the highest GWC change for the first wetting and drying. In Figure 76 only 10% single WTR2<sub>w</sub> (AM3) and 10% co-amendment with WTR2<sub>w</sub> (AM5) are statistically significantly different from unamended soil over the whole time series ( $p < 0.01$ ). AM4 (10% co-amendment with WTR2<sub>d</sub>) is not statistically different from unamended soil despite the WTR2<sub>w</sub> counterpart performing well. During the 2<sup>nd</sup> wetting and drying all amendments at 10% amendment or co-amendment had a higher GWC than unamended soil (with the exception of AM4), where the single amendment of compost held the most water during the second wetting.

The volume of samples shown in Figure 77 during the 1<sup>st</sup> wetting and drying phases are not significantly different from unamended soil for any amendment and all return to a similar volume at the end of the 1<sup>st</sup> drying phase. However, during the 2<sup>nd</sup> wetting and drying, 10% single amendments of compost (AM1) and WTR2

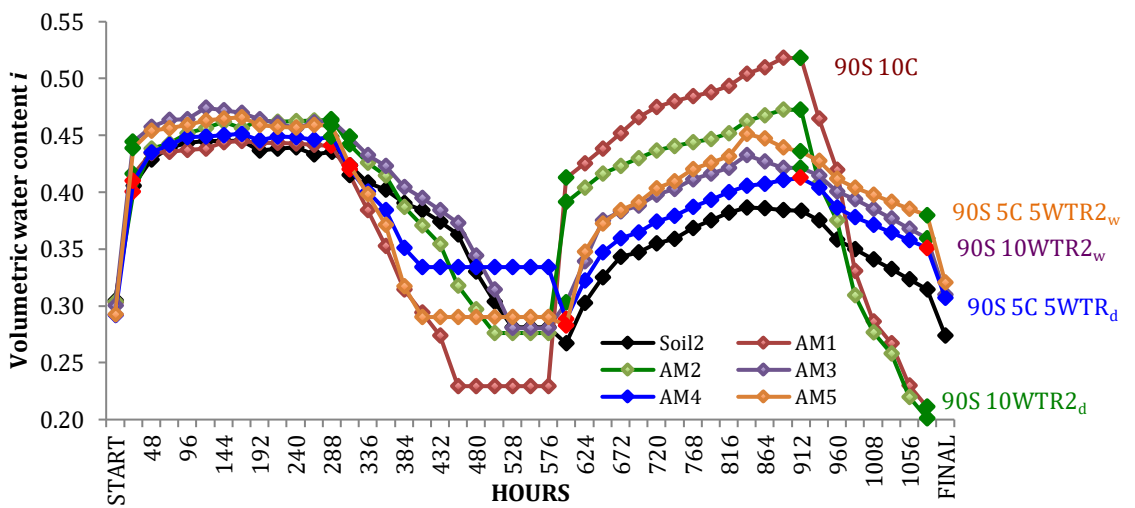
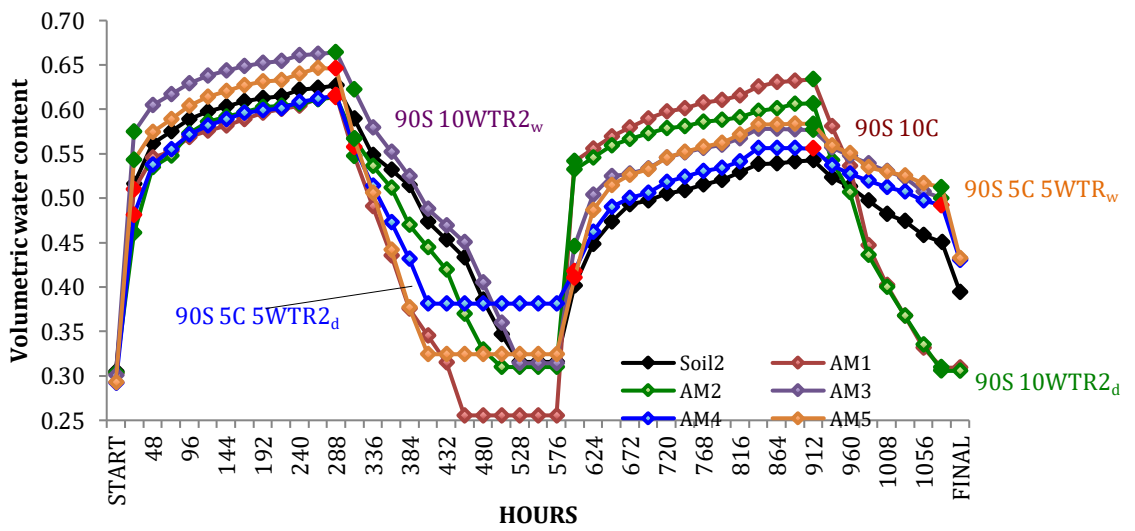
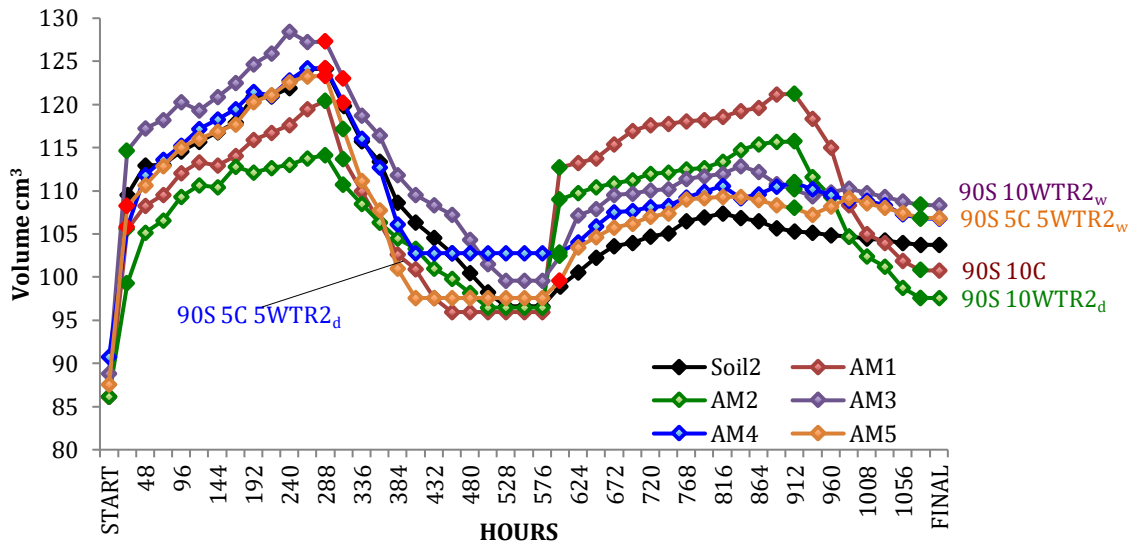


Figure 77: Average effect single amendment vs co-amendment at 10% amendment on VWC and VWC<sub>i</sub>. Green markers indicate statistical significance in the difference between amendments and Soil2 (unamended soil), red markers indicate there is no significance in the difference.

(AM2 and AM3) have a higher volume peak than co-amendments at the same ratio, which is a non sequitur to the effect of co-amendments at 30 and 20% proportions. Figure 77 also compares the VWC and VWCi of 10% amendment and shows that the VWC during the 1<sup>st</sup> wetting and drying is only significantly higher for one amendment (AM3, 10% WTR2<sub>w</sub>,  $p < 0.01$ ) in comparison to unamended soil. During the secondary wetting phase, all amendments except AM4 (10% co-amendment with WTR2<sub>a</sub>) are significantly greater than unamended soil ( $p < 0.01$ ). This provides further evidence that at a low amendment proportion (10%), due to the heterogeneous distribution of the amendment, the soil requires an initial wetting and drying period for these amendments to take effect whereas at higher amendment proportions of 20 and 30%, the effect is more readily measured. Similarly, Figure 77 shows that during the 2<sup>nd</sup> wetting and drying, the VWCi increases significantly, and as discussed previously, we know that the volume of the unamended soil is also lower than the amendments, meaning the increase in VWCi is not just a function of smaller samples.

#### **5.1.3.7 Concluding remarks: Trial 4**

This summary section concludes the major findings with a statement heading and subsequent bullet pointed supplementary information: (A) firstly from the average effect of single amendments on the gravimetric water content, volume, VWC and VWCi change over time, (B) secondly from the comparison of different amendment ratios of compost and then WTR, and (C) lastly major findings from the comparison of co-amendments against the control and the single amendments. All values are for an  $n = 12$  and where  $p < 0.01$ .

*(A) The single amendment of compost improves all parameters better than the single amendment using WTR.*

##### **1. GWC of single amendments at 30%**

- Single 30% compost results in a GWC that is 20% higher than single amendments of WTR or 34.7% higher than soil.
- 30% single amendments of WTR reached 93% of their GWC maximum within 24 hours
- 30% single amendment of compost reached 84% of maximum GWC within 24 hours

- 30% WTR2<sub>d</sub> dried slower than any other 30% single amendment.
- There is no statistical difference between WTR2<sub>w</sub> and WTR2<sub>d</sub> (until 912 hours)
- Compost is statistically better than single amendments of WTR ( $p < 0.01$ )
- During the second drying, WTR amended samples experience a 6% less reduction in GWC compared to compost at 16%.

## 2. GWC of single amendments at 20%

- Compost again has the highest GWC, and is significantly different from WTR2<sub>d</sub> 20% amendment.
- 20% single WTR2<sub>w</sub> is not significantly different from unamended soil.
- 30% single amendment of WTR2<sub>d</sub> performs equally well to 20% amendment of compost ( $p < 0.01$ )
- WTR amendment experiences 2% less reduction in GWC than compost at 12%.

## 3. GWC of single amendments at 10%

- Only 10% WTR2<sub>w</sub> is significantly higher than soil (0.39) during the first wetting
- During the second phase of wetting all amendments significantly higher than soil
- 10% WTR2<sub>w</sub> has a 13% reduction in GWC, 10% WTR2<sub>d</sub> only has a 3% reduction in GWC, and 10% compost achieves a 3% increase in the GWC in the second phase of drying,

## 4. Volumetric changes were higher for all single amendments, and largest for compost amended samples

- Compost had the biggest volume change (44.1%) compared to unamended soil
- WTR2<sub>w</sub> achieved up to 12% increase in volume compared to unamended soil, but WTR2<sub>d</sub> restricted swelling in the first phase of wetting and only reached 7.3% better volume than soil at 30% amendment.
- VWC/VWC<sub>i</sub> was higher than unamended soil for all 30% amendments.
- No difference between WTR2<sub>w</sub> and WTR<sub>d</sub> during the first phases for VWC/VWC<sub>i</sub>, but WTR2<sub>d</sub> was statistically significantly higher than WTR2<sub>w</sub> during second phases.

*(B) The GWC response to amendment was greater with higher proportions of material (compost), however the relationship was not as linear for the use of WTR.*

1. Single compost at increasing proportion:

- 30% increases the GWC by 34.7% and 20% by 13.2% but no significance for 10% during the 1<sup>st</sup> wetting
- 10% amendment increased the GWC by 83.3% compared to unamended soil in the second wetting, 20% by 29.3% and 30% by 59.2% compared to the control soil.

2. Single WTR<sub>2d</sub> and WTR<sub>2w</sub> at increasing proportion:

- 30% WTR addition gives the highest GWC increase, and is 16.5% higher than unamended soil
- No trends are apparent between addition of wet or dry for GWC change, where all amendments statistically significantly improve the GWC (except AM8)
- WTR amendments make no difference to GWC on the first wetting at 10% amendment ratio.
- All single WTR amendments improve the volume increase during second wetting (except AM7), and reduced shrinkage during drying.
- Despite this, only 30% WTR amendment has a significantly higher VWC/VWC<sub>i</sub> than unamended soil

*(C) Co-amendments perform better than single amendments of WTR, and in some cases perform equally well to the single amendment of compost for GWC, volume and VWC/VWC<sub>i</sub>*

- 30% single amendment of compost has the highest overall increase in GWC (34.7%), however co-amendments perform up to 24.7% better than soil and 9% better than single amendments of WTR.
- The trend in the volume change of samples is the same as found for GWC, but no overall trends separating the performance of WTR<sub>2d</sub> and WTR<sub>2w</sub>
- At 30%, the co-amendment improves the VWC<sub>i</sub> during the second wetting by up to 31%, which is equal to the VWC<sub>i</sub> achieved by single compost at 30%
- At 20% amendment there is no statistical difference in the improvement between co-amendments and single compost for maximum GWC.

- 20% amendment does not increase the volume of samples for any amendment during the 1<sup>st</sup> wetting, however all co-amendments are significantly higher in volume during the 2<sup>nd</sup> wetting than soil and the single amendments of WTR.
- Only 10% WTR2<sub>w</sub> amendment improves the VWC/VWC<sub>i</sub> during the 1<sup>st</sup> wetting, however all amendments improve the VWC during the 2<sup>nd</sup> phase of wetting but the WTR2<sub>d</sub> co-amendment is no longer better than the single amendment.

To summarise the findings from Trial 3 and 4, single amendment using WTR improves the GWC (with two exceptions) and volume change of samples (using WTR2<sub>w</sub> at 30% amendment) which may occur due to high surface area of the WTR particles as a result of fine particle size, as the material is not known to swell upon wetting despite high organic content. WTR also reduces the extent of shrinkage when the samples are dried, suggesting that the soil architecture has been beneficially changed due to WTR addition and ‘cementing’ occurs during drying (Moodley & Hughes, 2006). The single amendment of compost improves the maximum water content of the samples to the greatest extent as the material itself swells, opening up more space for water to be held water to be held within the sample, however upon drying this space shrinks and the porosity is lost to a large extent. The addition of co-amendment yields the benefit of both amendments, where the water holding capacity is increased as a function of improved maximum gravimetric water content, and improved structural characteristics that reduce shrinkage upon drying to maintain pore space for further wetting cycles. Stabilizing pore space during drying will allow better infiltration and percolation rates through the soil for future events, which improves a soil’s ‘flood holding capacity’ according to the definition by Kerr *et al.* (2016). Although a single compost amendment results in the best improvement of water holding capacity at high amendment rates, as discussed in Chapter 2 excessive application of compost has detrimental effects on the soil geochemical environment and on the geotechnical properties of the soil. At realistic field application rates (<10%), the difference in the effect of compost is not as pronounced as in 30% application rates and the co-amendment therefore performs equally well.

## 5.2 Erosional resistance

### 5.2.1 Drop testing (Veitch method)

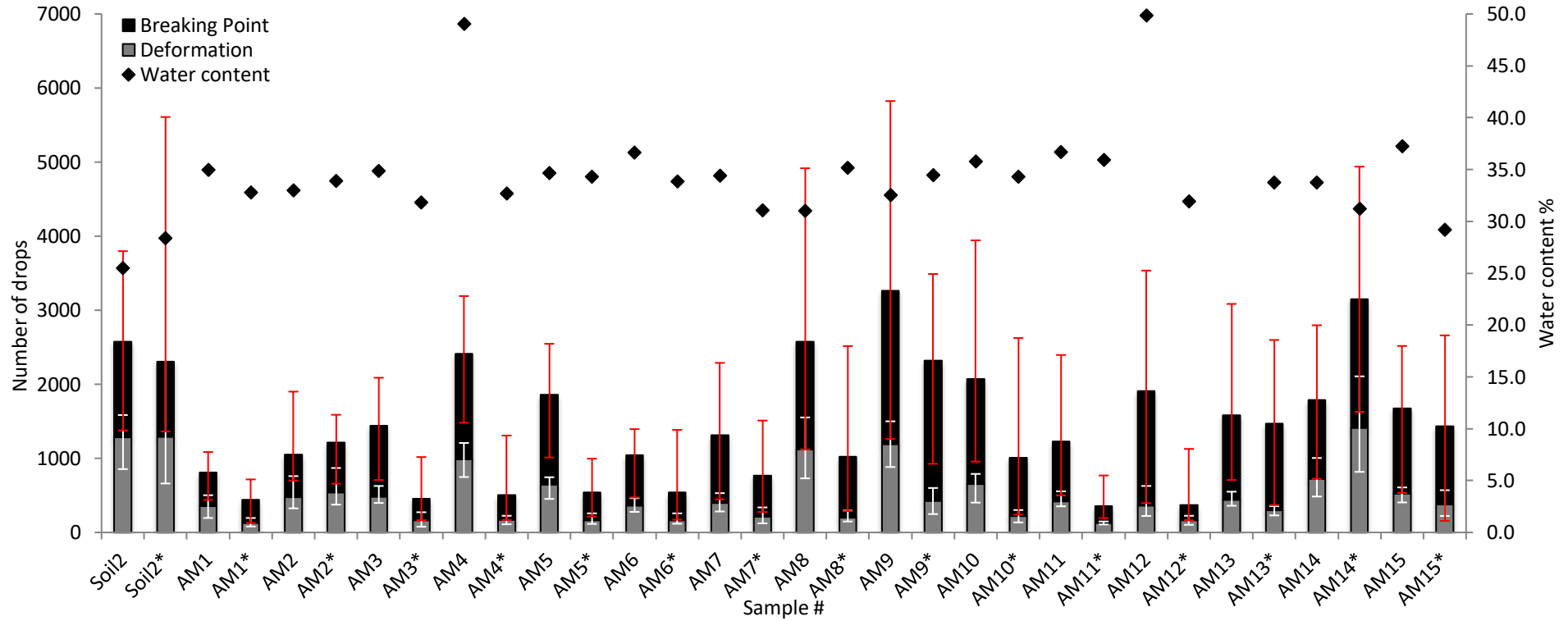


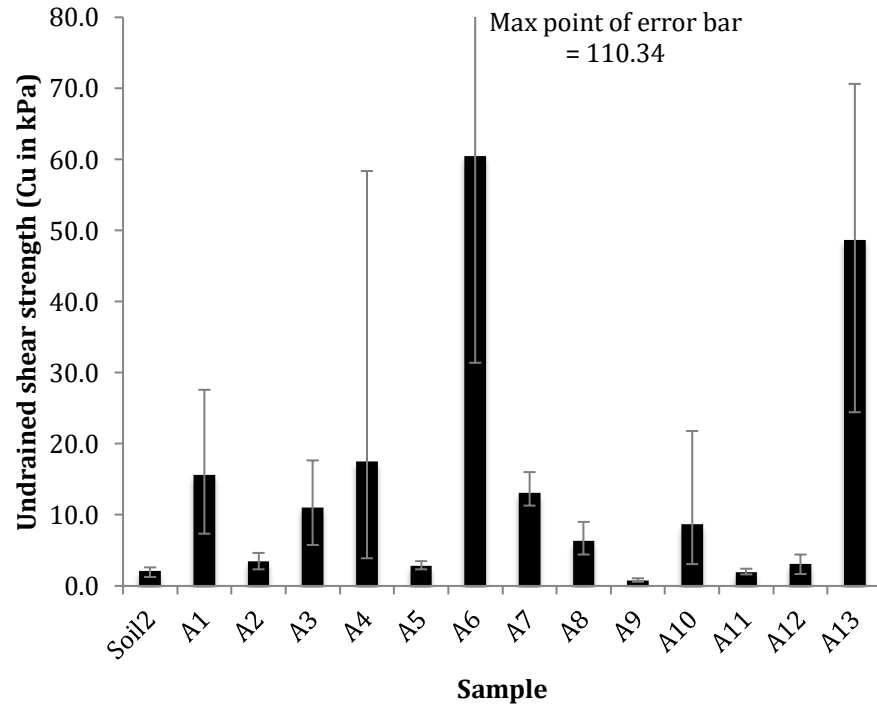
Figure 78: Average number of drops required for deformation and breaking for unamended soil and 15 amendments. Samples annotated with \* have been through one wetting and drying cycle prior to testing.  $n = 12$  and error bars present the maximum and minimum values in each data set. No statistical testing was performed on this data.

Figure 78 shows the average number of water drops until deformation and breakage for unamended soils and samples AM1-AM15. 'Deformation' occurred when the flat surface of the sample cracked, slumped or started to erode. 'Breaking' occurred when a piece of soil became detached from the soil. Each sample had one of two treatments, where one was tested 24 hours after production, and the other tested after one wetting and drying cycle as annotated with an \* on the X axis. No statistical analysis was performed on this data; however, the large error bars suggest that there may be no significance in any differences shown in the graph and trends are only apparent. All amended samples deformed with fewer drops than soil (1280 drops), however on average four amendments were able to sustain more than or as many drops as unamended soil before breakage, 10% co-amendment (WTR2<sub>d</sub>), 20% WTR2<sub>w</sub>, 20% co-amendment of WTR2<sub>d</sub> and WTR2<sub>w</sub> and 30% co-amendment with WTR2<sub>d</sub>. In general, samples that had been subjected to one wetting and drying cycle deformed and reached breaking point with fewer drops than the sample tested after production with the exception of 10% WTR2<sub>w</sub> and 30% co-amendment with WTR2<sub>d</sub> (AM2 and AM14)

The water contents of samples at the breaking point in Figure 78 (shown on the secondary Y axis) ranged between 29% and 37%, which indicates that the samples were fully saturated the point of breakage as these values were similar to the maximum water content achieved in Trial 4. It appears then, that samples broke when they reached full saturation, and those samples that took up water most quickly (as shown in the WHC trials in section 5.1 and subsequently in hydraulic conductivity testing in section 5.3.1), had the fewest drops required to breakdown. This presumably is a function of slaking whereby the rapid intake of water causes air bubbles to become trapped and pressurised which causes them to burst and breakdown aggregates. For samples that had a lower rate of wetting, this effect was less prominent and the samples remained intact for a longer period of wetting. For any robust conclusions to be drawn about the erodibility of samples after amendment, the Veitch method likely requires some further refinement and the number of samples needs to be greatly increased in an attempt to reduce the deviation between each test as a result of extreme heterogeneity in the material tested.

### 5.2.2 Fall cone testing

The following figures 79-81 show the undrained shear strength values obtained from fall cone testing on both samples from Trial 3 (conducted by Mansfield, 2015) and Trial 4.



<i>Sample</i>	<i>Soil</i>	<i>Compost</i>	<i>WTR2<sub>d</sub></i>	<i>Silica</i>
<i>N= 8</i>	<i>2%</i>	<i>2%</i>	<i>%</i>	<i>%</i>
Soil 2	100			
A1	50		50	
A2	50			50
A3	50	25	25	
A4	50	25		25
A5	60	40		
A6	60		40	
A7	60			40
A8	60	20	20	
A9	60	20		20
A10	70	30		
A11	70		30	
A12	70			30
A13	70	15	15	

Figure 79: Undrained shear strength ( $C_u$ ) for Trial 3 samples conducted according to BS1377: 1990 with reference table (right). Error bars denoted max and minimum values for each sample, where  $n = 5$ . (Left) Table with a summary of sample amendments.

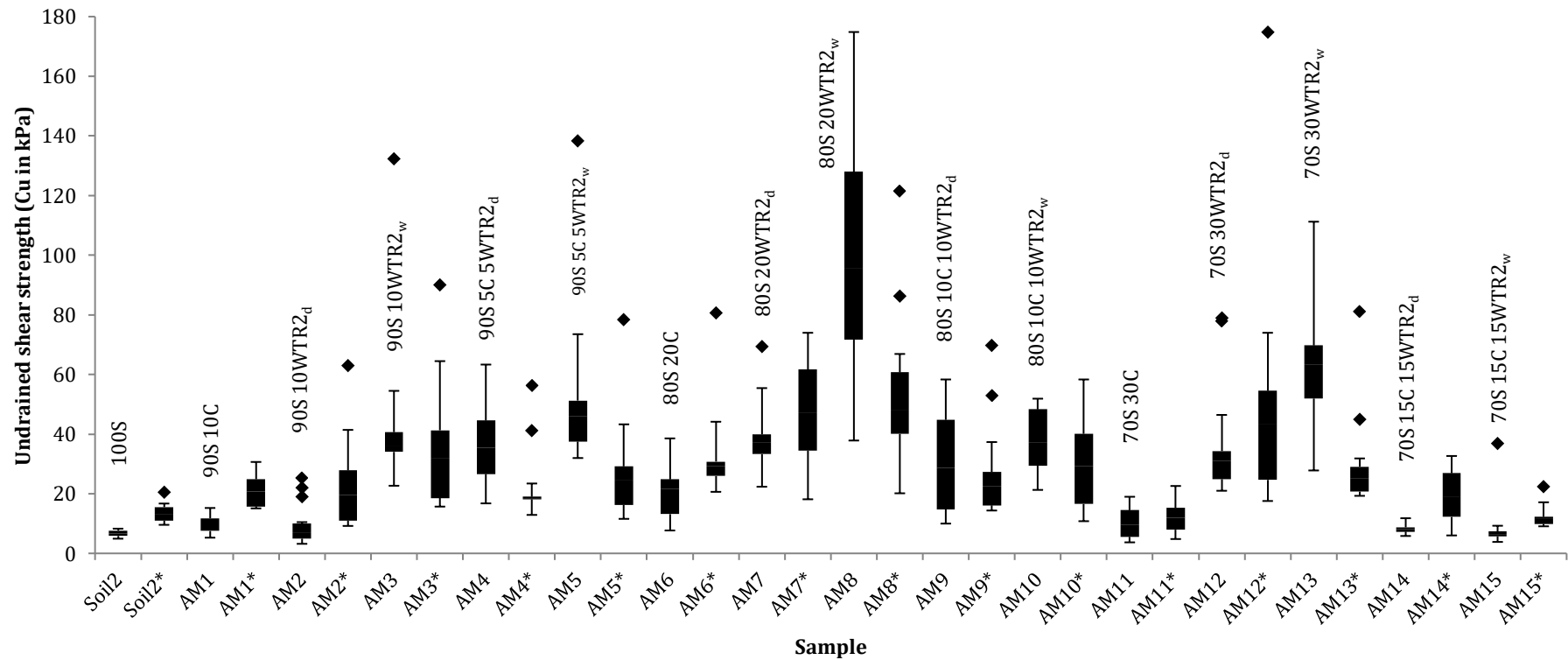


Figure 80: Undrained shear strength results from fall cone testing on samples from Trial 4 (BS1377) where  $n = 12$ . Samples annotated with \* have been fully saturated and dried to the same moisture content (0.16) as samples that were tested 24 hours after production. Black boxes are Q2-Q3 and whiskers on each box show the maximum and minimum values derived. Diamond markers indicate outliers in the data that cannot be omitted but would skew the data significantly.

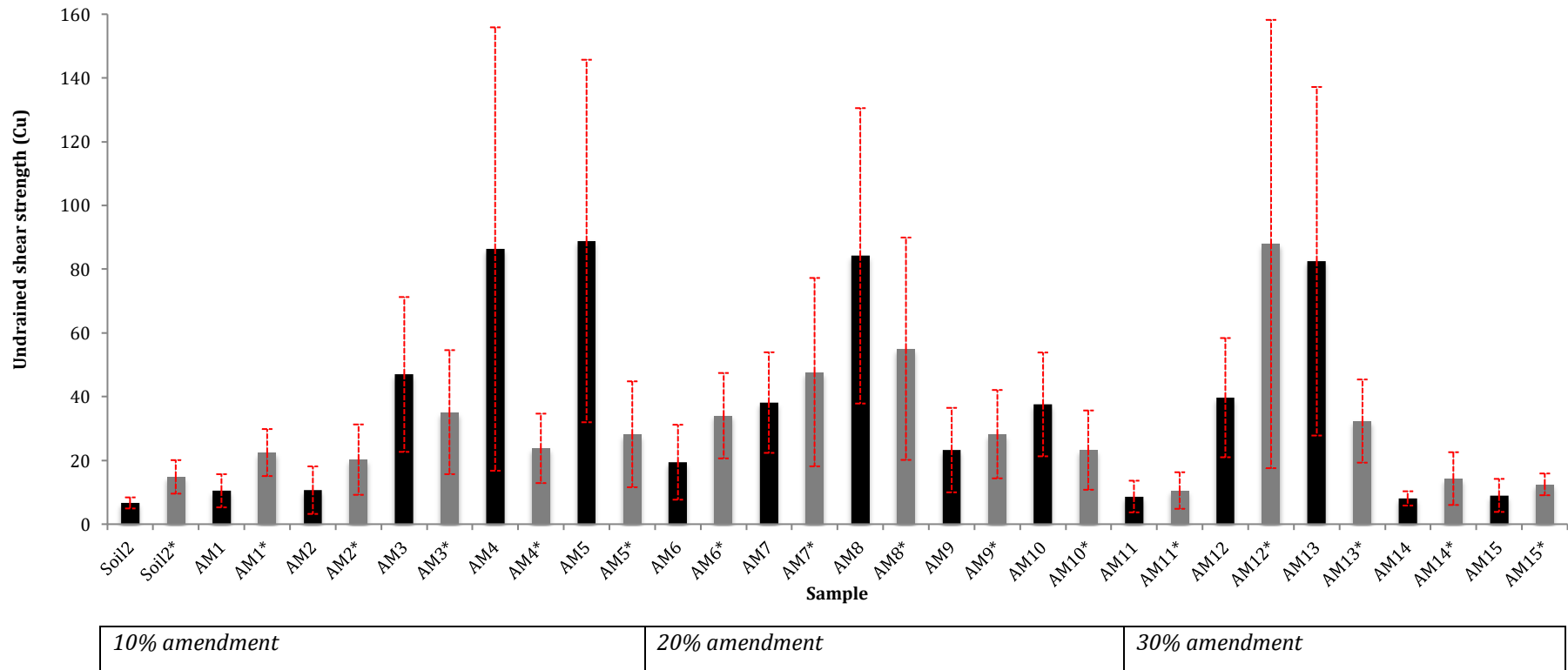


Figure 81: Average undrained shear strength values for Trial 4 samples from fall cone testing (BS1377). Red dashed lines indicate the error i.e. the minimum and maximum value obtained for the samples where  $n = 12$ . Samples annotated with \* have been fully saturated and dried to the same moisture content (0.16) as samples that were tested 24 hours after production. AM1, AM2, AM11 and AM15 are not significantly different from Soil2, and AM2\*, AM11\*, AM14\* and AM15\* are not significantly different from Soil2\*. The remaining amended samples have a significantly higher Cu (two tailed at alpha level of 0.05) than both Soil2 and Soil2\*.

Figure 79 presents results of fall cone testing using on samples from Trial 3 and shows that unamended has a low Cu (2.1) in comparison with the majority of amendments, where A2 (50% silica amendment), A5 (40% compost) A9 (40% co-amendment with silica), A11 (30% compost) and A12 (30% silica) have similarly low values of CU ranging from 0.9 to 3.5. There appears to be no trend between the addition of WTR2<sub>d</sub> in the undrained shear strength as A1 (50% WTR2<sub>d</sub>), A6 (40% WTR2<sub>d</sub>) and A11 (30% WTR2<sub>d</sub>) have values that do not correspond with the proportion of amendment. No trends are apparent between proportion of compost or silica either, however in general it appears that the addition of any material improves the undrained shear strength in comparison to unamended soil.

Figures 80 and 81 show the range of Cu (undrained shear strength) values obtained for fall cone testing on samples from Trial 4. Figure 80 shows the Q1-Q3 box plots; where whiskers show the remaining data, and diamond markers show apparent outliers. It is clear that there is a great degree of heterogeneity in the values of undrained shear strength within each set of 12 replicates per sample, as apparent in erosional testing. This may be accounted for by the heterogeneity of the sample surface, where in one test the fall cone may have struck soil and in another it strikes a piece of dried WTR (WTR2<sub>d</sub>) or a piece of compost, and the total effect of the amendment is not tested rather the direct shear properties of component itself. In general, it appears in Figure 80 that the single amendments using WTR2<sub>w</sub> had the highest shear strength

As shown in Figure 81, 10% co-amendments AM4 and AM5, AM8 (20% single WTR2<sub>w</sub> amendment), AM12\* and AM13 (30% single WTR2<sub>d</sub> and WTR2<sub>w</sub> amendments respectively) all have similarly high mean values of ~80 Cu, compared to unamended soil of 6.66 or 14.83\*. Mann Whitney U statistical testing was used to test for significant difference between samples, and despite large range of values (error) for each amendment, the majority of amendments are significantly better than unamended soil for undrained shear strength ( $p < 0.05$ ) The exceptions are AM1 (90S 10C), AM2 (90S 10WTR2<sub>d</sub>), AM11 (70S 30C) and AM15 (70S 15C 15WTR2<sub>w</sub>). There doesn't appear to be a single component that is responsible for insignificance in the difference between the amended sample and unamended soil. In addition, there does not appear to be an obvious difference between the shear

strength of samples tested just after production and the samples tested after one wetting and drying (annotate with \*), where Soil2\*, AM1\*, AM2\*, AM9\*, AM11\*, AM12\*, AM14\* and AM15\* have higher shear strength values than the same samples tested after production, and the remaining \*amendments have lower values for Cu than their respective samples.

Soil2 = 6.66					
Single amendment of WTR		Single amendment of C		Co-amendment	
Sample	Cu average	Sample	Cu average	Sample	Cu average
AM2	10.67	AM1	10.47	AM4	86.33
AM3	46.98	AM6	19.44	AM5	88.84
AM7	38.14	AM11	8.67	AM9	23.24
AM8	84.19			AM10	37.58
AM12	39.70			AM14	8.08
AM13	32.35			AM15	9.03

Table 26: Average Cu values for soils categorised into their amendment properties. Red titles indicate no significance between the value and unamended soil.

Soil2* = 14.83					
Single amendment of WTR		Single amendment of C		Co-amendment	
Sample	Cu average	Sample	Cu average	Sample	Cu average
AM2*	20.25	AM1*	22.47	AM4*	23.79
AM3*	35.14	AM6*	34.03	AM5*	28.20
AM7*	47.69	AM11*	10.55	AM9*	28.24
AM8*	55.04			AM10*	23.23
AM12*	87.90			AM14*	14.30
AM13*	32.35			AM15*	12.49

AM1 = 90S 10C, AM2 = 90S 10WTR<sub>2d</sub>, AM3 = 90S 10WTR<sub>2w</sub>, AM4 = 90S 5C 5WTR<sub>2d</sub>, AM5 = 90S 5C 5WTR<sub>2w</sub>, AM6 = 80S 20C, AM7 = 80S 20WTR<sub>2d</sub>, AM8 = 80S 20WTR<sub>2w</sub>, AM9 = 80S 10C 10WTR<sub>2d</sub>, AM10 = 80S 10C 10WTR<sub>2w</sub>, AM11 = 70S 30C, AM12 = 70S 30WTR<sub>2d</sub>, AM13 = 70S 30WTR<sub>2w</sub>, AM14 = 70S 15C 15WTR<sub>2d</sub>, AM15 = 70S 15C 15WTR<sub>2w</sub>

Table 27: Average Cu values for soils after one wetting and drying cycle (annotated with \*) categorised into their amendment properties. Red titles indicate no significance between the value and unamended soil.

Table 26 shows that the greater the proportion of compost, the lower the shear strength (which links with literature in Chapter 2) and in general the amendment using WTR2 increases the Cu of soil to the largest degree against unamended soil. Importantly the use of co-amendment at a rate of 10% (AM4 and AM5) increases the Cu more than higher application rates of co-amendment, presumably because of

the increase in compost, which reduces the shear strength at a greater rate than WTR increases it. The application of 10% or 20% co-amendments have higher mean  $C_u$  values than any single amendment of compost, however 30% co-amendments have similar values to single compost 30% amendment (8.08, 9.03 and 8.67 respectively). The amendment using WTR2<sub>w</sub> increases the shear strength compared to unamended soil to a higher degree than the addition of WTR2<sub>d</sub>, which can be attributed to the cementing properties of the wet WTR outlined by Moodley & Hughes. Table 27 indicates the same differences shown in Table 26, for samples subjected to a wetting and drying cycle (annotated with \*). For these samples, all single amendments of WTR2 have higher values than any single amendment of compost or a co-amendment at the same proportion of amendment.

### **5.2.3 Concluding remarks**

The erosional resistance testing in section 5.2.1 did not provide conclusive results due to large deviation in the values obtained via the Veitch method. All amendments required fewer drops to deform than unamended soil which suggests that aggregate stability is lower for amended samples. This is likely due to the erosional process associated with a rapid intake of water, reflected by the high-water content of samples at point of complete failure (breakage) as a result of better water holding capacity and infiltration rate. Further conclusions are difficult to make owing to the rudimentary nature of the Veitch method; therefore, refinements are required in the methodology and a greater number of samples are needed to improve the accuracy and reliability of results.

Fall cone testing in section 5.2.2 showed that in Trial 3 the 40% single amendment of WTR2<sub>d</sub> improved the  $C_u$  value by a factor of 28 (60.4) compared to unamended soil (2.1), although the 50% and 30% single amendments do not achieve such high improvement rates, their averages were 15.6 and 1.9 respectively. Samples that contained compost or silica as single or co-amendment had low undrained shear strength. Fall cone testing for Trail 4 samples showed that the amendment with compost at any ratio provided the lowest undrained shear strength, and were not significantly different from unamended soil. There does not appear to be a trend between the ratio of amendment and the improvement in shear strength, nor between the type of WTR added.

### **5.3 Triaxial testing**

Core samples synthesised during Experiment 4 were tested in a triaxial cell to measure their hydraulic conductivity and shear strength. All amendments were tested for their hydraulic conductivity, however only the largest amendment proportions (AM11-AM15) were tested for shear strength due to the long-term nature of the testing procedure.

#### **5.3.1 Hydraulic conductivity**

The saturated hydraulic conductivity of a soil is a function of many parameters including soil texture, clay content, organic matter, and soil aggregation (Hillel, 2008; Moutier *et al.*, 2000), and is widely used as an indicator of soil quality (Reynolds *et al.*, 2000; Lim *et al.*, 2016) as it reflects good soil structure. Figure 82 shows the variation in hydraulic conductivity measured on specimens of the unamended soil and soils with amendments, at three different confining stress levels (25 kPa, 50 kPa and 100 kPa), using a hydraulic gradient of 15 kPa. As expected, the hydraulic conductivities are lower at greater stress levels, as increasing the confining stress causes a reduction in porosity, making water flow more difficult. Silt and medium plasticity clays typically have a permeability of around  $10^{-10}$  m/s, however as shown in Figure 82 unamended soil (Soil2) has a high permeability of  $1.26 - 6.73 \times 10^{-7}$  m/s depending on confining pressure (which equates to 0.006-0.34 ml/hr, as shown in Table 28).

The 30% single amendment of WTR2<sub>d</sub> (AM12) has the highest hydraulic conductivity (up to  $1.77 \times 10^{-5}$  m/s at 25 kPa), followed by single amendments of 30% WTR2<sub>w</sub>, 20% WTR2<sub>d</sub> and WTR2<sub>w</sub> (reaching maximum conductivities of 7.22, 3.81 and  $5.32 \times 10^{-6}$  m/s at 25 kPa respectively). WTR2<sub>d</sub> is able to increase the hydraulic conductivity of unamended soil by a factor of 26, however the single amendment of WTR2<sub>w</sub> at the same rate increases the hydraulic conductivity by a factor of 11, compared to soil alone at  $6.72 \times 10^{-7}$  m/s. This difference in performance may be due to particle size differences, where WTR2<sub>w</sub> adds fines < 75  $\mu$ m, whereas WTR2<sub>d</sub> has a particle size distribution that includes larger grains of up to 3 mm; the addition of coarse material increases the hydraulic conductivity (Boadu, 2000).

