Impact of Biochar and Dairy Manure on the Hydraulic Properties and their Measurements in Boreal Agricultural Podzols

A Thesis
Presented to
The Faculty of Graduate Studies
of
Memorial University of Newfoundland

by

Wanniarachchige Dinushika Kumari Wanniarachchci

In partial fulfillment of requirements for the degree of
Master of Science
in
Boreal Ecosystems and Agricultural Sciences
September 2018

Abstract

W. D. K. Wanniarachchi
Memorial University of Newfoundland, 2018

Advisor:
L. W. Galagedara

Soil hydraulic properties such as hydraulic conductivity and volumetric soil moisture content are the basis for understanding flow and transport processes in the vadose zone. In addition, hydraulic properties of surface soils influence the partition of input water (by precipitation / irrigation) into runoff and soil water storage and are altered with the use of soil amendments in agricultural soils. Therefore, the present study has two different parts *i.e.* first section looking into hydraulic conductivity and second section into volumetric moisture content measurements. The first study was focused on field unsaturated (K_{unsat}) and near saturated (near K_{sat}) hydraulic conductivity of agricultural soil with an emphasis on amending the soil with dairy manure (DM) and biochar (BC). The study was conducted at both field and the laboratory scales using mini disk infiltrometer (a tension infiltrometer). The second study evaluated the effect of BC incorporation on TDR (Time Domain Reflectrometry) based soil moisture measurements. TDR is a well-established method for measuring volumetric soil moisture content (VSMC) at point scales using soil's dielectric properties. To calculate VSMC from dielectric constant obtained from a TDR cable tester (MOHR CT 100), Topp's equation–M1, mixing model–M2 and the forest soil model–M3 were used. The three models were compared with a standard (M0) VSMC calculated using gravimetric moisture and soil bulk density. According to the results, there was no significant effect of DM and BC on near K_{sat}. BC was observed to have no considerable influence, while IN+DM 1, IN+DM 2 and IN+DM 1+BC had significantly lower K_{unsat} under 2 cm suction (p=0.009, 0.002 and 0.031, respectively) compared to the control. The results from regression analysis showed the M1 and M2 reported significantly lower VSMC values, while M3 reported higher values than M0 for both powdered and granular BC treatments (p<0.001). However, for powdered BC treatment, the relationships between M1, M3 with M0 were not significant (p=0.228, 0.052), while it was significant for M2 with M0 (p=0.028). For granular BC treatments, the M2, M3 with M0 regressions have shown significant similarity (p=0.009 & 0.032); this was not true for the M1 to M0 comparison (p=0.571). These results show that the effects of types and rates of BC on VSMC prediction models based on soil dielectric constant need to be further studied under both laboratory and field conditions. Since these soil amendments can influence soil hydrology such as reduced infiltration and increased surface runoff, carefully monitored agronomic practices are recommended.

Key words: Dairy manure, Biochar, Unsaturated hydraulic conductivity, Saturated hydraulic conductivity, Volumetric soil moisture, Time Domain Reflectometry

Acknowledgements

I would like to express my sincere gratitude to my supervisor Dr. Lakshman Galagedara not only for giving me this opportunity, his gentle guidance, the useful comments, remarks and engagement through the learning process of this master thesis but also his help and encouragements during this whole time and when I couldn't find my confidence on my own. Furthermore, I would like to thank my co-supervisor Dr. Mumtaz Cheema and members of the thesis committee Dr. Adrian Unc and Dr. Raymond Thomas for the support along the way. Also, I would like to thank Dr. Michele Piercey-Normorethe Dean of School of Science and the Environment of Grenfell Campus.

Also, financial support from the Research and Development Corporation (RDC), of the Government of Newfoundland and Labrador (Ignite R&D – 5404-1962-101) and the Memorial University of Newfoundland is highly appreciated.

Next, I would like to thank my colleagues; Emmanuel Badewa, Kamalesh Sadathcharam, Chameera Illawathure, Marli Vermooten, Waqas Ali, Waqar Ashiq, Muhammad Zaeem who have willingly shared their precious time and help during field data collection and Abigail Owusuah in laboratory data collection.

Also, I would like to thank Krista Hansen, Roy Robitschek, Anna, Sarah, Joshua and Dayna for giving me all the support, love and warmth I needed during this two years and providing me a home away from home. Also, I would like to thank Dr. N.D.K. Dayawansa for her continuous motherly guide as always.

Finally, I must express my very profound gratitude and love to my parents, sister, niece, cousins and friends at home for providing me with unfailing support and continuous encouragement throughout my years of study. This accomplishment would not have been possible without them. I will be grateful forever for your love.

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A – A computed value related to the van Genuchten parameters for a specific soil type

BC – Biochar

BC_G – Granular Biochar

BC_P – Powdered Biochar

BD (ρ_b) – Bulk Density

C – Velocity of an EM wave in free space (2.988×10^8 m/s)

C_1 – Parameter related to hydraulic conductivity (ms^{-1})

C_2 – Soil sorptivity ($ms^{-1/2}$)

CEC – Cation Exchange Capacity

DM – Dairy Manure

EC – Electrical Conductivity

EM wave – Electromagnetic wave

h_0 – Suction at the infiltrometer's disk surface

I – Cumulative infiltration

K – Hydraulic Conductivity

Ka – Dielectric Constant

K_a – Dielectric Constant of soil air

K_b – Bulk Dielectric Constant

K_s – Dielectric Constant of soil solid

K_{sat} – Saturated Hydraulic Conductivity

K_{unsat} – Unsaturated Hydraulic Conductivity

K_w – Dielectric Constant of soil water

L – Length of the waveguide

l_a – Apparent length

n – van Genuchten parameter

n – Soil Porosity

pH – Acidity of a medium

r_0 – Radius of the infiltrometer's disk

SOM/OM – Soil Organic Matter/ Organic Matter

t – Time

TDR – Time Domain Reflectometer

v – Velocity

VSMC (θ) – Volumetric Soil Moisture Content

V_T – Total soil volume

V_w – Volume of water

WDPT – Water Drop Penetration Time

α – van Genuchten parameter

CHAPTER 1

INTRODUCTION

1.1 Overview

The unsaturated zone or zone of aeration or vadose zone is the porous soil subsurface above the groundwater table consisting of soil solid, air, and water. This zone is absent in some places such as in lakes, shallow in places such as in wetlands, and deeper in places such as soil in arid regions (Heinse & Link, 2013). In agricultural and arable soils, the unsaturated zone provides habitats; water and nutrients to plants and soil organisms (Tindall et al., 1999; Selker et al., 1999). Hydrologically, it is the main factor controlling water entry, storage and movement from the land surface to the aquifer/ groundwater (Li et al., 2017). Thus, understanding the dynamics of the unsaturated zone is critical for the use and management of groundwater. At regional scales, the processes and functions in unsaturated zone control both short and long-term dynamics in watershed and basin water balance (Harter & Hopmans, 2004). Also, this is the initial contact layer of contaminant transport to groundwater. The flow rates and chemical reactions in the unsaturated zone control where and how contaminants enter the groundwater, which is crucial in understanding groundwater quality (Heinse & Link, 2013; Harter & Hopmans, 2004).