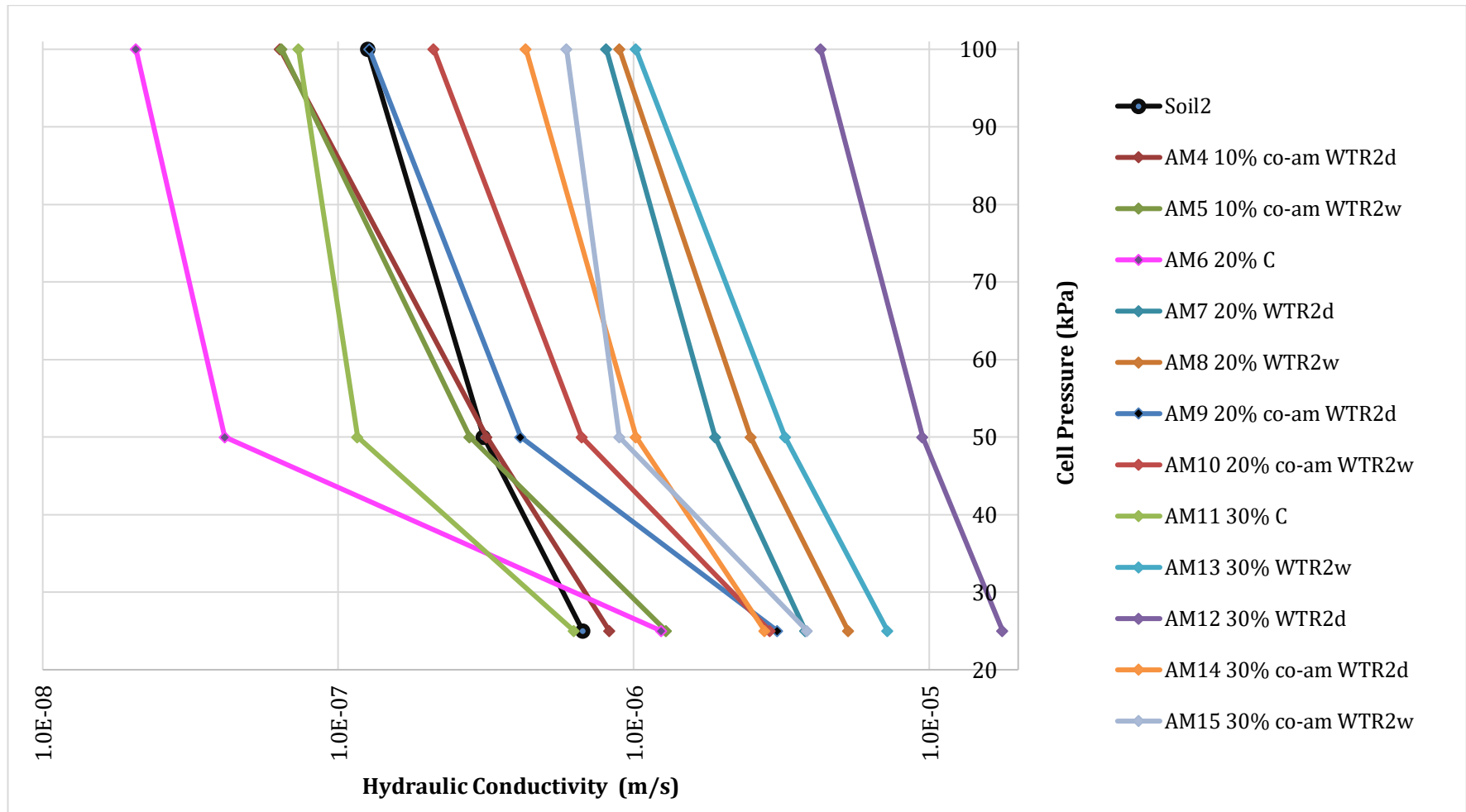


Figure 82: Hydraulic conductivity of samples conducted in a triaxial cell at pressures of 25, 50 and 100 kPa, on a log scale. Samples AM1, AM2 and AM3 were only tested at 25 kPa and do not feature on the graph; their  $k$  values were  $1 \times E-06$ ,  $6.48 \times E-07$  and  $4.06 \times E-06$  m/s respectively.  $n = 1$  for all samples. Tests on samples AM4, AM5, AM6, AM9 and AM10 were conducted for all cell pressures on the same specimen, all other samples used one specimen for each cell pressure as the sample was sheared to end the test.

The 20% and 30% single amendments of compost have the lowest overall hydraulic conductivity at the highest pressures (2.06 and  $7.33 \times 10^{-8}$  m/s or 0.6 and 0.32 ml/hour, respectively) compared to soil. However, at 25 kPa there is negligible difference between AM11 (30% compost) and soil, and the rate of water movement is twice as fast for AM6 (20% compost) compared to soil alone. This suggests that the addition of organic matter hinders the movement of water through a sample at high pressures due to increased tortuosity and increased compressibility of the sample (Lim *et al.*, 2016), but provides preferential flow path ways shown by higher hydraulic conductivity at low pressure. The 10% co-amendment of WTR<sub>2d/w</sub> (AM4 and AM5 respectively) perform marginally better than the unamended soil reaching maximums of  $8.27 \times 10^{-7}$  and  $1.29 \times 10^{-6}$  m/s respectively. With greater proportions of co-amendment, the hydraulic conductivity increases, where the 20% co-amendment (AM9 and AM10) perform better, i.e. have a higher hydraulic conductivity than the soil, by a factor of 5, and a 30% co-amendment by a factor of 6 compared to soil alone. With the exception of AM12, WTR added in the wet form gives a higher hydraulic conductivity than the addition of dried WTR, although it is unknown if the difference is of any statistical significance.

Table 28 presents the maximum water contents recorded in the triaxial cell at 100 kPa compared to the maximum water contents recorded in Trial 4. For the former, the water content is measured at the end of the triaxial test after the soil has undergone consolidation, and as such it is both compressed and restricted from swelling to the same degree as Trial 4 due to uniform external pressure, which in effect reduces the porosity. Therefore, as shown the maximum water contents are higher for samples in Trial 4 as these were allowed to swell without restriction. In general, samples with the highest proportion of amendments, and therefore the highest surface area (contributed by WTR, see chapter 6 for details) or the highest available pore space (contributed by compost) in comparison to volume of soil, had the highest water content. When comparing amendments at the same ratio, the single WTR and co-amendments have similarly high maximum water content to single compost amendments, which suggests that WTR is able to improve the hydraulic conductivity to the same extent if the swelling factor is removed. The reason for 10% amendments having a higher maximum water content than the 20% amendments

in the triaxial cell is unknown, although as discussed previously in the analysis of Trial 4 data, the 20% amendments did not perform as expected.

Sample	Max water content at 100 kPa	Max water content Trial 4
Soil2 (100S)	26.36	36.3
AM1 (90S 10C)	26.38	35.9
AM2 (90S 10WTR2 <sub>d</sub> )	26.09	35.7
AM3 (90S 10WTR2 <sub>w</sub> )	27.28	39
AM4 (90S 5C 5WTR2 <sub>d</sub> )	26.39	37.2
AM5 (90S 5C 5WTR2 <sub>w</sub> )	24	39
AM6 (80S 20C)	25.07	42.2
AM7 (80S 20WTR2 <sub>d</sub> )	23.78	38.2
AM8 (80S 20WTR2 <sub>w</sub> )	24.63	36
AM9 (80S 10C 10WTR2 <sub>d</sub> )	24.25	41.2
AM10 (80S 10C 10WTR2 <sub>w</sub> )	24.04	42.1
AM11 (70S 30C)	32.45	50.1
AM12 (70S 30WTR2 <sub>d</sub> )	31.45	42.3
AM13 (70S 30WTR2 <sub>w</sub> )	32.95	41.1
AM14 (70S 15C 15WTR2 <sub>d</sub> )	31.92	46.4
AM15 (70S 15C 15 WTR2 <sub>w</sub> )	33.5	46.5

*Table 28: Values of maximum water content in obtained from the triaxial cell apparatus, compared to the maximum water content achieved by samples in Trial 4.*

In summary, the hydraulic conductivity is increased with the addition of WTR by up to a factor of 28, where the use of WTR2<sub>d</sub> performs better than WTR2<sub>w</sub>, presumably as a function of the addition of coarse particles. Compost in general reduces the hydraulic conductivity of samples due to the compressibility of the material which reduces the porosity of compost amended samples as they are placed under stress in the triaxial testing equipment.

### 5.3.2 Shear strength

<i>Sample</i>	<i>Composition</i>	<i>25 kPa</i>	<i>50 kPa</i>	<i>100 kPa</i>	<i>Angle of friction</i>
Soil2	100S	69 kPa	123 kPa	<b>179 kPa</b>	33.7°
AM11	70S 30C	84 kPa	105 kPa	<b>165 kPa</b>	36.6°
AM12	70S 30WTR2 <sub>d</sub>	115 kPa	200 kPa	<b>174 kPa</b>	33.0°
AM13	70S 30WTR2 <sub>w</sub>	158 kPa	113 kPa	<b>174 kPa</b>	33.2°
AM14	70S 15C 15WTR2 <sub>d</sub>	106 kPa	53 kPa	<b>252 kPa</b>	37.3°
AM15	70S 15C 15WTR2 <sub>w</sub>	78 kPa	129 kPa	<b>249 kPa</b>	37.6°

*Table 29: Summary of triaxial cell data for unamended soil (Soil2) and five 30% amendments. Bold values indicate the highest shear stress value, and red values indicate erroneous values.*

From triaxial data produced in the Durham University laboratory by a skilled technician, stress paths and stress/strain graphs have been derived for Soil2 (unamended soil) and samples AM11- AM15. The three important parameters that we can analyse from triaxial testing are; the shear strength of a sample (shown by stress/strain paths), the tendency of a sample to dilate or contract, and the frictional resistance or angle of friction (both shown by stress paths). As shown in Table 29, unamended soil had a maximum shear strength of 179 kPa, which classifies the soil as ‘stiff’ and is typical for a sandy loam, and an angle of friction of 33.7° (where the maximum values obtained by sands are 40° and soft clays would be expected to have an angle of friction between 20° and 25°, Craig, 2004).

#### **5.3.2.1 Stress strain relationships**

Stress strain graphs show the relationship between increasing axial strain and the resultant deviator stress ( $q$ ) on the sample (the stress difference between the axial stress and the confining stress applied to the sample). Figure 83 provides an exemplary plot of this relationship for Soil2 at 25, 50 and 100 kPa. The slope of the stress ( $q$ ) versus axial strain plot indicates the stiffness of the soil. Samples having a soft response, would show greater strain as the stress ( $q$ ) increases, i.e. the slope of the curve would be shallower. The dashed line overlaid on the graph shows a typical stress strain curve for a dense coarse grained soil or an over consolidated fine

grained soil, where the peak indicates the point of maximum shear stress (the peak shear strength), after which the soil progresses to a lower 'critical state' where no further volume change or distortion occurs with increasing strain, as the soil flows as a frictional fluid and exhibits no cohesion (Schofield & Wroth, 1968). A loose coarse grained sand or normally consolidated fine grained soil will not demonstrate a peak but will reach the maximum shear stress at the ultimate (critical) state, as demonstrated by the data in Figure 83. At the Critical State there is a unique relationship between  $q$  (deviator stress in kPa),  $p'$  (mean effective stress) and the specific volume of the sample (volume of soil particles per unit volume). The majority of stress strain curves for the data presented subsequently display no distinct peak but instead plateau, suggesting that the shear strength is generally reached at the critical state (ultimate state).

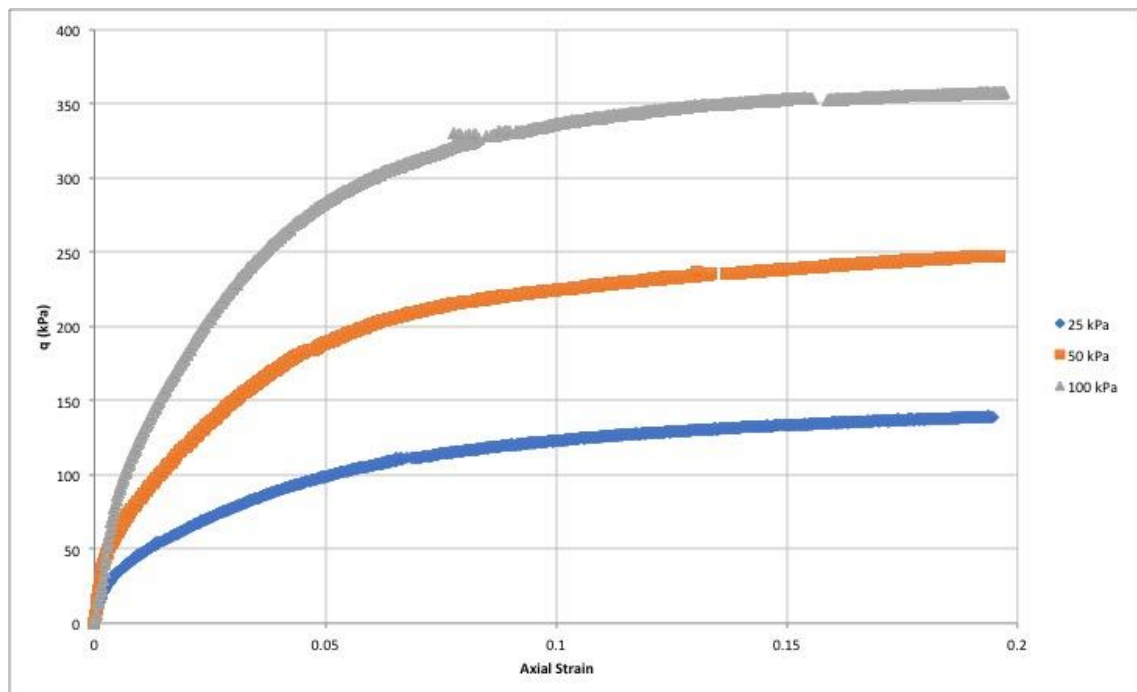


Figure 83: Stress strain curve for unamended soil at three cell pressures.

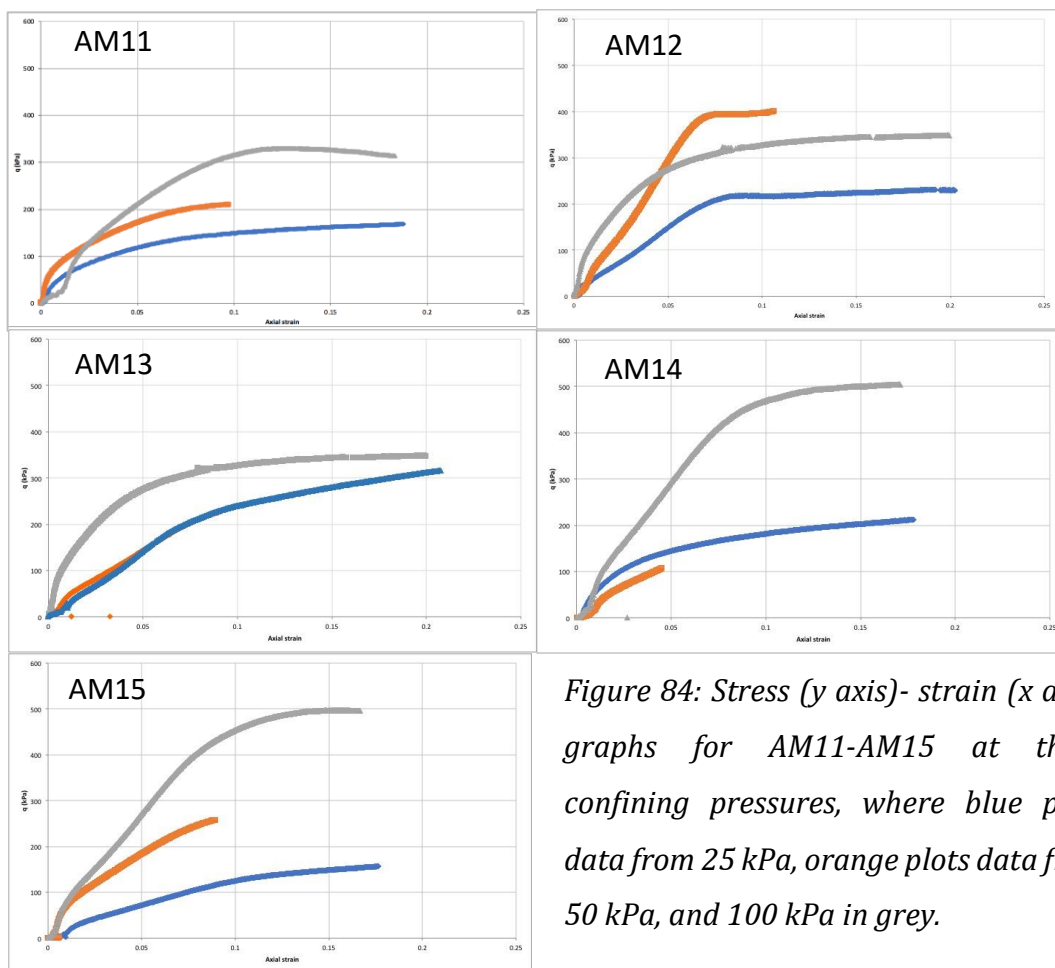


Figure 84: Stress (y axis)- strain (x axis) graphs for AM11-AM15 at three confining pressures, where blue plots data from 25 kPa, orange plots data from 50 kPa, and 100 kPa in grey.

All samples experienced their highest shear strength at 100 kPa and lowest shear strength at 25 kPa. Testing at 50 kPa produced erroneous results (shown in red in Table 29), which did not fall between the values of 25 and 100 kPa and therefore cannot be taken forward in this analysis. Figure 84 highlights differences in stress-strain behaviour between each amendment. It is clear from the comparison of orange data lines (50 kPa) with the 25 kPa (blue) and 100 kPa (grey) lines that these tests at 50 kPa were either incomplete (AM13 and AM15) and did not reach failure, taken to be at 20% axial strain (as can be seen for tests at 25 and 100 kPa where the stress-strain behaviour reaches a plateau by this value of strain) or had erroneous data as the values for 50 kPa should sit between those for 25 kPa and 100 kPa. The 50 kPa reading for AM11 almost reaches a plateau as it was only sheared to 15%. AM12 at 50 kPa appears to have higher shear strength than at 100 kPa, and AM14 at 50 kPa has a lower shear strength than the 25 kPa (although considering the trend of the line, should the test have been completed to failure, the value may have been

satisfactory). The subsequent figures compare the stress strain relationship at cell pressures of 25 kPa, 50 kPa and 100 kPa.

Figure 85 presents the 25 kPa tests, which indicates that unamended soil has the lowest shear strength (70 kPa). The reasons for these responses are discussed later. Figure 86 shows the stress strain relationship of amended soils compared to unamended soil at 50 kPa, however as mentioned previously these tests were incomplete or erroneous (with the exception of soil alone). AM11 (30% compost) and AM15 (30% co-amendment with WTR2<sub>w</sub>) are the only two data sets from which reliable conclusions can be drawn, as their values fall between those obtained for 25 and 100 kPa. Although the test has been cut short of sample failure, the trend line of 30% compost (AM11) suggests that it would not exceed the shear strength of soil at failure, where the maximum recorded value for shear strength was 105 kPa compared to unamended soil at 124 kPa. The projection for 30% co-amendment (AM15) suggests that the maximum shear strength would be higher than the 129 kPa maximum recorded. Figure 87 shows the stress strain of samples tested at 100 kPa. Unamended soil, shown in black is stiffer than any of the amended samples, shown by the steeper gradient in  $q$  (kPa) as the axial strain increases. Single amendments of compost, WTR2<sub>d</sub> and WTR2<sub>w</sub> do not show any improvements in shear strength compared to the unamended soil (165, 174 and 174 kPa respectively). However, both co-amendments have a higher shear strength than soil by a factor of 1.4, where soil reaches a maximum of 179 and co-amendments AM14 and AM15 reach 252 kPa and 249 kPa respectively. The reasons for this will be discussed after an interpretation of the stress paths and critical state lines.

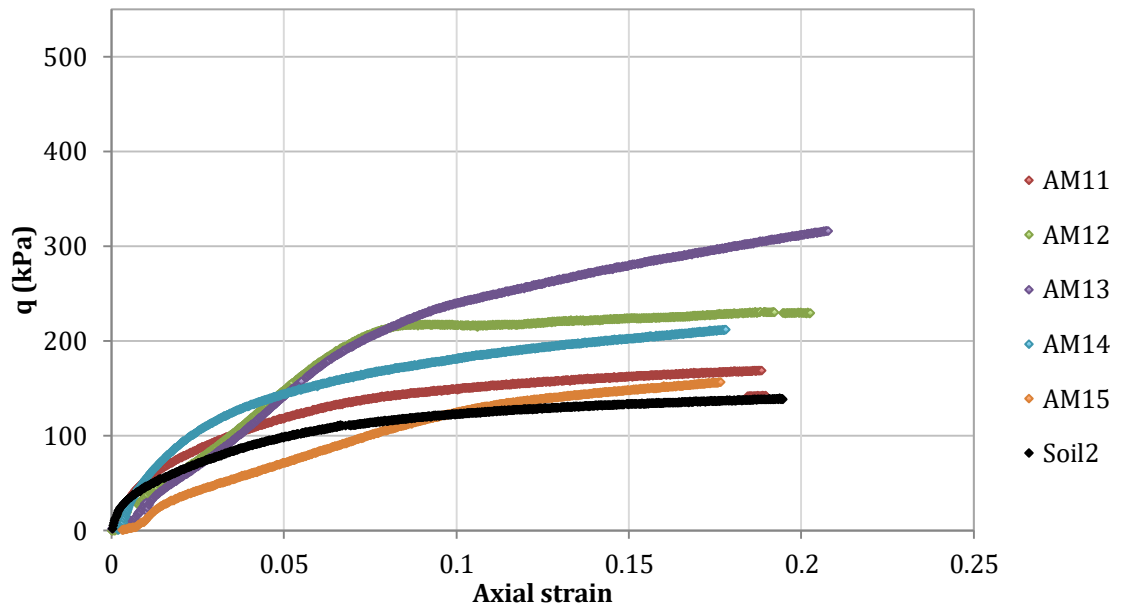


Figure 85: Stress strain curve for unamended soil and five amendments tested at 25 kPa where AM11 = 70S 30C, AM12 = 70S 30WTR<sub>2d</sub>, AM13 = 70S 30WTR<sub>2w</sub>, AM14 = 70S 15C 15WTR<sub>2d</sub>, AM15 = 70S 15C 15WTR<sub>w</sub>

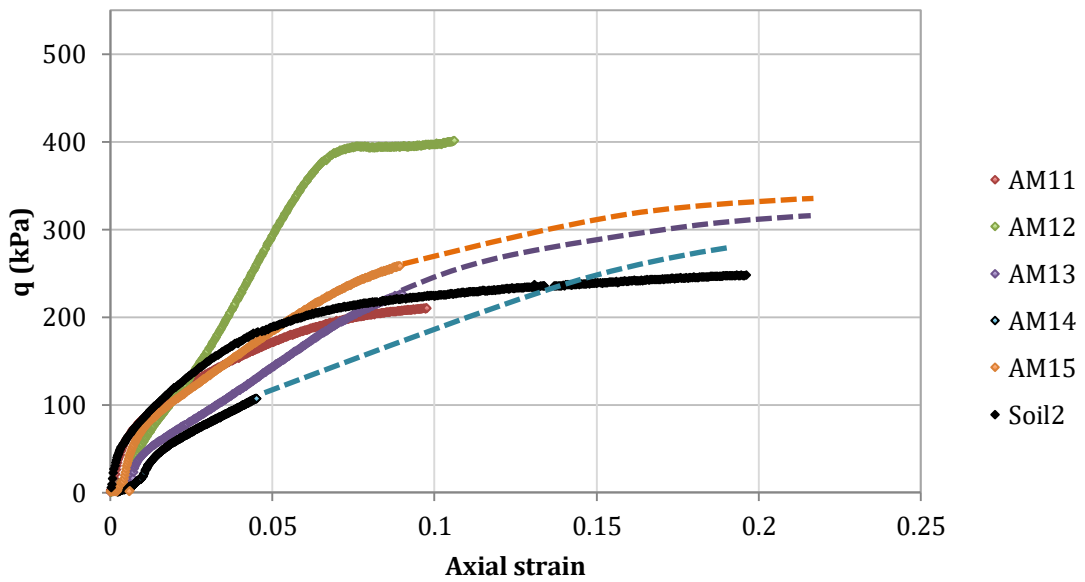


Figure 86: Stress strain curve for unamended soil and five amendments tested at 50 kPa where AM11 = 70S 30C, AM12 = 70S 30WTR<sub>2d</sub>, AM13 = 70S 30WTR<sub>2w</sub>, AM14 = 70S 15C 15WTR<sub>2d</sub>, AM15 = 70S 15C 15WTR<sub>w</sub>

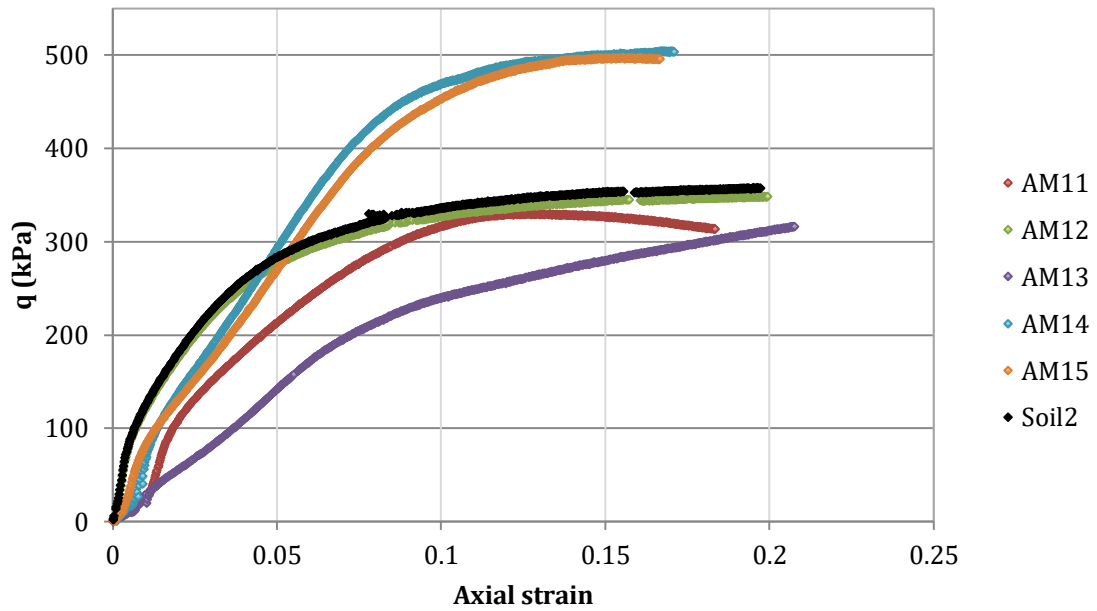


Figure 87: Stress strain curve for unamended soil and five amendments tested at 100 kPa where AM11 = 70S 30C, AM12 = 70S 30WTR<sub>2d</sub>, AM13 = 70S 30WTR<sub>2w</sub>, AM14 = 70S 15C 15WTR<sub>2d</sub>, AM15 = 70S 15C 15WTR<sub>w</sub>

### 5.3.2.2 Stress paths

The answer to why there seems to be a synergistic relationship between compost and WTR when added to soil lies in the analysis of the stress path data, from which we can determine contractive or dilatant tendencies of a sample when sheared, and  $M$  (the slope of the critical state line) can be calculated using Equation 19, from which the angle of friction is obtained using Equation 20.

$$M = \frac{q}{p'}$$

Equation 19: Calculation  $M$ , the slope of the critical state line.

$$\phi_c' = \sin^{-1} \left( \frac{3M}{M + 6} \right)$$

Equation 20: Calculation to obtain the angle of friction from  $M$  (Craig, 2004 pg 123)

Stress paths are used to represent the states of stress in a sample during loading until the point of failure (the failure envelope), by plotting the deviator stress ( $q$ ) against the mean effective stress ( $p'$ ). As the soil is sheared, it will move through different states of stress towards the Critical State Line. Figures 88, 89 and 90 show

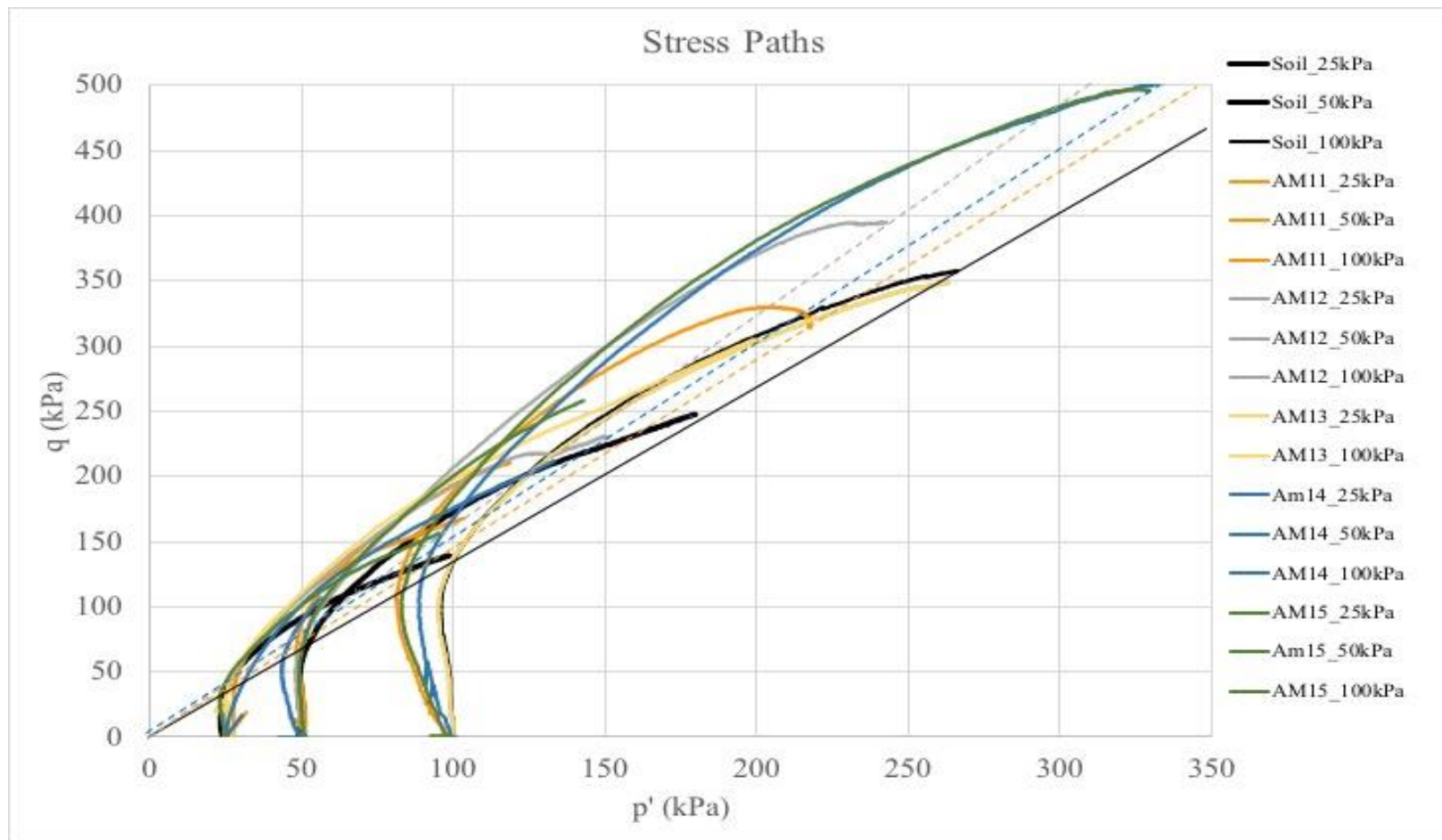


Figure 88: Stress paths and critical state lines for all samples tested in the triaxial cell. Data for AM13 at 50 kPa has not been included due to machine error giving spurious results.

the effective stress paths for unamended soil and five amendments. The strength of a soil is dependent on the slope of the failure envelope (angle of friction) and how far up the failure envelope a soil can travel (based on its tendency to dilate or contract). Therefore, the dilatant tendencies and angle of friction provide us information on why and how one soil has different shear strength to another as shown in the stress strain graphs above.

Critical State lines have been drawn through the endpoints of the stress paths in Figure 88. In all cases the lines pass through the origin (showing no cohesion) as would be expected for the Critical State. The Critical State stress ratio ( $M$ ) has been determined for each soil and amendment type (Eq. 19). Equation 20 has then been used to calculate the angle of friction for this state.

<b><i>Sample</i></b>	<b><i>Composition</i></b>	<b><i>Angle of friction</i></b>
Soil2	100S	33.7°
AM11	70S 30C	36.6°
AM12	70S 30WTR2 <sub>d</sub>	33°
AM13	70S 30WTR2 <sub>w</sub>	33.2°
AM14	70S 15C 15WTR2 <sub>d</sub>	37.3°
AM15	70S 15C 15WTR2 <sub>w</sub>	37.6°

*Table 30: Angle of friction values for unamended soil (Soil2) and five amendments, calculated from stress path graphs.*

Table 30 highlights the angle of friction achieved by each amendment, which has been determined from the angle of the critical state line. Unamended soil has a relatively high angle of friction of 33.7°. The single addition of WTR does not improve the frictional response of the soil as AM12 and AM13 have similar angles of friction (33.0 and 33.2°). However, all samples with compost amendment showed a significant increase in angle of friction. Single 30% compost amendment (AM11) has a higher angle of friction than unamended soil (33.7°) at 36.6°. The angle of friction of both the co-amended samples (AM14 and AM15) are also considerably higher (37.3° and 37.6°) than unamended soil. Therefore, it is clear that the compost produces a significant improvement in angle of friction (whether present at 15% or 30%), whereas the addition of WTR alone produces no benefit in terms of angle of

friction. We can now look at the stress paths to see how the pore water pressure responses influence the shape of the stress paths. Samples that have a more dilatant tendency can climb further up the failure envelope. Therefore, soils that have a low angle of friction can still show greater strength due to the generation of negative changes in pore water pressure.

In general, the curves in Figure 88 show an initial tendency for elastic compression, causing the generation of positive excess PWP in the samples. An excess of pore pressure indicates that pore water is exerting pressure on soil particles, which reduces the contact stress between the particles and makes the sample more susceptible to failure. If the sample continues to show a tendency to compress under load, the stress path will veer to the left and reach the failure envelope at a low stress. However, with continued shearing, the materials become dilatant in tendency, producing negative changes in PWP causing the curves to move to the right of the graph. As a general rule, the higher the dilatancy of a material, the higher the maximum shear stress that can be resisted by the material.

In Figure 89, at 25 kPa, all amended soils reach a higher stress before failure than unamended soil, which shows the lowest stress ratio (angle of friction). The WTR amended soils (AM12 and AM13) do show higher stress ratios than unamended soil at this stress level, implying there could be some curvature to the failure envelope (as this was not evident in the angles of friction calculated by considering the whole stress range). This could imply that particle crushing is taking place at the higher stress level (100 kPa) that restricts the development of frictional resistance.

AM12 and AM13 show the greatest dilative tendency at 25 kPa, therefore the pore water pressure drops and they climb the furthest up the critical state line and therefore have the highest shear strength at this pressure. The frictional response of single compost (AM11) and co-amendments (AM14 and AM15) appear to be very similar to each other and hence reach similar maximum shear strength (84.53, 106.17, and 78.35 kPa).

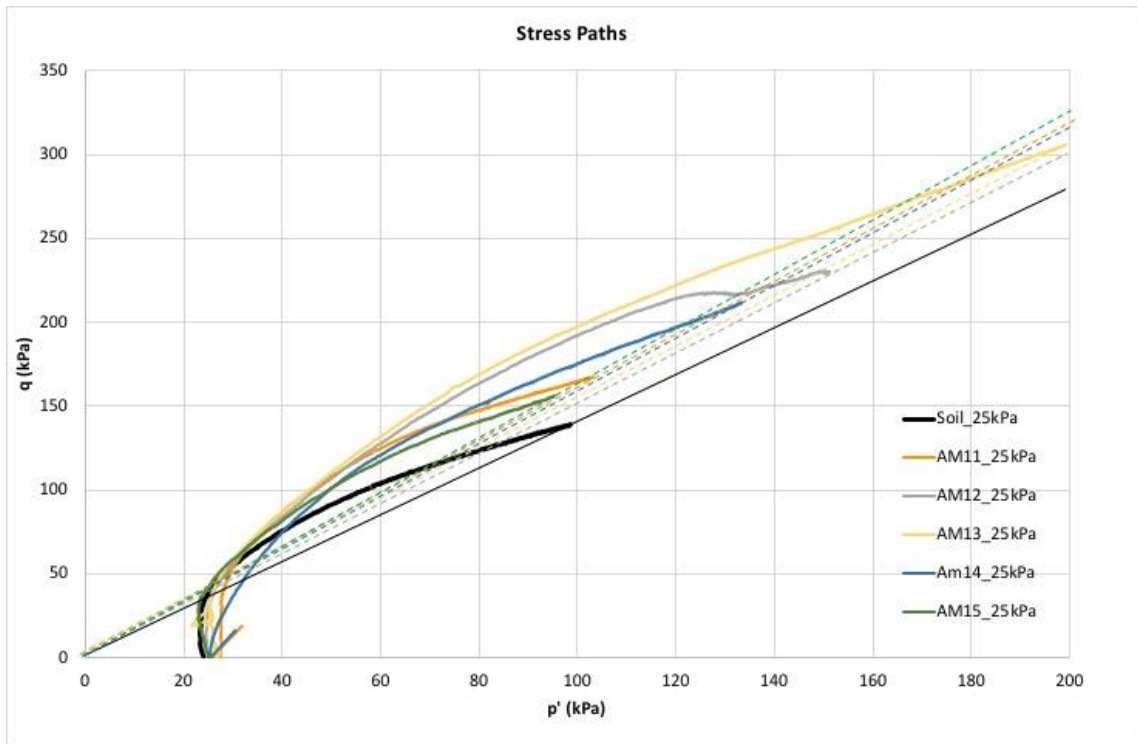


Figure 89: Stress paths for unamended soil, and five 30% amendments at a cell pressure of 25 kPa. Dashed lines indicate the critical state line, which coincides with the peak failure.

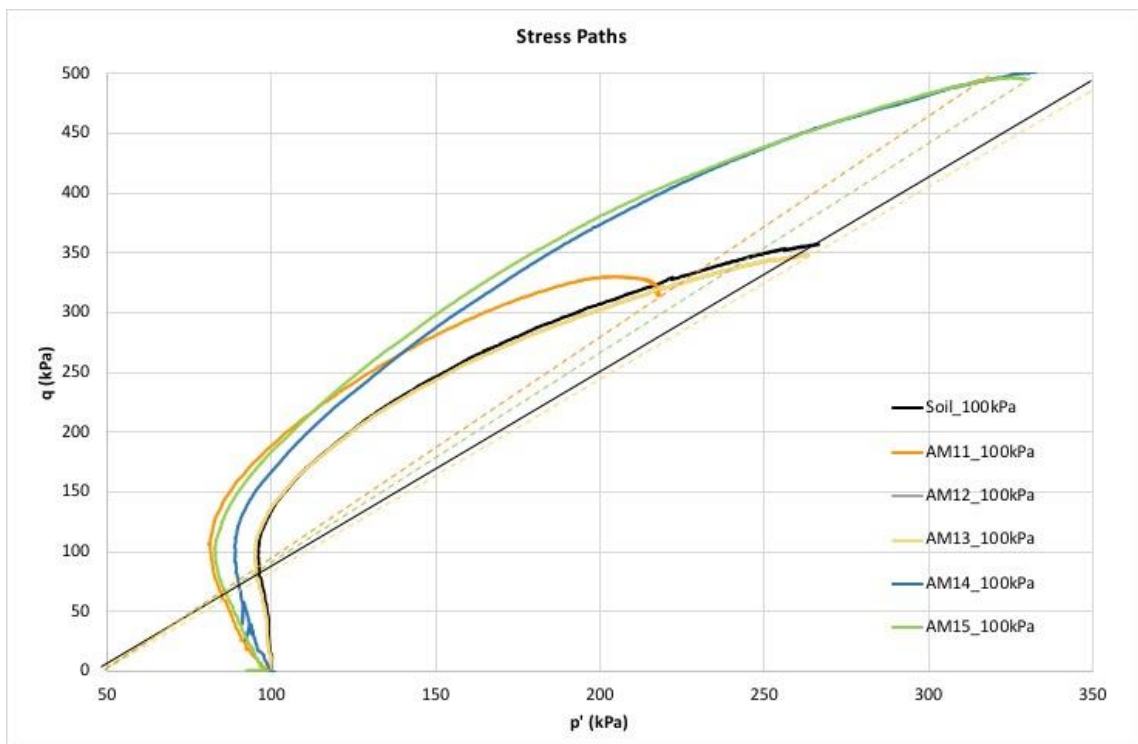


Figure 90: Stress paths for unamended soil and five 30% amendments & co-amendments at a cell pressure of 100 kPa. Dashed lines indicate the critical state line, which coincides with the peak failure. The AM12 and AM13 are almost identical and hence only AM13 may be observed.

It was difficult to analyse trends at 50 kPa as only unamended soil, AM11 (30% compost) and AM15 (30% co-amendment) appeared to have legitimate data, and these were stopped before reaching 20% strain, and therefore these data are not included here. Figure 90 shows the stress paths for unamended soil and amended samples at 100 kPa; it is clear that the responses of the samples are different to the lower cell pressure. The WTR amended soils, AM12 and AM13, show almost identical stress paths to unamended soil, so the benefits of improved dilation seen at 25kPa are not evident at this higher stress level. This might suggest that at the higher pressure the WTR becomes crushable and instead of particles being forced around one another, they are crushed and break under shearing. This would be consistent with the observation that the stress ratios are different at 25 kPa and 100 kPa.

At this higher stress level, the compost-amended sample (AM11) appears to be restricted from a tendency to dilate, due to the compressibility of compost. This means the PWP increases initially (shown on the graph as the green data line extends furthest to the left), after which the compost does dilates. However, overall the generation of negative pore water pressure is restricted, thus giving lower strength. The co-amendments (AM14 and AM15) also show increased contractive tendencies but with further shearing do generate more dilation and hence these result in the highest strengths.

From a geotechnical perspective, the results show that the addition of compost produces an increase in the angle of friction. However, the benefits of a higher failure envelope are cancelled out by the fact that compost amended soils generate more positive pore water pressure, so a single amendment of compost does not produce an overall increase in strength. The addition of WTR produces benefits in strength at low stress levels (25 kPa) due to increased dilatant tendencies, but these benefits are lost at the higher stress level (100 kPa). It is suggested that the higher stress level results in crushing of the WTR, thus restricting the dilatant tendency.

The co-amendment of compost and WTR provides the benefit of increased angle of friction while maintaining the dilatant tendency even at the higher stress level. It is possible that the compost provides some “cushioning” of the WTR, thus preventing

it from crushing (which limits the dilatant tendency in the WTR alone). Therefore, the combination of the higher angle of friction (due to the compost) supported by dilatant tendency (due to the WTR) gives the highest strengths.

### **5.3.2 Concluding remarks**

The shear strength, identified in the stress strain graphs, is dependent firstly on the slope of the failure envelope (angle of friction) and secondly how far up the failure envelope the soil can travel (as seen from the stress paths). The angle of friction represents the “intrinsic” strength of the soil, which is independent of the initial state (density). The dilative behaviour will depend on the initial state (higher density will mean more dilation) or stress level (higher stresses will suppress dilation). A soil that has a greater dilative tendency generates more negative pore water pressure and will climb further up the envelope to a higher strength. At low pressure, all amendments improve the shear strength and dilative tendency of the soil, however at high pressure the dilative tendency of the amendments is suppressed, and the angle of friction becomes more prevalent in how the soils respond. The co-amendment of compost and WTR provides the benefit of increased angle of friction while maintaining the dilatant tendency even at the higher stress level. Therefore, the combination of the higher angle of friction (due to the compost) supported by dilatant tendency (due to the WTR) gives the highest strengths.

## 6. X-ray Computed Tomography

### 6.1 Introduction to XRCT

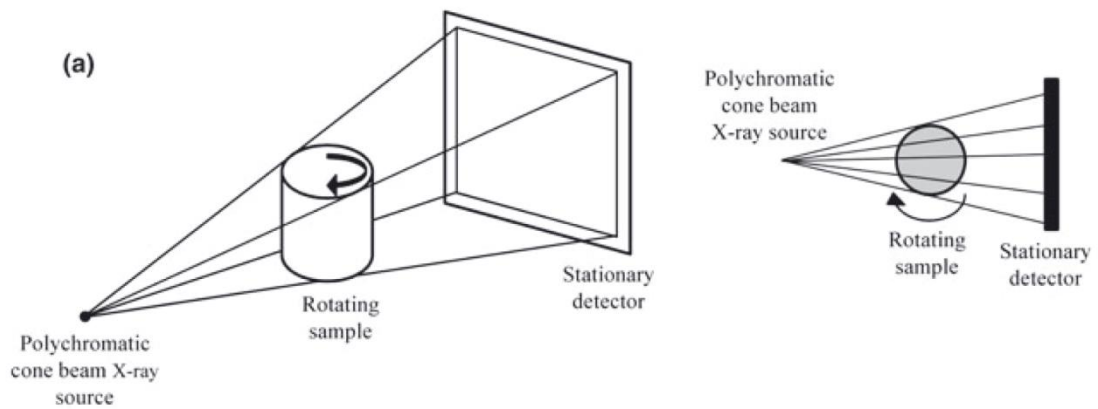


Figure 91: Schematic diagram of the XRCT system using cone-beam X-ray and a cylindrical core soil sample (Helliwell *et al.*, 2013, adapted from Wildenschild *et al.*, 2002)

X-ray computed tomography (XRCT) is the imaging of an object by acquiring images of an object through a series of x-ray radiograms after which tomographic reconstruction can occur in 3D (Figure 91). The key concept of this method is that imaging is achieved based on the different densities of materials; at the most basic level XRCT relies on the measurement of x-ray attenuation by detector after x-rays pass through an object, where the beam experiences progressive attenuation due to interaction with the atoms in the material (Taina *et al.*, 2008). Three components are required, the x-ray source (conventional x-ray tube or synchrotron light source), a sample stage that rotates the sample through 360°, and either an x-ray detector or a scintillator screen and charged couple device camera (Helliwell *et al.*, 2013; Smith *et al.*, 2014); for an overview of a detailed technical description of XRCT, readers are directed to Ketcham & Carlson (2001), Cnudde & Boone (2013), and Liu *et al.* (2016), all of whom give thorough summaries of the technology and applications. The greatest advantage of this technique is that it is a powerful non-destructive tool that allows an investigation of the internal structure of a material at high resolutions up to <5  $\mu\text{m}$  (Iassonov *et al.*, 2009; Peth, 2010). This allows comparisons of the same sample over time or for processes such as fluid movement (Rozenbaum, 2011; Van den Bulcke *et al.*, 2009), stress or strain (Zabler *et al.*, 2008) and allows time-lapse monitoring (Mokso *et*

*al.*, 2011). Technological improvements, particularly over the last decade, have meant this technology is widely used in engineering geology (Jacobs & Cnudde, 2009) and soil science (Helliwell *et al.*, 2013), although for soil research the visualisation of smaller particles and voids has been limited by spatial resolution. The information obtained from the scanners can be stored as individual JPEG or TIFF files for each slice, meaning that even the most basic software can be used to visualise the internal structure of an object. For quantitative analysis, 3D volumes can be analysed in specially developed software packages, ranging from open source and free programmes like Image J to high spec packages such as Avizo Fire and Volume Graphics (VG Studio Max) among many others. The caveat is that each package requires considerable training, they are generally expensive software packages and the data processing is subject to systematic and user based error.

Images from x-ray scanning are based on a grey-scale, where each pixel in the image is designated a shade of grey based on the attenuation coefficient recorded by the detector after x-rays pass through the material. Dense materials soak up more x-rays than less dense materials and therefore their attenuation coefficient is higher. Wildenschild *et al.* (2002) and Ketcham & Carlson (2001) provide comprehensive documents outlining the fundamentals of XRCT and readers are direct to these publications for further information. There are currently no standards for the application of XRCT and as with all research methods, XRCT is not without limitations. Image analysis is complicated by artefacts (system-based errors) that cause misinterpretation of data, such as image noise, partial volume effects, beam hardening, and ring artefacts. Limited correction of these known errors can be completed using filters and calibration of equipment. These issues will be briefly discussed in subsequent sections.

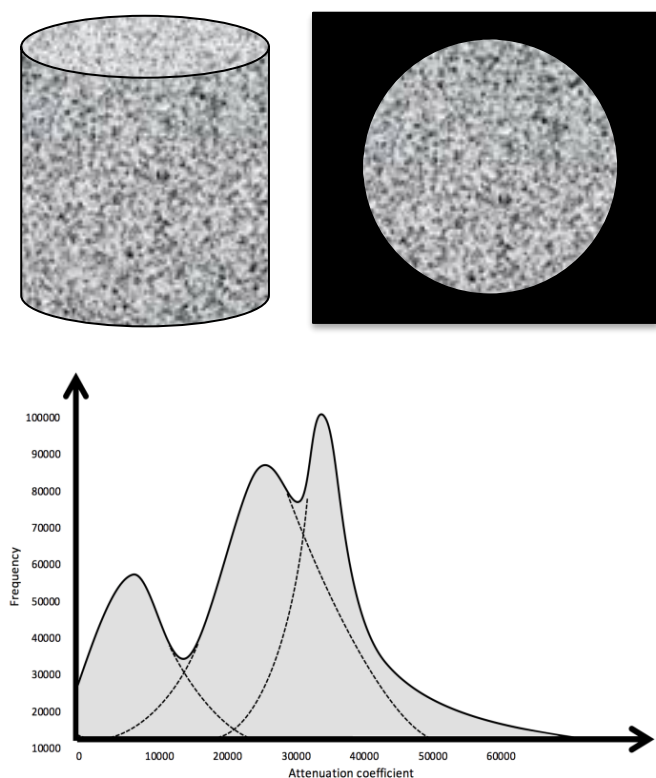


Figure 92: (top) Example image of a 3D sample and a single XRCT image 'slice' through the material. White represents a high attenuation coefficient ( $\mu$ ) of the material in that pixel, and black represents low attenuation coefficient; (bottom) an example histogram, giving the frequency of pixels with a given attenuation coefficient. Each bell curve corresponds to a single material.

XRCT data are commonly processed by thresholding, which is a segmentation technique that identifies two or more populations (of material) in the image, the simplest of which is global segmentation that separates materials based on the  $\mu$  values in the image histogram (Figure 92). Thresholding of an image therefore classifies all pixels below a given grey-value as one material, and all pixels with a grey-value above the selected threshold as a different material. Local thresholding techniques that take into account surrounding voxel intensities may provide the user with more accurate data (Al-Raoush & Willson, 2005), however there is still heated debate into the reliability of either types of thresholding owing to its fairly novel application to soil science. In general the thresholding of soil is particularly complicated as each voxel is likely to contain more than one material (due to low resolution scans and or infinitesimal material), resulting in their attenuation values being proportionally averaged, giving rise to the partial volume effect discussed subsequently (Clausnitzer & Hopmans, 2000; Ketcham & Carlson, 2001). There is currently a lag between technological improvement of XRCT and the methods with which to process, segment and refine 3D images (Iassonov *et al.*, 2009); the following sections will discuss the challenges faced with analysing data produced using XRCT.

### **6.1.1 Primary research into XRCT**

The first uses of XRCT after Hounsfield's development were for medical purposes for non-destructive visualisation of the human body (Gawler *et al.*, 1974; Lcdley *et al.* 1974; Paxton & Ambrose, 1974). Following this, the application of XRCT began in other research areas such as palaeontology (Conroy & Vannier, 1987), marine sciences (Boespflug *et al.*, 1995), industrial applications (Hopkins *et al.*, 1981) and geosciences (Vinegar & Wellington, 1987). The use of XRCT in soil science began with Petrovic *et al.* (1982) who used the technique to show a linear relationship between x-ray attenuation and bulk density of glass beads, from which they were able to determine high resolution soil bulk density to a precision of 19 mg/cm<sup>3</sup>. Hainsworth & Aylmore (1983) used XRCT to study the process of root-related water absorption, Crestana *et al.* (1986) used XRCT to observe changes in attenuation as a wetting front passed through the specimen, and Anderson *et al.* (1988) evaluated the potential for XRCT to reconstruct and analyse macro-pores in undisturbed soil cores. Although these scans were relatively coarse (~ 1 mm), this flurry of research was an important step in developing interest in this method for analysis. As Young *et al.* (2001), Helliwel *et al.* (2013) and Taina *et al.* (2008) describe, soil systems are fundamentally characterised by the complicated distribution of organic and mineral particles in their matrix. Therefore being able to place soil processes such as water movement in a hierarchical physical framework is critical for soil science. However due to great heterogeneity in soil characteristics, there have been significant limitations for experimental and theoretical based insights into a soil's spatial architecture; XRCT bridges this gap by moving away from high resolution thin sections and electron microscopy that are limited to 2D, into viewing soil structure in 3D which provides immediate valuable quantitative assessment rather than an extrapolation from 2D research (Anderson & Hopmans, 1994).

### **6.1.2 Recent applications and uses of XRCT**

Schulter *et al.* (2014), Helliwell *et al.* (2013), Jacobs & Cnudde (2009), Taina *et al.* (2009), Peth (2010) provide detailed reviews of the use of XRCT in soil, engineering and geology sciences. In the last 36 years XRCT has had drastic advances in the technological production of images, with increasing speed of acquisition and image quality and perhaps most importantly vastly improved

computer processing abilities, such that high resolution XRCT (or commonly called micro-CT or  $\mu$ CT, based on the very high resolution), capable of a resolution of  $<200 \mu\text{m}$  and use desktop based controlling & analysis systems are now commonplace (Baraka-Lokmane *et al.*, 2009; Cnudde & Boone, 2013). A number of studies produced between 1992 and 2007 have been based on data with voxel sizes of 10 – 100's of  $\mu\text{m}$ , which reflects on-going refinement over time (Taina *et al.*, 2008). Practical limitations of technology meant that modern micro-CT, with resolutions in the order of  $<10 \mu\text{m}$  was not achieved until 1995 (Lindquist *et al.*, 1996), and at this scale of observation of particles is limited to sand and silt sized fractions. Ketcham & Carlson (2001) provide a thorough review of the cutting edge imaging at the time, but today it still proves to be a critical text for practitioners (Wildenschild & Sheppard, 2013); Cnudde & Boone (2013) state that improvements in the technology are reaching the current physical limits of development based on cost, sample size and computer power; to image a  $1 \text{ cm}^3$  sample to  $2 \mu\text{m}$  requires almost 1TB of data to process (Young *et al.*, 2001).

To date there have been many published papers on the use of XRCT or  $\mu$ CT for soil-based applications which include;

- macro-pore characterisation ( $>75 \mu\text{m}$ ) in large soil cores (Elliot & Heck, 2007; Devereux *et al.*, 2012; Parvin *et al.*, 2017; Pierret *et al.*, 2002 and 1999; Schlüter *et al.*, 2011 and 2014; Wang *et al.*, 2012),
- micro-pore (5 – 75  $\mu\text{m}$ ) characterisation in samples  $< 3 \text{ mm}$  (Nunan *et al.*, 2006; Peth *et al.*, 2008),
- pore networks (Munkholm *et al.*, 2012; Peth *et al.*, 2015; Sleutel *et al.*, 2008; Sok *et al.*, 2010),
- aggregate characterisation (Feeney *et al.*, 2006; Garbout *et al.*, 2013; Helliwell *et al.*, 2013; Nunan *et al.*, 2006; Papadopoulos *et al.*, 2009; Peth *et al.*, 2008; Voltolini *et al.*, 2017),
- porosity and hydraulic conductivity (Anderson *et al.*, 2003; Clausnitzer & Hopmans 2000; Elliot *et al.*, 2010; Hamamoto *et al.*, 2016; Heijs *et al.*, 1995; Ketcham & Carlson, 2001; Mooney 2002; Peyton *et al.*, 1992, Tracy *et al.*, 2015),
- pore diameter (Anderson *et al.*, 1990),
- pore tortuosity (Moldrup *et al.*, 2001; Perret *et al.*, 1999)

- fracture analysis (Kling *et al.*, 2016),
- fluid flow analysis (Mooney *et al.*, 2012),
- visualisation of organic matter (Bouckaert *et al.*, 2009; Peth *et al.*, 2014; Sleutel *et al.*, 2008 and 2010),
- soil biological experiments (Feeney *et al.*, 2006; Van den Bulcke *et al.*, 2009; McNamara *et al.*, 2003),
- soil bulk density analysis (Anderson *et al.*, 1990; Jenessen & Heyerdahl, 1988; Petrovic *et al.*, 1982),
- soil compaction and consolidation (Keller *et al.*, 2013; Ma *et al.*, 2015; Pires *et al.*, 2007, 2010; Tracy *et al.*, 2012),
- strain mapping (Bay *et al.*, 1999; Higo *et al.*, 2011; Peth *et al.*, 2010b),
- soil structure (Pagenkemper *et al.*, 2014; Rogasik, *et al.*, 2003),
- root analysis (Kuka *et al.*, 2013; Mooney *et al.*, 2012),
- water content (Crestana *et al.*, 1985; Hopmans *et al.*, 1992),
- gas transport parameters (Naveed *et al.*, 2013),
- chemical distribution patterns (Clarke *et al.*, 2016),
- the effects of agricultural practises (Gantzer & Anderson, 2002; Olsen & Borreson, 1997; Rogasik *et al.*, 2003),
- surface sealing and raindrop impact (Fohrer *et al.*, 1999; Macedo *et al.*, 1998;).
- multiscale variation in Fe and C within aggregates to characterise pore structure and cementation (Yu *et al.*, 2017)

Critically to the study of these phenomena we may also view the soil in real-time so that dynamic processes that influence key structural parameters can be quantified and explored. Despite a wide range of applications, current segmentation and processing methods to identify various parameters pores are user dependant and have a large impact on results obtained from XRCT data (Beckers *et al.*, 2014).

Owing to the relative novelty of the science, there are a number of research gaps in the application of XRCT, particularly in respect to soils. There has been little work on the organic components of soil in comparison to mineral solids, where most has focussed on the analysis of non-mineral solids such as roots (Blais, 2005; Heck & Elliot, 2006; Heeraman *et al.*, 1997; Pierret *et al.*, 1999; Steppe *et al.*, 2004;). This is

likely due to the difficulties in differentiating water, organic matter and pore space; Heck and Elliot (2006) found that organic materials such as wood and lignite had HU values of 0-1000, which is within the range of attenuation values for water, owing to organic matter's typical high water content, porous nature, and the low density of carbon. Digital images of thin slices have long been the traditional method with which to quantify soil structure (Drees *et al.*, 1994; Pagliai *et al.*, 2004; VandenBygaart *et al.*, 1999), but there are drawbacks in the destructive and 2D nature of this method (Hazlett, 1997; Øren & Bakke, 2002). Resulting from the evolution of CT systems to include micro scale study, the visualisation of complete 3D pore networks at sub-micron scales has been possible (Brunke *et al.*, 2007 & 2010; Weinekotter 2008). Pores or voids have a significant role in soil health and soil function, therefore visualisation and quantification of porosity improves our understanding (Perret *et al.*, 1999; Peth *et al.*, 2008). The application of XRCT means that one can, in theory, directly quantify and analyse the structure of soil in 3D and discriminate between soil phases (air, water and mineral) and organic matter (Bouckaert *et al.*, 2009; Rogasik *et al.*, 1999; Sleutel *et al.*, 2008). Discrimination between pores and mineral matter is relatively easy, but as will be discussed subsequently, the addition of other matter (organic matter, WTR, water) makes discrimination between phases more difficult.

### **6.1.3 Applications of XRCT for soil research**

Two paradigms exist in the analysis of soil structure (De Gryze *et al.*, 2006), one is the measurement of pores (Young *et al.*, 2001) and the other is the measurement of soil particles or soil aggregates, which is arguably much easier to accomplish in comparison (Six *et al.*, 2004). The basic structural characteristics of soils, aggregate size and shape, compaction, and water distribution highly influence the pore network (Hamamoto *et al.*, 2016), where the pore network in turn influences the properties of soil. The following section describes the benefits, limitations and gaps in current research surrounding porosity measurements in XRCT.

A comprehensive definition of a pore appears to be “a part of the pore space, homotopic to a ball, bounded by the solid and connected to the other pores by throats of minimal surface area” (Plougonven, 2009). Values for the size of a macro-pore range from > 1 mm (Perret *et al.*, 1999), to >80 µm (Brady & Weil, 2016) to >60 µm (Ghezzehei & Or, 2005), therefore >0.75 mm/ 75 µm is used here

as the boundary of a macro-pore, although the nomenclature for the size of pore in this research is of little consequence. While macro-pores only constitute a relatively small proportion of overall pore volume within a soil, it is well recognised that they dominate the rate of flux near saturation and control the speed at which solutes, water, microorganisms and air move through the soil to lower within the profile (Luxmoore *et al.*, 1990; Perret *et al.*, 1999; Pierret *et al.*, 2002; Wildenschild *et al.*, 2002). Despite continued efforts to review traditional models of water movement, they do not provide adequate descriptions of the complex process involved in heterogeneous soils and there is a lack of detailed or quantitative relationships between pore structure and flow dynamics in soil (Luo *et al.*, 2008; Lin *et al.*, 2005; Šimůnek *et al.*, 2003; Köhne & Mohanty, 2005).

Until the 1990s the characterisation of macro-pores ( $>75\ \mu\text{m}$ ) in undisturbed soils was hindered by a lack of non-destructive methods (Anderson *et al.*, 1990). Although pores may be visualised well in XRCT for homogeneous materials such as rock and fine sands, the quantification of pores in more complex material is much less simple. Much of the published work that traces water movement through soil columns using medical CT is limited to a resolution of 1 mm, however industrial CT scanners may reach  $5\ \mu\text{m}$ ; there is a considerable trade-off between resolution, sample size and the number of possible images taken over time. Currently the smallest pores that researchers are able to quantify are in the order of  $15\text{-}20\ \mu\text{m}$  considering that the highest resolutions being used are in the order of  $5\ \mu\text{m}$  (see section 6.2.1), hence an emphasis on macro-pores.

Traditional 2D destructive methods have quantified pores using dye tracers (Flury & Wai, 2003) and indirect methods involved the collection of leachate through gravity or capillary suction to measure preferential flow, however these 2D methods don't offer information about flow pathways or structural organisation (Luo *et al.*, 2008). For example, by using XRCT Perret *et al.* (1999) were able to detail the geometry and topology of macropore networks, and found that 80% of the network was comprised of just one independent pore path. As well as enabling the pore volume to be calculated, XRCT also provides information about the distribution, shape, and connectivity of pores within a sample (Anderson *et al.*, 1990; Gantzer & Anderson, 2002; Peyton *et al.*, 1992; Rachman *et al.*, 2005). Attempts to model pore geometry and networks often simplify the shape of pores

into spherical or cylindrical representations (Lin *et al.*, 1999), however recently more realistic models are being produced using information from XRCT that account for irregularities between pore shape, pore throat diameters and pore topology (e.g. Mooney & Korosak. 2009; Vogel *et al.*, 2005)

CT imaging permits actual characterisation of pores and porosity rather than inferred values, although there are a number of machine parameters such as resolution and sample volume that subtly affect the quantification of porosity in the same sample (Rab *et al.*, 2014; Wildenschild *et al.*, 2002). The caveat to using XRCT to wholly characterise the pores and porosity of a material is that the porosity values derived from CT scanning are heavily dependant on user bias during thresholding (as shown by Baveye *et al.* (2010) and Iassonov *et al.* 2009)), therefore there must be some validation of the values obtained, and only by comparing them with real values obtained from thin slices, or comparison with a known maximum volume of voids at saturation such as recorded in a triaxial test, can the accuracy of porosity measurements be determined.

There are a number of papers that have attempted to validate porosity measurements derived from XRCT by comparing them with either empirical or experimentally determined porosity values. Iassonov *et al.* (2009) compared directly measured and image analysis derived porosities on a range of natural and artificial porous media, all of which yielded different results depending on the thresholding technique and operator. Given the same volume of soil scanned at a resolution of 180  $\mu\text{m}$ , porosity values ranged from 0.019-0.110% depending on the thresholding method used. At a resolution of 5.9  $\mu\text{m}$ , the void ratio measurement of glass beads ranged between 0.499 and 0.576, where the measured (real) value was 0.508. They suggest local image information and the application of locally adaptive techniques to be the only promising options. Iassonov *et al.* (2009) concluded that although manual segmentation is time consuming, has operator bias and thresholding inconsistencies, unsupervised processing is not yet viable due to the inconsistency of results even when analysing different images of an identical material, and for materials with clear discretisation between pore and media. An option that is currently being explored for the characterisation of pore structure is the use of contrast agents, e.g. Luo *et al.* (2008) used a high resolution industrial CT scanner and digital radiograph to quickly collect images over time,

allowing them to observe flow processes effectively, similarly to Bresson *et al.* (2004) and Maruyama *et al.* (2003). A 60g /L KI solution was used (as used in Perret *et al.*, 2000 and Clausnitzer & Hopmans, 2000). From this research, Luo *et al.* (2008) suggest that an optimal approach to accurately quantify porosity for a soil is to scan the soil completely saturated and completely dry, however these extremes are difficult to reach and between the two scans, and the soil is subject to strain during swelling.

Echoing outcomes of research by Rab *et al.* (2014), Parvin *et al.* (2017) used XRCT to study macropores and the dynamic of soil drying close to saturation, in comparison with an evaporation method to determine porosity using a soil water retention curve (SWRC). The use of XRCT allowed them to make better quantification of macropores than interpretation from a SWRC. This is as a result of more extensive macropore identification in XRCT, where all pores can be visualised but evaporation methods only identify connected macro-pores. The precision of XRCT porosity analysis methods has also been determined by comparing values derived for rock or bentonite mixture samples from CT with the results of vacuum saturation and Hg-porosimetry tests (Farber *et al.*, 2003; Hall *et al.*, 2013; Hashemi *et al.*, 2015; Van Geet *et al.*, 2003). As CT determined porosity includes both connected and unconnected pores, values are typically higher than vacuum or Hg-p as these only determine the connected pores.

It is clear from the previous discussions that XRCT presents huge opportunity to characterise soil based on the porosity and pore structure, for which there are number of ways to determine values of porosity. Limitations arise due to inadequate research on threshold validation, as these tend to be user biased and have large discrepancies depending on how each user decides to process their data. The use of contrast agents, comparisons with SWRC and other secondary methods appear to be the foremost suggestions. In this research, we may be able to compare porosity/pore thresholding to the maximum porosity in the triaxial tests in combination with empirically derived void space from particle density as a validation for porosity gained from image analysis, as well as providing insight into the effect of co-amendment on the location of water uptake between wet and dry samples (there are limitations to this, which are discussed subsequently in section 6.2.2).

## 6.2 Limitations of XRCT technology

The greatest limitation of this technology is that despite the provision of recommended default settings by the manufacturers or software companies, there are no fixed or standard accepted protocols for XRCT scanning or the analysis of the data produced and many of the parameters critical to the output of the scan, e.g. tube voltage, exposure time, filters and sample size, are often arbitrarily chosen by the user and therefore extremely subjective (Baveye *et al.*, 2010). As many images are prone to technical issues (Iassonov *et al.*, 2009) there are a variety of user dependent pre-processing steps required, all of which affect the end picture and quantification obtained from processing.

There are as many papers on XRCT data processing as there are about soil tomography itself, which has produced an enormous grey area in the field of data processing (Beckers *et al.*, 2014). Segmentation of the volume into phases based on the grey value (attenuation coefficient,  $\mu$ ) is user biased and not comparable between systems or between different scans on the same machine in some instances. Segmentation methods for analysis of material phases in scanning are currently lagging behind improvements in the technical ability of scanners and computer capabilities for processing the data. The subsequent sections briefly describe the issues with using XRCT in general and with an application to soils.

### 6.2.1 Sample size and resolution stand off

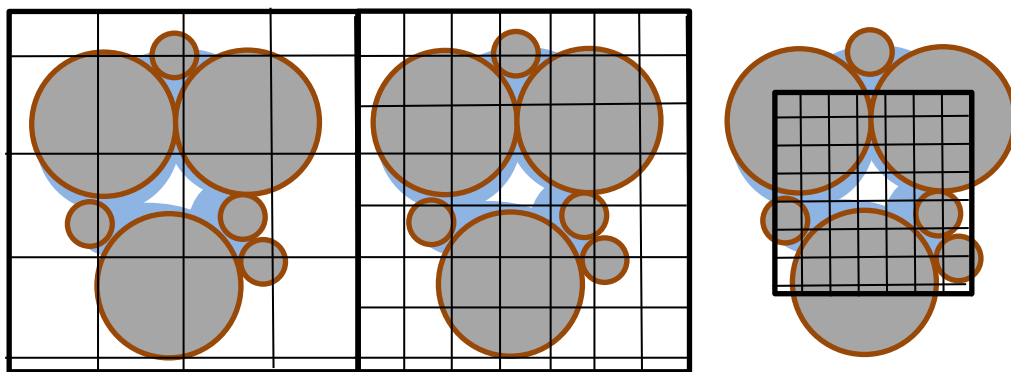


Figure 93: An example of sample size against resolution standoff, a lower resolution is required to capture a whole sample, and for higher resolution only a small portion of a sample is able to be captured.

As a general rule, the smaller the object to be scanned the greater the resolution achievable, although with heterogeneous material there are significant issues with

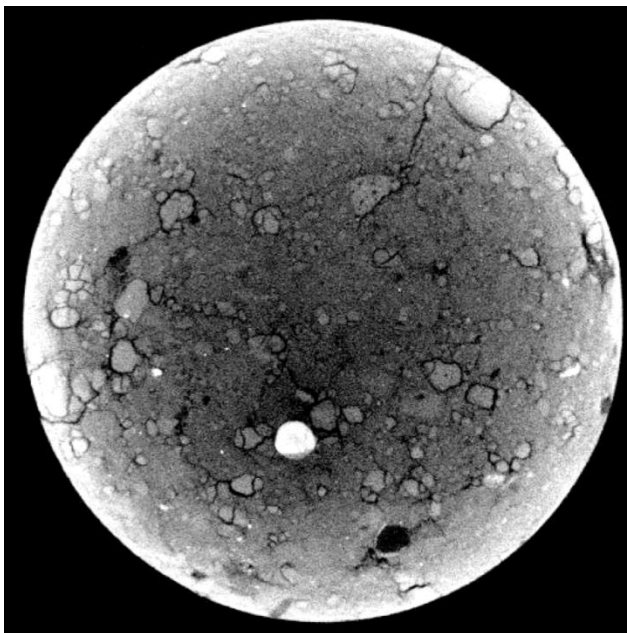
representativeness of the subsample (Baveye *et al.*, 2002), which is a critical consideration of research across the entire spectrum of soil studies and not limited to XRCT. Although large-scale processes are governed by small-scale process, a representative elementary volume (REV) is needed to accurately extrapolate the information in one sub-sample, whereby repeated measurements of a small area across a larger sample have an acceptably low variance in values. Scanning at a resolution that is too coarse for relative size of material grains of interest provides problems for latter analysis of the images, as shown in Figure 93, giving rise to issues such partial volume effects (see section 6.2.2.2). High resolution images, in the order of 5 -10  $\mu\text{m}$ , can only be obtained with samples that are < 2 mm in diameter (Mees *et al.*, 2003). However as Baveye *et al.* (2002) indicate from their research, for samples that are less 10 mm<sup>3</sup> the values derived for volumetric air content, volumetric water content, gravimetric water content and dry density each exhibit significant and erratic fluctuations. Values derived for volumetric air content stabilise as the sample size increases, however dry density values do not reach a stable value suggesting that even the 70 x 70 x 30 mm<sup>3</sup> reference volume is still insufficiently large to determine it as an REV. Generally speaking for industrial CT machines, for a single scan or sub-volume, samples of <18 mm in diameter may achieve a resolution of 10  $\mu\text{m}$ , and samples of <45 mm diameter may achieve a resolution of 25  $\mu\text{m}$ .

Resolution is important when analysing porosity as the quantification of the volume or frequency of micro-pores (5-75  $\mu\text{m}$ ) is reliant on the resolution; should one wish to identify a single pore, according to Ketcham (2005), it requires 21 voxels (3 x 3 x 3) to wholly identify the spherical shape. Given a resolution of 5  $\mu\text{m}$ , the smallest pore one could effectively quantify is 15  $\mu\text{m}$ . To resolve the issue of resolution vs sample size, multi scale imaging is gaining importance (Cnudde & Boone, 2013; Wildenschild & Sheppard, 2013; Sok *et al.*, 2010), as a high-resolution scan of a large object may be achieved with repeated sub-volume scans. The drawback of this method is that is it very time consuming and therefore expensive, and produces vast data sets that require large memory banks and high spec computers to process the data.

## 6.2.2 Imaging artefacts and corrections

Imaging artefacts, subtle complications of computer tomography, occur due to machine limitations and are problematic for quantitative analysis of the CT image (Ketcham & Carlson, 2001). There are a number of methods that can remove the effects of machine-derived errors, but any filtering or processing applied to images can remove true features or in some cases, produce more artefacts. Many of these steps are taken to aid segmentation and other processes required for quantitative analysis (Glasbey & Horgan, 1995), however the choices made at this point by the user have a great impact on the performance of subsequent analysis, particularly with highly heterogeneous materials such as soil (Kaestner *et al.*, 2008). Little work has currently been done to estimate the impact of image acquisition and reconstruction settings on the quality of 3D soil images and subsequent analysis upon them (Houston *et al.*, 2013). Vaz *et al.* (2011) provide a detailed current assessment of the effect of various machine and reconstruction settings on subsequent soil images, which goes towards a guideline for XRCT users.

### 6.2.2.1 Beam hardening and ring artefacts



*Figure 94: Beam hardening example on a cylindrical sample of soil, taken from data processing on AM13 (70S 30WTR2<sub>w</sub> redried), the light halo is evidence of beam hardening on the edge of the material.*

A commonly experienced issue with XRCT is beam hardening, due to the use of bench-top polychromatic x-ray beam, and occurs when the beam is passed through an attenuating material (Cnudde & Boone, 2013; Ketcham & Carlson, 2001; Wildenschild & Sheppard, 2013). Lower (soft) energy X-rays are progressively attenuated through the material volume at a greater rate than higher (hard) energy x-rays, giving a higher effective attenuation at the surface of the material than in the centre. As the lower energy beams are depleted from the spectrum, the beam is 'hardened'. For a cylindrical sample, a beam-hardening artefact would present itself as shown in Figure 94, where the edges of the material appear have a higher attenuation than the centre. Problematically, there isn't a way to prevent beam hardening from occurring as heterogeneous density materials harden the beam to different degrees, so the path divergence from Lambert-Beer law (from which tomographic reconstruction is calculated) will be different in each case, however there are many pre-scanning filters and post-scan software methods that provide correction of beam hardening; these are thoroughly summarised by Ketcham and Carlson (2001) and Iassonov & Tuller (2010). Beam correction is often applied during the reconstruction step, or in some cases where the sample is uniform in shape the exterior of the sample may be trimmed in post-processing to remove the light ring, although this strategy is prone to error if the sample is not uniform as the effect of beam hardening influences the CT number to a less degree through smaller diameter areas of a sample. A simple method to avoid beam hardening is to increase the beam energy sufficiently such that the effect can be ignored (although this is problematic in samples with a mixture of highly attenuating and lesser attenuating materials), or a pre-hardening filter may be applied (although this provides more image noise).

Other effects caused by beam hardening or attenuation issues are ring artefacts, presented as full or partial circles on a sample image, which occur due to partial failure of the detector, which causes anomalous CT values. In addition materials cause streak artefacts if they have a particularly high attenuation relative to the remaining material, and movement of the sample during scanning also generates artefacts. Similarly to beam hardening, these issues can be addressed pre-scan with filters or sufficiently high x-ray energy use.

### 6.2.2.2 Partial volume effects

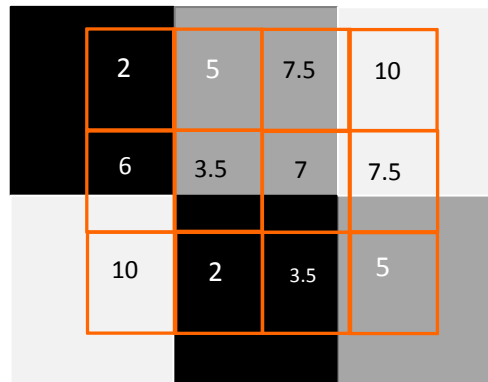


Figure 95: Partial volume effect, where low resolution scanning includes more than one material in a single voxel, thereby averaging the two values of attenuation as a result.

A partial volume effect occurs when a single pixel or voxel covers an area that includes more than one material, resulting in a CT value that is an average value of the two or more materials (Figure 95). This produces errors when thresholding as some pixels may be misclassified as one material, when in fact it is a mixture of two, sometimes classified as 'mixels' (Choi *et al.*, 1999; Hussein *et al.*, 2015). Should the averaged value be sufficiently similar to the range of values for a given material (such as water) then thresholding will overestimate the presence of that material. Due to the resolution limitations of CT technology, all particle edges in a material will be blurred to some extent, making the images difficult to quantitatively interpret with a high degree of certainty. Although the partial volume effect is a limitation in the broader scope of XRCT studies, the effect has been used advantageously in medical CT to trace two-phase fluid flow in soil (Wellington & Vinegar, 1987). The movement of fluid can be identified at a resolution considerably finer than the image resolution by detecting subtle attenuation changes within the material (Ketcham & Carlson, 2001), even if the resolution is too low to observe the change directly.

### 6.2.2.3 Image noise

The scattering of x-rays as they pass through the material being scanned causes image noise, which may also occur as a result of reflected x-rays that reach the detector. This results in random variations in brightness on the image and reduces the image quality by producing a mottled or grainy appearance. Noise may also be

produced by detector drift, but should a sample be mounted correctly this is less common. Image noise disrupts manual thresholding as there are erratic values in the image histogram, however this can be resolved by either noise-reduction before segmentation, morphological noise-reduction on post thresholding segmented data, or by using a method that tolerates noise (Wildenschild & Sheppard, 2013). As there is no *a priori* knowledge of material data in soil analysis, the first option is omitted. Disturbance of an image by scatter and noise usually requires a smoothing filter to be applied to suppress the noise during the scanning process e.g. Gauss filters, although this process is detrimental to resolution and sharp edged features. A thorough review of filtering and image enhancement methods can be found in Kaestner *et al.* (2008), which covers the use of pre-scan filters, noise reduction, post processing operations such as erosion and dilation to remove noise and artefacts in the images (Soille & Vogt, 2009). The caveat to these enhancement processes is that they may remove true features and therefore caution is advised whenever altering the image.

### **6.2.3 Water content and volume change of samples**

Although XRCT provides a fantastic opportunity to analysis materials over time, or through wetting and drying cycles, there are limitations that hinder the direct comparison of scans. In theory the correct alignment of dry and wet scans allows subtraction analysis to separate fluid phases and visualise phase distribution in the structure (Schnaar & Brusseau, 2005 and 2006; Wildenschild *et al.*, 2002). However, when soils are wetted there may be considerable movement of particular markers within the sample (key particles on which one image is registered with another) due to density changes as the bulk density is reduced. Where flow is involved, the analysis of soil between dry and wet states tends to ignore any swell/shrink behaviour and simply (incorrectly) assumes that any change is due to water (Young *et al.*, 2012). Even in samples that undergo no swelling under wetting, the presence of water exacerbates the partial volume effect; Higo *et al.* (2011) took a sample of sand 18 mm  $\varnothing$  by 17.7 mm in height and observed the water retention behaviour during a drying and wetting process using XRCT. Images were trinarised (three phase separation) into the soil phase, pore water phase and pore air phase. The thresholding technique was required to account for the partial volume effect as voxels sharing both soil phase and air

phase were incorrectly identified as a water phase as the value of the former is an average of solid and air phases, thus giving a similar value (Hashemi *et al.*, 2015; Higo *et al.*, 2011).

#### 6.2.4 Thresholding

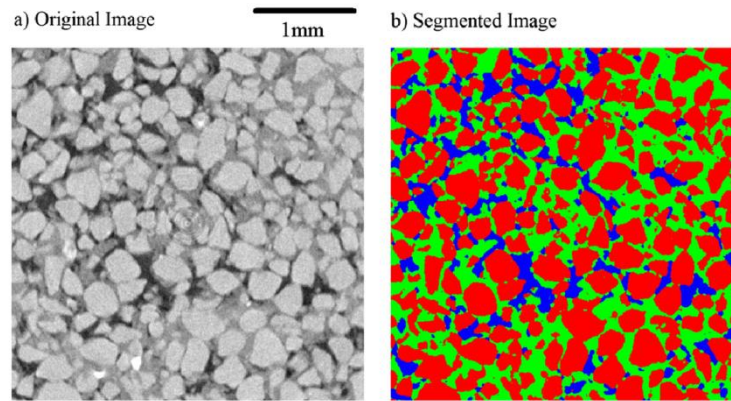


Figure 96: Segmented image of a lime treated sand bentonite mixtures from Hashemi *et al.* (2015), where macro-voids are shown in blue, bentonite shown in green and sand in red

Thresholding (also called binarisation or segmentation) is crucial for the analysis of three phase systems such as soil, particularly where the same sample material is scanned through drying or wetting stages and later compared in order to separate fluid phases, perform geometric analysis and identify phase or pore distribution through the material (Iassonov *et al.*, 2009; Wildenschild *et al.*, 2002). As images of the scanned material are presented as a range values on grey scale histogram, which represents the relative attenuation of a particle, thresholding is a technique used to separate phases in a material in order to quantify some aspect of space e.g. porosity, whereby a grey value is used as the cut off between one material and another. This is shown in Figure 96, where the grey image has been segmented into three materials based on the attenuation characteristics. The choice of thresholding or segmentation technique entirely controls the resulting measurements (Baveye *et al.*, 2010, Taina *et al.*, 2008; Zhang, 2001), however there are currently countless methods from which to choose (Dey *et al.*, 2010; Sezgin & Sankur, 2004), none of which are the 'correct' or ideal method as each is particular to the research on which it was based. Readers are directed to Tuller *et al.* (2013)

for an extensive review and comparison of global and locally adaptive methods for segmentation of XRCT data of porous materials.

For fairly homogenous materials such as sand the process of thresholding is relatively easy as void space and material have distinctly different ranges of attenuation, however two-way or three-way segmentation of soil provides a substantial challenge. As discussed, most thresholding methods are not suited to a highly heterogeneous material such as soil and as such require highly skilled operators to provide acceptably accurate segmentation (Iassonov *et al.*, 2009). Baveye *et al.* (2010), Helliwell *et al.* (2013), Iassonov *et al.* (2009), Kaesnter *et al.* (2008) and Wang *et al.* (2011) all concur that thresholding is the most important step in data analysis and affects all subsequent processing, where subjective decisions during the thresholding processes critically determine outputs such as porosity, surface area and network structure.

Briefly, thresholding techniques are divided into global or local methods and further into fully automated or manual user dependent categories. Traditional global methods use a cut off value between clear peaks in a histogram to distinguish between two or more phases, however in general the value of the cut off is arbitrary and inadequate (Lindquist *et al.*, 1996) and results in truncation of the histogram. Global thresholding, which although rudimentary, is the most common approach for quantitative space analysis and fluid mechanics, and few studies implement more advanced or complicated techniques despite growing technology to do so (Iassonov *et al.*, 2009; Sezgin & Sanker, 2004). Local thresholding may provide the user with more accurate results as instead of using one value, a variable (adaptive) threshold is used based on local characteristics (Pierret *et al.*, 2002) such indicator kriging, which classifies each a pixel/voxel as either pore or solid based on its greyscale value and local spatial correlation structure. This method requires two thresholds, one threshold below which all pixels are pores, and one value above which all pixels are solids (Al-Raoush & Willson, 2005; Houston *et al.*, 2013; Oh & Lindquist, 1999; Wang *et al.*, 2011; Zhou *et al.*, 2013). This method is an optimal segmentation tool when considering bimodal histogram patterns, however it is somewhat limited for use on unimodal histograms or in materials where visual inspection cannot sufficiently assess the

appropriate thresholds, and instead the global Otsu method (1979) is preferable for unimodal histograms (Wang *et al.*, 2011).