The fundamental measure of water content and the flow in an unsaturated zone is volumetric soil moisture content (VSMC) or wetness (θ), defined as the ratio of water volume to the total soil volume. Amount of moisture in agricultural soil plays a critical role in a number of biophysical processes from seed germination to crop harvest. Not only it is a key factor in plant growth and nutrition uptake, also it is important in soil microbial growth, organic matter decomposition, nutrient transformation in the root zone and heat transfer at the land-atmosphere interface etc. (Dari et al., 2018; Schwingshackl et al., 2017; Champagne et al., 2012; Koster et al., 2010). Therefore, the measurement of VSMC is necessary for agricultural and horticultural field monitoring as well as for large scale climate models.

The rate of water movement through soil is also important in water entry into soil, evaporation from the soil surface, flow to wells and drains, water movement to plant roots etc. Therefore, the second important characteristic govern water movement is the hydraulic conductivity (K), which is the ability of the soil to transmit water (Perkins, 2011). It is a measure of how easily water moves under a given driving force and is highly sensitive and nonlinearly depend on the water content. Also, knowledge of soil K helps in improved and accurate runoff and flood forecasting, proper designing of water control structures, earthen storage facilities etc. In unsaturated soils, the K highly depends on the VSMC since water flows through the soil pores which are filled with water (Gallage et al., 2013). Conversely, as the soil dries, the total number of flow paths that water can travel decreases. Higher VSMC in coarser soils have higher K whereas finer soils have lower K. The relationship between VSMC and K_{unsat} (K-function) shows the change in flow rate and gradient in the soil (McCartney et al., 2007). The rate of decrease in K with decreasing VSMC is steeper in coarser soils.

The vadose zone is the mostly accessible zone for human activities such as agriculture, construction, mining and waste disposal (Nimmo, 2009). Therefore, various constituents, processes and participants modify this environment making it very complex and complicated to study and manage (Heinse & Link, 2013). In agricultural soils, this zone is continuously being altered with the increase of use of soil amendments, as well as other management practices such as tillage, irrigation etc.

Soil amendments are the materials added to soil to improve it's physical, chemical and biological properties (Traunfeld & Nibali, 2015). Unlike inorganic fertilizer which is added to soil to improve soil fertility, the main purpose of using soil amendments is not necessarily to improve soil fertility, but to improve overall soil properties (Page-Dumroese et al., 2018; Zhou et al., 2016). Dairy manure (DM) and biochar (BC) are two such amendments used by most farmers in tropical countries. While DM is commonly used in temperate regions, use of BC to improve soil properties is also gaining attention among farmers in cool climates.

DM is the dairy cattle waste including their urine and bedding material that is used for fertilizing cultivated lands. Addition of DM increased soil organic matter (SOM) and soil aggregation (Jiao et al., 2006), improved soil tilth and porosity (Whalen & Chang, 2002), decreased soil bulk density (BD) hence increased soil infiltration rates and K_{sat} (Eghball 2002; Benbi et al., 1998; Darwish et al., 1995) and enhanced crop yields (Jarvis et al., 1995). Also, a very important and indirect effect reported due to high rates of manure amendment is that the soil can become water-repellent (Olsen et al., 1970) due to production of water-repellent organic substances following decomposition by fungi (Weil

& Kroontje, 1979) and gradual intermixing of hydrophobic substances or hydrophobic coatings formed on the mineral soil particles (Leelamanie, 2016; Hallett, 2007; DeBano, 2000). This cause reduction in infiltration rates or uneven patterns of infiltration (Rodny et al., 2015; de Jonge et al., 1999), influences water flow in the soils through phenomena such as bio-crust formation (Lichner et al., 2012). This leads to increased surface runoff and overland flow intensifying soil erosion (Pires et al., 2006; Goebel et al., 2004a; Doerr et al., 2000).

BC is biomass pyrolyzed under low or no oxygen and very high temperature conditions and used as a soil amendment and growing media in agriculture. The addition of BC also provides suitable habitats to microorganisms. Therefore, facilitating aggregate formation by stimulating microbe and fungal activity, increasing their exudate production, and providing greater binding agents between particles (Six et al., 2002). It is also possible that aromatic components in BC contribute to the stabilization of microaggregates (Brodowski et al., 2006; Tisdall & Oades, 1982). Earthworms mix BC through the soil profile and assist aggregate stabilization (Topoliantz et al., 2006) and soil aggregates prevent rapid biodegradation of SOM and enhance the soil structure, overall surface area, the porosity and reduce bulk density (Abel et al., 2013; Mukherjee & Lal, 2013; Novak et al., 2012; Chan et al., 2007; Tisdall & Oades, 1982). This result in increased soil moisture contents, infiltration, soil water flow and improvement in soil aeration and soil strength (Major et al., 2010; Downie et al., 2009). Also, these studies showed that BC improved soil chemical properties such as pH and cation exchange capacity (CEC) (Liang et al., 2006).

However, effects of BC and DM on K_{unsat} and K_{sat} vary in the literature and studies reporting the effects of soil amendments on K_{unsat} are very few (Villagra-Mendoza, 2017; Miller et al., 2002), and the reports are even fewer in relation to podzolic soils common in boreal ecosystems. Generally, podzolic soils are formed in coarse- to medium-textured, acidic parent materials, under forest or heath vegetation in cool climates. However, it can occur in wet sandy sites in areas of sub humid climates and can be formed from calcareous parent materials. Podzols are distinctively characterized by illuviated B horizons where humified organic matter combined with Al and Fe accumulation, often overlain by a light colored eluviated (Ae)horizon (Soil Classification Working Group, 1998). Despite of the growing need for food production and expanding agriculture in boreal cool climates and specially in Newfoundland, there is limited information available on hydraulic properties and water management on podzolic soils for effective agricultural production (Badewa et al., 2018).

One study found that addition of BC enhances soil microporosity, hence enhancing K_{unsat} at higher matric potentials and rapidly decreasing towards lower potentials of sandy and sandy loam soils (Villagra-Mendoza, 2017). Also, some studies have reported higher K_{sat} when BC is applied because of improvements in the structure and the porosity of the amended soil (Herath et al., 2013; Lei & Zhang 2013; Uzoma et al., 2011b; Major et al., 2010; Asai et al., 2009; Oguntunde et al., 2008). Conversely, some researchers reported that the addition of BC might significantly decrease (Barnes et al., 2014; Githinji, 2014; Deveraux et al. 2012; Uzoma et al., 2011a, b; Brockhoff et al., 2010) or has no effects

(Rogovska et al., 2014; Hardie et al., 2014; Ouyang et al., 2013; Busscher et al., 2010; Laird et al., 2010) on the K_{sat} of sandy, loamy-sand and loamy soils. Another study found that long-term manure application had little or no effect on K_{unsat} (−0.3, −0.5, −0.7, and −1.0 kPa) (Miller et al., 2002); while short-term applications increased soil infiltration rates and K_{sat} (Eghball, 2002; Benbi et al., 1998; Darwish et al., 1995).

Despite of extensive research studies carried out to investigate the utilization of BC as a soil amendment to improve soil's physical properties (Chan et al., 2007; Novak et al., 2012; Abel et al., 2013; Mukherjee and Lal, 2013) and VSMC and water availability for plants (Kameyama et al., 2014), they have not necessarily offered any evidence for thorough evaluation of techniques and measurements of VSMC. Even though Time Domain Reflectometry (TDR) is the most widely used method in agriculture, forestry, soil science, hydrology etc., to measure VSMC of mineral soils, based on our understanding, it's applicability and accuracy of TDR based estimation models in agriculturally important BC amended soils is not studied and evaluated extensively.

Therefore, application of BC and DM as soil amendments in agriculture or to soil based or non-soil based growing media requires further understanding of its effects on the physiochemical properties of the soil or the media. Also, VSMC, K_{unsat} and K_{sat} and their variabilities, are essential to describe the infiltration capacity and flow and solute transport in such soils with different soil amendments. This includes both studying the effects on

VSMC, K_{unsat} and K_{sat}, and developing accurate methods for the measurement of these soil properties in amended soils including podzols characteristics of boreal ecosystems.

1.2 Objectives

Therefore, to achieve the overall goal of this thesis, two different studies were designed to address the impact of soil amendments on 1) Hydraulic conductivity 2) VSMC

The first study evaluated the effect of the application of DM and BC as soil amendments on soil K_{unsat}. Thus, this study focuses on K_{unsat} and near K_{sat}, with an emphasis on the amending podzolic soil with DM and BC at both the field and the laboratory scales using mini disk infiltrometer (a tension infiltrometer).

The objective of the second study was to determine the effects of BC incorporation on TDR measurements. I hypothesized that the incorporation of BC affects the measurements of dielectric constant as obtained with TDR. The study evaluated three existing calibration models that link VSMC and the TDR obtained dielectric constants, and the applicability of these models for agriculturally important BC amended soils.

1.3 Theoretical Background

1.3.1 Volumetric Soil Moisture Content and Measurements

Among the techniques used in measuring VSMC, TDR has been widely used as a method with enormous potential and minimum soil destruction (Yu & Yu, 2006). The technique is based on the travel time analysis of electromagnetic (EM) wave propagation in a metallic waveguide of a known length inserted into the soil. The EM frequencies range from 1 MHz to 1 GHz at a speed of 30 cm ns^{-1} (Yu & Yu, 2006; Jones et al., 2002; Cassel et al., 1994; Topp et al., 1980).

The travel velocity, *v*, of an EM wave through media can be calculated as:

$$v = \frac{c}{\sqrt{K_a}} \qquad \text{Eq. 1}$$

Where; *c* is the velocity of an EM wave in free space (2.988×10^8 m/s) and K_a is the dielectric constant. The time, *t*, for the EM wave travel the length (*L*) of the waveguide and back, *2L* is given by:

$$t = \frac{2L}{v} \qquad \text{Eq. 2}$$

Substituting Eq. (1) in (2):

$$K_a = \left(\frac{ct}{2L}\right)^2 \qquad \text{Eq. 3}$$

By defining (*ct/2*) as the apparent length l_a, the K_a can be calculated as:

$$K_a = \left(\frac{l_a}{L}\right)^2 \qquad \text{Eq. 4}$$

In the TDR signal, the l_a is determined from analyzing the time elapse between reflections of top and end of waveguide/cable (Figure 1.1).

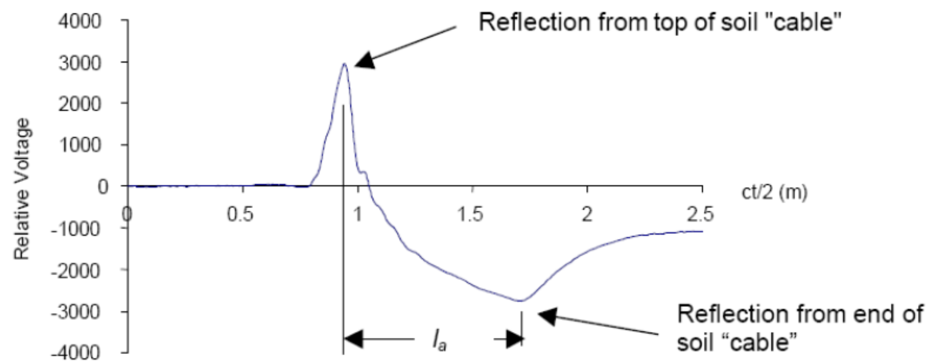

Figure 1.1: A typical TDR curve for soil showing measurement of apparent length l_a (Drenvich et al., 2001)

The VSMC, (θ_v), frequently used by agronomists, can be defined as the ratio of the volume of water, V_w, to the total soil volume, V_T.

$$\theta_v = \frac{V_w}{V_T} \qquad \text{Eq. 5}$$

There are a number of factors that influence measurements of soil K_a, most of them are soil physical properties such as soil porosity, BD, temperature, soil water (free and bound), soil air and mineral/clay fractions (Jones et al., 2002; Schaap et al., 1996; Roth et al., 1990; Topp et al., 1980). Considering the above factors, several empirical and mixing models have been developed to relate soil moisture content to soil K_a (Jones & Or, 2002).