Other local thresholding techniques include edge detection (Sheppard *et al.*, 2004; Schlüter *et al.*, 2010), however this method requires the careful manual selection of seed regions to enable satisfactory thresholding and is therefore a very labour intensive method (Kaestner *et al.*, 2008). Region growing methods are based on the assumption that should one select a single voxel or region of interest (ROI), then all surrounding and connected voxels that have sufficiently similar  $\mu$  value must belong to the same object, scaled relatively by the mean intensity of the sample (Ketcham, 2005). This method would be highly applicable in determining the surface of complicated materials, and determining the extent to which a substance such as water would penetrate a material upon submersion.

Baveye *et al.* (2010) compared the outcomes and approaches to thresholding of 13 XRCT experts on three test images and found that each analysis was unique to the individual, with no single method yielding the 'best' results. There is no way to select the best method as each could be approximately correct or best suited to a given application or desired outcome. Baveye *et al.*, (2010) therefore stress the need for physical standards (Thompson *et al.*, 2012; Sleutel *et al.*, 2008), adoption of specific procedures for accuracy assessment, and a development in the processing of data that does not require a binary image. Similarly, Iassonov *et al.* (2009) compared 14 segmentation methods on macro porous soils, sand-bentonite mixtures and precision glass beads and found that only a handful of the methods performed well for all three materials, where only the Otsu (1979) method and CL-Ridler (Ridler & Calvard, 1978) gave 'adequate' binarisation for global methods. Locally adaptive methods had more accurate performance than the range of global thresholding methods used but had significant limitations; the indicator kriging method by Oh & Lindquist (1999) overall had the best segmentation quality, however this method was labour intensive even for a skilled operator and the outcome is not only sensitive to the thresholds chosen (Zhou *et al.*, 2013), but is really only effective on bimodal histograms.

The conclusion from a review of current research is that there are countless methods for segmentation but there is little to determine their relative 'success' in giving real values for parameters such as porosity and bulk density, and as such

the rationale for choosing a particular method is down to the user's preference. Due to vastly different scales, resolutions and processes, the results from different studies are very difficult to compare (Zhou *et al.*, 2013). There are benefits and limitations to both approaches; local methods allow some small variations to be taken into account through the sample, however global methods are easier to conduct and require less computational power. Wang *et al.* (2011) show that the performance of a thresholding technique is not always based on the complexity of the method, where a global method (Otsu, 1979) is more efficient than the local method employed by Oh & Lindquist (1999). The underlying reason for uncertainty in the methods is that validation and performance assessment is a missing element for this type of research due to its relative infancy. Validation only occurs should thresholding techniques give values close to an empirically or experimentally derived value. There is an overwhelming need for reliable and consistent automated algorithms that do not yield any operator bias, which in turn may be applied to a large range of materials (Iassonov *et al.*, 2009).

Regardless of the method chosen, the problem with thresholding soil is that due to the volume of sample and resolution achievable (10's of  $\mu\text{m}$  for samples up to 40 mm  $\emptyset$ ), many voxels will be vulnerable to partial volume effects and there will be a great deal of noise in the image due to the extreme heterogeneity of soil samples, which is attributed to different mineralogy of soil matter, the presence of low density organic matter and the presence of both water and air in pore space. In addition many of the important structures in soil that are critical to their function e.g. micro-pores (5-75  $\mu\text{m}$ ) are lost completely within voxels should the image scale exceed 5-10  $\mu\text{m}$ . Sub-micron & single value micron scanning is achievable but requires very small samples, however the heterogeneity of soil means that sub-sampling to this extent may not be at all representative. There are a number of options available to threshold soil for the purposes of analysing porosity, water movement, stress and strain, which shall be discussed subsequently in section 6.4.

### ***6.3 Overcoming issues of segmentation in heterogeneous materials***

Distinguishing between pore space, soil OM, soil mineral matter and water is currently limited by both the technology of CT and the software with which to process acquired data, despite increasingly high resolution and image quality. There are many publications that attempt to use different methods of thresholding

to identify pore space or water in both easily segmentable bimodal images and in images where there are significant attenuation similarities between phases that result in unimodal histograms (Peth *et al.*, 2008). Baveye *et al.* (2010) propose that porosity measurements may be a fast and ready way to validate the accuracy of thresholding, as its measurement it does not affect the procedure of scanning (Al-Raoush & Willson, 2005). Research by Beckers *et al.* (2014) questions if visible porosity provides a useful and robust benchmark for segmentation and analyses if it can be used for the basis of thresholding by comparing to 4 other methods; (Ostu, 1979), Schlüter *et al.* (2010), Hapca *et al.* (2013) and Houston *et al.* (2013). They found that global segmentation methods provide good agreement between XRCT derived information on retention and SWRC derived from macroscopic measurements. The porosity-based method is robust as it agrees with macroscopic measurements regardless of the pre-processing of images, however there is still a need in the field of XRCT to be able to effectively quantify the images without the need for comparison of data from secondary methods.

### **6.3.1 Tracers in solution**

One method of overcoming the subjective and complicated process of differentiating between solid phases, water, and air in highly heterogeneous materials is the use of contrast agents by addition of compounds to water or other liquids (Kaestner *et al.*, 2008; Luo *et al.*, 2008; Van Loo *et al.*, 2014; Wildenschild *et al.*, 2002). As discussed previously, pore sizes range from centimetres down to the nano-scale level, whereby the spatial location of pores through a soil matrix has a profound impact on the location of water, microorganisms and soil organic matter (Ekschmitt *et al.*, 2008). The technique of using tracers (contrast agents) has great potential for phase and porosity identification and has been used for many years in medical applications, where heavy elements are used to improve the diagnostic capabilities of low resolution scanners (Caltagirone *et al.*, 2015; Granton *et al.*, 2008), and increasingly in geological and petroleum science applications (Alajmi *et al.*, 2009; Hirono *et al.*, 2003; Yang *et al.*, 2016). The drawback of using any form of tracer is that it may change the density, viscosity, contact angle or surface tension of the fluid in which the agent is immersed and users may experience issues with crystallisation, therefore this must be acknowledged should it be used for modelling flow behaviour. The concentration of tracer is also important, such that

it gives sufficiently high attenuation that exceeds the distribution of the solid phases in non-absorptive materials. For materials that will allow water to enter, such as organic matter, the addition of contrast agent causes simultaneous attenuation increases in the pore space and absorptive structures (Kaestner *et al.*, 2008).

There is a growing body of literature on the use of contrast agents to measure porosity or hydraulic properties in real-time in rocks or generally uniform sandy soils whereby a solution containing a contrast agent is used to fill pores within a sample, typically sodium iodide (Alajmii *et al.*, 2009), potassium iodide, potassium bromide, xenon (Akin & Kovscek, 2003; Wildenschild *et al.*, 2002) and calcium iodide used as a tracer by Kaestner *et al.* (2008). However, the use of tracers in heterogeneous soils is currently very limited despite a number of papers that have explored the theory of contrast application; Hussein *et al.* (2015) suggest the use of a contrast agent to completely remove the need for thresholding and therefore provide valid porosity values without secondary assessment of accuracy. One sample that has been fully saturated using a soluble contrast agent (which has a higher attenuation coefficient than the solid material), can be compared with a sample that has been full saturated using water alone, which has a zero attenuation coefficient. As the values of soil mineral matter (impenetrable solid material) do not change when the sample is wetted, these voxels can be matched between the two scans. The voxels fully occupied by pores will also (in theory) be identical, therefore the total pore space can be identified, as any remaining voxels that have been subject to partial volume effects and contain a mixture of solid and pore space can be classified as mixels (Choi *et al.*, 1999). This therefore automates the process of thresholding as the pore space is clearly identified by the tracer; however this requires a priori knowledge of the CT values of the solid material, therefore its application for extremely heterogenic materials such as soil is less effective, especially when organic matter with a similar attenuation value to pore space is added.

There is also a lack of data surrounding the study of soils containing significant proportions of organic matter, including the investigation of biochemical interactions (Voltolini *et al.*, 2017) and the importance of pore geomorphology in the processes of aggregation and decomposition of OM (Peth *et al.*, 2014; Sleutel *et*

*al.*, 2012), which comes as a direct result of technique limitations with which to determine the location of OM (Young *et al.*, 2001). The identification of organic matter is especially difficult as the x-ray attenuation coefficients of OM fall between the range of values typical of pores (water or air) and the mineral component of soil (Kaestner *et al.*, 2006; Peth *et al.*, 2014), therefore creating unimodal histograms. Thresholding of samples with overlapping  $\mu$  values is further complicated by factors such as partial volume effects. The current research gap in the study of OM in XRC is slowly being filled; there is growing interest in using agents to stain the OM directly.

### **6.3.2 OM identification**

It is still well recognised that discriminating between soil phases and the quantification of organic matter, particularly at high water contents, remains a challenging prospect (Helliwell *et al.*, 2013), however there have been some attempts at identifying organic matter without the use of tracers; De Gryze *et al.* (2006) attempted to visualise OM in 3D using CT data at a resolution of 13.4  $\mu\text{m}$  without the use of tracers (although effective optical resolution was 27  $\mu\text{m}$  due to removal of all single voxel data points). They were able to identify the histogram as bimodal through modelling and were able to produce the histograms for each constituent. A threshold attenuation coefficient was determined by manual selection of pore areas in 5 random locations, although the quality of the tomographic images didn't allow organic matter to be located based on the attenuation coefficients alone. In this case manual selection of pores and subsequent thresholding was used, as automated three-phase classification was too insensitive to pick up on the very subtle differences in contrast between OM and pore space or water. However, De Grzye *et al.* (2006) stated at the time that CT scanning cannot be used together with staining techniques that are commonplace in thin sections e.g. Chenu & Plante (2006), but instead suggest that porosity and pore morphology can be directly observed.

Sleutel *et al.* (2008) attempted to segment pore space, OM and mineral particles in sand/OM mixtures but found that there were large overlaps in both the  $\mu$  values of OM and mineral and (more problematically) between pore space and OM. Quinton *et al.* (2009) similarly found significant challenges in distinguishing between water and organic matter, especially when saturated as the attenuation values are very

similar; Ketteridge & Binley (2011) experienced the same issue when trying to quantify characteristics of peat and resorted to using lead nitrate to flush the sample in order to contrast the components of interest. Elyeznasni *et al.* (2012) were able to detect coarse organic matter in XRCT images at a resolution of 68-88  $\mu\text{m}$  in samples that had a very high concentration of OM, but had difficulty in quantifying fine sized OM and in identifying any OM when mixed with a sandy soil. They suggest that comparing XRCT images with observations from thin slices would aid in determining a suitable threshold and also aid in standardising protocols for obtaining sensible porosity values in OM rich materials. As a concluding remark to the research by Elyeznasi *et al.* (2012), they also suggest the extended use of staining techniques (e.g. Peth *et al.*, 2010a). Chenu & Plante (2006) used lead, silver and uranium in transmission electron microscopy to stain 2D thin sections of soil and OM, the theory being that colloidal soil OM has a strong affinity for heavy metal cations (e.g.  $\text{Fe}^{3+}$ ) due to ligands that form chelates and then complexes with the metal (Van Loo *et al.*, 2014), therefore the use of compounds containing heavy metal which preferentially adsorb to OM and not to soil minerals, is ideal for this application.

Recent literature goes towards providing evidence that staining has potential within XRCT research and removes issues of unimodal histograms and the need for modelling to find suitable thresholds, or comparisons with 2D thin sections for validation of chosen thresholds, in order to identify organic matter (Peth *et al.*, 2014). Peth *et al.* (2010a) for example used osmium as a staining agent due to its interaction with C-bonds of organic compounds (Hayes *et al.*, 1963), allowing visualisation of even fine OM that has been adsorbed onto clay minerals and cannot be observed discretely (Chenu & Plante, 2006). Osmium was used to stain OM in air dried samples (in vapour form) and is distinguishable from other material in XRCT images due to its high atomic number, although bright spots attributable to other soil minerals and compound such as Fe-Oxides may have complicated the quantitative analysis.

One of the first (known) publications that has investigated the effect of using different stain on organic matter and mineral samples rather than using tracer to identify the spatial location of water, was Van Loo *et al.* (2014). In their research, 52 compounds containing heavy elements were dissolved in deionised water and

their attenuation was assessed for suitability to be used as a contrast agent. Secondly their suitability to enhance the x-ray attenuation of pure OM and respective impacts on soil mineral matter (MM) were taken into account. Preferable contrast agents either had good relative effect on the attenuation of water without staining the SOM significantly ( $C_{\text{water}}$ ) or a high ability to stain SOM compared to air ( $C_{\text{SOM}}$ ). The best four agents for staining SOM were chosen for further testing on three samples, OM alone, MM alone and an OM/MM mix; these were phosphomolybdic acid (PMA), silver nitrate, lead nitrate and lead acetate. Osmium tetroxide, used by Peth *et al.* (2010a) performed similarly well to the four best agents chosen by Van Loo *et al.* (2014) in terms of  $C_{\text{water}}$  and  $C_{\text{SOM}}$ , but was not very soluble in water (maximum of 6.5 g/100 ml compared to 44-216 g).

Samples were forcibly saturated to avoid crystal formation, and excess solution was removed before sealing the sample to permit a reaction between the sample and the contrast agent. Samples were then dried at 20°C before scanning. A reference sample was included for each sample that followed the same procedure, with the use of deionised water alone. Van Loo *et al.* (2014) evaluated the shift in x-ray attenuation between the control and each contrast agent and found in the control sample there was little contrast between the air and SOM (as found by Sleutel *et al.*, 2008). When contrast agents were used, the air and MM values remained the same but the value of the SOM particles were then higher on average than the MM particles, therefore the contrast agent must increase the U values of OM sufficiently that it can be then distinguished from the MM. KI (potassium iodide) in Van Loo *et al.* (2014) had one of the greatest effects on the relative x-ray attenuation of water (0.88) but very little attenuation change for SOM (0.09), where iodine on its own makes negligible difference. They also found that using staining agents has no impact on the u value of air or mineral matter.

Other work that has used contrast agents includes Heijs *et al.* (1995) who took one sample and compared the dry and wet scans. They followed the procedure of Booltink & Bouma, (1991) and used 15 mm of water with kalium iodide solution (HU value of 2900), scanned a sample 25 cm x 12.3 cm to a resolution of 0.27 mm. This work allowed the binary image of the water content distribution to be compared with the air filled macropores. Similarly, Luo *et al.* (2008) used KI solution at 6.6 ml/min for 23 hours through a saturated soil column. The soil column

was 10 cm  $\varnothing$  x 30 cm height and scanned to a resolution of 0.1 x 0.1 x 0.13 mm. Solute transport was measured in real time by scanning two critical positions, after which the whole column was scanned to obtain the solute distribution. Similarly Wildenschild *et al* (2002) used KI Iodine (absorption edge at 33.7keV), allowing water to be clearly distinguished from the air and solid phases. Kaestner *et al.* (2008) used CaI (chosen for its preservation of clayey soil aggregates) at a concentration of 4% for optimal contrast, although this was not tested by Van Loo *et al.*, (2014), therefore a comparison of its effectiveness cannot be made.

The use of contrast agent (KI) was attempted for visualising porosity in Soil2 and AM14 (70S 15C and 15WTR2a) at concentration of 1 mol (166 g/L or 16% %w/v), which although more than KI solutions used in Clausnitzer & Hopmans, 2000, Luo *et al.* (2008) and Perret *et al.* (2000), was used in order to rapidly stain the soil pores. It became apparent in the initial analysis of scan results and in personal communication with Stephan Le Roux (an expert XRCT technician at Stellenbosch scanning facilities) that the use of iodine as a tracer creates more problems than it aims to solve. The high density of the iodine creates a high number of artefacts, image noise, and due to partial volume effects, increases the attenuation of the entire material such that the distinction between phases and different materials within the sample is actually more difficult than when no tracer was used. A great deal of investigation into optimal tracer concentrations and machine parameters to produce images that can be readily analysed is required. The use of tracers for phase identification has considerable potential, but the complexity and labour intensive nature of this process of investigation is well beyond the scope of this thesis.

#### **6.4 XRCT methodology and results**

The previous discussions have outlined the benefits and flexibility of using XRCT for high-resolution imaging, modelling, and quantification of porous media and fluid distributions in 3D (Werth *et al.*, 2010). Despite a growing range of possibilities using XRCT and increasing sophistication in scanning and analysis, there are a number of issues with the use of this technology that need to be addressed in order for it to be used as a comparative and reliable tool. System faults (such as partial volume effect, beam hardening and other artefacts) and user-based faults with thresholding limit the application of XRCT; the resolution/sample size trade off remains problematic when studying heterogeneous material. Given the rapid development of this method of 3D representation since its beginning, it is likely that these issues will be resolved sooner rather than later using new technologies such as dual energy. Once standardisation occurs in the set up and processing of data, XRCT will continue to improve material research.

The following section discusses the different methods used for this thesis to obtain critical information on the effect of using WTR and co-amendment on soil. The aim of using XRCT was in an attempt to answer questions about the effect of WTR on soil, particularly why it changes the water retention properties of soils and how it may interact with compost. The research explores a currently sparse area of study, and to the authors knowledge there are no XRCT papers that have looked in detail at WTR or attempted to analyse soils with co-amendments. The aims of using high-resolution imaging were to;

- 1) determine structural changes as a result of WTR<sub>2d</sub> and WTR<sub>2w</sub> amendment and co-amendment with compost,
- 2) determine if WTR can be characterised in the images,
- 3) determine the internal structure & porosity of WTR<sub>2d</sub>,
- 4) determine how WTR reacts when exposed to water,
- 5) test the ability of tracers to enhance our ability to characterise pore space.

The machine settings and data outputs of XRCT scanning retrieved for this thesis were in all cases fully directed by highly skilled technicians, both in Durham and Stellenbosch. Given that this area of research is novel, the methods explored and subsequent results presented here are to illustrate the potential of using XRCT to

reconstruct and quantify particular parameters in soils that have been amended with highly organic components, and to view the internal structure of WTR<sub>d</sub> at high resolution. The approach is very well suited to the complex analysis of highly heterogeneous material that are less well served by 2D analysis.

#### **6.4.1 Machinery and settings**

Three machines were used for the production of XRCT images, an Xradia/Zeiss VersaXRM 410 system (Durham University), Phoenix VtomeXC series machine, and a Phoenix VtomeXC series machine (both at Stellenbosch University). The following gives a very brief outline of their capabilities. Data from the scans was stored as stack of TIFFs after reconstruction, to enable any program to be subsequently used for analysis. The data from the Xradia machine was processed using Avizo 9.4.0 and data from the Phoenix systems were processed using Volume Graphics 3.2.0 software. Both of these software packages are highly complicated and advanced, but for the research analysis presented here the computation outcomes are likely to be negligibly different. All data processing was under the supervision of a skilled technician.

##### **Xradia/Zeiss Versa XRM 410:**

- 150kV 10W microfocus x-ray source
- 2k x 2k 16bit CCD x-ray camera
- A single macro lens (0.4X) for large field of view scans and three high resolution lens (4X,10X&20X) for region of interest scans.
- 4 axis motorised sample stage with 15 kg load capacity and 360 degree rotation, and maximum sample size of 300 mm
- Up to 0.9 um resolution
- A single 3D volume takes between 4 and 24 hours to scan depending on resolution

Sample diameter ( $\emptyset$ )	Full sample scan voxel size	High resolution scan voxel size
<5 mm	3um	1-2um
<18 mm	10um	5um
<45 mm	25um	10um

*Table 31: Example size vs resolution capability of the Xradia/Zeiss XRM 410 machine [23, Xradia]*

As shown in Table 31, the resolution of the scanned images range from 1-40  $\mu\text{m}$ , depending on a range of factors including the sample size and the required field of view and the time taken to perform the scan.

### **Phoenix VTomeXC series**

- 450kV, fast cone beam CT with scatter correct
- ASTM E1695 guidelines for scanning
- Robust, small footprint
- Maximum sample size 500 mm  $\emptyset$  x 1000 mm
- Up to 5  $\mu\text{m}$  resolution

### **Phoenix VTomeXM series**

- 300kV/500W scatter correct microfocus CT
- Industry leading magnification dual tube configuration for  $\mu\text{CT}$
- 2  $\mu\text{m}$  maximum resolution
- Maximum sample size up to 500 mm  $\emptyset$ , 600 mm height
- Maximum 3D sample size 290 mm  $\emptyset$ , 400 mm
- Maximum sample weight 50 kg, high accuracy up to 10 kg
- Scan time is <4 hours

### **6.4.2 Initial scanning**

Initial scans were conducted in 2013 on field moist, loosely packed samples of 50S, 25C, 25C (by dry mass), 100% soil, 100% compost and 100% WTR1<sub>w</sub>, at a resolution of 50  $\mu\text{m}$ , using 140kV/10W power to ensure sufficient x-rays passed through the samples considering the material density and size of the samples being scanned (38 mm  $\emptyset$  x 50 mm). These scans were exploratory in their nature and were used to test if distinct structures were identifiable and decide if

differentiation between WTR, compost and soil fractions was visually possible, considering the limitations experienced by many others in the identification of organic matter and material of similar attenuation coefficients to water. Figure 8 presents a single slice of the 3D volume of the various materials tested. All images are presented in greyscale, whereby the white end of the scale presents materials with high attenuation, and the black end of the scale presents material with no attenuation (void space). In theory, for scans conducted with constant parameters (beam energy, filters etc) a single material should have the same grey scale value across all the scans; however in practise the x-ray flux wavers slightly and there will be a resultant small discrepancy between images.

The grey-scale presented has a direct correlation between its value and the x-ray absorbance of a material, as the system is designed such that a single x-ray passing through a sample generates a single photon, which is captured in the grey-scale value for that pixel on the detector. For materials with a low atomic number/particle density (which includes all the materials used in this research), the attenuation is linear with the mass. However, considering the density similarities of soil mineral matter ( $2.65 \text{ g/cm}^3$ ) and WTR ( $2.11 \text{ g/cm}^3$ ), and the density of organic matter ( $1.65 \text{ g/cm}^3$ ), it is likely that differentiating between materials on the grey-scale values will be exceptionally difficult and only visual analysis is appropriate. Figure 97 shows a single ortho slice ( $38 \text{ mm } \emptyset$ ) from each scan of soil (A), compost (B), WTR<sub>1w</sub> (C), and Figure 9 shows a mixture of 50% soil, 25% compost and 25% WTR<sub>1w</sub>. Each image has been cropped such that the outside casing that housed the sample to prevent movement has been removed.

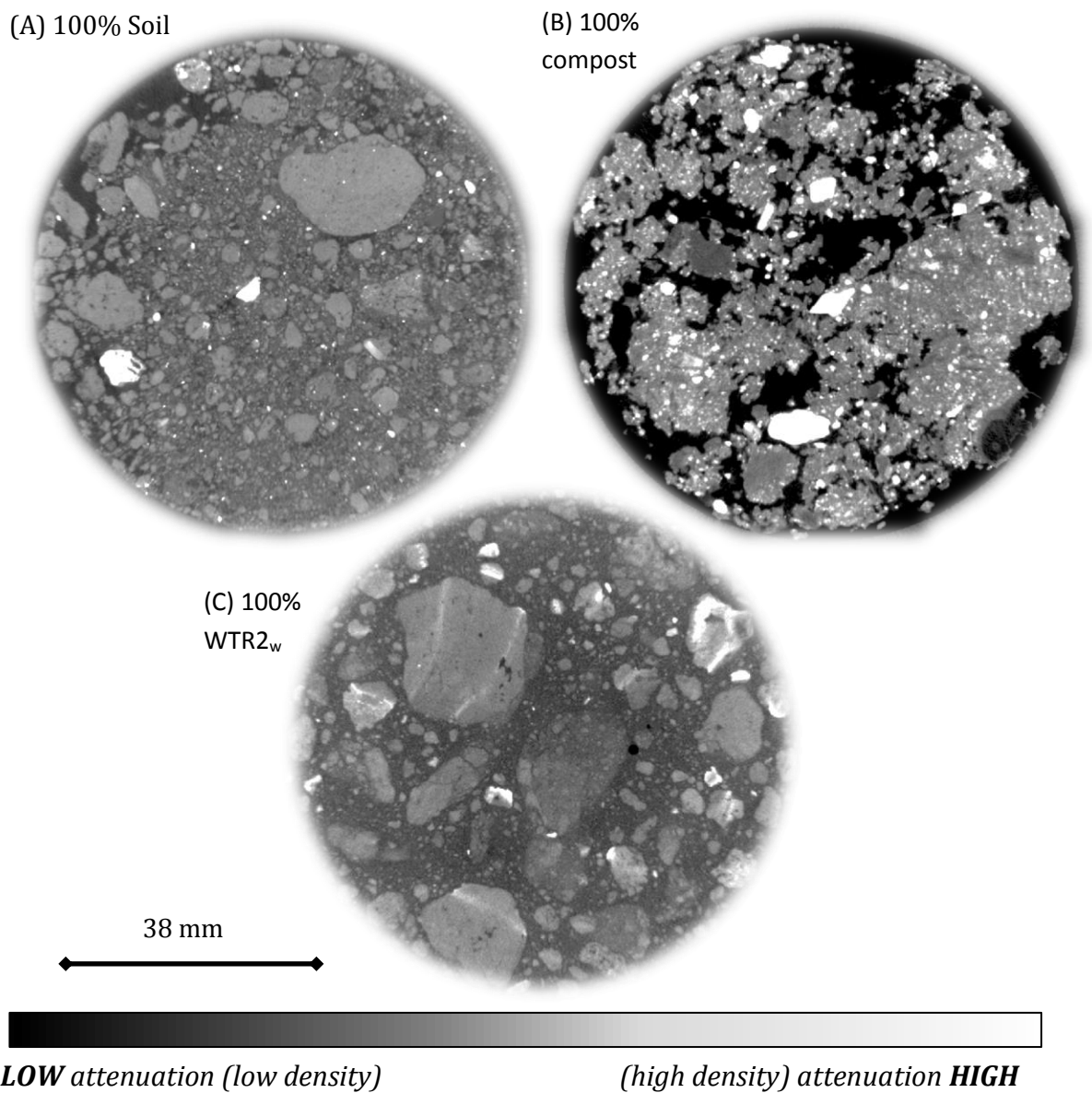
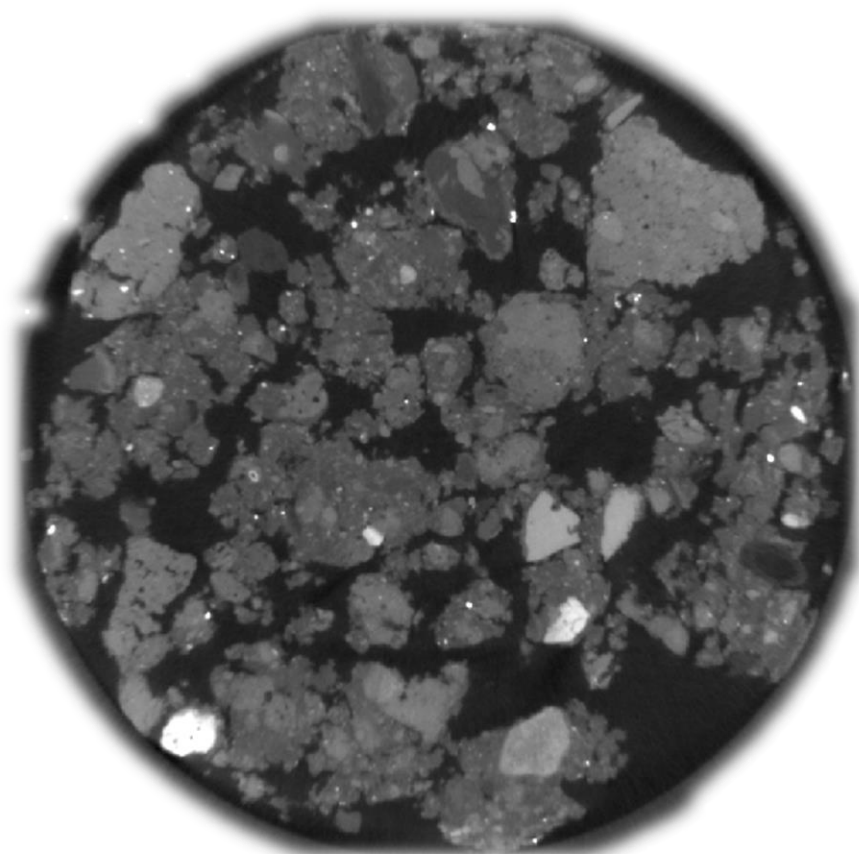


Figure 97: XRCT scans of (A) 100% soil, (B) 100% compost, (C) 100% WTR<sub>1w</sub> at 50  $\mu\text{m}$  resolution

In these images we can see that at 50  $\mu\text{m}$  resolution the individual particles, macro pores and voids can be identified in the soil (Figure 95a) where the densest minerals such as Fe oxides ( $\sim 5 \text{ g/cm}^3$ ), clays and quartz ( $2.65 \text{ g/cm}^3$ ) appear as bright white spots and voids are black. In Figure 97B, dark grey areas (organic matter) are irregularly arranged with large black void spaces. There appear to be a number of darker grey areas, which may be areas of high water content (due to reduced attenuation coefficient). There are a number of dense particles (white)

that are presumably small pieces of soil mineral or other contamination in the compost, but go some way to providing us with an idea of the contrast between soil minerals and organic matter. In Figure 97C, the metal and organic elements of WTR1<sub>w</sub> are clearly contrasting; concentrated volumes of Fe oxides of WTR were apparent when oven drying the material for characterisation (Figure 14, Chapter 3). The image in Figure 97C provides further evidence to the uneven distribution of Fe in the WTR; in the largest solid particle we can identify streaks of higher density material (presumably iron) perhaps formed due to gradual settling of the WTR in the tank. The presence of other heavy elements, such as Cd, Cr, Cu and Mg (Table 8, Chapter 3) in the WTR may also account for some of the bright spots. The surrounding free fluid has a higher attenuation than water, suggesting the presence of low-density suspended solids (organic matter). The black colour of the sludge provides further evidence that organic matter is present in the free fluid.



*Figure 98: XRCT image at 50  $\mu\text{m}$  resolution on a 38 mm  $\varnothing$  sample of 50% soil, 25% compost and 25% WTR1<sub>w</sub>.*

As the sample shown in Figure 98 was mixed by dry mass of each material, there is a greater volume proportion of both compost and WTR1<sub>w</sub> to soil, owing to their

high water content and lower particle densities, the majority of the image appears as the mixture of compost and WTR1<sub>w</sub> (Table 14, Chapter 4). The darker pore space is still relatively easy to determine, and the frayed edges of the compost allows us to identify the larger clumps of aggregates of organic matter. Small bright grains are likely to be quartz minerals although these could equally be iron rich deposits in the WTR. Discriminating between the WTR and compost is now exceptionally tricky as they have similar attenuation coefficients, and at this resolution, the particle shape and porosity are not clear. Subsequent to initial scanning, over the remaining period of research, three scanning investigations were completed;

1. Durham (2016) – investigation of samples from Experiment 4 (Chapter 4, 4.4.5) to a resolution of 25  $\mu\text{m}$ ,
2. Stellenbosch (2016) –WTR2<sub>d</sub> to a resolution of 6  $\mu\text{m}$
3. Durham (2018) – 15  $\mu\text{m}$  resolution scanning of Soil2 and AM14 to investigate optimum machine parameters and the effects of sieving to different sizes.

#### **6.4.3 Exploratory use of XRCT, Durham (2016)**

Moving forward from initial scans, in 2016 single soil cores from each amendment in Experiment 4 were scanned in three states; ‘dry’ (as produced at 17.5% GWC), ‘wet’ (fully saturated), and redried” (air-dried back to 14 or 25% GWC). The cores were 38 mm  $\varnothing$  x 76 mm length and were sealed for the duration of the scan to avoid moisture loss in the warm scanning environment. The images were taken at a power of 140kV and current of 71W, using an HE2 filter (for beam hardening correction) and a 3 second exposure time. The field of view was 40 x 40 x40 mm, giving a voxel size of 25  $\mu\text{m}$  for data sets that were 2004 x 2048 pixels (X, Y) and 2007 projections (Z). The data were reconstructed using appropriate centre shift (ranging between -0.4 and 0.3) and beam hardening correction (0.1). Having underestimated the time required to process each data set, only Soil2 and A10-A13 were processed using Avizo 9.4.0, as these contained the highest concentration of amendment, under the direction of a highly skilled technician.

Briefly, each stack of TIFFs was imported into Avizo for a particular sample e.g. Soil2 dry, Soil2 wet and Soil2 re-dried. The original dry data set was trimmed

vertically (Z axis) to remove roughly 5 mm from ends of the sample such that subsequent analysis did not include edge effects and any breakdown of the ends under wetting. Using the cropped dry data as a basis, a distinct pattern of bright particles were identified in each sample, to which the wet and re-dried samples were registered by rotating the data appropriately and then resampled using Lanzos interpolation. Finally each data set was finely cropped vertically to match the end points of the dry data. This was to allow a better visual comparison between the data, although during the wetting and re-drying there was a small shift in the location and orientation of some particles due to swelling and shrinkage. In order to threshold the data, the data volumes were cropped into cubes (removing the edges of the samples), which measured between 1200 x 1200 x 1200 to 950 x 950 x 950 pixels/voxels and equates to samples of 25 mm<sup>3</sup>. For each material, auto-thresholding low was conducted, where a suitable upper threshold value to select all the dark pore space was chosen manually. These values are present in Table 32 and reflect the difference in relative attenuation of each scan.

	Soil2	AM11	AM12	AM13	AM14	AM15
	<i>100S</i>	<i>70S 30C</i>	<i>70S</i>	<i>70S</i>	<i>70S 15C</i>	<i>70S 15C</i>
			<i>30WTR2<sub>d</sub></i>	<i>30WTR2<sub>w</sub></i>	<i>15WTR2<sub>d</sub></i>	<i>15WTR2<sub>w</sub></i>
Dry	7200	13500	11500	9000	5600	8700
Wet	7200	13000	9000	9000	9500	14000
Redry	9700	14600	13500	12500	7000	8500

*Table 32: Lower auto-thresholding values chosen for Experiment 4 samples*

The values presented in Table 32 are derived from porosity analysis based on data from XRCT thresholding, volume of pores at the end stage of triaxial testing and values derived using information on the bulk volume and particle density for core samples. The void ratio of each sample gives an indication of how the void space changed between the sample in its dry state, having been saturated and then re-dried.

Before the discussion of results, it must be noted that void space as defined by the XRCT only captures the macro-pores; therefore if a sample has a high porosity but these pores are very fine, the void ratio & volume of voids will be low in comparison with a sample that has a greater proportion of large pores. This

analysis therefore favours samples with greater macro-porosity and under values samples with micro-porosity. The void ratio is also, as a result, much lower for XRCT than the values derived from triaxial testing and calculated porosity. The volume of voids is an absolute value in  $\mu\text{m}^3$ , and the void ratio is a ratio of the volume of voids to the total volume (which in this case is the cubic cropped region of each sample). Pagliai (1988) suggested that typical porosity values (void ratio) for very compact soil is  $<0.005$ , compact soil 0.05 -0.1, moderately porous 0.1-0.25 where 0.1 is the lower limit for 'good' soil structure (Pagliai & Vignozzi, 2002), highly porous 0.25-0.40 and extremely porous when porosity is  $<0.4$ . It must be accounted for that estimates of porosity become less precise in highly compacted soils (Periard *et al.*, 2016). Table 33 and Figure 99 show the results of porosity determination. When compared with the values of Pagliai (1988), it is apparent that the relative degree of compaction of samples is variable between amendments.

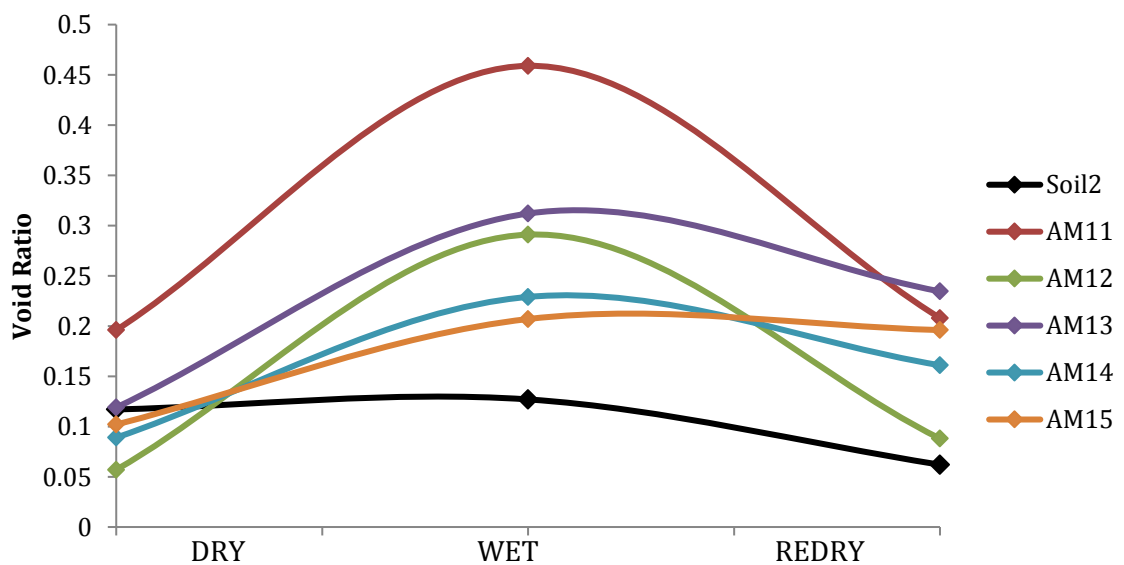


Figure 99: Void ratio change of Soil2 and AM11-AM15 derived thresholding of XRCT data in Avizo.

Sample	Volume of voids (Vv)	Void ratio (Vr)
Soil2 dry	1858 $\mu\text{m}^3$	0.117
Soil2 wet	2127 $\mu\text{m}^3$	0.127
Soil2 re-dried	1052 $\mu\text{m}^3$	0.062
Soil2 dry (calc)	(25.97) $\text{mm}^3$	0.315
Soil2 wet (Triax)	36.1 $\text{mm}^3$	0.390
AM11 dry	2566 $\mu\text{m}^3$	0.196
AM11 wet	7810 $\mu\text{m}^3$	0.459
AM11 re-dried	3195 $\mu\text{m}^3$	0.208
AM11 dry (calc)	(23.97) $\text{mm}^3$	0.376
AM11 wet (Triax)	45.34 $\text{mm}^3$	0.442
AM12 dry	788 $\mu\text{m}^3$	0.057
AM12 wet	4390 $\mu\text{m}^3$	0.291
AM12 re-dried	1289 $\mu\text{m}^3$	0.088
AM12 dry (calc)	(19.99) $\text{mm}^3$	0.347
AM12 wet (Triax)	42.38 $\text{mm}^3$	0.422
AM13 dry	1781 $\mu\text{m}^3$	0.119
AM13 wet	3936 $\mu\text{m}^3$	0.312
AM13 re-dried	2631 $\mu\text{m}^3$	0.235
AM13 dry (calc)	(23.50) $\text{mm}^3$	0.382
AM13 wet (Triax)	44.38 $\text{mm}^3$	0.433
AM14 dry	1504.47 $\mu\text{m}^3$	0.089
AM14 wet	4594.43 $\mu\text{m}^3$	0.229
AM14 re-dried	2886.45 $\mu\text{m}^3$	0.161
AM14 dry (calc)	(18.88) $\text{mm}^3$	0.330
AM14 wet (Triax)	43.88 $\text{mm}^3$	0.433
AM15 dry	1342.24 $\mu\text{m}^3$	0.102
AM15 wet	3583.83 $\mu\text{m}^3$	0.207
AM15 re-dried	3081.27 $\mu\text{m}^3$	0.196
AM15 dry (calc)	(20.96) $\text{mm}^3$	0.264
AM15 wet (Triax)	44.83 $\text{mm}^3$	0.435

*Table 33: summary of porosity values derived from XRCT for samples Soil2, and AM11-AM15, and comparisons with calculated porosity for dry samples based on particle density and volume measurements, and triaxial derived porosity for wet samples.*

In Figure 99, unamended soil (Soil2) has a fairly low relative change in void volume and void ratio between dry and wet states, followed by a reduction in void ratio after re-drying due to unrecoverable shrinkage (0.117 - 0.127 - 0.06). In general, the values of porosity derived from the triaxial test are considerably higher than for XRCT derived porosity (Table 33), but this reflects the pore sizes present in the soil, as smaller pores cannot be characterised by XRCT and therefore the dry porosity is underestimated by XRCT for all samples. For example, the triaxial derived wet porosity of AM11, AM12 and AM13 are negligibly different from one another (0.442, 0.422 and 0.433) however the XRCT derived wet porosity values show a larger difference (0.459, 0.291, 0.312).