Topp et al. (1980) proposed one of the most widely used calibration equations (Eq. 6) for estimating VSMC.

$$\theta_v = 4.3 \times 10^{-6}K_a^3 - 5.5 \times 10^{-4}K_a^2 + 2.92 \times 10^{-2}K_a - 5.3 \times 10^{-2} \qquad \text{Eq. 6}$$

Where θ_v is the VSMC and K_a is the apparent dielectric constant.

Although this relationship estimates the VSMC with an error of 0.013 m^3/m^3, it fails to describe the $\theta_v - K_a$ relationship for moisture contents higher than 0.5 m^3/m^3, as may be found in organic soils, and in soils with high clay contents (Jones et al., 2002).

The mixing models are based on dielectric constants and volume fractions of solids, water and air of soils to obtain a relationship, and therefore it introduces a composite (bulk) dielectric constant (K_b) (Friedman, 1998; Roth et al., 1990; Dobson et al., 1985; Birchak et al., 1974).

According to Roth et al. (1990), K_b is expressed as:

$$K_b = \left[\theta K_w^{\beta} + (1-n)K_s^{\beta} + (n-\theta)K_a^{\beta}\right]^{1/\beta} \quad \text{Eq. 7}$$

Where, n is the soil's porosity, $1\text{-}n$, θ, and $n\text{-}\theta$ are fractions of volume for each component and, K_s, K_w, and K_a are the dielectric constants of soil solids, water and air respectively.

Rearranging Eq. 7 and solving for θ:

$$\theta = \frac{K_b^{\beta} - (1-n)K_s^{\beta} - nK_a^{\beta}}{K_w^{\beta} - K_a^{\beta}} \quad \text{Eq. 8}$$

Many studies have used β=0.5 (Roth et al., 1990) to obtain a calibration curve for VSMC.

For example, introducing known values (β=0.5, K_a =1, K_s =4 and K_w =81) for the various constituents, into Eq. 8:

$$\theta = \frac{\sqrt{K_b} - (2-n)}{8} \quad \text{Eq. 9}$$

Soils with higher OM content and many horticultural growth media have properties distinct from typical mineral soils such as higher porosity, low BD, substantial amounts of bound water fraction, and limited clay amounts, all of which affect the dielectric signatures. Schaap et al. (1996) obtained an empirical expression for organic forest soil horizons (Eq. 10). I therefore have also tested this model given that; 1) BC has been proposed as an amendment for improving horticultural media, particularly under greenhouse conditions, and 2) TDR is the most commonly used method to measure VSMC in this industry. Also, the estimation of VSMC and EC with very high accuracy is essential for the efficiency level expected in this industry compared to field crop production.

$$\theta = (0.133\sqrt{K_b} - 0.146)^{0.885} \qquad \text{Eq. 10}$$

Although, consideration of the soil bulk density should improve calibration models, the influences of bound water fraction, which mostly correlated to the surface area of the soil/ media particles, maybe not easier to be resolved due to large variations of each soil type and local conditions including origin of the organic matter and degree of decomposition (Oleszczuk et al., 2007).

1.3.2 Soil Hydraulic Conductivity and Measurements

There are number of experimental and empirical methods used in both field and laboratory to determine hydraulic conductivity of soil. The use of tension disk infiltrometer is one such method that has been developed to measure field hydraulic conductivity (Angulo-Jaramillo et al., 2000; Ankeny et al., 1991; Perroux & White, 1988).

In this study, mini-disk tension infiltrometer was used to estimate K_{unsat} and near K_{sat} of soil surfaces (Figure 1.2). This instrument has been used to determine K_{unsat} of soils under various conditions including different plant covers and soil types, and in soils after wildfires (Dohnal et al., 2010; Homolak et al., 2009; Lewis et al., 2006). The tension infiltrometer measures the K_{unsat} at different applied tensions. The instrument consists of a water reservoir, a mariotte tube, a bubble chamber, a suction control tube and a porous sintered stainless-steel contact disc 4.5 cm in diameter and 3.0 mm thickness (METERGroupInc., 2017).

The principle is based on maintaining the water under a controlled tension using the mariotte tube in the bubble chamber. Therefore, only pores with matric potential lower than the applied tension (or higher soil suction) can be filled. Infiltration is realized until it reaches a constant infiltration rate (Siltecho et al., 2015). K_{unsat} is calculated using the method proposed by Zhang (1997).

With this technique, K_{unsat} in the soil matrix can accurately be estimated eliminating preferential flow caused by cracks, bio-macropores and other structures (Siltecho et al., 2015; Simunek & van Genuchten, 1996).

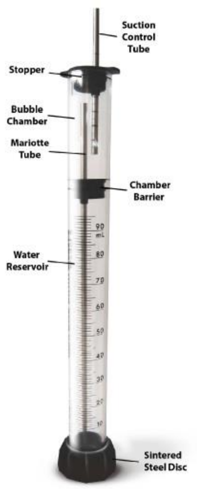

Figure 1.2: Mini Disk Infiltrometer (Source: www.metergroup.com)

Cumulative infiltration (I) is calculated using Eq. 11.

$$I = C_1 t + C_2\sqrt{t} \qquad \text{Eq. 11}$$

Where, C_1 is a parameter related to hydraulic conductivity (ms^{-1}) and C_2 is soil sorptivity ($ms^{-1/2}$). The hydraulic conductivity (k) for the soil is then computed from Eq. 12.

$$k = \frac{C_1}{A} \qquad \text{Eq. 12}$$

Where, C_1 is the slope obtained from the relationship between cumulative infiltration and the square root of time. A is computed as in Eq. 13 and 14 and it is related to the van Genuchten parameters for a specific soil type to the suction rate and the radius of the infiltrometer disk.

$$A = \frac{11.65\ (n^{0.1}-1)\ \exp[2.92\ (n-1.9)\propto h_0]}{(\propto r_0\ )^{0.91}} \qquad \text{Eq. 13}$$

$$A = \frac{11.65\ (n^{0.1}-1)\ \exp[7.5\ (n-1.9)\propto h_0]}{(\propto r_0\ )^{0.91}} \qquad \text{Eq. 14}$$

Where, n and α are the van Genuchten parameters (Carsel & Parrish, 1988), r_0 is the radius of the infiltrometer's disk (2.25 cm), and h_0 is the suction at the infiltrometer's disk surface.