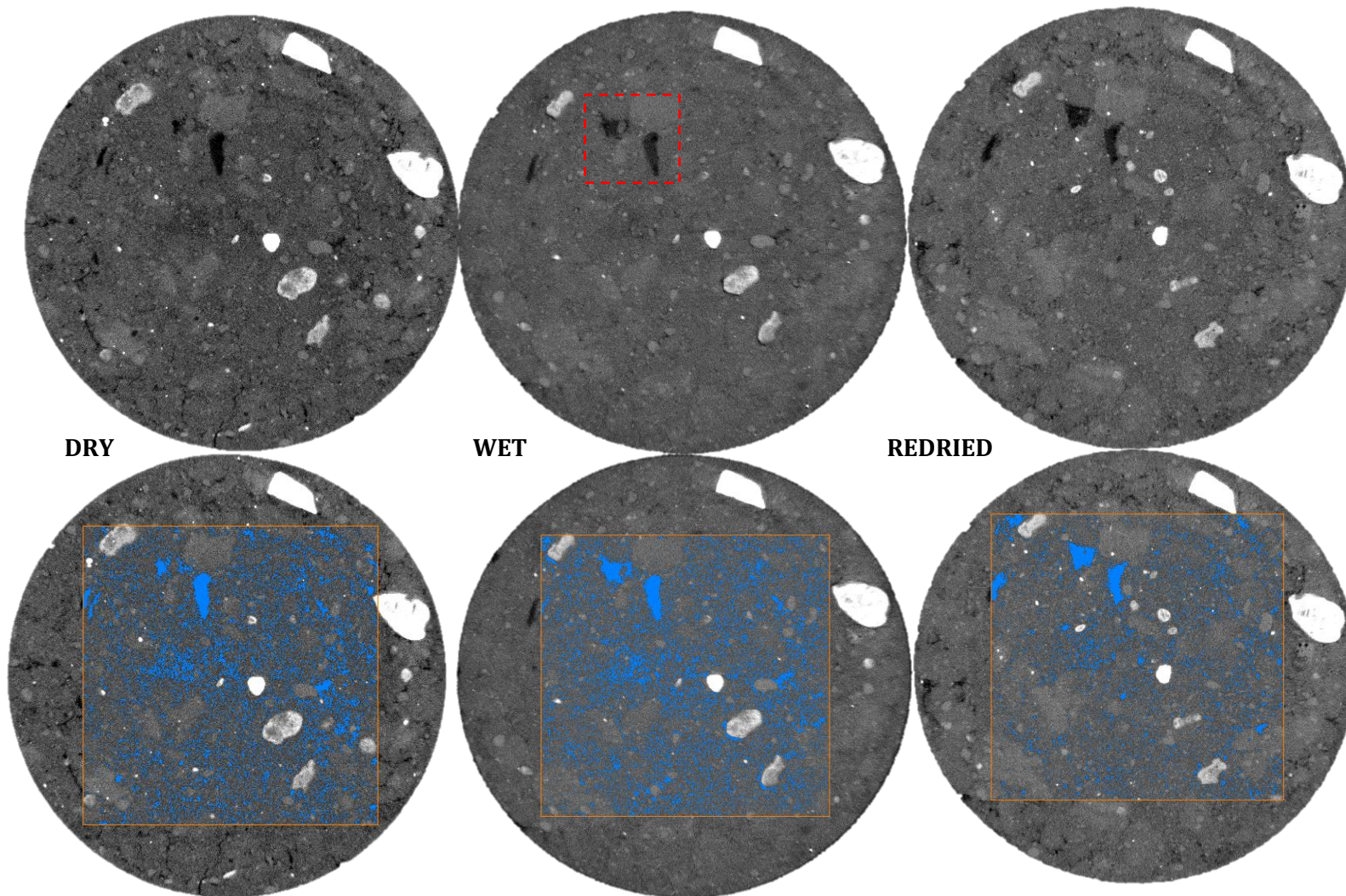
AM11 (70S 30C) has the highest initial void ratio, and largest porosity change (+0.26 compared to soil of +0.01), which is perhaps a reflection of the large pores in the sample as a result of the addition of compost. For the 30% single compost amendment (AM11) the large increase in  $V_v$  and  $V_r$  is explained by decreasing bulk density and increase in sample volume when it is wetted as described in analysis in section 5.1.3.3. Although there is a large change in void ratio between dry and wet states, the compost amended sample returns to a similar void ratio when redried, suggesting that the compost may operate in isolation to improve the water holding capacity by increasing the void volume but it has no considerable effect on the soil structure.

The 30% single addition of WTR2<sub>d</sub> and WTR2<sub>w</sub> (AM12 and AM13 respectively) increases the pore volume (4390 and 3937  $\mu\text{m}^3$ ) in comparison to unamended soil (2127  $\mu\text{m}^3$ ) when the sample is wetted. Although both have a lower void ratio (0.291 and 0.312) than the single amendment with compost (0.459) when wet, it appears that the void ratio remains higher when the sample is re-dried when using WTR2<sub>w</sub> (0.235) in comparison with the use of WTR2<sub>d</sub> (0.088) or compost (0.208) as an amendment, where the void ratio almost returns to the starting value with the use of dried WTR. This effect may occur due to the cementing factor of wet WTR as described in Chapter 3 (Basim, 1999; Moodley & Hughes, 2006), which means that once the soil is wetted, the soil architecture is preferentially changed such that it experiences less shrinkage and retains porosity when redried.

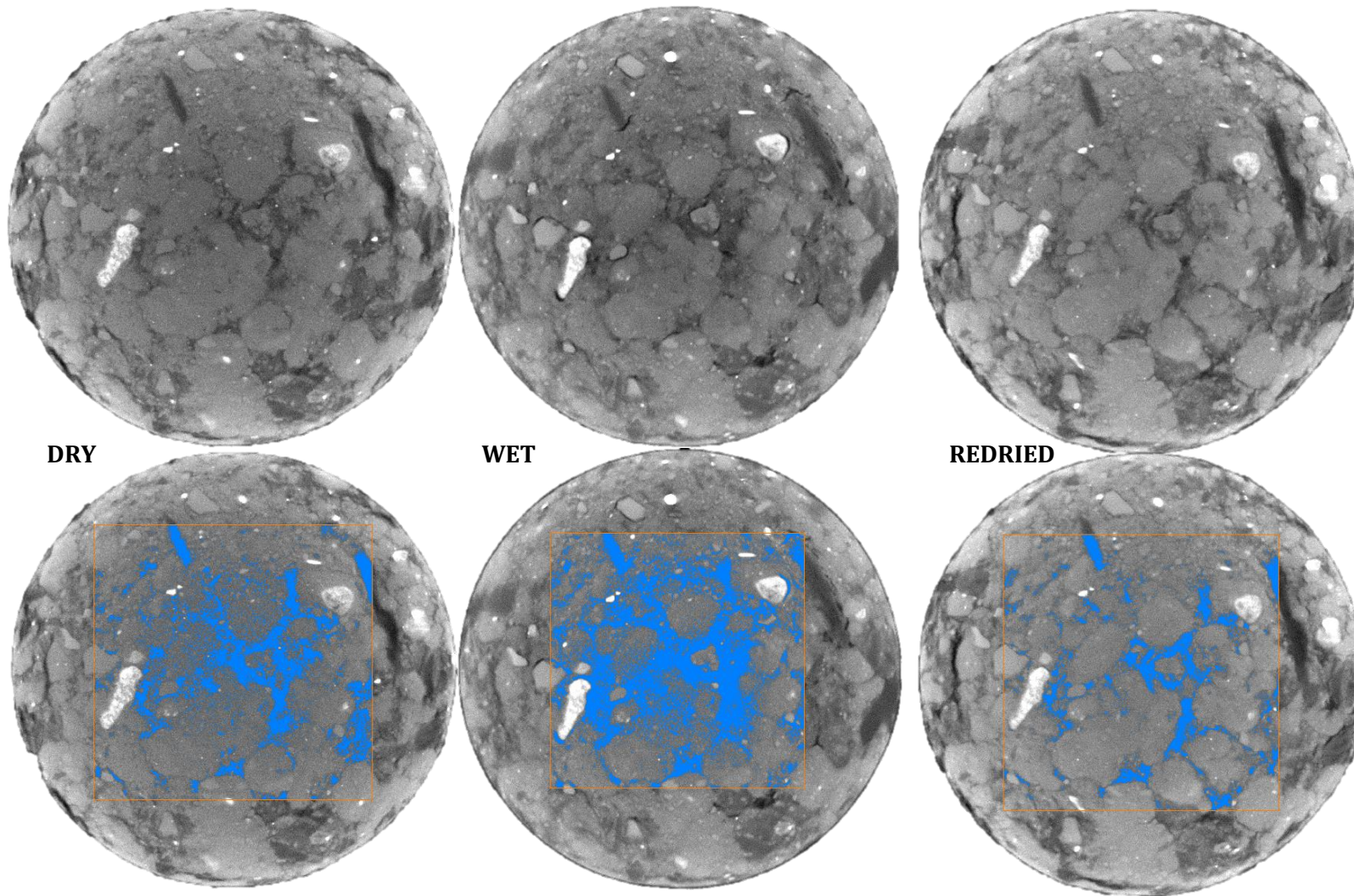
Despite co-amended samples having the highest wet void ratio (0.229 and 0.307) in comparison to unamended soil (0.127), the dry XRCT derived porosities for both

AM14 and AM15 (0.089 and 0.102) are lower than unamended soil (0.117). In addition, the wet XRCT derived porosity values for co-amendments are 0.06 and 0.005 lower than the values for single amendments. One might expect the co-amendment of WTR and compost to have a void volume & void ratio between the values of compost and single amendments. Nonetheless, as discussed for Trial 4 WHC results (section 5.1.3), the co-amended samples importantly do not lose porosity as a result of volume decrease when redried, unlike the unamended soil, single compost or WTR2<sub>d</sub> amendments. The low void volume and void ratios values derived for co-amendment (AM14 and AM15) may be due to thresholding issues as the higher proportion of low density materials makes the selection of a 'sensible' threshold difficult as a greater proportion of the sample is highlighted. As the attenuation of organic matter and water is fairly similar, due to the resolution, separating pores in these materials is far more subjective than more homogeneous materials (unamended soil) or in samples where there are clear boundaries between each material (such as AM12, Figure 102).

The porosity values derived from XRCT in Table 33 are based on the thresholding of the entire 3D volume i.e. >2000 slices. Figures 100-105 show a single representative slice from each sample in their dry, wet and re-dried states as well as the same slice with the auto-thresholded pore space overlain to demonstrate the differences between each sample. For each image, the dry sample slice has a diameter of 38 mm and wet and re-dried samples have a diameter of between 38 and 42 mm. The size differences in samples at each stage of wetting has been clearly outlined in Chapter 5 (section 5.1.3), therefore these images are sized uniformly for visualisation purposes. To provide a comparison between scans, the contrast in each image was adjusted such that each bright spot (soil mineral) has an approximately equal white value. Dried samples appear lighter due to a generally higher attenuation of the sample as the majority of the sample is comprised of soil mineral with small volumes of air or water, which are darker. In wet samples, the water has a lower attenuation value than solid particles, thereby giving the entire image a dark appearance.



*Figure 100: Unamended soil (Soil2) images from XRCT scanning at 25  $\mu\text{m}$  resolution (top) soil in dry, wet and re-dried states (bottom). Blue shading indicates areas selected by auto thresholding using a manual threshold.*



*Figure 101: 30% single compost amendment (AM11) images from XRCT scanning at 25  $\mu\text{m}$  resolution (top) soil in dry, wet and re-dried states (bottom). Blue shading indicates areas selected by auto-thresholding using a manual threshold.*

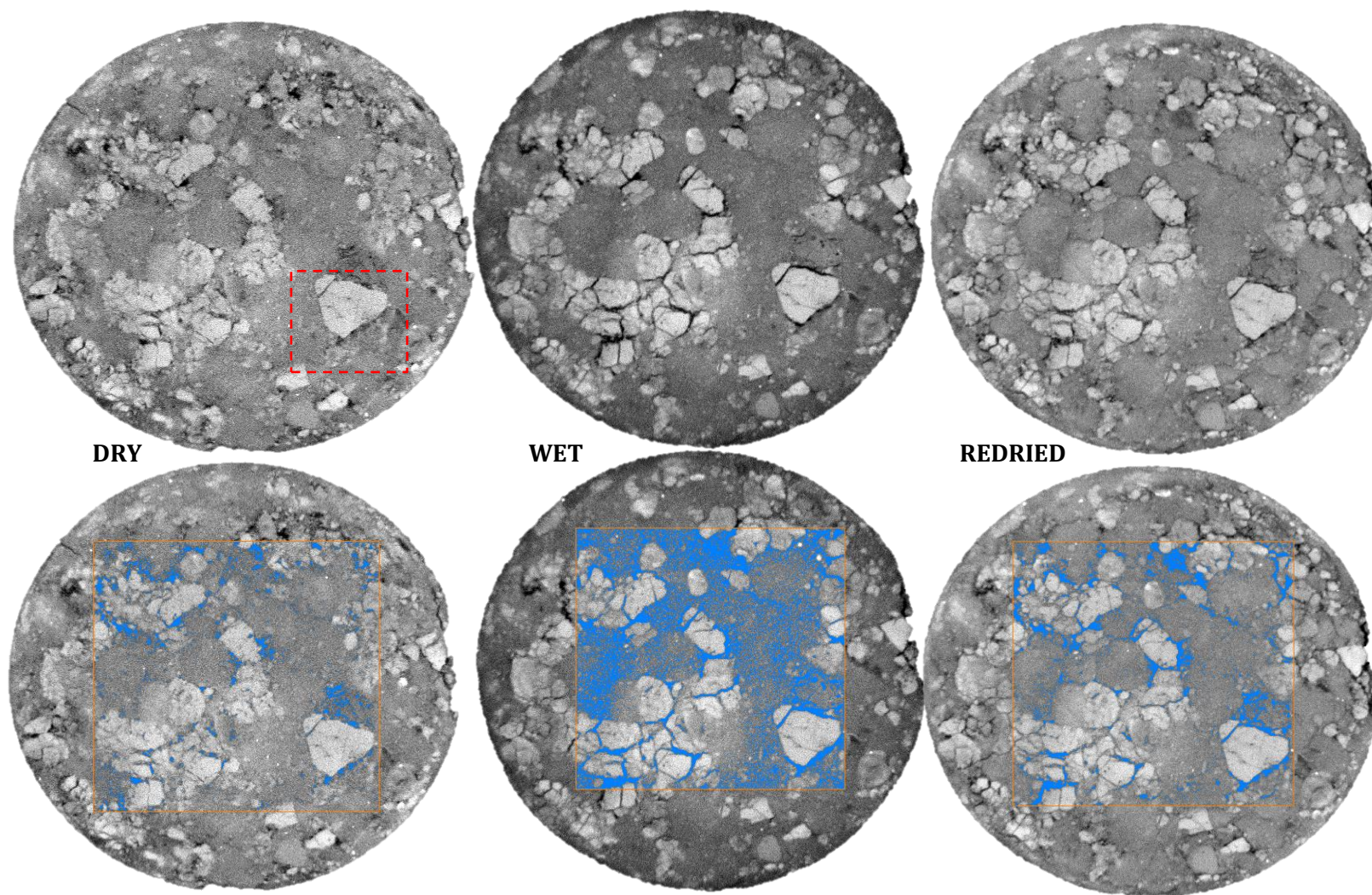
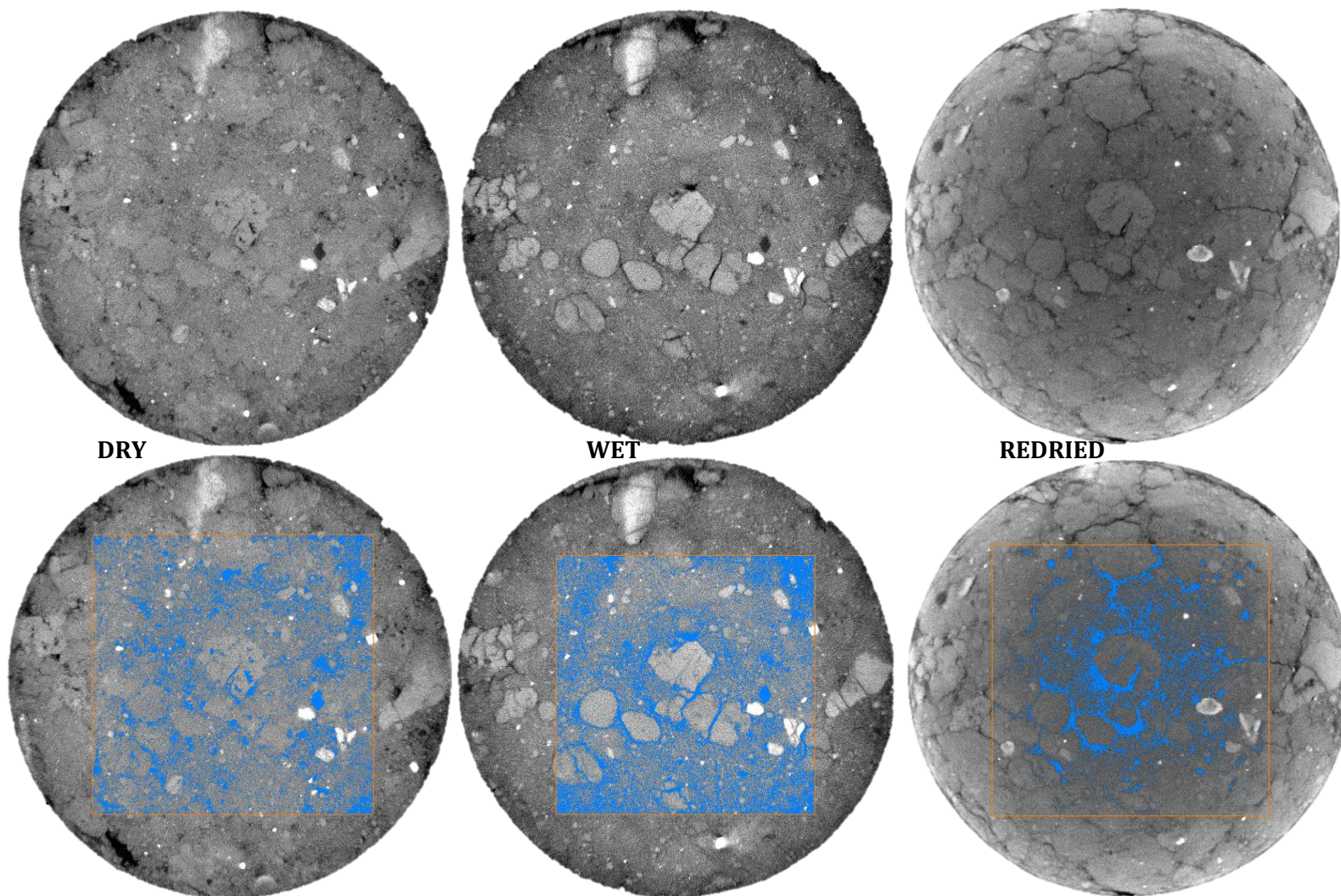


Figure 102: 30% single  $WTR_{2d}$  (AM12) images from XRCT scanning at  $25\ \mu\text{m}$  resolution (top) soil in dry, wet and re-dried states (bottom). Blue shading indicates areas selected by auto-thresholding using a manual threshold.

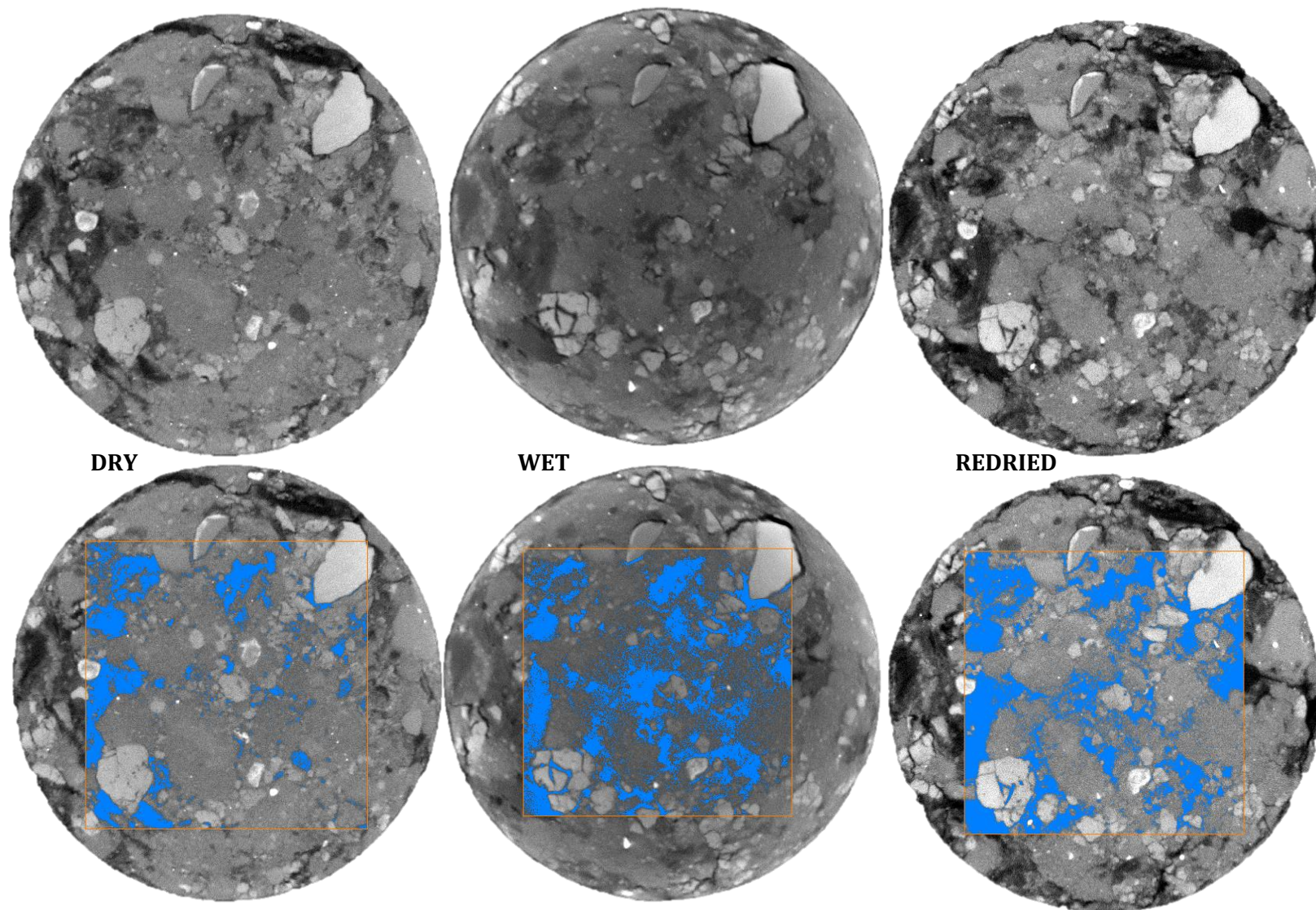
XRCT images of Soil2 are shown in Figure 100, where the top row shows a single slice of the 3D volume for the dry, wet and re-dried sample. In these images, large individual particles can be easily identified as well as pores, presented as dark spots. When the sample is wet, the image becomes much less clear as the water has in effect blurred the image; the partial volume effect means that voxels containing both water and higher attenuating material have been averaged to a lower value and voids have increased in their attenuation due to water presence. This gives the image an overall narrower histogram. Small pieces of organic matter present in the soil sample (red box) have clearly swelled when water has been added, and remained at a higher volume & lower bulk density than the original sample.

In Figure 101 (AM11, 70S 30C), the presence of organic matter is clear to see, where there are large portions of dark, irregularly shaped pieces of material which surround soil aggregates. In the wet image, these patches of organic matter swell and due to an increase in water in these materials they have a higher attenuation than in the dry image. The difference can also be easily identified when viewing the three images at the bottom of the figure with auto-thresholding overlay.

Figure 102 (AM12, 70S 30WTR2<sub>a</sub>) is an exceptionally informative series of images, and provides an indication of what occurs inside the sample as it goes through a wetting phase. In the dry images solid pieces of WTR2<sub>a</sub> are clearly visible and are to some degree distinguishable from soil mineral matter due to their irregular shape, such as the triangular piece highlighted in the red box. Small cracks in the dried WTR are apparent, presumably from the process of core production, and once the sample has been wetted these defects are then filled with water and expand. Interestingly once the sample is redried, the voids created when the sample was wet remain considerably wider than in the original state, despite the values in Table 33 suggesting that this porosity is lost upon drying. It is difficult to determine why such large voids are created around the dried WTR but as discussed in Chapter 3 (WTR) once dried WTR is immersed in water, gas is emitted while the dried WTR breaks down into smaller fragments.



*Figure 103: 30% single WTR<sub>2w</sub> amendment (AM13) images from XRCT scanning at 25  $\mu\text{m}$  resolution (top) soil in dry, wet and re-dried states (bottom). Blue shading indicates areas selected by auto-thresholding using a manual threshold.*



*Figure 104: 30% co-amendment with compost and WTR2<sub>a</sub> (AM14) images from XRCT scanning at 25  $\mu$ m resolution (top) soil in dry, wet and re-dried states (bottom). Blue shading indicates areas selected by auto-thresholding using a manual threshold.*

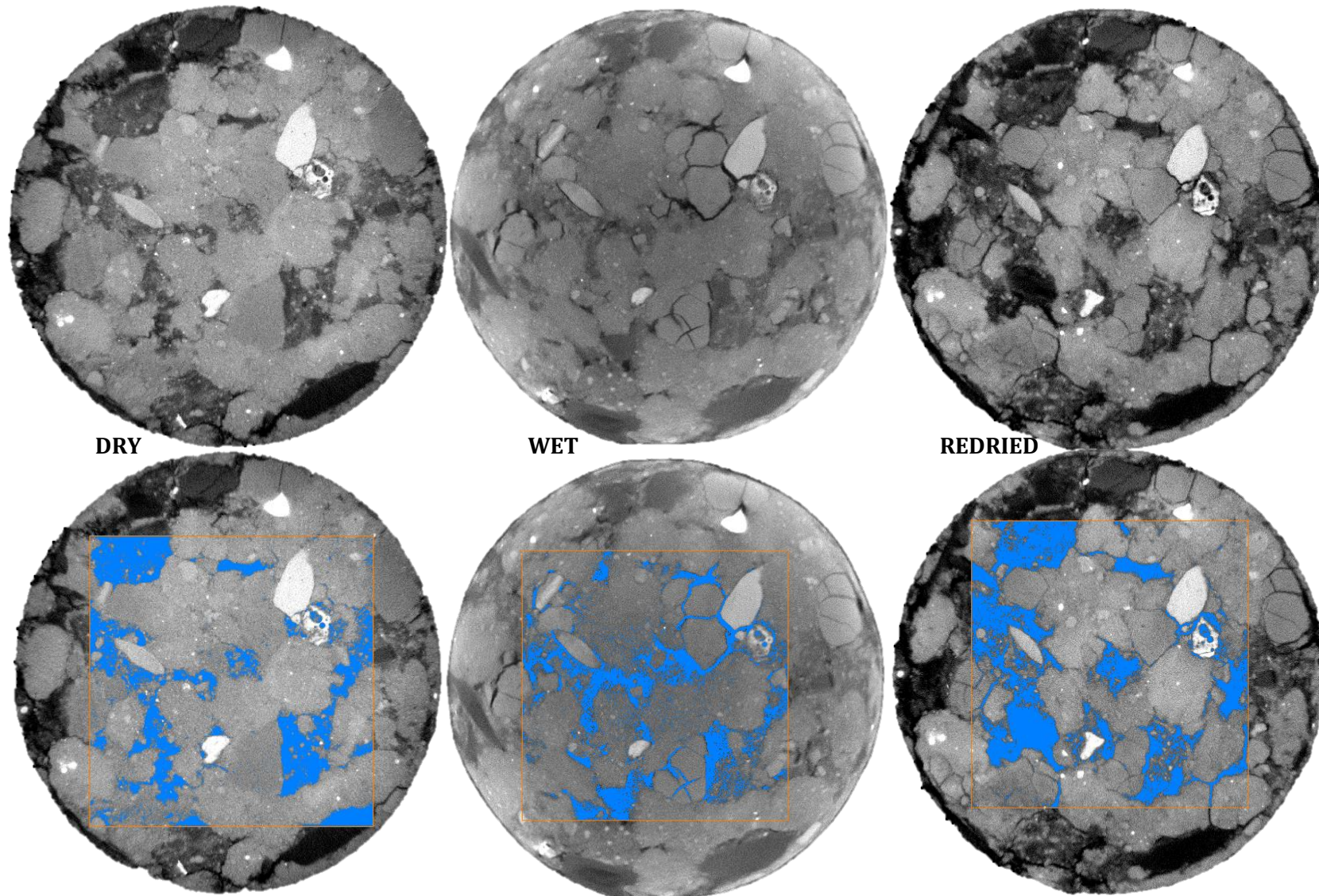


Figure 105: 30% co-amendment with compost and  $WTR2_w$  (AM15) images from XRCT scanning at  $25\ \mu\text{m}$  resolution (top) soil in dry, wet and re-dried states (bottom). Blue shading indicates areas selected by auto-thresholding using a manual threshold.

Figure 103 shows the 30% single amendment of WTR2<sub>w</sub>, where the sludge has been mixed through soil and then air-dried to 17.5% water content before being compacted into a core. WTR2<sub>w</sub> tested immediately after export from the water treatment works, as discussed in Chapter 3 (3.2.1) comprises particles ranging between 1nm and 1 µm in size (Bohn *et al.*, 1995) and Chapter 4 (4.3.2) where 75% of particle pass a 74 µm sieve (Raghu *et al.*, 1995). However, importantly there appear to be small dried pieces present in the sample, suggesting that the mixing process doesn't necessarily spread the fine grained material homogeneously through the soil, and over time clumps of WTR are able to dry as larger aggregates which appear otherwise identically to the dried WTR in Figure 100. As a result, the XRCT image shows that the WTR2<sub>w</sub> is mixed thoroughly with the soil, with some areas of larger WTR particles. This may indicate why, in the triaxial testing in the previous chapter, that WTR amendments have similar frictional response but different dilatancy behaviours at lower pressures.

The attenuation of water in the sample in the wet image in Figure 103 is clear, as non-absorptive materials stand out against a darker background. Again, the dried WTR pieces have fractured into smaller sized particles and created a small void between the pieces. Although the image quality of the re-dried material is compromised by beam hardening (giving an artificially higher attenuation coefficient on the outer surface of the core), it appears that the channels opened up by water during the wetting have remained open, supporting the hypothesis that WTR added in a wet form may create a stronger soil architecture. The analysis of Figure 102 and 103 go some way into describing why we saw such a difference in the hydraulic conductivity of samples with both WTR2<sub>d</sub> and WTR2<sub>w</sub> in comparison with samples containing compost. It is apparent that during wetting, the effect of WTR is to open up large flow paths, through which water may flow with greater speed than through varying size of pore throats.

Figures 104 and 105 show the effect of wetting and drying on 30% co-amended samples using WTR2<sub>d</sub> and WTR2<sub>w</sub>, respectively. In Figure 102 large pieces of compost are still visible in the dry, wet and redried sample; however identifying the WTR in the dry sample is much more difficult. Manual identification is required to recognise the WTR pieces by their shape and fracturing characteristics when they are wetted. Attempts to threshold the histogram to identify WTR in this

instance does not work due to the similar attenuation with soil mineral. The wet image in Figure 104 reflects the high water holding capacity of AM14 as shown in GWC testing in section 5.1.3, where again large voids appear around the dried pieces of WTR and the attenuation of compost increases significantly as it takes in water. When AM14 redries, the void spaces remain apparent and soil aggregates appear slightly more distinguishable from organic components than in the original image. In Figure 105, the wet WTR has effectively smoothed the image as it has been evenly mixed with the soil and compost (with the exception of some dried pieces) and as a result the differentiation between the three materials in the sample is very difficult.

#### **6.4.4 High resolution scanning, Stellenbosch**

In August 2016, single micron resolution images of WTR2<sub>a</sub> were obtained using a Phoenix VtomeXM machine, using a power of 150kV and current of 90W. The scan took approximately 2 hours and gave a resolution of 6  $\mu\text{m}$ . Five small >6.3 mm pieces of the air-dried WTR2<sub>a</sub> were scanned in a small plastic vial for the first scan, after which the WTR2<sub>a</sub> was completely submerged in water and sealed for a 3 hours before the sample was rescanned. This was, in practice, sufficient time for any immediate water movement into the specimen, without compromising the integrity of the solids. As discussed in Chapter 3, in previous submersion tests, the WTR breaks down into smaller fragments while emitting air bubbles, after which the smaller pieces remain unchanged in solution.

High resolution scanning of the WTR2<sub>a</sub> revealed some key features of this material in its dried form, and what changes occur once the dried piece of WTR is then submerged in water. Image processing had few steps;

1. Firstly the image had an adaptive gauss filter applied to remove image noise and the background (air or water).
2. To calculate the pore volumes, the surface of the particles was determined using the erode/dilate tool.
3. Defect surface determination was calculated.
4. Regions of interest were extracted and inverted from steps 2&3, on which pore/inclusion analysis was run.

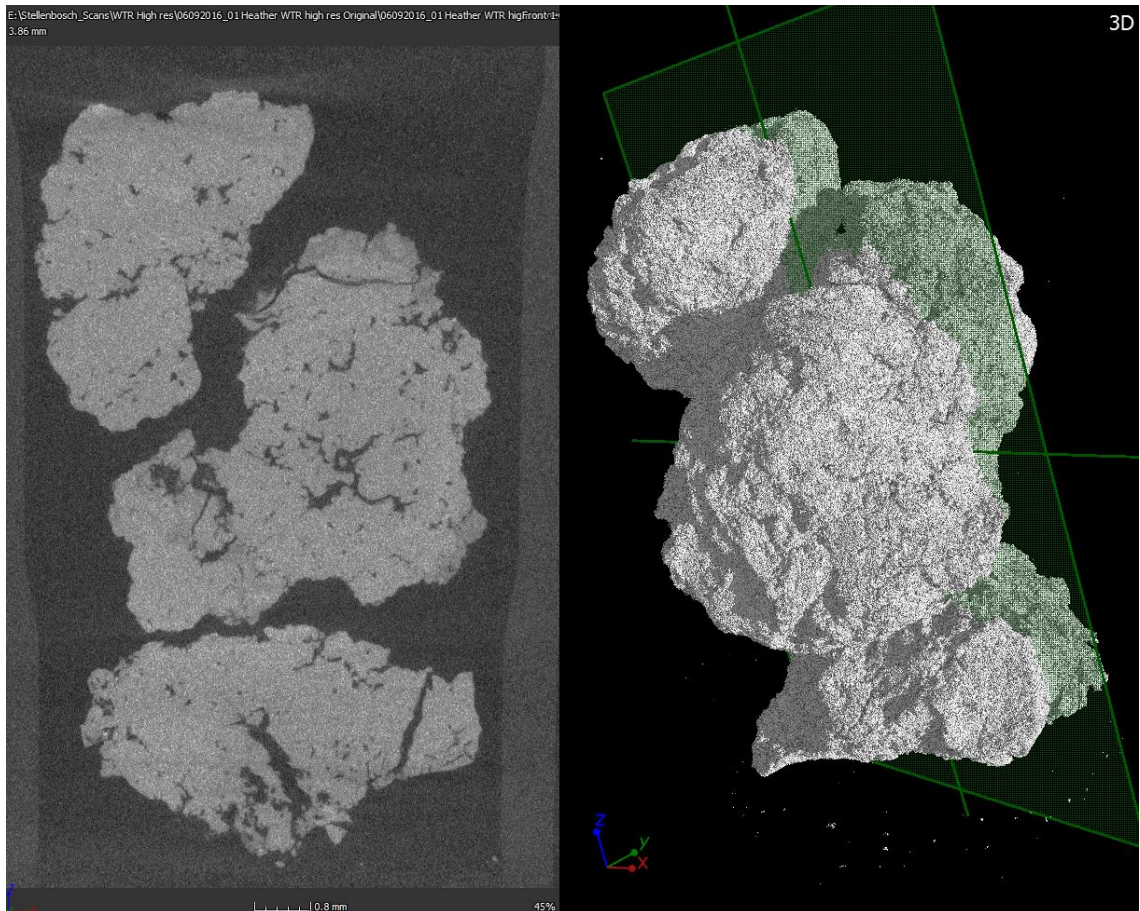
	WTR2 <sub>a</sub> 6 μm	WTR2 <sub>a</sub> WET 6 μm
Total volume of material (mm <sup>3</sup> )	136.81	103.55
Total volume of pores (defect volume, mm <sup>3</sup> )	13.45	11.29
Defect volume ratio %	8.95	9.83
Volume of voids connected to outside(mm <sup>3</sup> )	13.34	10.64
Volume of voids disconnected (mm <sup>3</sup> )	0.112	0.683
Surface of voids (mm <sup>2</sup> )	1499.34	1373.42
Number of defects	32438	18264
Number of defects per volume	237.10	176.38
Maximum defect size (mm <sup>3</sup> )	7.83	9.76
Surface of voids to volume	10.96	13.26
Pore size diameters	<i>Count</i>	<i>Count</i>
<14.756/14.77 μm	27964	15725
<92.86/112.22 μm	3040	1805
<170.97/209.67 μm	782	394
<249.07/307.12 μm	279	186
<327.175/404.47 μm	150	59

*Table 34: Summary statistics of WTR analysis from Volume Graphics 3.2 porosity/inclusion analysis*

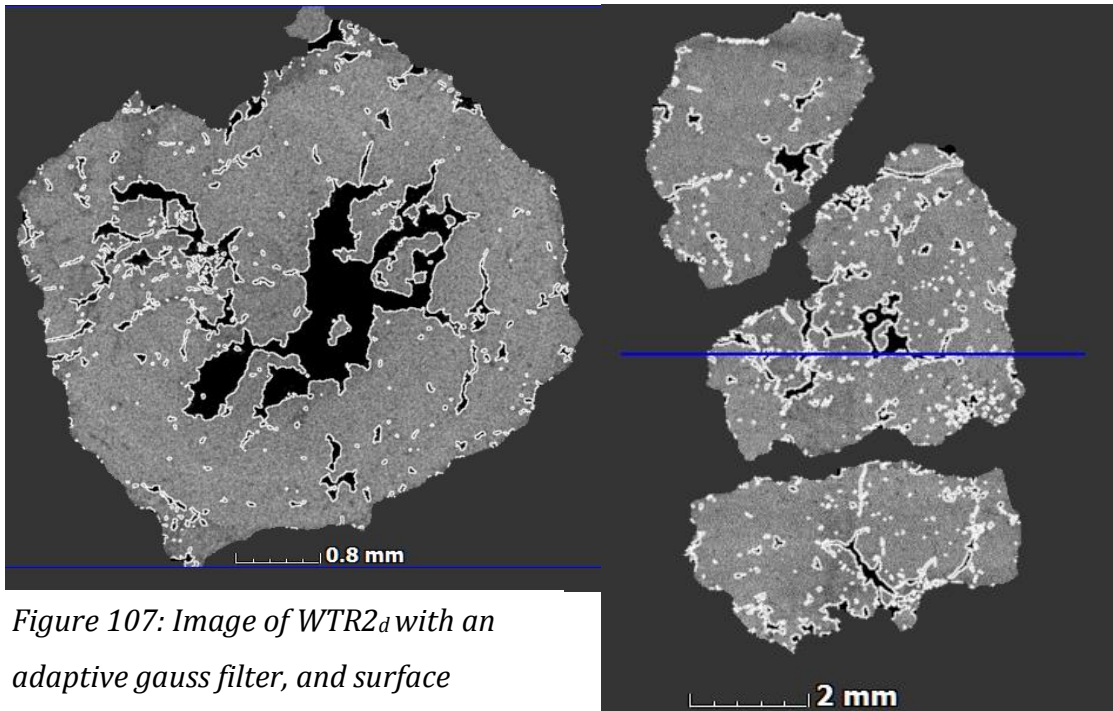
As shown in Table 34, the majority of pores or defects present in the WTR2<sub>a</sub> are connected to the outside of the particle and are essentially cracks into the centre of the material. A very small proportion of pores (0.008%) are entirely disconnected from the surface in the dried state, but when wetted the volume of disconnected pores increases by a factor of 10 (to 0.06%), which is likely to be because of mass fracturing and breakdown of the original dried pieces, which leaves a greater proportion of disconnected volume of pores. The total volume of the ‘wetted’ WTR is lower than the original volume due to pieces breaking off into very fine fragments. In comparison a uniform block of material, the effective surface area of the WTR2<sub>a</sub> particle is exceptionally large due to the effect of surface connected voids; 1499 cm<sup>2</sup> when dry and 1373 mm<sup>2</sup> when wetted where in comparison a cube of equal volumes would have surface areas of 159.15 cm<sup>2</sup> and 131.98 cm<sup>2</sup> respectively. This high surface area is important for chemical interactions as well

as providing a surface on which to hold water, increasing the water holding capacity of the soil.

Figure 106 shows an unprocessed image of the WTR2<sub>a</sub> in high resolution (6 μm) and provides a good indication of the crack structure within the sample. As outlined in Table 33, 99.2% of the identifiable voids (shown in black) are connected to the outside surface of the material, also highlighted in Figure 107. The irregular external shape of the particle as a result of drying is well presented.



*Figure 106: Unfiltered XRCT image of WTR2<sub>a</sub> at a resolution of 6 μm (left) and a 3D reconstruction of the 3D surface of the particle, where the green slice indicates the position of the left image*



*Figure 107: Image of WTR2<sub>a</sub> with an adaptive gauss filter, and surface determination of pores/cracks; (right) single X axis slice and (left) single Y axis slice, where the blue line indicates the position of the X axis slice*

Figure 107 shows the surface determination of voids/defects; interestingly, although the large pore (~1 mm) in the centre of the particle appears isolated in this single 2D slice, 3D analysis reveals that it is connected to the external surface of the WTR2<sub>a</sub> by tiny cracks (<10 μm) in the solid material. The variable size and shape of pores (defects) in the sample is more clearly shown in Figure 108, which highlights the maximum diameter of each defect. In this image we can see that defects that almost span the entire width of the particle are present, suggesting that as the WTR2<sub>a</sub> dries it traps air within its structure, which may slowly escape once the material is solidified. This may also explain why the material emits bubbles when submerged, as the cracks to these pockets are expanded and the total fracture causes the air to be released.