1.3.3 Water Drop Penetration Time (WDPT)

The hydrophobicity was determined according to Leelamanie et al. (2008) using the water drop penetration time (WDPT). The method is to place a drop (about 2.5 ml) of de-ionized water on a flat soil (air dried for 72 h) surface using a dropper and time taken by the water drop to fully penetrate the surface is recorded using a stopwatch. The degree of water repellency was determined according to Table 1.1 (Leelamanie et al., 2008; Chenu et al., 2000; Bisdom et al., 1993; King, 1981).

Table 1.1: Water drop penetration time classes and corresponding repellency rating.

WDPT class (s)	$0 \leq 1$	1- 60	60 - 600	600 - 3600	>3600
Repellency rating	Non-repellent	Slightly Repellent	Strongly Repellent	Severely Repellent	Extremely Repellent

1.4 Format of the Thesis

The thesis is organized as four chapters.

Chapter one provides an overall overview with theoretical backgrounds and overall thesis objectives.

Chapter two discuss the study on impact of soil amendments on hydraulic conductivity of loamy sand soils in western newfoundland, with relevant literature.

Chapter three describes the effect of biochar on TDR based volumetric soil moisture measurements in a loamy sand soil, with relevant literature.

Chapter four provides an overall discussion to the thesis, conclusions and future recommendations.

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1.6 Co-authorship Statement

A manuscript based on the Chapter 2, entitled “Impact of Soil Amendments on the Hydraulic Conductivity of Boreal Agricultural Podzols” has been submitted to Canadian Biosytems Engineering Journal (Wanniarachchi, D., Cheema, M., Thomas, R., Kavanagh, V. and Galagedara, L., 2018). Dinushika Wanniarachchi, the thesis author was the primary author and Dr. Galagedara (supervisor), was the corresponding and the last author. Dr. Cheema (co-supervisor) and Dr. Thomas (committee member) were second and third authors, respectively. Dr. Kavanagh, a collaborative researcher of this project from the Department of Fisheries and Land Resources of the Government of NL was the fourth author. All authors were part of the same research project on “Hydrogeophysical Characterization of Agricultural Fields in Western Newfoundland using Integrated GPR-EMI”, which was led by Dr. Galagedara. For the work in Chapter 2, the design of the study was developed by Dinushika Wanniarachchi with input from Dr. Galagedara. Ms. Wanniarachchi was responsible for the data collection, analysis, and interpretation and writing of the manuscript. Dr. Galagedara provided specific guidance on statistical analysis, data interpretation and manuscript writing. Drs. Cheema, Thomas and Kavanagh provided inputs for the field experiment, data interpretation and manuscript editing. For the remainder of the thesis including introduction, literature review, data collection, analysis and interpretation and writing, was performed by Ms. Wanniarachchi. The Chapter 3 is on “Effect of Biochar on TDR based Volumetric Soil Moisture Measurements in a Loamy Sand Podzolic Soil”. has been submitted to Transactions of the ASABE

(Wanniarachchi, D., Unc, A., Cheema, M. and Galagedara, L., 2018). Dinushika Wanniarachchi, the thesis author was the primary author and Dr. Galagedara (supervisor), was the corresponding and the last author. Dr. Unc (committee member) and Dr. Cheema (co-supervisor) were second and third authors, respectively. All authors were part of the same research project on "Hydrogeophysical Characterization of Agricultural Fields in Western Newfoundland using Integrated GPR-EMI", which was led by Dr. Galagedara. For the work in Chapter 3, the design of the research was developed by Dr. Galagedara and Dr. Unc with input from all members of the group. Ms. Wanniarachchi was responsible for the data collection, analysis, and interpretation and writing of the manuscript. Dr. Unc provided specific guidance on statistical analysis, data interpretation and manuscript writing. Dr. Cheema provided inputs for the field experiment, data interpretation and manuscript editing. Dr. Galagedara as the project lead and the main supervisor, provided research plans and guidance for the entire process.

CHAPTER 2

IMPACT OF SOIL AMENDMENTS ON THE HYDRAULIC CONDUCTIVITY OF BOREAL AGRICULTURAL PODZOLS

Dinushika Wanniarachchi, Mumtaz Cheema, Raymond Thomas, Vanessa Kavanagh, Lakshman Galagedara (2018). Impact of Soil Amendments on the Hydraulic Conductivity of Boreal Agricultural Podzols (Submitted to Canadian Biosytems Engineering Journal).

Abstract

Hydraulic properties of soil are the basis for understanding the flow and transport through the vadose zone, and different soil amendments can alter soil properties affecting soil hydrology. The aim of this study was to determine the effect of soil amendments on soil infiltration under both unsaturated and near-saturated conditions of agriculturally used loamy sand (73.7±4.1 % sand + 23.0±3.8 % silt + 3.3±0.3 % clay) soils. Both field and laboratory experiments were conducted using two soil amendments: dairy manure (DM) and biochar (BC). DM and BC were incorporated up to a depth of 20 cm at a rate of 30,000 L ha^{-1} and 20 t ha^{-1}, respectively. The experimental design was a randomized complete block and the plots were planted with silage-corn (*Zea mays*), and not irrigated. The treatments were: control without amendment (0N), inorganic N fertilizer (IN), two types of DM (IN+DM1 and IN+DM2), IN+BC only and IN+DM1+BC. Soil samples collected from these treatment plots were used in the laboratory experiment. Infiltration data were collected using a mini disk infiltrometer under three suction levels in the field study, where 4 and 2 cm was ascribed as unsaturated and 0.1 cm ascribed as near-saturated condition. Five suction levels (6, 4, 2, 1, 0.1 cm) were used in the laboratory study. Based on the measured infiltration rates, unsaturated (K_{unsat}) and near-saturated (near K_{sat}) hydraulic conductivities were calculated. There were no significant effects from DM and BC on near K_{sat}. Treatments IN+DM1, IN+DM2 and IN+DM1+BC under field condition and only IN+DM2 under laboratory conditions significantly reduced the K_{unsat} compared to the control. Since these soil amendments can influence soil hydrology such as reduced

infiltration and increased surface runoff, carefully monitored application of soil amendments are recommended.