Figure 108 highlights the porosity in dried WTR, where red pores are at the larger end of the scale (6-7 mm maximum diameter) and blue pores are 0.01-1.6 mm. The central right photo shows the 3D sample as a whole, and shows that there is a heterogeneous spread of pore sizes across the sample. Figure 107 shows what happens to the dried particle once it has been submerged in water, critically the expansion of internal pores. A direct comparison between scans is difficult as the

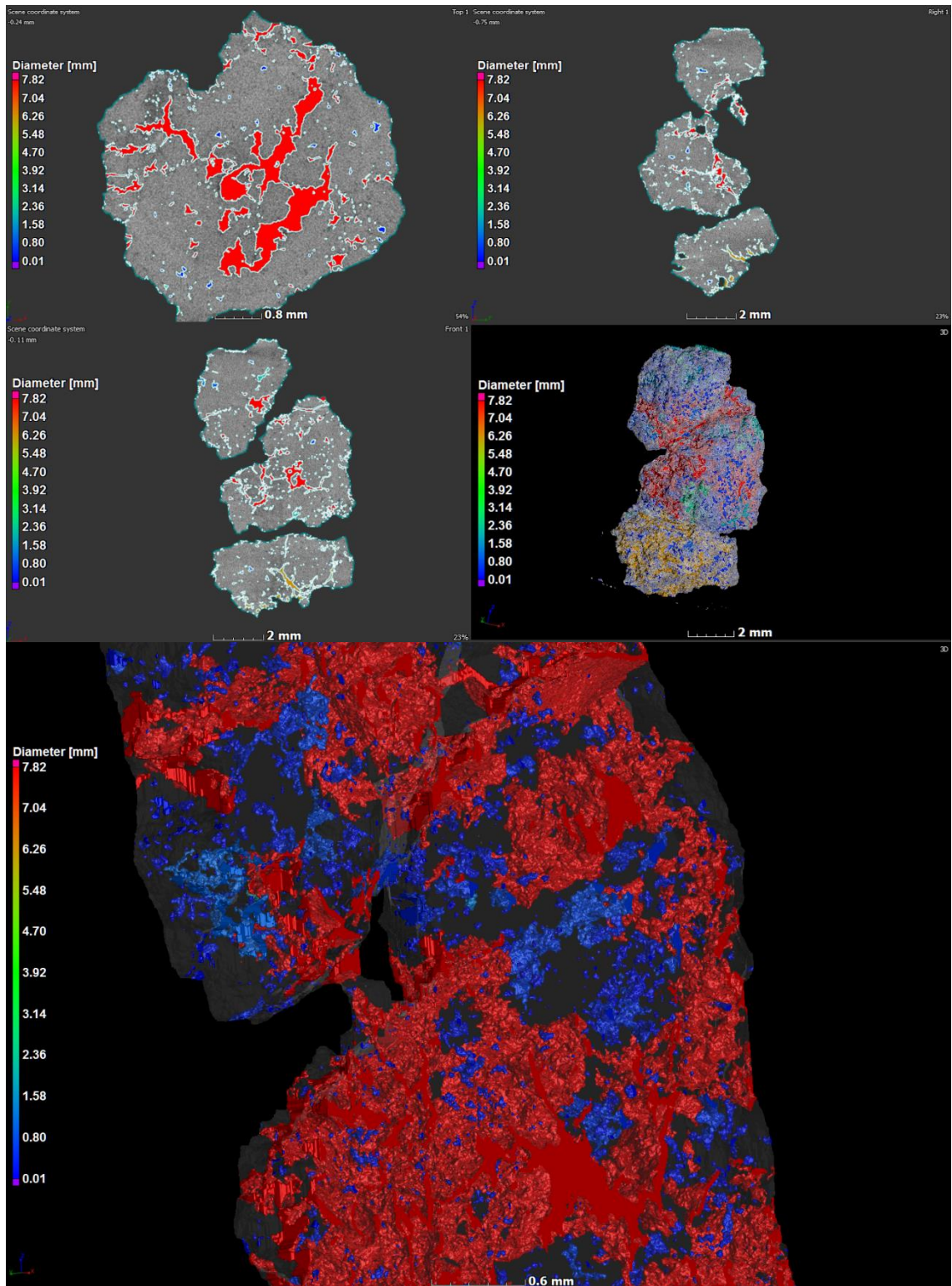


Figure 108: XRCT porosity/inclusion analysis on WTR2d

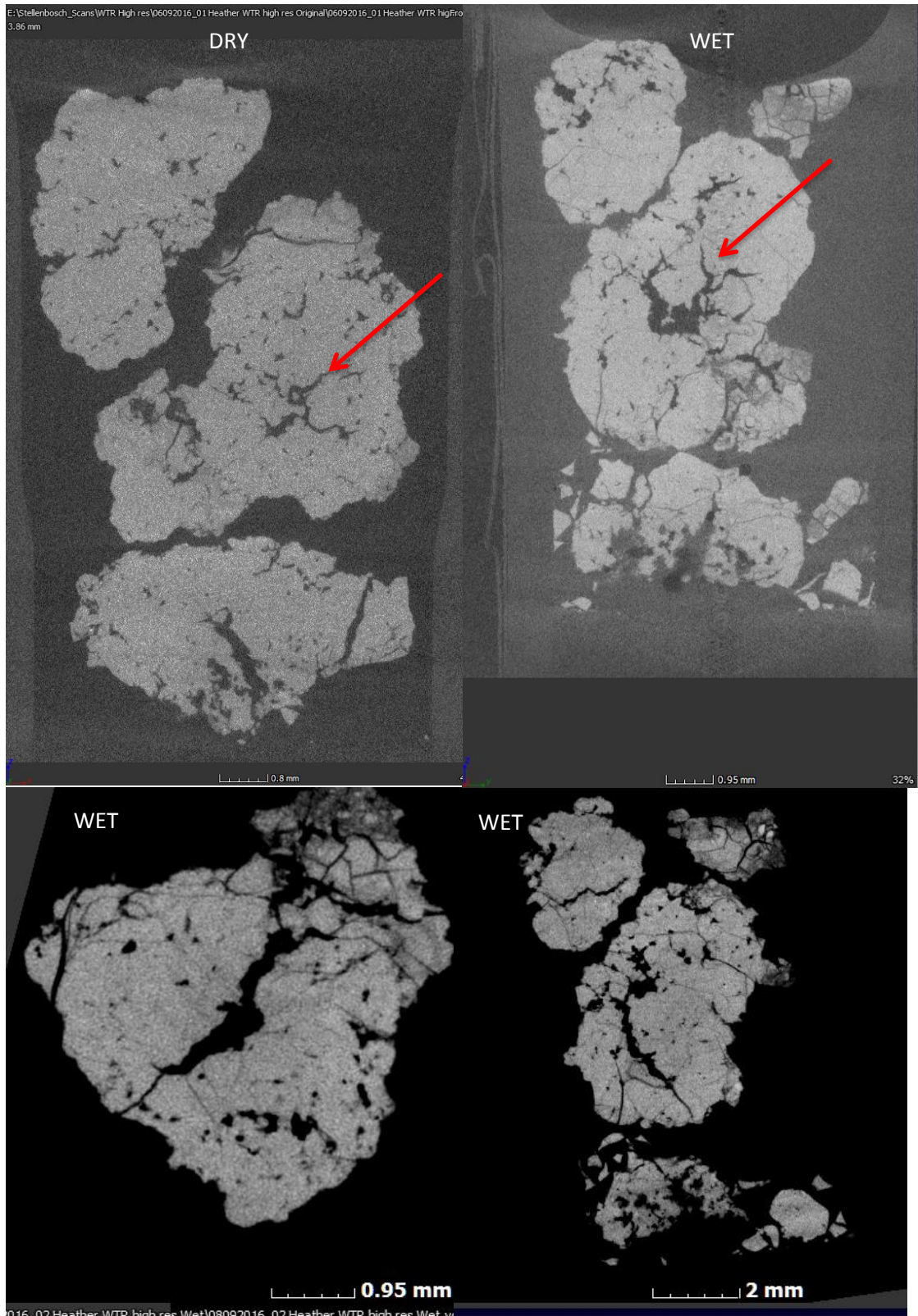


Figure 109: (top left) Unprocessed image of WTR2a and (top right) unprocessed image of WTR2a having been submerged in water. Images with adaptive gauss filter applied to remove background noise, (bottom left) single X axis slice of submerged WTR2a and (bottom right) single Y axis slice of submerged WTR2a. Red arrow indicates the growth of a central defect when the sample is wetted.

particles moved and broke apart after wetting and therefore the image shows a representative slice from both the dry and wet WTR that show the largest defect in the centre of the particle. It is difficult to ascertain if water has entered the defect, although considering the difference in attenuation value between the surrounding water and inside the defect it appears not; therefore, this increase in defect size is not an influence of water pressure. At the bottom of Figure 109, the image has been filtered to remove water noise and the defects are more apparent. The XRCT scan shows that the three main particles of the WTR2<sub>d</sub> have fractured and created a number of smaller pieces at the base of the material. Statistics from the defect analysis shows little difference between the size distribution of the defects in the particles in the dry state and submerged state, where 80% of the pores were <0.5 mm and the remaining 20% are the large defects easily identifiable in the XRCT images.

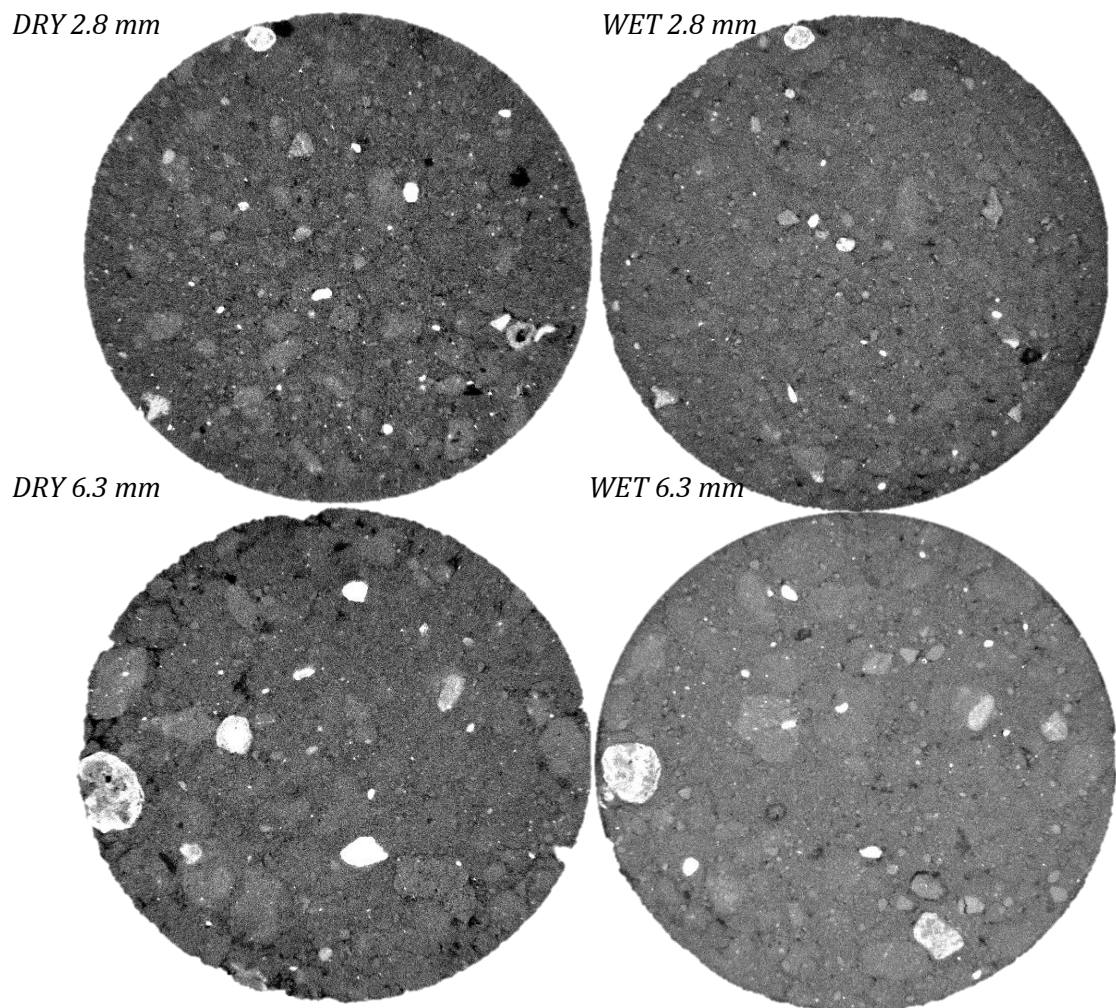
#### **6.4.5 High resolution scanning in Durham**

Having scanned samples from Experiment 4 at a resolution of 25 µm, freshly produced samples of Soil2 and AM14 (70S 15C 15WTR2<sub>d</sub>) were scanned in 2018 with the objective of achieving the highest resolution possible and to optimise machine settings and enable better visualisation. In order to achieve this, samples of Soil2 and AM14 were produced using the materials from Experiment 4 (17.5%WC and sieved to 6.3 mm), according to the split mould method (section 4.4.1). The core diameter remained the same at 38 mm, but the height was halved to reduce the size of samples (giving 38 mm Ø x 38 mm height). In addition, two further samples of Soil2 and AM14 were produced to the same dimensions, where the pre-mixed material was further sieved to 2.8 mm. This was to test the effect of different particle size on the packing of grains and pore size distribution.

Under the supervision of Dr Kate Dobson (independent research fellow in Earth Sciences, Durham University), the samples were scanned to a resolution of 20 µm while dry, and then scanned again after being saturated with water. The machine parameters were changed to 90V and 10 W so that lower density materials were better represented. The images were processed using the same method as described in section 6.4.3. Void ratios, calculated as a ratio of the volume of the cropped cube (from the centre of the sample) to the volume of material classified

as pores by thresholding. Figures 110-112 show the high image quality obtained at a better resolution using smaller samples and different machine parameters.

In Figure 110, the images show a distinct difference in the structure of soil sieved to 2.8 mm and 6.3 mm, with larger pores and soil mineral particles present in the latter. In the images of 2.8 mm soil small aggregates are more apparent than in the coarser sieved material. The materials appear much better defined in the image of 6.3 mm sieved material in comparison with 2.8 mm, which may be a function of material size vs scan resolution. However, we are still not able to visually discriminate between organic matter and pore space, and between WTR2<sub>a</sub> and soil due to similar attenuation values, nor are we able to use the unimodal histogram to aid discrimination.



*Figure 110: XRCT image of Soil2 in dry and wet states, sieved to 2.8 mm compared to Soil2 sieved to 6.3 mm at a resolution of 20  $\mu$ m.*

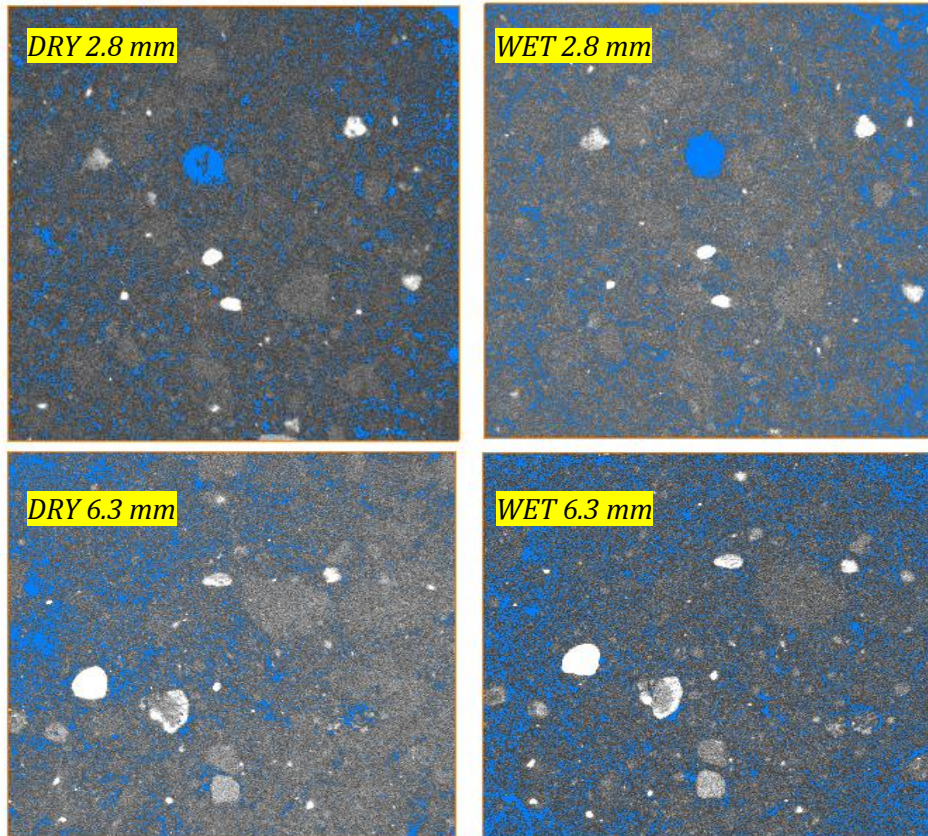


Figure 111: Soil2 2.8mm (dry vs wet, top) and Soil2 6.3 mm (dry vs wet, bottom) thresholding

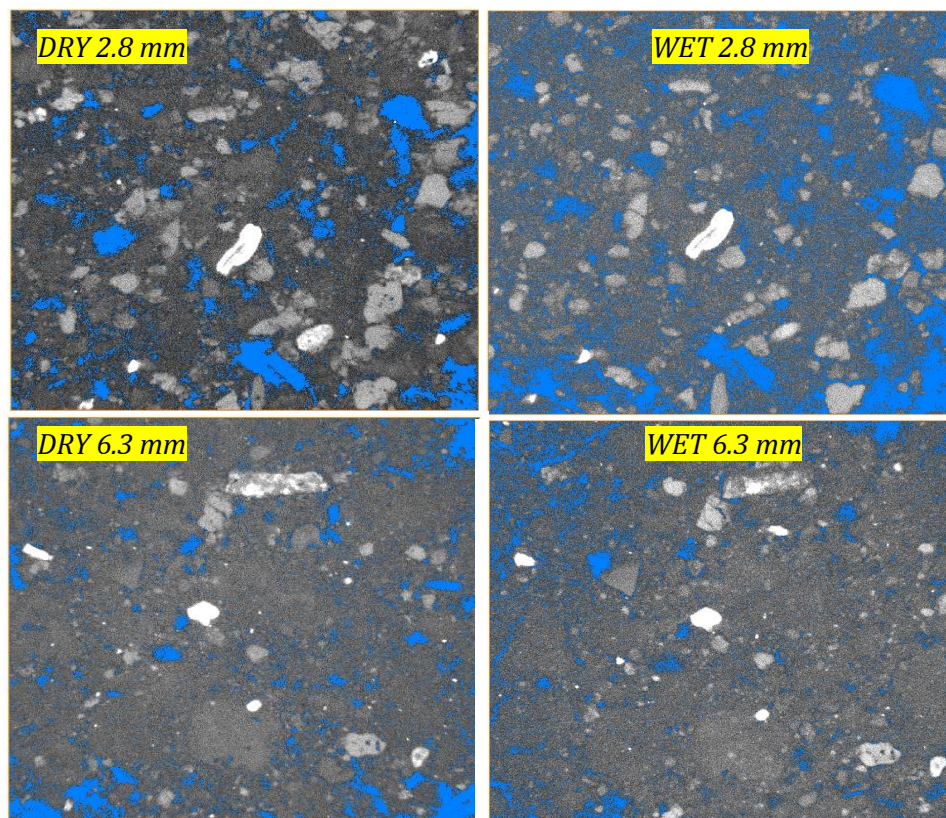


Figure 112: AM12 2.8mm (dry vs wet, top) and AM14 6.3 mm (dry vs wet, bottom) thresholding

Figures 111 and 112 show the thresholding process on Soil 2 and AM14 in wet and dry states. In each case the wet sample has a greater number of voids due to swelling of the samples as water fills each pore, however there is a large difference in values of pore volume and void ratio obtained between 2.8 mm sieved samples and 6.3 mm samples. This can be attributed again to the fact that as we threshold the samples, the thresholding process favours samples with large and obvious pores as the selection of a critical value is user based. Figure 113 provides a graphical representation of this difference, where void ratio values are compared with the values derived from triaxial testing. As discussed previously, the values for triaxial void ratio are higher as they include the micro-pores that are not quantifiable by XRCT, and are at full saturation, whereas samples in the XRCT have only been saturated under gravity. Figure 113 clearly shows that in a dry format, the void ratios of samples at 6.3 mm are higher than at 2.8 mm, whereas when the samples are wet the void ratio is higher in 6.3 mm samples, presumably because the pores are larger and therefore we can better visualise the water.

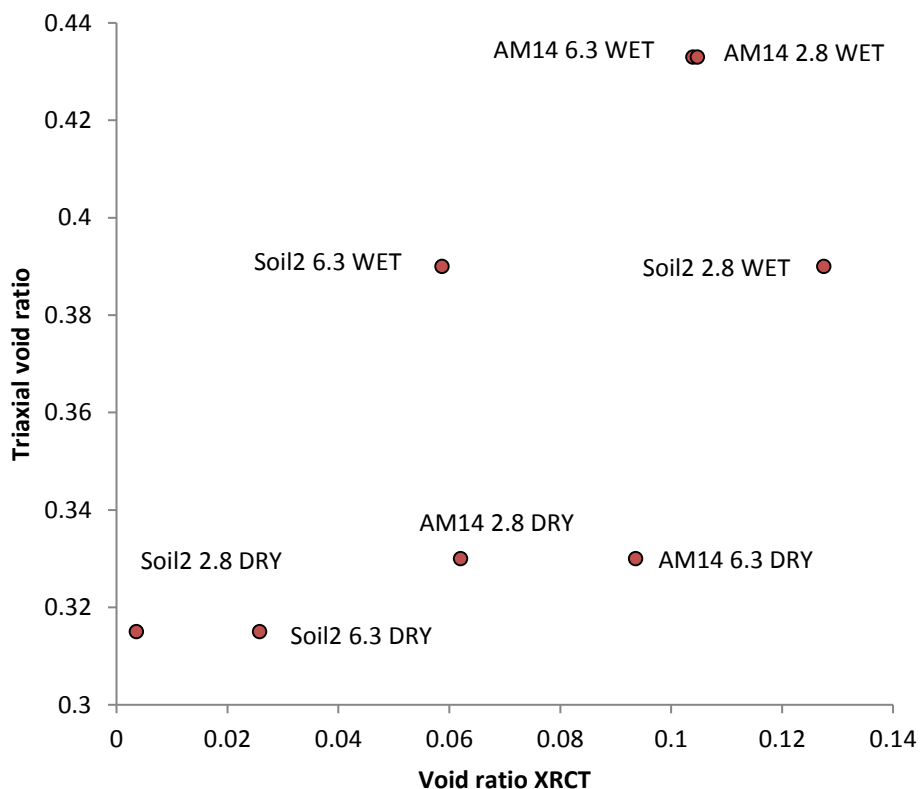


Figure 113: Comparison of void ratios derived from XRCT and triaxial cell, where Soil2 is unamended soil and AM14 is a 30% co-amendment with WTR2<sub>a</sub>

### ***6.5 Discussion and concluding remarks***

This chapter has demonstrated the potential of the application of XRCT to the study of soils. The method provides a non-destructive measurement tool for the visualisation of 3D internal soil structures including the pore network, and arrangement of soil particles, and allows us to view the same sample at different moisture contents. The use of XRCT on soil is currently limited by the pitfalls of the technology (artefacts, image noise etc), the stand-off between resolution, sample size and representative volumes, and a lack of clarity in guides to processing the vast data sets produced by CT scanners, owing to the sparse but growing field of related literature. Although researchers can readily complete the segmentation of relatively binary images to a high degree of accuracy with validation from empirical measurements, the results obtained from attempts to binarize or segment the soil volume into three phases are lacking. Although the set up of samples, scan settings and initial analysis required a skilled technician, with a little guidance this technology is accessible to new users and therefore the research gaps identified in the previous discussion should close as rapidly as the technology was adopted for this novel area of research.

The use of XRCT in this thesis was exploratory and focused on discovering which important soil parameters could be calculated or determined using the technology. The aims set out at the beginning of the research firstly revolved around characterising soil structure with the addition of WTR2<sub>a</sub> and WTR2<sub>w</sub> as single amendments and as part of a co-amendment. Secondary to this, the research was aimed to determine if WTR could be characterised in the images, including visualisation of the internal structure & porosity of WTR2<sub>a</sub>, how WTR reacts when exposed to water and in testing the ability of tracers to enhance our ability to characterise pore space. Initial scans conducted to a resolution of 25 µm over a single wetting cycle, provided some critical information about the structural characteristics of amended soil, and in particular the behaviour of WTR2 through this process. It is clear that WTR2<sub>a</sub> experiences a failure that can be best described as fracturing when exposed to wetting, which creates voids around the particle that remain open when the soil is redried and may provide preferential flow paths that aid in infiltration and percolation should the sample be wetted again. Although the differentiation between organic matter and WTR was unsuccessful

from a computing point of view, we are able to see the aggregation of materials once the sample has been redried in co-amended samples.

In very high resolution scans of WTR2<sub>d</sub> conducted in Stellenbosch, it was determined that >99% of the defects or voids in the material were connected to the external surface of the material, providing the dried material with an exceptionally high surface to volume ratio (10.96 when dry and 13.26 when wet). It became apparent from the visual comparison of dry and submerged scans that the dried WTR fractures along the larger cracks and as a result the defects are widened. This may also explain why, upon submersion, dried WTR emits bubbles as air may become trapped in pockets as the WTR dries.

The use of iodine as a contrast agent was unfortunately not successful, and hence the reason that current research into the use of tracers in soils is sparse. The addition of highly attenuating contrast agent KI in water indiscriminately increased the attenuation of the entire soil mass such that porosity could not be effectively measured. Staining the organic matter or WTR directly with a gas or liquid prior to its inclusion in the co-amendment, or using a staining agent that only targets OM is likely to be one of the only methods to easily segment organic fractions from the remainder of the material. The combination of low-density organic matter and Fe oxides will further complicate attempts to identify the material based on the attenuation value of the material.

The final scans conducted on samples sieved to different sizes revealed little more information on the use of XRCT and confirmed that, even at the optimum machine settings and highest resolution currently possible for samples of the dimensions used, an in-depth analysis of porosity is only achievable for the largest of pores in the sample. In addition, the values in Table 33 highlight the vast differences in void ratio based on the analysis of scans of a similar material by the same user.

## **7. Weetslade Field Trial: Engineering soil for flood resilience proof of concept at the field scale**

### **7.1 Introduction**

As discussed in Chapter 3, although a number of studies have looked into the use of WTR as a soil amendment for the improvement of a variety of soil parameters, these values have only been determined in the laboratory or in small artificial plots on a singular soil type (Elliot & Dempsey, 1991), therefore their effect on different soil types on a field scale are currently unknown. Chapters 5 and 6 have outlined the effects of WTR on the water retention of soils and shear strength properties of soils with the application of single WTR<sub>d</sub> and WTR<sub>w</sub> and co-amendment of both these materials with compost. In the majority of testing, there was little difference in the performance of using WTR in the form in which it is exported from the water treatment plant (~20% solids, WTR2<sub>w</sub>), compared to the air dried and sieved format used for trials 3 and 4 (~ 20% water content, WTR2<sub>d</sub>). This suggests that there is no need for further processing of the WTR for use in this application, and that WTR can be used in its raw format without any detriment to the soil engineering properties in comparison to WTR that has been further processed. To the greatest extent possible, initial factors in the laboratory experiments, such as density, moisture content and sample mixing, have been thoroughly controlled in order to gain a meaningful understanding of the changes imparted. In reality the documentation of any beneficial or detrimental changes in a field setting is much more difficult to obtain. In order to validate the effects of WTR that have been observed in the laboratory, a full-scale field experiment was conducted at Weetslade Country Park, Northumberland in the early months of 2017.

#### **7.1.1 Background**

The field study, much like the rest of this thesis was built upon the ROBUST project, which provides a framework for community led regeneration of brownfield sites using sustainable technologies. Waste materials (such as the Fe based WTR used for research in Chapters 3-6) may be used in conjunction with compost to transform contaminated wastelands, which in turn improves the health and wellbeing of the local community. Weetslade Country Park is already a prime example of this, having been turned into a large public amenity from the remnants

of a coal-mining pit. The value of natural environments and the health benefits associated with green spaces is well known (Mitchell & Popham, 2007), however the value of soil as a critical partner in the implementation of sustainable green spaces is overlooked. As discussed in Chapter 1 soil degradation costs England and Wales up to £1.2 billion/year (National Audit Office, 2014) as a result of detrimental land practises and climate change, and as such soil regeneration and protection is high on the Government's environmental agenda; Michel Gove MP (Environment Secretary) recently announced that, as a result of a soil health inquiry, soil health targets are now implemented by the a parliamentary group called the Sustainable Soils Alliance. Northumbrian Water, among many other water companies, is keen to expand their knowledge of the application of WTRs on land to enable this method to develop as a long term, sustainable disposal option.

### **7.1.2 Project overview**

The Weetslade field trial aimed to build on successful laboratory results (Chapter 5), which have shown that on a small scale the addition of WTR can improve soil structure, water-holding capacity and as such improve the flood holding capacity of a loam topsoil (Kerr *et al.*, 2016). The Weetslade trial was designed to provide 'proof of concept' such that the amendment technology could be applied to other sites across the North of the UK within the NWL catchment area, where flooding and soil erosion are particularly prevalent. The field trail also aimed to inform the partners on possibilities of using the waste amendment technology for engineering flood resilience in South African cities that produce similar waste materials.

The projected timeline for the project was briefly:

*16th Sept – 1st October 2016* – the Researcher spent time working with NWL initially to facilitate sharing of data on this project and establish which are the best by-products to use for the field trial. In addition, the plans for the field trial were finalised and a contractor identified (with help from the Land Trust). The output is a database summarising the properties of NWL by-products with a cost benefit analysis of using them as soil amendments carried out. In addition, plans for the field trial were finalised in conjunction with NWL and other academic partners to ensure that this facility is a long-term resource for urban soil engineering research.

*1st October 2016 – 1st March 2017* – 2 months were dedicated to project management of the site working to establish the field trial on site. This constituted a long-term resource for Durham in the field of urban soil engineering.

### **7.1.3 Beneficiaries and project aims**

Not including academics, there are four benefactors of this research. First and foremost, Northumbrian Water is the main partner to benefit from the reuse of their waste material. As discussed extensively in Chapter 3 increasing costs and production of WTR have pushed water companies ever further into finding long-term sustainable disposal avenues. This project looks at cost-effective sustainable ways of maintaining and enhancing soil health and improving flood resilience within an urban country park, but results are relevant to both urban and agricultural settings. Northumbrian Water will benefit as the technology uses by-products, which have no current market value and therefore in doing this it closes the loop by reusing wastes as resources for the iron rich water industry waste products. NWL currently spends significant funds on disposing of waste products to landfill. For example, in just this small project we will be diverting 30 tonnes, which is equivalent to £2400 in potential disposal costs (should landfill be the disposal route, as typical for water treatment works). On a longer-term basis NWL is interested in improving water quality across the catchment (chemicals and suspended solids in particular), which in turn may also reduce the volume of treatment waste. Flooding issues are also key on the agenda, where Northumbrian Water along with other industries such as property development, land-holding (The Land Trust), insurers in flood risk, tourism and recreation boards, agriculture and food, are interested in reducing flood risk across the UK. The Land Trust is particularly interested in the long-term management of public spaces to improve resilience to flooding and as such allowed the research to be carried out at Weetslade Country Park.

The second beneficiaries are national policy makers in governmental departments such as the Department for Environment, Food & Rural Affairs (DEFRA), the Department of Energy and Climate Change (DECC), the Environment Agency, the Scottish Environment Protection Agency (SEPA), the Forestry Commission, Natural England and Public Health England. This work may also have consequences for the

Water Framework Directive and legislation surrounding groundwater abstraction licenses, as the Environment Agency are increasingly looking towards immobilising pollutant using soil amendments while increasing transmission of overland flow into aquifers. Thirdly, local policy makers (local planning authorities) and LEPs such as Newcastle City Council closely follow research of this nature to consider these amendments on brownfield sites in Newcastle to improve local flood resilience. The final beneficiaries are organisations that influence policy making or are engaging in the implementation of the policy at a sub-national level through a variety of environmental programmes, e.g. National Trust, River & Wildlife Trusts and the RSPB.

There are additional impacts to the field trial at Weetslade that go beyond direct advantages to the beneficiaries identified and include the effect on engineering and geography research as well as economic and societal impacts. The direct impact on an engineering basis is the empowerment of communities to use water industry by-products as soil amendments to regenerate land, using the ROBUST methodology, which can both improve soil health and provide flood resilience to transform wasteland into green space. The 'geography impact' is that by enhancing urban resilience to flooding and improving soil health, communities are likely to see an increase in their health and wellbeing. This 'proof of concept' project may allow researchers (e.g. Dr Johnson) to continue policy work with the Government's Environmental Audit Committee exploring how urban soils can be better protected and is relevant not only to both the Engineering and Geography departments at Durham University, but also countrywide. In addition, there is global potential as the research extends to Stellenbosch University soil science, looking at how to increase urban flood and drought resilience using similar waste minerals in Southern African cities.

Although the economic benefits to Northumbrian Water are clearly identified, where the sustainable incorporation of WTR into soil will reduce disposal costs, it is as yet too early for commercial opportunities to be closely defined. Once a proof of concept exists from laboratory work and the wide scale field application, as well as continuing with NWL as a partner, talks can begin with the insurance industry about the potential of urban flood resilience technologies to reduce flooding risks.

The contaminated land industry and developers are also as potential end-users (as well as farmers in agricultural settings), where engineers are looking for sustainable technologies to stabilise ground, which ensure geotechnical strength for our built infrastructure at the same time as maintaining soil functions.

The work has important societal impacts; without making soil a priority and protecting this resource we risk losing the ecosystem services and functions that soils offers us in both urban and rural environmental settings. If we lose these services, then soil is not being protected for future generations and hence we are not treating this resource sustainably. The soil amendments trialled in this proposal will help place the UK at the forefront of soil amendment techniques to enhance soil health and therefore generate potential economic impact through uptake by the existing contaminated land industry. Rolling these interventions out across both urban and rural hinterlands will help enhance soil health and increase water holding capacity and shear strength where it most needed, helping future-proof communities against flooding and therefore potential generating both economical and societal impact. By further developing the ROBUST methodology to include flood resilience as one the many advantages of transforming wasteland into green space, we will be enabling a range of communities to work with local authorities and industry to identify and regenerate local land using soil amendments (sourced from industry) to enhance flood resilience in our cities.

In summary, the overall aim of the field trial is to develop a suite of technologies to maintain and enhance soil health and soil structure in urban environments by providing proof of concept (improvement in flood holding capacity) from laboratory work to the field scale. This is both timely and essential as in approximately 25 years there won't be any urban soil left to protect, due to soil sealing with tarmac and concrete during urban development. The use of NWL by-products (common across the Water Industry) allows the company to explore the potential to close the loop (recycling) for this product, thereby saving money (diverting waste from landfill) and making their processes more sustainable. Improving soil's ability to retain water during flooding events and improve water quality in the NWL catchment would also be beneficial, although this is a long-term

goal as opportunities for using these technologies strategically in more rural catchments would need to be explored in conjunction with hydrological modelling.

The objectives of this field trial were:

- *Objective 1:* Determine if there is a change in the water holding capacity of a water treatment residual amended soil in a field environment
  - o Using soil moisture and soil potential sensors in the field.
- *Objective 2:* Assess the shear strength and erosional resilience of water treatment residual amended soil
  - o By taking soil samples from the field and reforming them in a lab for triaxial testing

## 7.2 Site overview and methods

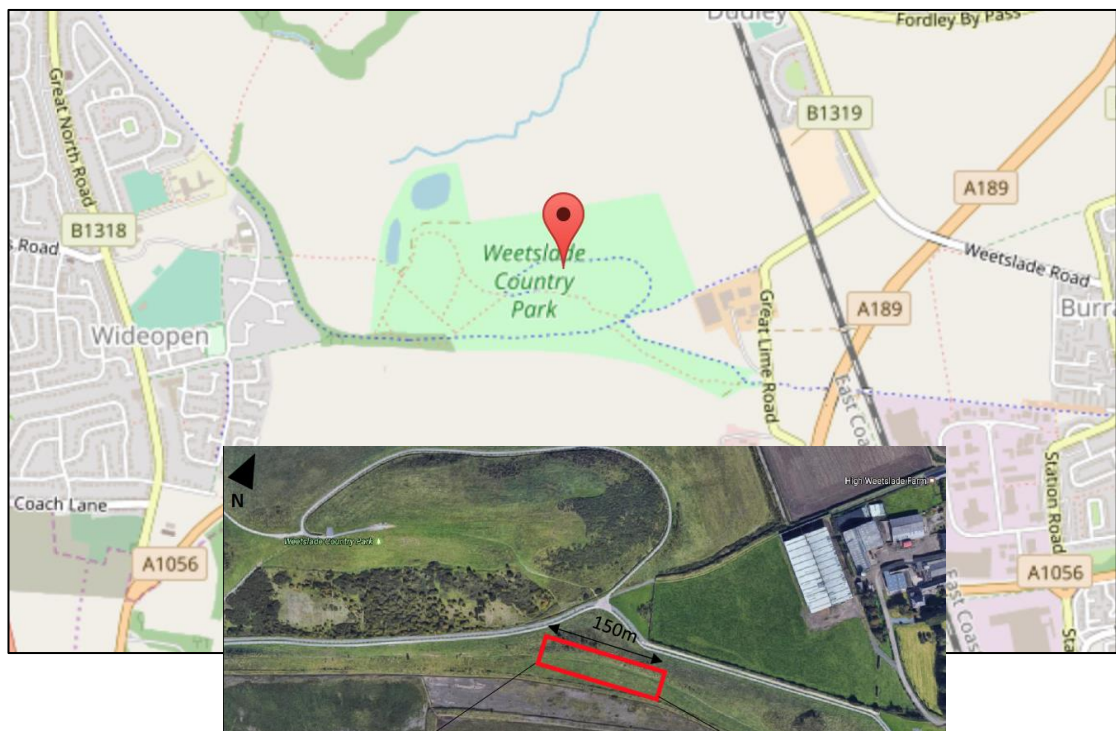


Figure 114: Ordinance Survey map of the Weetslade Country Park with an aerial Google Maps image of the study area (in red) [24].

The Northumberland Wildlife Trust manages the 38 ha Weetslade Country Park, which belongs and is managed by the Land Trust (shown in Figure 114), with help from volunteers from the local community. The park is located in the Seaton Burn catchment in Northumbria, North East England, to the north of Gosforth Park and

is within a 'strategic wildlife corridor'. The local area was used for mining from the 1800's and Weetslade Colliery, upon which the park was built, was an active coalmine from 1903 to 1967. Mining operations have left the local soil heavily degraded with the contamination of arsenic and lead in the topsoil (Baker *et al.*, 2004), where Weetslade Park soil is classified as slowly permeable wet slightly acid but base-rich loamy and clayey. After its closure the site was left to recover naturally and in the 1990s was designated as a Site of Nature Conversation Interest. In 2006 the site was transformed into the Green Flag award winning green space it is today, and contains important habitats for scrubs, hedgerows, marsh, wetland and reed beds, which accommodate otters and kingfishers among other species. Today the Land Trust estimate that the green space at Weetslade has contributed £1.48 million to the local health sector and £1.14 million toward crime reduction (Land Trust, 2015 [25]), however these benefits are at risk on account of local flooding (Figure 115). In addition, there is considerable potential for contaminants within the soil to be mobilised in flood events, such as those that occurred in September 2008, June 2012 and September 2012. These events were experienced due to increasingly extreme weather events in the last decade (Murphy *et al.*, 2009) and exacerbated by soil compaction and urbanisation (Environment Agency, 2013[26]).