Keywords: Biochar, Dairy manure, Hydraulic conductivity, Infiltration, Soil amendments

2.1 Introduction

The unsaturated zone, also called zone of aeration or vadose zone, is the porous soil subsurface above the groundwater table consisting of solids, air, and water. In agricultural and arable soils, the unsaturated zone provides air, water, and nutrients to plants and soil organisms (Tindall et al., 1999; Selker et al., 1999). Hydrologically, it is the main factor controlling water and contaminant entry, storage, and movement from the soil surface to the aquifer/groundwater (Li et al., 2017). Thus, understanding the dynamics of the unsaturated zone is critical for the use and sustainable management of groundwater. Hydraulic properties of soils such as hydraulic conductivity and the moisture retention function are fundamentals for understanding flow and transport through the soil matrix and are important inputs in vadose zone simulations (Ouyang et al., 2013; Zhang, 1997). In addition, saturated and unsaturated hydraulic properties of any soil including cultivated surface soils influence the separation of input water (i.e. precipitation/irrigation) into runoff and soil water storage (Eusufzai & Fujii, 2012). Unsaturated (K_{unsat}) and saturated (K_{sat}) hydraulic conductivity is a measure of how water flows through an unsaturated and saturated soil profile respectively and is one of the main properties considered in governing water flow (Perkins, 2011).

Soil amendments are materials added to soil to improve its physical, chemical and biological properties (Traunfeld & Nibali, 2015). Unlike inorganic fertilizer which is added to improve soil fertility, the main purpose of using soil amendments is not necessarily to improve soil fertility, but to improve overall soil properties (Page-Dumroese et al., 2018; Zhou et al., 2016). Dairy manure (DM) and biochar (BC) are two such amendments used by farmers worldwide to improve soil properties. While the addition of DM increases soil organic matter (SOM) and soil aggregation (Jiao et al., 2006), the addition of BC also changes soil properties and provides suitable habitats for microorganisms (Lopez, 2014). Therefore, both DM and BC facilitate aggregate formation by stimulating microbial and fungal activity, increasing their exudate production, and providing greater binding agents between soil particles (Six et al., 2002). In addition, aromatic components in BC can also contribute to the stabilization of microaggregates (Brodowski et al., 2006; Tisdall & Oades, 1982). Earthworms mix BC through the soil profile and assist aggregate stabilization (Topoliantz et al., 2006). Soil aggregates are very important in soil property determination because it prevents rapid biodegradation of SOM, thus enhancing the soil structure and porosity (Sarker et al., 2018; Chaplot & Cooper, 2015).

The physical and hydraulic properties of agricultural soils are continuously being altered with the increasing use of these soil amendments in addition to other management practices. Previous studies have reported that DM amendments improved the soil tilth and porosity (Whalen & Chang, 2002), increased SOM, decreased soil bulk density, increased soil infiltration rates, K_{sat} (Eghball, 2002; Benbi et al., 1998; Darwish et al., 1995), and increased crop yields (Jarvis et al., 1995). Also, studies showed that BC improved soil chemical properties such as pH and cation exchange capacity (CEC) (Liang et al., 2006), as well as physical properties such as porosity and bulk density (Abel et al., 2013; Mukherjee & Lal, 2013; Novak et al., 2012; Chan et al., 2007). Collectively, both the chemical and physical properties are known to increase the infiltration and soil water flow. In addition, the increased surface area and porosity have been shown to influence the soil structure through changing the overall surface area, amount, size, and distribution of soil pores, and bulk density, thus improving soil aeration and soil strength (Major et al., 2010; Downie et al., 2009). Another advantage of BC application to agricultural soils is reduction in greenhouse gas emissions (Bamminger et al., 2017; Agegnehu et al., 2016; Sackett et al., 2014) and carbon sequestration (Du et al., 2017; Nanda et al., 2016; Lin et al., 2015; Lorenz & Lal, 2014).

It is important to note that amending the soil with different types and rates of DM and BC may have varying as well as specific influences on soil properties due to the composition and inherent properties of DM and BC. The properties and composition of DM can be affected by several factors such as the stage of growth and health conditions of the herd and feeding practices. These factors can determine the quality and quantity of excretion as well as the amount and type of bedding, amount of water used in barn or added to manure, type and duration of storage of manure, and weather conditions (Larney et al., 2006). The type of biomass used as the feedstock and pyrolysis conditions such as temperature and charring time can affect BC properties. The effects of BC on the soil properties may also change according to the type of soil and climatic conditions (Lopez, 2014; Herath et al., 2013; Mukherjee & Lal, 2013). Additionally, the maturity of these amendments, especially after field application, will have different and complex effects on soil properties (Neufeld et al., 2017; Burrell et al., 2016; D'Amours et al., 2016; Zrim, 2016; Min et al., 2003).

A very important and indirect effect reported due to high rates of manure application is that the soil can become water-repellent (Olsen et al., 1970). This may be caused from the production of water-repellent organic substances following decomposition by fungi (Weil & Kroontje, 1979) and/or the gradual intermixing of hydrophobic substances or hydrophobic coatings formed on the mineral soil particles (Leelamanie, 2016; Hallett, 2007; DeBano, 1981). This can reduce infiltration rates or result in uneven patterns of infiltration (Rodny et al., 2015; de Jonge et al., 1999) that can influence water flow in the soil through phenomena such as bio-crust formation (Lichner et al., 2012); thus, increasing surface runoff and overland flow, thereby intensifying soil erosion (Pires et al., 2006; Goebel et al., 2004a; Doerr et al., 2000).