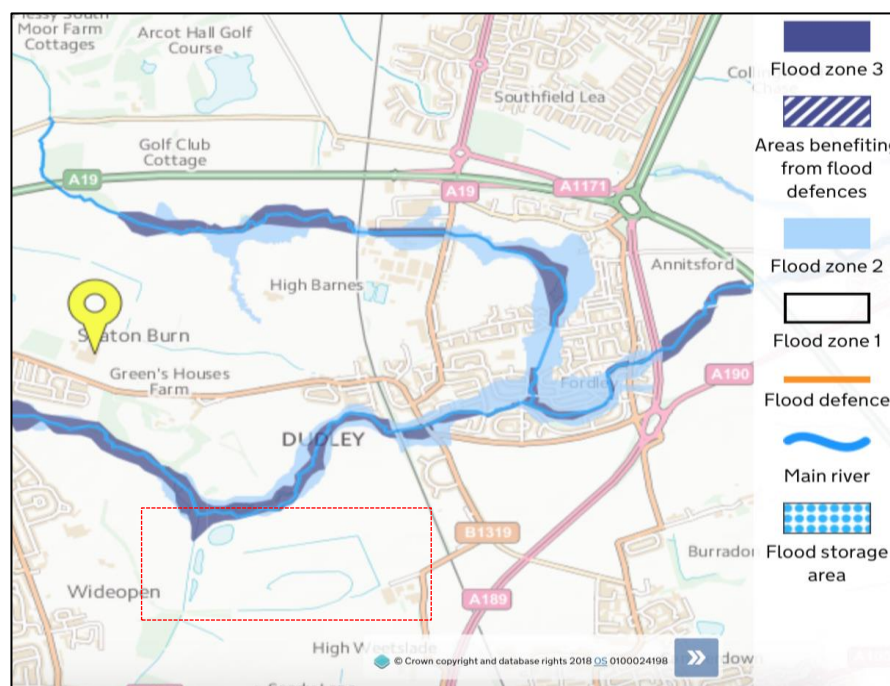


Figure 115: Flood map for the Seaton Burn [24] of the area local to Weetslade (identified in the red box)

In the area rainfall varies seasonally, with the majority of the annual 698.6 mm precipitation falling in the winter (Met Office, 2017a [27]). The area consists predominantly of agricultural land with small urban settlements. The Seaton Burn itself is heavily modified as a result of mining in the area, which has had severe impacts on the water quality due to agricultural and urban inputs containing silt, sediment, oils and pesticides (Baker *et al.*, 2004). As a result, the river has been classified as moderate for ecological and chemical qualities by the EA (2016 [28]). River modification and the presence of compacted agricultural soils from intensive farming in the area have contributed to surface water flooding. Recent flooding in the Seaton Burn catchment has caused a number of properties to be affected, loss of income to the agricultural sector and the mobilisation of contaminants into the local watercourse (Jarvis & Mayes, 2012).

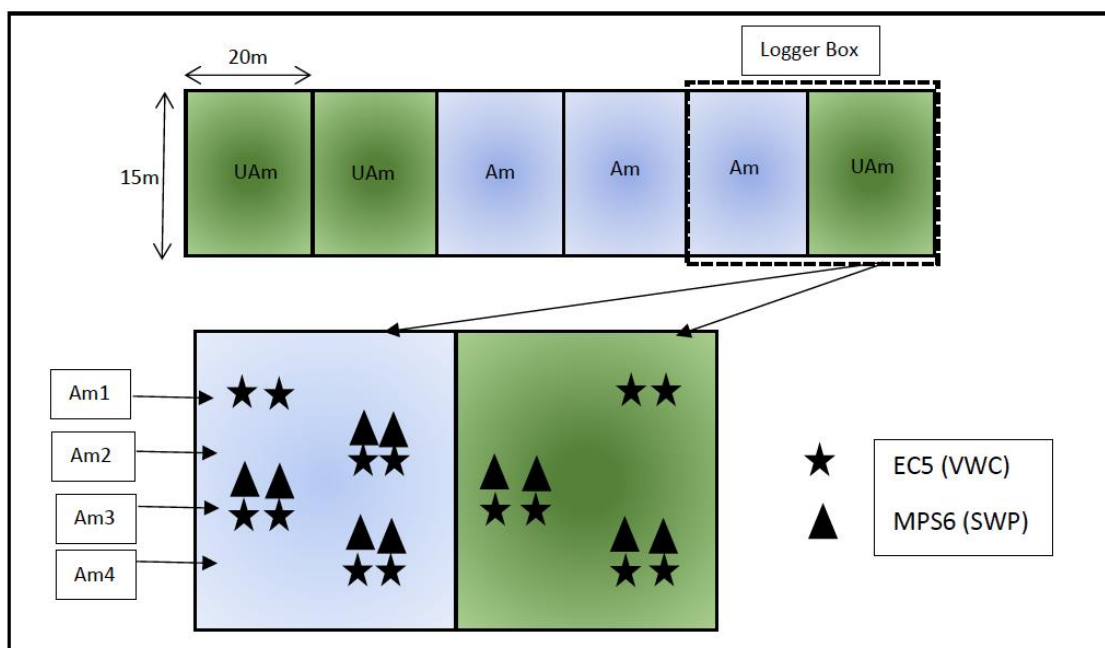


Figure 116: Schematic of field trial plots, location of amended plots and location of sensors on the two amended plots.

In March 2017, works to transform a SW facing embankment within Weetslade Park (red box in Figure 114 and Figure 115) were conducted by WL Straughan & Sons Ltd (a local landscape and environmental contractors). Briefly, the works consisted of marking out 6 plots, 20 m in width and 15 m in length down the embankment (Figure 116). All six plots were treated in the same way so that the results between three unamended plots and three amended plots soil could be

directly compared. Using a large JCB, firstly the top 10 cm of soil that was vegetated with grass was removed and stored for subsequent replacement on the field plots. Secondly each bare soil plot was dug to a depth of 30 cm, which was the maximum depth possible due to the presence of heap spoil underneath the ‘clean’ soil cap. Three ‘unamended’ plots were lined with an impermeable membrane (Visqueen) and the soil was returned to the plot, this was to provide some degree of control on the inputs to the plot – where only rainfall could provide the soil with moisture, and to avoid any cross contamination of ‘groundwater’ between amended and unamended plots. Grass removed in the first stage was then returned to the surface.

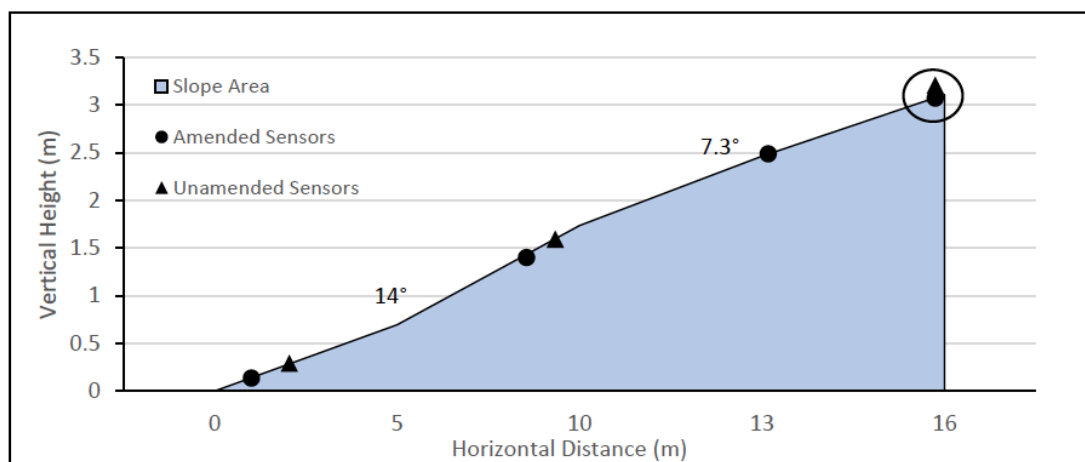


Figure 117: Slope profile average for the Weetslade embankment test site, where the location of sensors in Figure 2 are shown. Data from Ellis (2017).

The remaining three plots were amended with a 10% co-amendment (mass by wet weight). The three 20 x 15 m plots had a total of ~600-630 tonnes of soil removed during the excavation (given a bulk density of the soil at 1.2 – 1.4 g/cm<sup>3</sup>). Therefore for a 10% amendment, 27.5 tonnes of water treatment residual (delivered by James Enderby, a contractor of Northumbrian Water) and 27.5 tonnes of compost (from Straughan & Sons Ltd) were delivered to the site immediately prior to their use, and mixed together on site before application. The empty plots were lined with Visqueen, before being filled with a mixture of soil and the co-amendment, after which they were recovered with the grass layer removed in the initial stages. Each plot (~200 tonnes of soil) had 20 tonnes of co-amendment applied; however unlike the laboratory application of co-amendment,

the spreading of the co-amendment within the plots was very coarse due to the use of the JCB.

### **7.2.1 Measuring VWC and soil suction**

The following sensors were installed on two plots, one amended and one unamended as per Figure 115 at depths of 10 cm and 20 cm. A weather station was also set up at the base of the embankment at the centre of the 6-plot area. Each sensor is maintenance free but requires data to be collected manually from the loggers on a 30-day basis, and from the weather station on a quarterly basis. Weekly data collection started in May of 2017, which captured two wetting and drying cycles, however loggers were water damaged after one month due to an unexpectedly heavy two-day period of rain. Replacement loggers were installed in August, where data was initially taken in September. As a result, there is a gap in data between 1<sup>st</sup> June and 9<sup>th</sup> August for SWP probes (MPS6, SWP) and between 1<sup>st</sup> June and 1<sup>st</sup> September for VWC probes (EC-5, VWC).

*Volumetric water content: TDR (Decagon ECH20) Probe EC-5 and loggers (Decagon Em50-Em5b)*

- 14 probes and 3 loggers
- The EC-5 delivers research-grade VWC accuracy at a price that makes large sensor networks economically practical. Accuracy: 0.1% VWC (mineral soil)
- 15 minute interval logging

*Soil water potential:*

- 10 MPS6 sensors (Decagon) and 2 loggers (Em50)
- Matric water potential sensor that provides long term, maintenance free soil water potential and temperature readings at any depth without sensitivity to salts. Range is from field capacity to air dry.
- Accuracy SWP:  $\pm(10\% + 2 \text{ kPa})$  from -9 to -100 kPa, resolution of 0.1 kPa and 0.1°C

*Weather station*

- Solar panel, tipping rain gauge, net radiometer, wind gauge

### 7.2.2 Measuring geotechnical soil parameters

In addition, three supplementary laboratory tests were conducted by an MEng student (Ellis, 2017) to provide information on the soil type, bulk density and soil shear strength of the site. Firstly, simple Mason jar tests (crude versions of a hydrometer test described in section 4.3.1) were conducted on the unamended soil to classify the soil (Saxton & Rawls, 2006), which required the addition of soil, a dispersant such as Calgon (sodium hexametaphosphate) and water to a jar. These were shaken and left to settle for 24 hours, after which the depth of soil settled at various time points after a second mixing is recorded. Weetslade soil was characterised as a clay loam, with high clay content (38% sand, 33% silt and 29% clay).

Secondly, 20 random soil cores were taken from across the field plots in June 2017 to ascertain the average bulk density and water content of the amended and unamended plots, where unamended plots averaged  $1.15 \text{ g/cm}^3$  and amended plots averaged  $1.06 \text{ g/cm}^3$  (the statistical significance of the difference is unknown). These low values of bulk density are expected, as the plots were not compacted after being re-laid, with the exception of the JCB used to excavate the site. Lastly, triaxial testing was conducted on reformed soil cores to obtain values for shear strength. 30 amended and 30 unamended cores were prepared using the split mould method (see Chapter 4, section 4.4.1), to a bulk density of  $1.8 \text{ g/cm}^3$ ; as discussed previously this value was chosen to represent a compacted soil and was a density at which cores were sufficiently robust for testing (in addition to being comparable to previous triaxial testing). Here the soils taken from each plot were sieved to 6.3 mm before being brought to 15% water content for compaction. An undrained, unconsolidated triaxial (often called the 'quick') test was conducted on each core after 24 hours, where the test procedure is the same as the consolidated test in section 4.6 but without the consolidation stage. Ten cores were tested at each confining pressure for both amended and unamended samples (25 kPa, 50 kPa and 100 kPa).

### 7.3 Field trial results

The following section briefly presents both initial and longer-term findings from the field site. Immediately following the construction of the trial plots, the topsoil was mostly bare as the grass/vegetation layer removed by the JCB at the beginning was replaced only loosely. However, as the root network was preserved, the vegetation present was re-established relatively quickly as shown in Figure 118.



Figure 118: Photos of the field site on week post construction, and in the 2 months following for both an amended plot and an unamended plot

Although no quantitative data were taken, it appeared that there was more growth on the amended plots (with the exception of the unamended plot nearest the Weetslade meadow). A brief study was undertaken by Ellis (2017) on 6<sup>th</sup> June 2017 which identified common wasteland soil flora (buttercups, perennial rye grass, sheep fescue, quaking grass, dandelion, barron brome, alsike clover, common bent grass, Shasta daisies, and mushrooms (exclusive to amended plots).

	June 2017 <i>n</i> =10	February 2018 <i>n</i> =3
Unamended	1.15 g/cm <sup>3</sup>	1.27-1.41 g/cm <sup>3</sup>
Amended	1.06 g/cm <sup>3</sup>	1.13-1.35 g/cm <sup>3</sup>

Table 35: Values of bulk density for unamended and amended plots over time

In February 2018, cores were taken by another MEng student (Lamb-Camarena, 2018) to retest the bulk density of the amended and unamended plots, which gave values of 1.13-1.35 g/cm<sup>3</sup> for the amended plot and values of 1.27-1.41 g/cm<sup>3</sup> for the unamended plots, although according to Student t-test statistical test the difference between the two was not significant (this may be a reflection of the low number of repetitions where n = 3). It appears that in the 11 months subsequent to trial completion, the soil had settled and the bulk density of both plots had increased, likely due to the effects of wetting and drying that settle the soil to increase bulk density (shown in Table 34). No bioturbation was observed in the sample pits, where the crude mixing of organic matter and WTR was still apparent. It is unknown how long it will take for the plots become homogenised by bioturbation and other natural processes therefore it must be noted that any single sample taken from the field trial or any probe placed to data may not capture the properties of the entire body of soil, and may provide erroneous data in non-representative areas of the plot.

### 7.3.1 Water holding capacity

<b>AMENDED</b>	<i>Zone 1</i>	<b>UNAMENDED</b>
Am1 0.2		Um1 0.2
Am1 0.1		Um1 0.1
<i>Zone 2</i>		
Am2 0.2		<i>No sensors</i>
<b>Am2 0.1</b>		
<i>Zone 3</i>		
Am3 0.2		Um3 0.2
Am3 0.1		Um3 0.1
<i>Zone 4</i>		
Am4 0.2		<b>Um4 0.2</b>
Am3 0.1		<b>Um4 0.1</b>

Figure 119: Schematic of EC5 sensor locations by zone. Sensors in **red** did not return any data (probe fault).

EC-5 sensors recorded the volumetric water content (VWC) of soils in amended and unamended plots, where sensors were labelled as per Figure 119. Amended

sensors were labelled Am1 – Am4 with two depths of 0.2 m and 0.1 m, and similarly unamended sensors were labelled Um1 – Um3 again at two depths of 0.2 m and 0.1 m. The data is presented based on the location on the slope (shown in Figure 116), as sensors from the top of the slope are likely to provide different readings for the same rain event due to the gradient on the slope for, where water will preferentially flow to the bottom. Due to sensor malfunction, only in Zone 1 and Zone 3 can we compare the unamended and amended plots. There are fewer sensors on the unamended plot due to budget constraints. The two data periods of 3<sup>rd</sup> May – 30<sup>th</sup> May 2017 and 1<sup>st</sup> Sept 2017 – 04<sup>th</sup> March 2018 are presented separately due to the large time gap between the two. It must be noted that the weather station appears to have recorded rainfall events incorrectly, beginning on the 22<sup>nd</sup> May. According to hourly data obtained from Albemarle weather station, 11 miles west of Weetslade (Met Office, 2017b [29]), two distinct periods of precipitation occurred 13-16<sup>th</sup> May (~14 mm) and 19-20<sup>th</sup> May (~3 mm), neither of which are recorded by the weather station installed onsite, but are picked up by the EC-5 sensors as shown by an increase in VWC. Two precipitation events have been recorded by the weather station on 22<sup>nd</sup> and 23<sup>rd</sup> May, however the lack of response from the sensors and lack of supplementary data from Albemarle indicates that this may be a false reading. Additionally, the rainfall event at the start of June that flooded the sensors has not been recorded by the weather station, despite ~47 mm of rain falling within a few days. For the second data period, the weather station recorded extremely high rainfall values (up to 157.3 mm/hour) however according to Met Office data (2017a), average monthly rainfall typically does not exceed 60 mm, therefore any values above this for a single event have been removed. It is apparent that the calibration performed on the rainfall gauge is incorrect and so, although rainfall events are recorded, the numeric quantities associated are not accurate. It is for this reason that data from both rainfall stations are displayed on subsequent figures for the period of May.

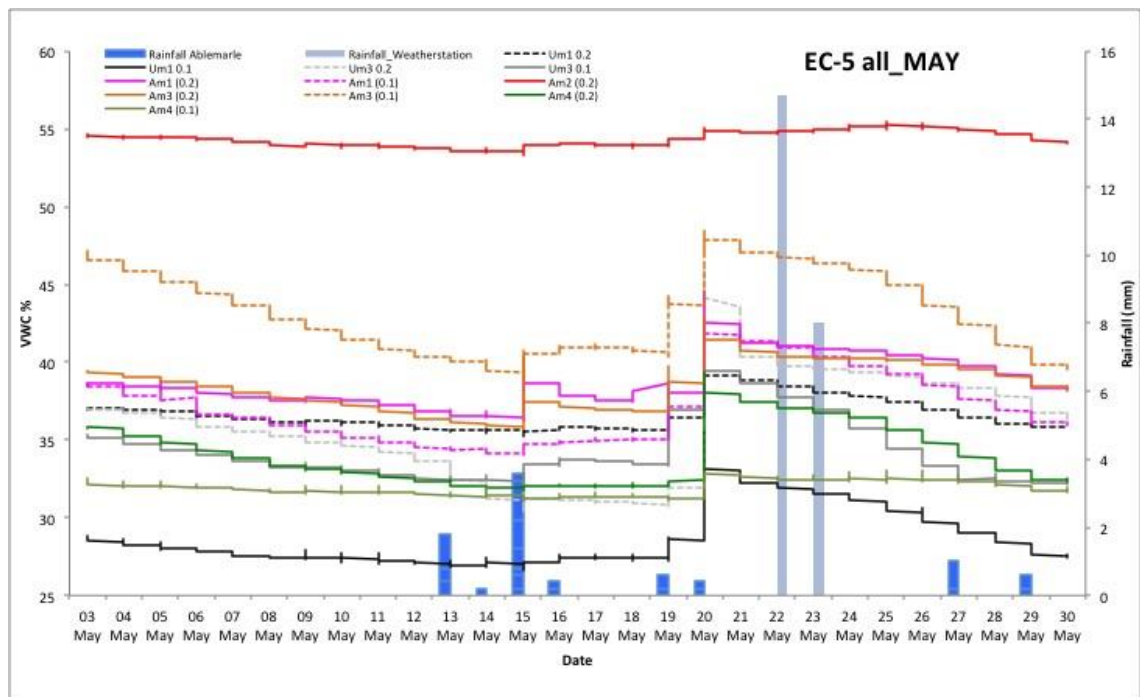


Figure 120: All EC-5 sensor data displaying VWC for May. Rainfall data presented in blue and grey blue are from Ablemarle and the onsite weather station respectively.

In Figure 120, the volumetric water content of the unamended and amended plots is shown for the first period of data collection for all sensors. There is negligible response to the 13 mm (total) rainfall event 13<sup>th</sup> – 16<sup>th</sup> May the majority of sensors (with the exception of Am1 0.2), however on the 20<sup>th</sup> May all sensors recorded an increase in the VWC on the both amended and unamended plots despite a much lower total precipitation of 3.2 mm. This may be a result of hydrophobicity of the clayey soil in the period preceding 13<sup>th</sup> May, where soils were dried by high ambient temperatures (Met Office, 2017a). There are two rainfall events recorded by the onsite weather station during May, on the 22<sup>nd</sup> and 23<sup>rd</sup> May, however these appear to have no effect on the volumetric water content of the samples (despite being quite significant rainfall totals of 14.7 mm and 8 mm) and are suggested to be false readings. This is a large range of VWC values, due to the gradient of the slope, where the values for sensors in Zone 3 are in general higher than those in Zone 1 as a result of water movement downslope.

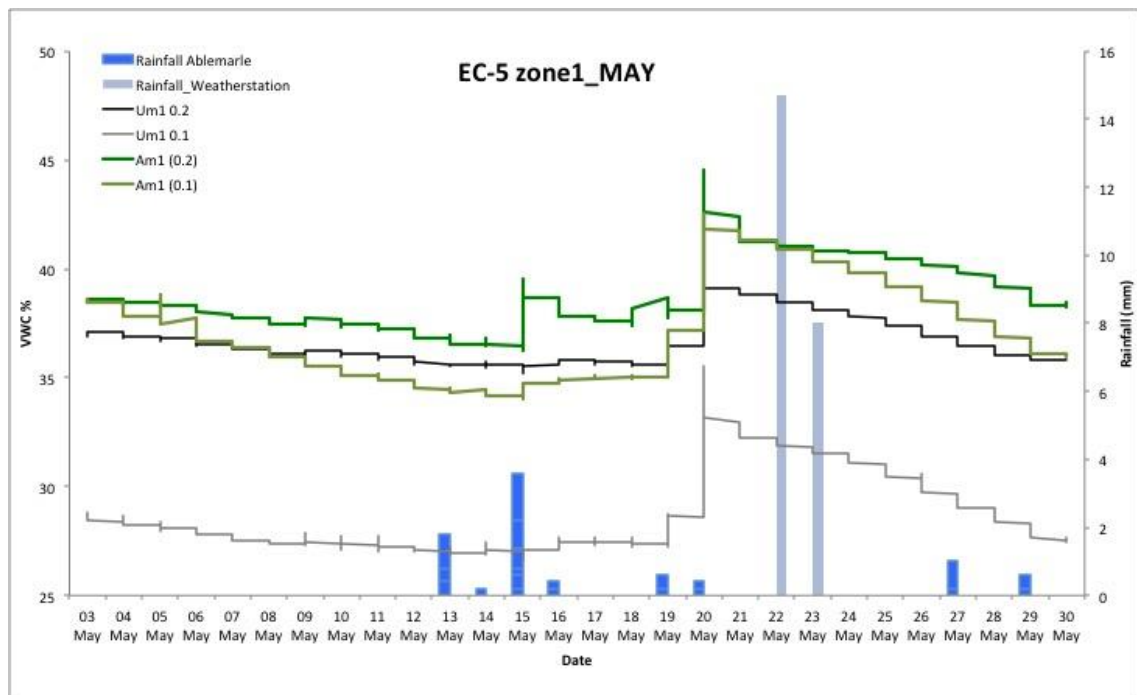


Figure 121: Volumetric water content of sensors in Zone 1 at Weetslade during May, with rainfall data obtained from an installed weather station (light blue) and from Ablemarle (dark blue).

In Figure 121, the unamended soil in slope Zone 1 has in general a lower VWC than the amended plot. All sensors in this zone have a similar VWC increase for the rainfall event 19-21<sup>st</sup> May of around 5%. Both of the shallow sensors experience a faster reduction in VWC after the rainfall event. There is little observable difference in the rate of VWC change. Figure 122 shows the VWC for amended soil during May in zone 3, which is approximately 4 m downslope from Zone 1. The VWC is expectedly generally higher than at the top of the slope (Zone 1), where again the amended plot has a higher VWC than the unamended plot. It appears that the shallower sensor has recorded a higher value than the deeper sensor in the amended plot, which may be down to sensor error or its placement in a 'wetter' part of the amendment, but could also suggest water retention is high in the upper layers of the amended soil. In the unamended plot, the deeper sensor has a higher VWC at the beginning and end of the month, however drops below the value for the shallower sensor before the rainfall events on the 13<sup>th</sup> -16<sup>th</sup> May and 19-21<sup>st</sup> May, after which it has a higher VWC.

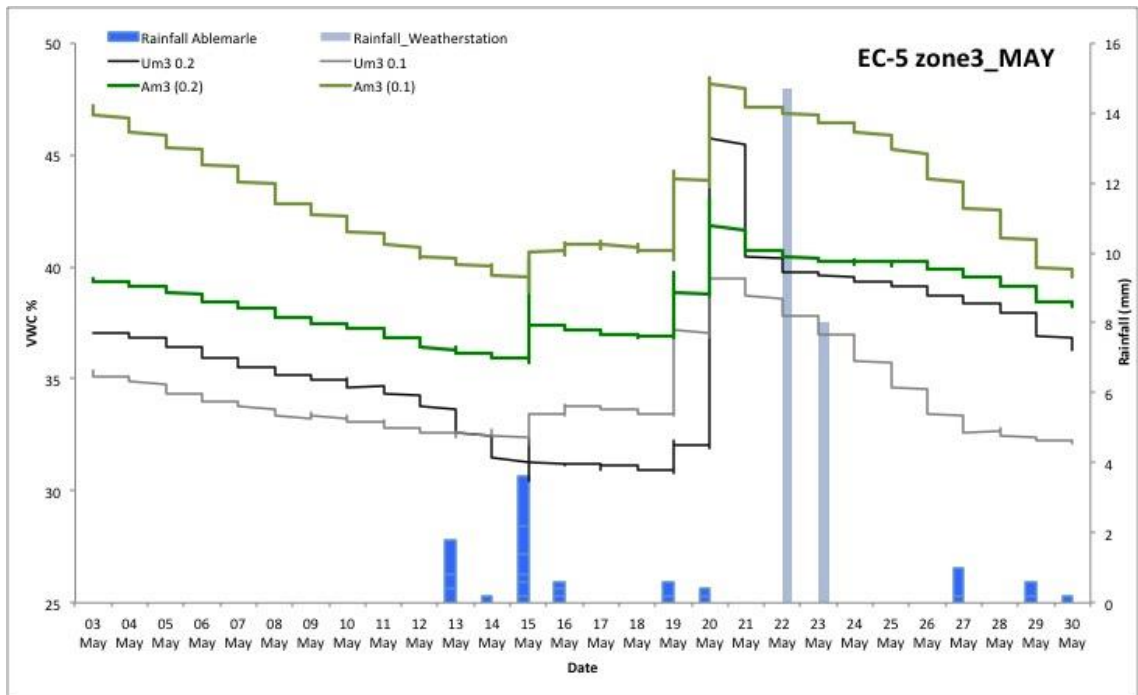


Figure 122: Volumetric water content of the soils in Zone 3 at Weetslade during May, with rainfall data obtained from an installed weather station (light blue) and from Ablemarle (dark blue).

After the rainfall event on the 20<sup>th</sup> May, no further rainfall occurred before the end of the data period and therefore drying characteristics could be evaluated. Figure 123, produced by Ellis (2017), shows the % of VWC lost after the peak VWC of each sensor shown in the previous figures, which is calculated over time to show the rate of drying between amended and unamended. The graph in Figure 123 shows that unamended soils (triangle points along the drying trendlines) in general release water more quickly than their amended counterparts, where the table below the graph compares the sensors based on their location in the slope. In the first 24 hours, the loss of soil moisture was faster in the unamended soils, where Am3 (0.2) declined 9% less than the unamended equivalent Um3 (0.2) after 216 hours. Additionally, sensors on the steeper part of the slope (lower half) recorded 5% faster VWC reduction than the remainder of the sensors, suggesting that these areas drained faster.

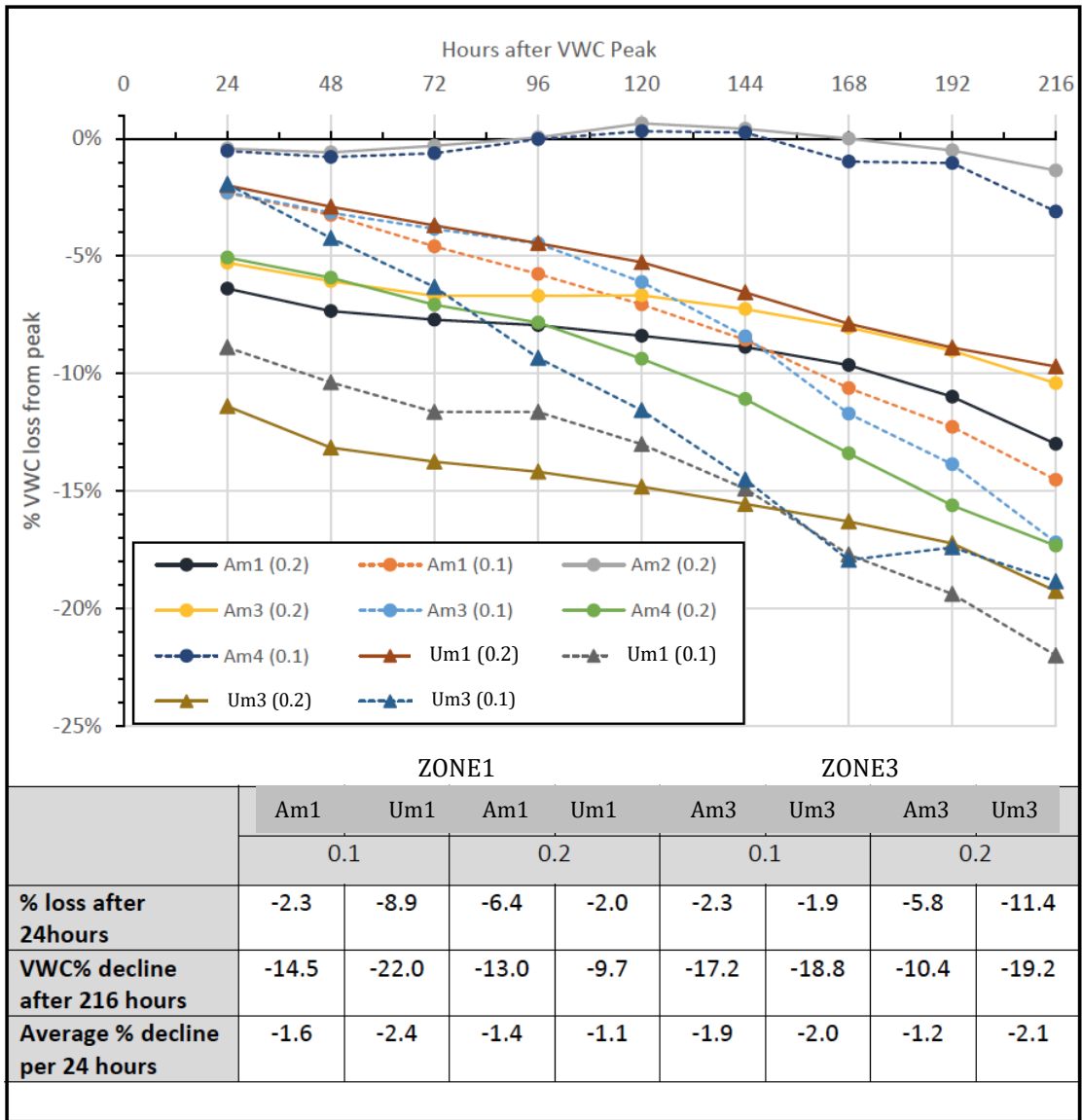


Figure 123: VWC decline in % for each sensor from the peak of their VWC during a rainfall event. The table below compares the sensors based on their location on the slope.

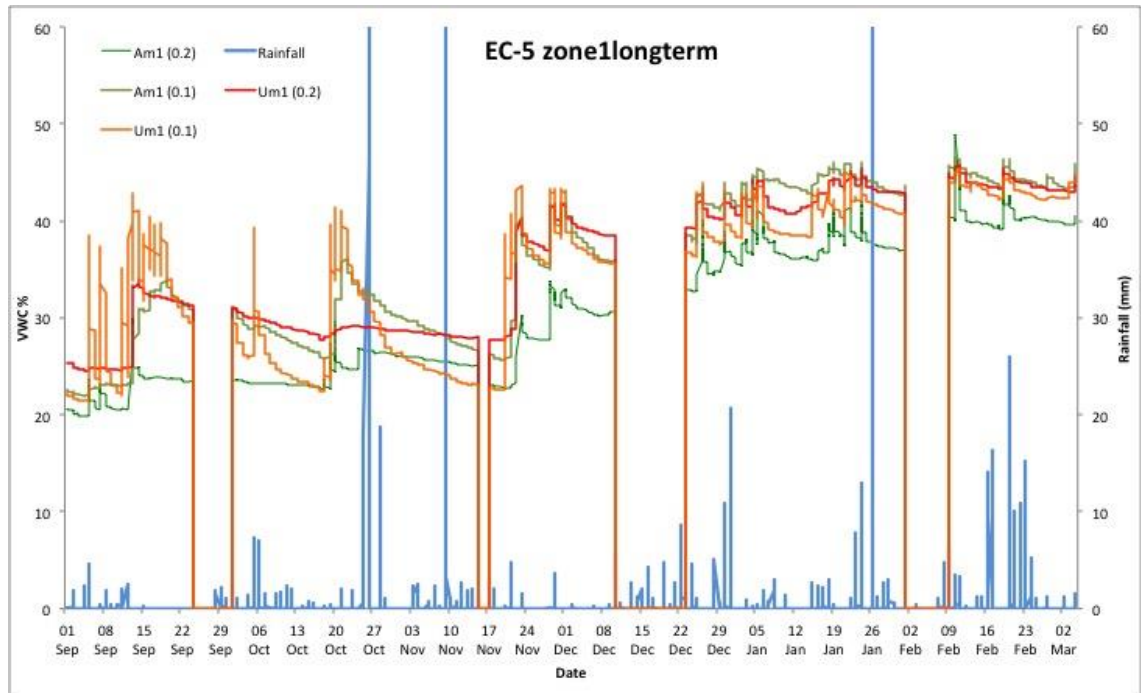


Figure 124: Long term VWC of amended and unamended plots in Zone 1 as recorded by EC5 sensors. Rainfall data is from the onsite weather station.

Figure 124 shows the long-term trends in VWC recorded by sensors in the amended and unamended plots in zone 1. As discussed above, it appears that the rainfall gauge installed at the base of the slope has recorded some values for rainfall but there appears to be very inconsistent correlation between rainfall events and an increase in VWC for any of the sensors. For example, the rainfall event at the beginning of August only elicited a response in Um1 (0.1), but on the 27th November, all VWC values increased as a result of ~ 5 mm total rainfall. The most obvious errors in recording are the three rainfall events recording more than 60 mm in either a single day or over a period of a few days. These have been included to provide an indication of the magnitude of error. Data from the rainfall gauge clearly needs calibrating, as the response of soil to different intensities of rain is important; we need an idea of how rapidly soil can incorporate a volume of water to enable an assessment of its ability to cope with rainfall intensities that are likely to cause flooding.

In general, there is only a small numerical difference between the VWC values of amended and unamended plots, although it appears that the unamended plot values are mostly higher than the amended plot, with the exception of January

2018 onwards were Am1 0.1 has the highest VWC. It appears the from the 20<sup>th</sup> November onwards that the plots near their maximum respective VWC as a result of persistent rainfall events during wet winter months, and the difference in VWC between amended and unamended plots is reduced. As discussed in Chapter 2, the VWC is not a reflection of the total amount of water within a body of soil, but a relative value that compares the volume of water to the volume of soil. For two soils that are unsaturated and hold the same amount of water on a gravimetric basis, a soil with a lower bulk density (assuming the same particle density) will have a lower VWC as the volume of the water remains constant but the volume of soil is higher for the lower bulk density soil. This means that without a priori knowledge of the bulk density or the gravimetric water content, we are still able to assume that the soil with a lower VWC (amended) than another soil (unamended) may still hold more water. The bulk density measurements in Table 35 show that amended soil has a lower bulk density than unamended soil, which given the same gravimetric water content, would result in a lower VWC for amended soil. However as these bulk density measurements are not statistically significant further bulk density measurements are required in addition to ascertaining the gravimetric water content. The VWC is higher for unamended soil when the soils are unsaturated as the amended soil experiences greater volume change (reducing bulk density), however, the values of VWC for amended soils reaches a similar value to the unamended soil when the soils are saturated in winter months as the proportion of water to volume of solids is greater. This may also indicate that it holds more water than the unamended soil, as if the VWC value is similar and the volume of the amended soil is greater, then this means the volume of water is greater than in unamended soil.

Figure 125 shows the long-term trends in VWC recorded by sensors in the amended and unamended plots in zone 3. The malfunction of sensors Am3 (0.2) and Um3 (0.2) mean we can only take information from the 0.1 sensors in Zone 3. Between September and November, the VWC of amended soil (Am3 0.1) was higher than the unamended soil (Um3 0.1) at its peaks, but has a lower VWC after long drying periods. The response of amended soil to moisture is clearly much greater, as the unamended soil experience much less fluctuation in VWC values during this period, however from the 22<sup>nd</sup> November onwards the response of

both soils to rainfall events is similar and the VWC of the unamended soil becomes higher than the amended soil as they reach near saturation.

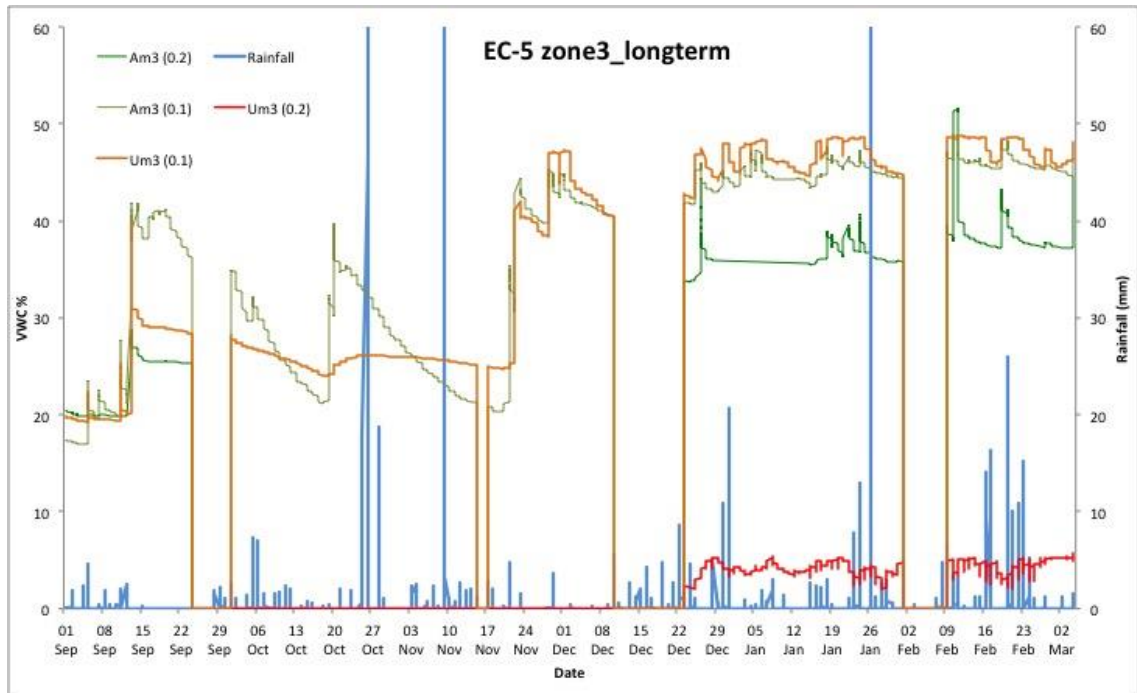


Figure 125: Long term VWC of amended and unamended plots in Zone 3 as recorded by EC5 sensors. Rainfall data are from the onsite weather station.

### 7.3.2 Soil water potential (SWP)

AMENDED	Zone 1	UNAMENDED
No sensors		No sensors
Zone 2		
Am2 0.2 Am2 0.1		No sensors
Zone 3		
Am3 0.2 Am3 0.1		Um3 0.2 Um3 0.1
Zone 4		
Am4 0.2 Am3 0.1		Um4 0.2 Um4 0.1

Figure 126: Schematic of MPS6 sensors location by zone on the slope. Sensors in red did not record data.

The formal definition of soil water potential is “the amount of work per unit quantity of pure water that must be done by external forces to transfer reversibly and isothermally an infinitesimal amount of water from the standard state to the soil at the point under consideration” (Or *et al.*, 2002). MPS6 sensors recorded the soil water potential (SWP), which is a measure of soil energy state, i.e. soil’s potential to take up more water, measured in kPa using a moisture content sensor. For this quantification a reference state is required, which in this case is pure water, where the SWP is 0 kPa. There is a direct relationship between VWC and SWP, where the higher the water content, the lower (closer to 0) the SWP as the soil has less potential to take on more water. For the period of May, three MPS6 sensors did not record data (Am4 0.1, Um4 0.1 and Um4 0.2) hence their exclusion from the data set, and due to this only Zone 3 can be used for direct comparison of amended and unamended plots (Figure 126).

In Figure 127, Um 0.1 shows an increasingly high SWP (shown by a reduction in the kPa) during the first 12 days of May and reached -0.47 kPa due to drying of the soil. Um 0.2 in contrast has very little change in SWP during this period, suggesting that water from this layer of the soil is not moved through evaporation. During the rainfall event on 13<sup>th</sup> May, the soil took a couple of days to respond to the wetting, and although there was little VWC change shown by EC5 sensors in Figures 120-124, Um 0.1 experienced a reduction in SWP. The rainfall even on 19<sup>th</sup> May caused a reduction in SWP for both sensors.

In Figure 128 there is a large range of values for SWP depending on which sensor is selected in the amended plot. Figure 128 shows that the SWP is highest in the shallower sensors (Am3 and Am2 0.1), which agrees with data in Figure 126, again due to this layer drying out more efficiently than the lower layer during a period without rainfall. Conversely to the VWC data, sensors higher up the embankment have less SWP, which suggests that these areas are less easily able to take up water.

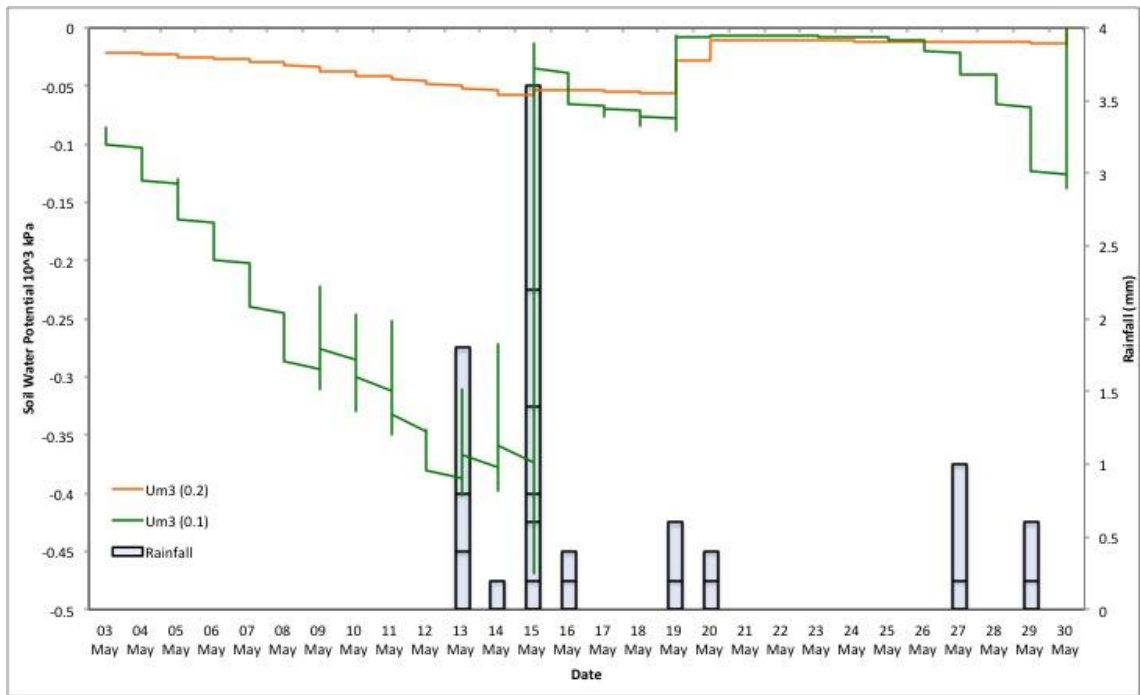


Figure 127: SWP in unamended soil for the period of May (Um4 0.1 and Um4 0.2 did not collect data during this period). Rainfall data sourced from Ablemarle weather station.

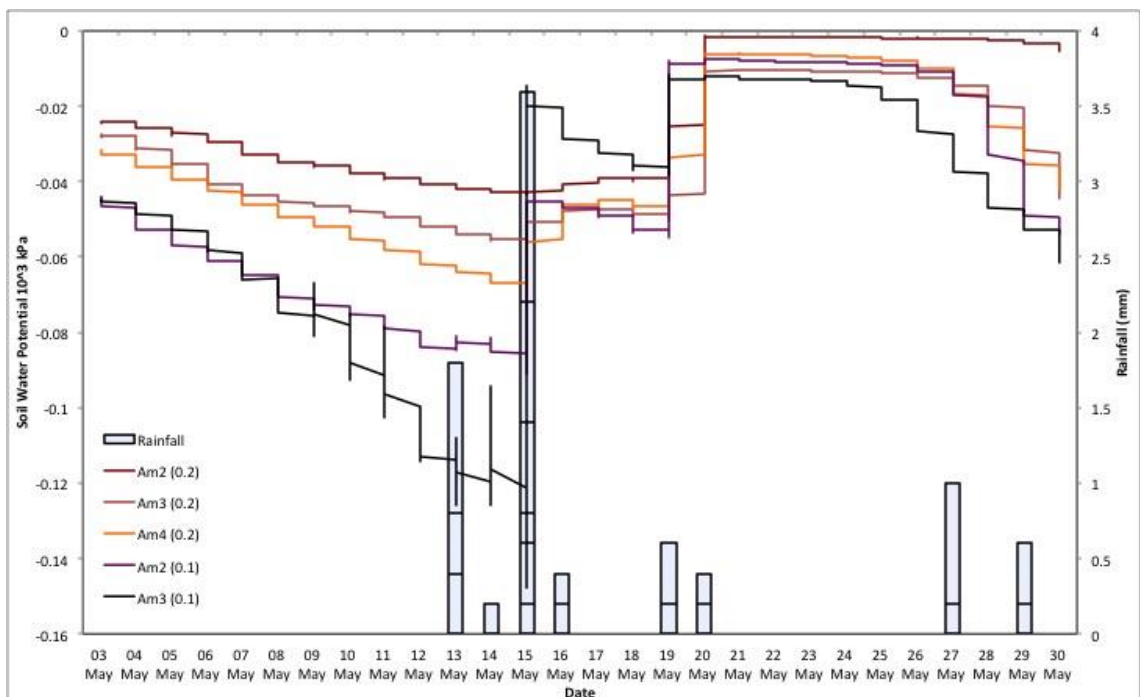


Figure 128: SWP in amended soil for the period of May (Am4 0.1 did not collect data during this period). Rainfall data sourced from Ablemarle weather station.

Figure 129 compares the values for SWP between unamended and amended plots in Zone 3 on the slope. The suction values for Um3 (0.1) are very high relative to the other sensors, with suctions of up to -400 kPa, suggesting that there is a very high soil potential which reflects the long period of drying identified in the VWC figures previously. There also a high degree of 'noise' (measurement error) shown by the vertical fluctuation of the Um3 (0.1) data line over a single day. After the rainfall event on the 15<sup>th</sup> May, all sensors reach a SWP of between -0.1 and 0.02 (-100 and -0.20 kPa) due to wetting of the soil. The amended soils have a lower SWP than unamended soil after the rainfall event, however this is likely to reflect the suction properties of soil rather than the capacity of each soil to take up water.

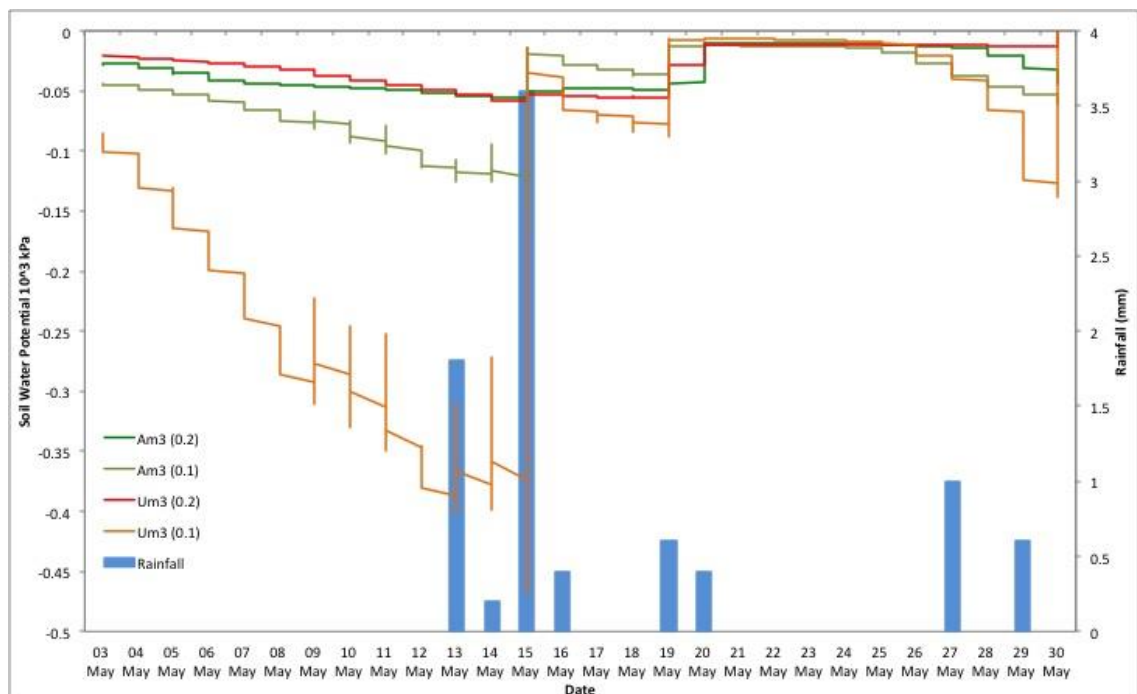


Figure 129: SWP for sensors in Zone 3 on the Weetslade field trial.

Figures 130 and 131 present the long-term data from MPS6 sensors on the SWP of soils on the amended and unamended plot respectively and Figure 132 compares the two data sets in Zone 3 on the slope. It appears in Figure 130 that the sensors higher on the slope (Am2 and Am3) have a lower SWP than the sensor at the base of the slope (Am4), which is a non sequitur as this suggests that base of the slope has drier soils than the top. This however may be a measurement error considering the high volume of noise for the Am4 sensor, where the probe is not fully in contact with the soil for example.

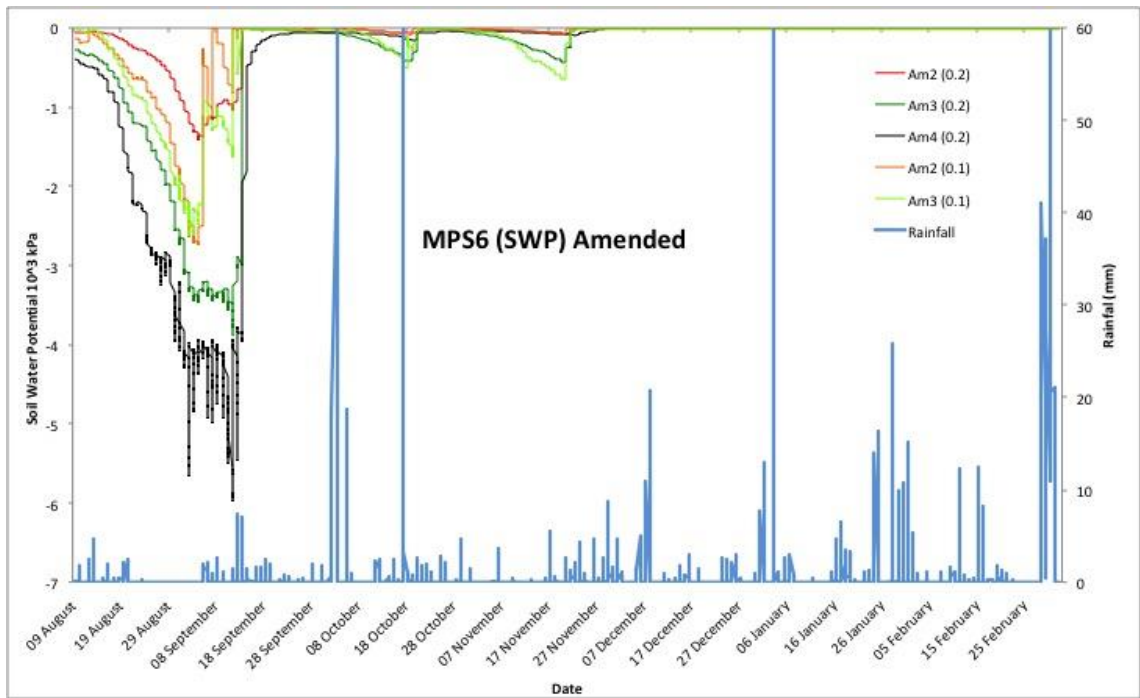


Figure 130: SWP of amended soil plots for the second data period.

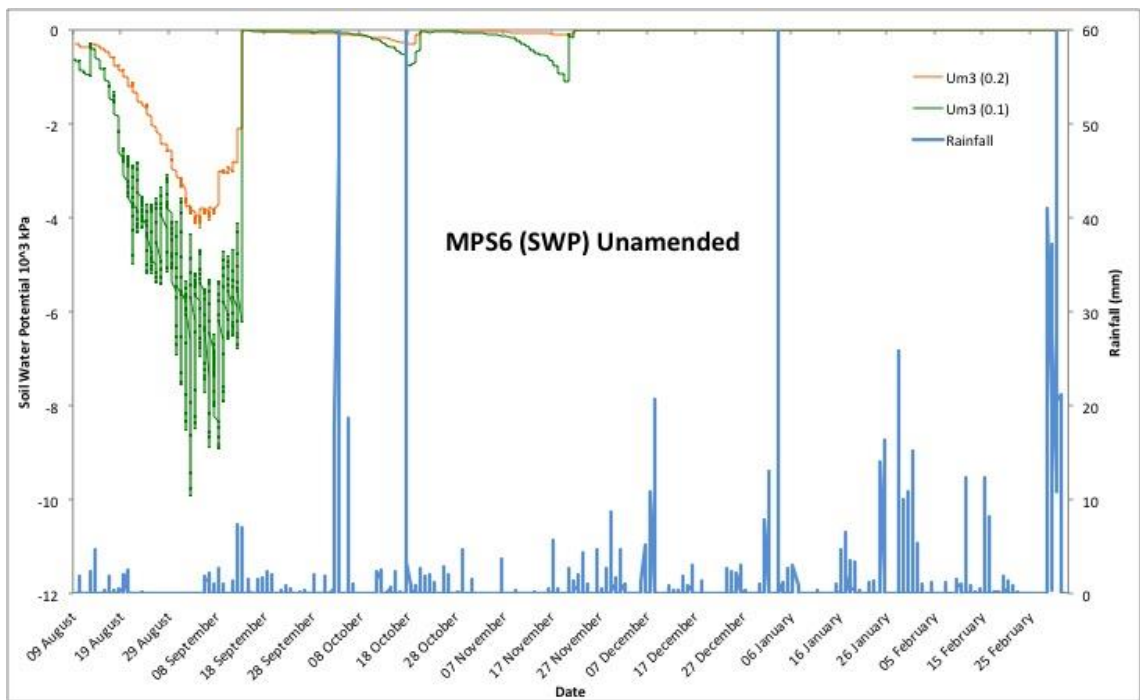


Figure 131: SWP of unamended soil for the second data period. Um4 data is not available due to sensor failure. Rainfall data is from the onsite weather station.

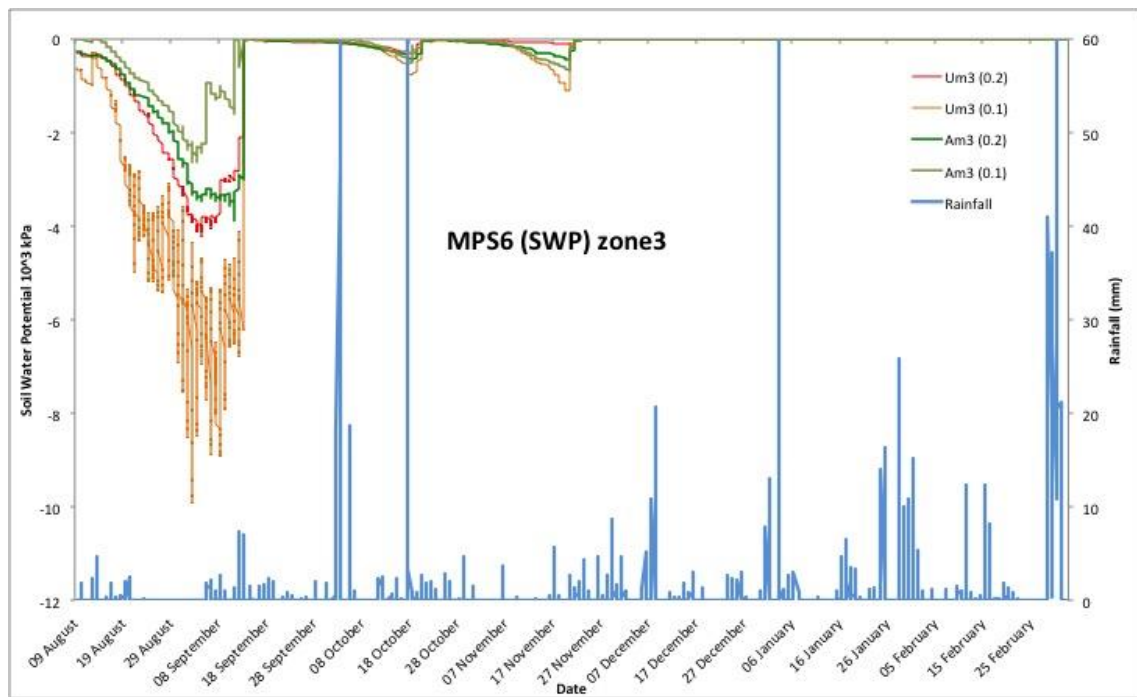


Figure 132: SWP of amended and unamended plots in Zone 3 on the Weetslade field Rainfall data is from the onsite weather station. -12.00 on the Y axis corresponds to a -1200 kPa suction

### 7.3.3 Soil strength

Triaxial testing was conducted by Ellis (2017) on 30 amended and 30 unamended reformed soil cores to obtain values for shear strength using an undrained, unconsolidated triaxial (often called the ‘quick’) test at cell pressures of 25, 50 and 100 kPa, the results of which are shown in Table 36. The average shear strength of unamended soil was determined to be  $124.02 \pm 20.7$  kPa (using Tresca’s theory), which is typical of soils of clay loam (with high clay content) and determines it as a ‘stiff’ soil (Craig, 2004). The maximum shear strength of 168.51 kPa of unamended soil is at 25 kPa, where cohesion was 99 kPa and the angle of friction was  $7.4^\circ$ . Amended soil had a maximum shear strength of 185.64 kPa, achieved during the 100 kPa test, where cohesion was 112 kPa and the angle of friction of  $20.3^\circ$ . These results echo those from Chapter 5, where the amended samples had better shear strength at the highest pressure compared to unamended soil.

There is not a significant difference in the shear strength of amended soil at the lowest confining pressure (25 kPa), however there is a statistical improvement in the maximum shear strength of amended soil at higher pressures in comparison to unamended soil, as well as improved cohesion and angle of friction, which suggests

that the soil aggregate stability is greater in the amended sample and as such is better able to resist shear forces.

	Unamended (n=10)	Amended (n=10)
25 kPa average <i>(not significant)</i>	146.55 ± 22 kPa	143.27 ± 29 kPa
50 kPa average	110.78 ± 27 kPa	163.69 ± 15 kPa
100 kPa average	114.73 ± 10 kPa	162.73 ± 18 kPa
Average shear strength	124.02 ± 20.7 kPa	156.6 ± 20.6 kPa
Maximum shear strength	168.51 (25 kPa)	185.64 (100 kPa)
Cohesion	99 kPa	112 kPa
Angle of friction	7.4°	20.3°

*Table 36: Summary of triaxial cell strength testing on samples from Weetslade Country Park.*

#### **7.4 Discussion and catchment implications**

The re-establishment of vegetation on the soil plots was very quick due to preservation of the root network during the construction of the plots. A quantitative analysis is required for any assumptions to be validated, however the brief analysis of vegetation by Ellis (2017) suggested that there was a greater amount of growth on amended plots. Soil bulk density was tested in June 2017 and February 2018, and showed that there was a small increase in the bulk density of both amended and unamended plots in the 11 months succeeding the implementation of the field plots (however no statistical testing was performed between unamended and amended values of initial bulk density, nor between the bulk density values at obtained at different times). In addition, the data from February indicated no statistical significance in the difference between the unamended and amended plots, although this may be due to a low number of repeats. It is very clear therefore that a thorough study with numerous repeats is

required to draw concrete conclusions on the bulk density characteristics of both the unamended and amended plots.

Assessment of data from EC5 (VWC) and MPS 6 (SWP) sensors installed on two plots on the Weetslade embankment trial site has revealed that in general the VWC is dependent on the depth of the sensor and the location of the sensor on the embankment. During the first period of data collection (May, 2017) the VWC of amended plots were 5.4% (at a depth of 0.2) and 22% higher (at a depth of 0.1) than unamended plots (no comparison could be made in Zone 2 as there were no sensors in the unamended plot at this point on the slope, and in Zone 4 there were malfunctions in the unamended sensors). Data produced by Ellis (2017) shows that in this time period the unamended soils released water at a faster rate (up to 9%) than amended soils in the period immediately after a rainfall event, suggesting that amended soils have better water retention than the unamended soil. Additionally, sensors lower on the slope recorded a 5% faster VWC reduction than sensors at the top of the slope, showing the effect of gradient on the movement of water through the field plots.

However looking at the long-term data between September 2017 and March 2018, the VWC of the unamended soil is in general higher than the amended soil. This may be as a result of two factors, the difference in water retention for different soil types, and/or the method of measurement. Weetslade soil was classified by Ellis (2017) as a clay loam with high clay content; it is well known that clays hold more water than other soil types due to the high surface area of clay particles and high number of micro-pores (Craig, 2004). By adding co-amendment to the Weetslade soil, the soil particle size has been altered to include a greater proportion of coarser particles, which although improve the soil structure, reduce the amount of water that the soil can hold under a particular suction while in an unsaturated state as the macro-pores present in the amended soil are not able to hold onto the water as well as micro-pores.

The average SWP of unamended soil was higher than the amended plot, due firstly to the difference in VWC and secondly due to the different textural composition of the soils as the results of the amendment. As discussed in Chapter 2, the

relationship between water content and suction is simple, the lower the water content, the higher the suction, although this relationship affected by the texture of a soil. Larger negative suctions, as shown by negative SWP (kPa) are more typical of clays in comparison to sandy soils due to the difference in water retention properties. Therefore at the same water content the clay will have a higher SWP. The differences in SWP may therefore be a reflection on the texture of the soil rather than on its ability to take up more water, as we have seen that water movement through the amended soil is faster in laboratory tested soils. Nonetheless, it is difficult to compare the effects of amendment on different soils, where a clay soil behaves differently to the loam soils tested in Trials 1-4.

High suctions are indicative of high shear strength and although Weetslade soil is classified as a stiff soil due to its highly cohesive aggregates (Craig, 2004) with a maximum shear strength of 168.51 kPa, the shear strength of amended soil was higher at 185.64 kPa. The increase in shear strength of amended soils, shown by this data, is positive for the soil's erosional resistance in a flooded state, as soils more resistance to shearing forces are less likely to breakdown as a result of raindrop and runoff erosion (as discussed in Chapter 2, section 2.3.5).

The wider implications of the field application of WTR are difficult to assess for the Seaton Burn catchment and requires the application of WTR co-amendment to a range of different of soils such that the effect of co-amendment can be determined for a range of soil textures. According to laboratory results, should a WTR co-amendment be applied to a soil, in general it will reduce the bulk density and compactibility of a soil. For a granular soil, the addition of fine particles and organic matter are likely to increase the water holding capacity, water retention and shear strength and improve hydraulic conductivity of the soil (as shown by testing results in Chapter 5). It appears that conversely to laboratory results the application of the co-amendment to a highly clay soil is likely to reduce the water content at field capacity (i.e. water held against the force of gravity after a rainfall event) in the soil based on the VWC, but these results must be discussed with reference to bulk density and gravimetric water content values that can be derived by taking samples from the field and assessing them according to the appropriate tests (discussed in Chapter 4, section xxx). It must also be noted that the soils at

Weetslade have not been subjected to the same degree of saturation as those in the lab, which were submerged for a long period of time.

### ***7.5 Concluding remarks***

The VWC of unamended soils are on average 18% higher than amended soils at a depth of 0.2, and around 3% lower than amended soils at a depth of 0.1 for long-term data despite initial trend to suggest that the amended plot had a higher VWC. To determine if this difference is due to volume change of the soils (as described in Chapter 2), further field samples are required to be taken for analysis of GWC and density. Triaxial results from reformed soil samples show that the shear strength of amended soil (185.64 kPa) is statistically significantly higher than unamended soil (168.51 kPa). Further laboratory tests will be required to test the erosional resilience. The discussion has attempted to explore further benefits of water treatment amended soil to the local environment through assessments of the field site, including vegetation surveys and consideration of the wider catchment, but this is a challenging process due to the inconclusive results of current data. A number of supplementary tests are required to wholly assess the effect of WTR co-amendment on Weetslade soil, along with continued monitoring of the field trial over a longer period of time.

## 8. Conclusions and Recommendations for Future Work

This thesis has outlined novel methods with which to quantify soil functions, which allow us to assess their flood holding capacity. This includes the water holding capacity, the hydraulic conductivity, the shear strength and erosional properties of the soil. Current methods of measuring water in soils need to include both an assessment gravimetric water content and volume change, in order to accurately assess the ability of soils to hold water and so volumetric water content does take swelling into account (and can lead to an underestimation of water holding capacity). This thesis has begun to bridge the gap between geotechnical and geoenvironmental perspectives of analysing soil with the by exploring the following avenues:

- 1) Development of novel water holding capacity experiments to assess the maximum gravimetric and volumetric water content of amended and unamended soils over at least one wetting and drying sequence.
- 2) Erosional resistance testing of amended and unamended soils through fall cone penetrometer and a newly development method (Veitch, 2016).
- 3) Assessment of hydraulic conductivity of amended and unamended soils using triaxial cell.
- 4) Assessment of shear strength properties of amended and unamended soils using triaxial cell.
- 5) Analysis of amended and unamended soils using X-ray Computed tomography, in order to understand the effect of amendment on soil structure.
- 6) Field trial application of the co-amendment at Weetslade Country Park.

Water Treatment Residual (WTR), a by-product of the clean drinking water industry has been used in conjunction with compost as a co-amendment, using the WTR at 20% solids ( $WTR_w$ ) and air-dried at 20% water content ( $WTR_d$ ). The reuse and or recycling of this clean waste is an important avenue of research for water companies in the UK and across the world due to increasing costs of disposal. Simultaneously there is a timely need to improve the health of the UK's soils in order to mitigate the historic effect of degrading practises. Improvements in

important soil functions as a result of both single WTR and the co-amendment of WTR and compost have been highlighted in the previous chapters. The significant findings are summarised as follows: (a) maximum gravimetric water content, (b) sample volume increase, (c) shrinkage, which refers to the difference in maximum sample volume between the 1<sup>st</sup> wetting and 2<sup>nd</sup> wetting sequences (d) maximum void ratio (porosity) as determined by either XRCT or triaxial testing (d) hydraulic conductivity and (e) shear strength. Values for a-c are derived from Trial 4, d from XRCT and d,e & f from triaxial testing. These are summarised in Table

<b>Soil Function</b> - Indicates no significance in improvement. Red indicates significant negative influence	<b>% amend-ment</b>	<b>Control (Soil2)</b>	<b>Single WTR<sub>d</sub></b> <i>20% water content</i>	<b>Single WTR<sub>w</sub></b> <i>20% solids</i>	<b>Single compost</b>	<b>Co-amendment WTR<sub>d</sub></b>	<b>Co-amendment WTR<sub>w</sub></b>
<b>1<sup>st</sup> wetting &amp; drying</b>							
Maximum gravimetric water content <i>(0.18 starting value)</i>	10		-	0.390 (4.9%)	-	-	0.390 (4.8%)
	20	0.372	0.382 (2.7%)	-	0.421 (13.2%)	0.412 (10.8%)	0.412 (10.8%)
	30		0.423 (13.7%)	0.410 (9.9%)	0.501 (34.7%)	0.464 (24.7%)	0.465 (25%)
Volume (change) in cm <sup>3</sup> <i>(86.2 cm<sup>3</sup> starting value)</i>	10	+37.9	+27.9 (-26.4%)	-	-	-	+35.7 (-5.8%)
	20		+31.7 (-16.4%)	-	+39.9 (5.3%)	-	+38.8 (2.4%)
	30		+36.3 (-4.2%)	+39.9 (5.3%)	+54.6 (44.1%)	+47.7 (25.9%)	+57.5 (51.7%)
Shrinkage (1 <sup>st</sup> volume peak- 2 <sup>nd</sup> volume peak)	10	-18.8 (15.2%)	+1.6 (1.4%)	-17.1 (13.5%)	+0.77 (0.65%)	-15.3 (10.7%)	-15.3 (12.4%)
	20		-13.8 (11.5%)	-15.9 (12.8%)	-15.3 (11.7%)	-19.6 (15.3%)	-20.2 (15.1%)
	30		-15.7 (12.6%)	-18.8 (14.5%)	-24.2 (16.2%)	-17.8 (12.8%)	-21.1 (14.6%)
Maximum void ratio (XRCT/Triax)	30	0.127/0.390	0.291/0.422	0.312/0.433	0.459/0.442	0.229/0.433	0.207/0.435
Hydraulic conductivity (25 kPa)	30	6.73 x E-07	1.77 x E-07	7.22 x E -06	6.28 x E-07	2.77x E-06	3.86 X E-06
Shear strength (25/100kPa)	30	69/179	115/174	158/174	84/165	106/252	78/248
<b>2<sup>nd</sup> wetting and drying cycle</b>							
Maximum gravimetric water content	10	0.314	0.353 (12.4%)	0.339 (8%)	0.369 (17.5%)	-	0.35 (11.5%)
	20		0.331 (5.4%)	-	0.375 (19.4%)	0.339 (8%)	0.361 (14.9%)
	30		0.378 (20.4%)	0.351 (11.8%)	0.417 (32.8%)	0.391 (24.5%)	0.391 (24.5%)
Volume (change) cm <sup>3</sup> (from original)	10	+19.1	-	+21.4 (12%)	+35.0 (83.3%)	+20.2 (5.8%)	+20.5 (7.3%)
	20		+17.9 (-6.3%)	+17.5 (-8.4%)	+24.7 (29.3%)	+19.1	+23.3 (22%)
	30		+20.5 (7.3%)	+21.1 (10.5%)	+30.4 (59.2%)	+29.7 (55.5%)	+36.4 (90.6%)
Original – dried porosity (XRCT)	30	-47%	+54.4%	+97.5%	+6.1%	+80.9%	+92.2%

Table 37: Summary of soil function data, comparing the control (Soil2) with single amendments of WTR<sub>d</sub>, WTR<sub>w</sub>, and compost, and against co-amendment of WTR<sub>d</sub>/WTR<sub>w</sub> with compost at ratios of 10, 20 and 30%. Data are absolute values of change and (the percentage increase or decrease compared to the control)

## 8.1 Concluding summary

1. Water treatment residual as a single amendment imparts important physical changes to soil and can beneficially modify the soil to increase the maximum gravimetric water content, sample volume, maximum void ratio (porosity), reduces shrinkage (loss of soil volume/porosity), increase hydraulic conductivity and shear strength at low stress. There is no statistically significant advantage in processing the wet WTR<sub>w</sub> (20% solids) to lower the water content (WTR<sub>d</sub>, 20% gravimetric water content).

a) WTR<sub>d</sub> statistically significantly improves the maximum gravimetric water content of samples by between 2.7% and 13.7% (20 and 30% amendment) during the 1<sup>st</sup> wetting cycle, and by between 5.4 and 20.4% (10-30% amendment) during the 2<sup>nd</sup> wetting cycle, against the control soil (n=12, p<0.05). 10% amendment did not yield a statistically significant improvement during the 1<sup>st</sup> wetting cycle.

WTR<sub>w</sub> statistically significantly improves the maximum gravimetric water content of samples by between 4.9% and 9.9% (10 and 30% amendment) during the 1<sup>st</sup> wetting cycle, and by between 8 and 11.8% (10 and 30% amendment) during the 2<sup>nd</sup> wetting cycle, against the control soil (n=12, p<0.05). 20% amendment did not yield a statistically significant improvement.

b) Only the addition of WTR<sub>w</sub> at 30% amendment statistically significantly improves the volume increase (5.3%) compared to the control soil during the 1<sup>st</sup> wetting cycle (n = 12, p<0.01).

WTR<sub>d</sub> (30% amendment) had a statistically significant increase (7.3%) in sample volume compared to the control soil during the 2<sup>nd</sup> wetting cycle (n =12, p<0.01).

WTR<sub>w</sub> had a statistically significant increase of between 10.5 and 12% (30 and 10% amendment) in sample volume compared to the control soil during the 2<sup>nd</sup> wetting cycle (n =12, p<0.01).

c) WTR<sub>d</sub> amended samples experienced between 1.4 and 12.6% (10-30% amendment) reduction in maximum sample volume (shrinkage) between the 1<sup>st</sup> wetting and 2<sup>nd</sup> wetting sequences, compared to the control soil which had a 15.2% reduction in maximum volume (shrinkage). (n = 12, p<0.01).

WTR<sub>w</sub> amended samples experienced between 12.8 and 14.5% (20 and 30% amendment) reduction in maximum sample volume (shrinkage) between the 1<sup>st</sup> wetting and 2<sup>nd</sup> wetting sequences, compared to the control soil which had a 15.2% reduction in maximum volume (shrinkage). (n = 12, p<0.01).

d) WTR<sub>d</sub> increased the maximum void ratio by 129% (XRCT)/8.2% (Triaxial), compared to the control soil (n=1).

WTR<sub>w</sub> increased the maximum void ratio by 145% (XRCT)/11% (Triaxial) compared to the control soil (n=1).

e) WTR<sub>w</sub> as a single amendment improves the hydraulic conductivity of soil by a factor of 28 (30% amendment), by a factor of 10 for WTR2<sub>d</sub> (30% amendment), a factor of 6 for 20% WTR2<sub>d</sub> and 8 for 20% WTR2<sub>w</sub>, a factor of 6 for 10% WTR2<sub>w</sub>, and doubles *K* with 10% WTR2<sub>d</sub>.

f) WTR<sub>d</sub> improves the shear strength of soil at low testing pressure (25 kPa) by 67%, but reduces the shear strength at high testing pressure (100 kPa) by 3% compared to unamended soil.

WTR<sub>w</sub> improves the shear strength of soil at low testing pressure (25 kPa) by 129% but reduces the shear strength at high testing pressure (100 kPa) by 3% compared to unamended soil.

2. Compost as a single amendment increases maximum gravimetric water content, sample volume, maximum void ratio (porosity), and reduces shrinkage (loss of soil volume/ porosity) on drying at low amendment ratios, however the amendment increases shrinkage (at high amendment ratio), reduces hydraulic conductivity and saturated shear strength of the soil. The effect of compost was greater during the 2<sup>nd</sup> wetting cycle compared to the 1<sup>st</sup> wetting cycle.

a) Compost statistically significantly improved the maximum gravimetric water content of samples by between 13.2 and 34.7% (20 and 30% amendment) during the 1<sup>st</sup> wetting cycle, and by between 17.5 and 32.8% (10-30% amendment) during the 2<sup>nd</sup> wetting cycle, against the control soil (n=12, p<0.05). 10% amendment did not yield a statistically significant improvement during the 1<sup>st</sup> wetting cycle.

b) Compost amendment statistically significantly improved the volume increase during wetting by between 5.3 and 44.1% (20 and 30%

amendment) compared to the control soil during the 1<sup>st</sup> wetting cycle (n = 12, p<0.01).

Compost amendment statistically significantly improved the volume increase during wetting by between 29.3 and 83.3% (20 and 10% amendment) compared to the control soil during the 2<sup>st</sup> wetting cycle (n = 12, p<0.01).

- c) Compost amended samples experienced an increase in sample volume during wetting (0.65% at 10% amendment) and between 11.7 and 16.2% (20 and 30% amendment) reduction in maximum sample volume (shrinkage) between the 1<sup>st</sup> wetting and 2<sup>nd</sup> wetting sequences, compared to the control soil which had a 15.2% reduction in maximum volume (shrinkage) between the two wetting phases (n = 12, p<0.01).
  - d) Compost increased the maximum void ratio on wetting by 261% (XRCT)/8.2% (Triaxial), compared to the control soil (n=1).
  - e) Compost as a single amendment reduces the saturated hydraulic conductivity by 3.65% (10 amendment), 84.5% (20% amendment), 6.6% (30% amendment) compared to the control soil (n=1)
  - f) Compost improves the saturated shear strength of soil at low testing pressure (25 kPa) by 21.7%, but reduces the shear strength at high testing pressure (100 kPa) by 2.8% compared to unamended soil.
3. Co-amendment of WTR with compost appears to provide soil with the well-known benefits of compost addition (enhanced water holding capacity and swelling) in addition to the geotechnical improvements of the WTR (structural stabilisation through reduced shrinkage on drying, improved saturated hydraulic conductivity and saturated shear strength at 100 kPa). Co-amendments therefore perform better than single amendments of compost or WTR in terms of their 'flood holding capacity'.
- a) Co-amendment using WTR<sub>d</sub> statistically significantly improved the maximum gravimetric water content of samples by between 10.8 and 27.4% (20 and 30% co-amendment) during the 1<sup>st</sup> wetting cycle and between 8 and 24.5% (20 and 30% amendment) during the 2<sup>nd</sup> wetting cycle (n=12, p<0.01), compared to the control soil.  
Co-amendment using WTR<sub>w</sub> statistically significantly improved the maximum gravimetric water content of samples by between 4.5 and

25% (10 - 30% co-amendment) during the 1<sup>st</sup> wetting cycle and between 11.5 and 24.5% (10- 30% amendment) during the 2<sup>nd</sup> wetting cycle (n=12, p<0.01), compared to the control soil.

- b) WTR<sub>d</sub> co-amendment statistically significantly improved the volume increase by 25.9% (30% amendment) during the 1<sup>st</sup> wetting cycle and between 5.8 and 55.5% (10 and 30% amendment) during the 2<sup>nd</sup> wetting cycle compared to the control soil (n = 12, p<0.01).

WTR<sub>w</sub> co-amendment statistically significantly improved the volume increase by between 2.4 and 51.7% (20 and 30% amendment) compared to the control soil during the 1<sup>st</sup> wetting cycle and by 7.3 and 90.6% (10-30% amendment) during the 2<sup>nd</sup> wetting cycle compared to the control soil (n = 12, p<0.01).

- c) WTR<sub>d</sub> amended samples experienced between 10.7 and 12.8% (10 and 30% amendment) reduction in maximum sample volume (shrinkage) between the 1<sup>st</sup> wetting and 2<sup>nd</sup> wetting sequences, compared to the control soil which had a 15.2% reduction in maximum volume (shrinkage). (n = 12, p<0.01).

WTR<sub>w</sub> amended samples experienced between 12.4 and 14.6% (10 and 30% amendment) reduction in maximum sample volume (shrinkage) between the 1<sup>st</sup> wetting and 2<sup>nd</sup> wetting sequences, compared to the control soil which had a 15.2% reduction in maximum volume (shrinkage). (n = 12, p<0.01).

- d) WTR<sub>d</sub> co-amendment increased the maximum void ratio on wetting by 80% (XRCT)/11% (Triaxial), compared to the control soil (n=1).

WTR<sub>w</sub> co-amendment increased the maximum void ratio on wetting by 63% (XRCT)/11.5% (Triaxial), compared to the control soil (n=1).

- e) WTR<sub>d</sub> co-amendment increases the saturated hydraulic conductivity by 23% (10 amendment), 355% (20% amendment), 313% (30% amendment) compared to the control soil (n=1).

WTR<sub>w</sub> co-amendment increases the saturated hydraulic conductivity by 91% (10 amendment), 330% (20% amendment), 475% (30% amendment) compared to the control soil (n=1).

- f) WTR<sub>d</sub> co-amendment improves the saturated shear strength of soil at low testing pressure (25 kPa) by 53.6% and increases the shear

strength at high testing pressure (100 kPa) by 40.8% compared to unamended soil.

WTR<sub>w</sub> co-amendment improves the saturated shear strength of soil at low testing pressure (25 kPa) by 13%, and increases the saturated shear strength at high testing pressure (100 kPa) by 38.5% compared to unamended soil.

4. Erosional testing was inconclusive and requires method refinement to allow sufficient analysis.
5. Visualisation of WTR amended samples using XRCT allowed the identification of void spaces around WTR<sub>d</sub> (20% GWC) pores in saturated samples that remained present once the sample was dried, i.e. the porosity was retained. As  $n < 2000$ , this provided quantifiable evidence of soil architecture change with the addition of WTR by analysis of void ratios.
6. Data from the Weetslade field trial has thus far provided inconclusive data on the field application of a 10% co-amendment, and further investigation of supplementary soil parameters such as bulk density and gravimetric water content is needed to quantify changes in soil properties.

## ***8.2 Recommendations for future work***

Taking into account the effect of co-amendment on essential soil functions that determines a soil's flood holding capacity (maximum gravimetric water content, volume change, resistance against shrinkage, void ratio (porosity), hydraulic conductivity and shear strength) and the economic and environmental sustainability issues, it is worth considering a co-amendment over the single amendment of compost or WTR. The research contained within this thesis has shown that on a short-term basis at a small scale, that the application of compost and WTR has considerable influence on the flood holding capacity. In order for this information to effectively feed back into the policy framework of companies such as Northumbrian Water and the Environment Agency in regard to waste management, the following research questions need to be addressed;

- Can we use WTR as a single amendment in soil on a long-term basis without negative geochemical consequence, e.g. detrimentally affecting P availability for plants?

- What are the long-term effects, of co-amendment with WTR and compost over numerous wetting and drying cycles, on the flood holding capacity of soils?
- What is the bio-chemical effect of the addition of WTR to soils in either wet or dry forms?
- Considering 'cementation effects' suggested to occur to soils with the addition of WTR in Trial 4, what are the long-term structural effects of WTR/compost addition on soil, with respect to volume changes and shear strength?
- What is the maximum quantity of either single amendment or co-amendment that is permissible on land (as current guidelines are based on EU regulations on sewage sludge and EA discretion), without detrimental effects on the soil's plant functions and water chemistry?
- Should research determine that the long-term application of WTR/compost amendment has no implications, can we use land spreading/soil amendment to completely close the loop on WTR waste? Are there enough brownfield sites or areas of flood vulnerability to incorporate the waste?

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