In addition to increased water repellency caused from high DM application, localized patches with higher water infiltration potential can also formed, that allow selective water entry into the soil stimulating preferential flow paths, hence bypassing the complete wetting of the soil matrix. This can cause microbial, nutrient and agrochemical leaching, subsequently increasing groundwater contamination (Kodesova et al., 2015; Hallett, 2007; Bauters et al., 2000). Also, this localized leaching can create problems for crops due to nutrient deficiency and decreased availability of soil moisture in the rhizosphere (Ward et al., 2015; Madsen et al., 2012).

The knowledge of the K_{unsat} and K_{sat} and its variability, therefore, is essential to describe the infiltration capacity, flow and solute transport in such soils where different soil amendments are added. As such, the main objective of this study was to evaluate the effect of the application of DM and BC as soil amendments on infiltration capacity under both unsaturated and near-saturated conditions. This study focuses on estimating K_{unsat} and near K_{sat}, using a mini disk infiltrometer at both the field and the laboratory scales with an emphasis on amending agriculturally used podzolic soil with DM and BC.

2.2 Materials and Methods

The study was conducted using different treatments of DM and BC in a field-based silage corn experiment. Infiltration experiments under controlled conditions were conducted in the laboratory using treated soil samples collected from the silage corn experimental field.

2.2.1 Field study

The study was conducted at Pynn's Brook Research Station (PBRS) of the Department of Fisheries and Land Resources, Government of Newfoundland and Labrador, Pasadena (49°04'22.6"N 57°33'38.9"W), Canada. Based on 30-year data (1986–2016) from the Deer

Lake weather station of Environment Canada (http://climate.weather.gc.ca/), the area receives an average precipitation of 1113 mm per year with less than 410 mm falling as snow, and has an annual mean temperature of 4 °C and based on a handheld GPS, the elevation is between 43 and 50 m a.s.l. The soil tested is classified as a loamy sand podzol (73.7±4.1% sand + 23.0±3.8% silt + 3.3±0.3% clay) (Badewa, 2017).

Experimental design and land preparation

The experiment was conducted using a randomized complete block design containing 32 experimental plots each having dimensions of 1 m width and 5 m length and planted with silage-corn (*Zea mays*). There were eight treatments in the main project and each treatment was replicated four times. However, only six treatments were considered for the present study to evaluate DM and BC effect as shown in Fig. 2.2. The treatments included amendments of two types of dairy manure according to their total nitrogen (N) and total phosphorous (P) contents (DM1 with high N, P and DM2 with low N, P) and granular BC produced by slow pyrolysis of Yellow pine wood (*Pinus taeda*). The Tables 2.1 and 2.2 provide some basic properties and characteristics of BC and DM used in the present study.

Table 2.1: Basic properties of biochar used in the study

Feedstock	Yellow pine wood (*Pinus taeda*)
Particle size	1–6 mm
Bulk Density (g cm^{-3})	0.20
Moisture	15.2 %
pH (1: 10 BC: Water)	9.0
EC_a at 21 – 22 °C (dS m^{-1})	5.2
Fixed carbon	87.3 %
Volatile Carbon (600 °C)	12.7 %
Ash	6 %

DM application rates were based on their equivalent N content with inorganic N fertilizer. The field was ploughed, with a spring disc plough for seed bed preparation. DM and BC were incorporated at a rate of 30,000 L ha^{-1} and 20 t ha^{-1}, respectively, and thoroughly mixed within the top 20 cm of the soil. DM application was according to the local dairy farmer practices applied as a liquid slurry and BC was applied via surface broadcasting. Silage corn was seeded with SAMCO system (SAMCO Agricultural Manufacturing, Ireland) having the ability to simultaneously cover the seeded fields with plastic sheets. The plastic sheet provides a cover to enhance the heat units during early growth stages of corn in the cool climate production system. The field was not irrigated, thus the crop relied solely on seasonal precipitation. The infiltration tests were carried out in the middle of the growing season when the crop was at the tasseling stage (August 4 - 22, 2017).

Table 2.2: Basic characteristics of two types of dairy manure used in the study (Ashiq, 2018)

Characteristic (as received basis)	DM 1	DM 2
Dry matter (%)	10.90	1.70
pH	6.80	7.10
Total Nitrogen (%)	0.44	0.12
Total Phosphorus (%)	0.08	0.01
Total Potassium (%)	0.37	0.12
Total Calcium (%)	0.19	0.04
Total Magnesium (%)	0.07	0.01
Total Iron (ppm)	68.00	7.00
Total Manganese (ppm)	21.00	5.00
Total Copper (ppm)	4.50	20.00
Total Zinc (ppm)	21.00	5.00
Total Boron (ppm)	3.40	0.50
Total Sodium (ppm)	904.00	241.00

2.2.2 Laboratory study

The experiment was conducted at the laboratory of the Boreal Ecosystem Research Facility, Grenfell Campus, Memorial University of Newfoundland, Canada.

Collection and preparation of soil samples

Disturbed composite soil samples from three locations of each of the treatment plots cultivated with silage corn (middle of the growing season) were collected at a depth ranging from 0 (surface) to 10 cm. The samples were air-dried for 72 h, pulverized, sieved (< 4 mm) and homogenized. The < 4 mm sieve was used to ensure that the finer BC particles (≤ 2mm) remained in the soil. At the same time, the bulk density of each plot sampled was determined using the collected undisturbed soil samples and by dividing the mass of the oven dried soil by the total sample volume.

The soil samples were packed into 750 mL plastic containers with drainage holes in the bottom to facilitate free liquid drainage, but lined with a piece of cloth to avoid removal of soil. The mass of soil required to fill each respective container was determined prior to the packing process based on the calculated average bulk density of each treatment plot. The packing method was consistent and approximately 30 g of soil were added for every packing segment. Following packing, a flat spatula was used to level the surface of the soil. After packing, the infiltration tests were carried out in each sample using the mini disk infiltrometer.

2.2.3 Infiltration tests and measurements

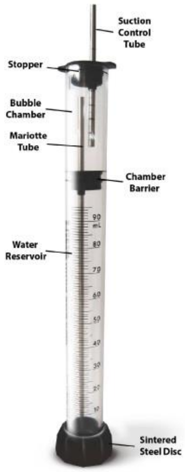
There are a number of experimental and empirical methods used on both field and laboratory scales to determine the soil hydraulic conductivity.

In this study, the mini disk infiltrometer (METERGroupInc.) was used to estimate K_{unsat} of the surface soil (Figure 2.1). The theoretical background and the use of mini disk infiltrometer is described in the Chapter 1.

When the field observations indicated the presence of hydrophobicity in any of the treatments, additional laboratory experiments were conducted to evaluate the hydrophobicity of these treatments. The hydrophobicity was determined by a simple method proposed by Leelamanie et al. (2008) as described in Chapter 1.

2.2.5 Statistical analysis

Statistical analyses were carried out using Minitab 17® statistical software package (©Minitab Inc. at http://www.minitab.com/en-us/). The data were checked for normality (Anderson-Darling test) and outliers (Grubb's test). To identify differences in computed K_{unsat} (cm s^{-1}) among the six treatments under three suction levels, one-way analysis of variance (ANOVA) and Tukey's tests were carried out at 95% confidence level.



IN+DM1	ON	IN+DM1	
IN+DM2	IN+BC		
	IN		IN+BC
	IN+DM1+BC	IN+DM1+BC	ON
IN+DM1+BC		IN	IN+DM2
IN	IN+DM2	IN+BC	IN+DM1+BC
IN+BC	IN+DM1	IN+DM2	IN+DM1
ON		ON	IN

Figure 2.1: Mini Disk Infiltrometer

(Source: www.metergroup.com)

Figure 2.2: Randomized block design representing the experimental layout used in this study - Six treatments and four replicates (0N - zero Nitrogen (control), IN - Inorganic Nitrogen, DM1 and DM2 - Dairy Manure 1 and 2 respectively and BC - Biochar)

2.3 Results and Discussion

The average bulk density of the field plots under different treatments ranged from 1.19 – 1.49 g cm^{-3} (Figure 2.3). Only IN+DM1 and IN+BC plots had significantly lower ($p=0.017$ and $p=0.013$, respectively) bulk density compared to the control. This was approximately a 20% (IN+DM1 – 19.7% and IN+BC – 20.3%) reduction in bulk density compared to the control treatment. The treatment containing IN+DM1+BC reduced the bulk density by 11%, but this was not statistically significant (p=0.302). Also, the change in wet bulk density due to alternate wetting and drying was observed during the laboratory experiment. However, it was relatively constant or quite low and practically negligible for a statistical comparison. Because of significant changes of bulk density among treatments, the change in bulk density was also considered when comparing the treatment effect on K. The observed effect of bulk density on the K_{unsat} and near K_{sat} was not significant for all the suction levels in both field and laboratory experiments. Therefore, any effect among treatments could not be attributed to the bulk density range observed in this experiment.

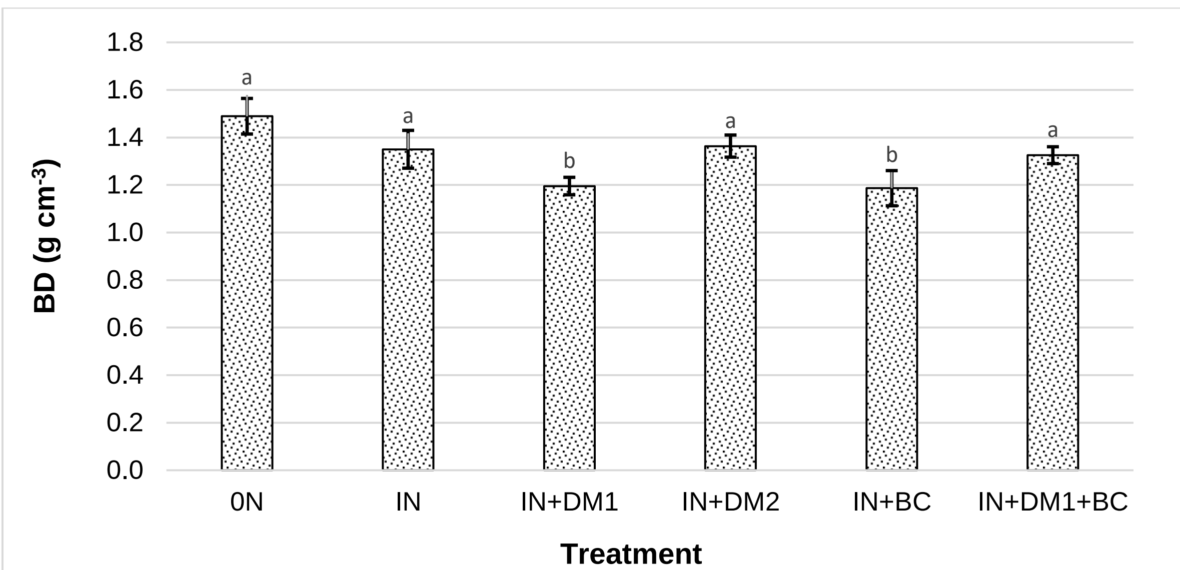


Figure 2.3: Average bulk density (BD) for field plots for different treatments (error bars show standard error of the mean; N = 36, alpha = 0.05)

As shown in Figure 2.4 (field experiment), the K increases with decreasing suction level regardless of the treatment as expected. The treatments containing IN+DM1 showed significantly reduced K values compared to the control under 4 and 2 cm suctions (IN+DM1: $p=0.005$ and 0.001; IN+DM1+BC: $p=0.006$ and 0.001, respectively). Also, IN+DM2 significantly reduced ($p<0.001$) K under 2 cm suction. There were no significant changes in K under 0.1 cm suction, which was assumed as the near-saturated K at field conditions. However, a relatively high variability of K at 0.1 suction for IN treatment was found. The tested soil was a loamy sand with higher sand content (73.7±4.1%) which generally has more macropores. The reduction in K in DM treatments might be due to liquid dairy manure clogging the soil pores or changes in soil porosity (Fares et al., 2008; Roberts & Clanton, 2000). However, amended BC did not reduce K significantly and this might be due to its granular nature (1 – 6 mm particle size). Hence, the soil porosity may be less affected by the BC amendment compared to when DM was applied as a soil amendment. However, the effect of SMC on K_{unsat} is not clear in this experiment, since the SMC variation was not measured.

According to the laboratory experiment (Figure 2.5), the same trend of increasing K with decreasing suction was observed as expected. Interestingly, only IN+DM2 showed a significant reduction of K_{unsat} under 6, 4 and 2 cm suctions ($p=0.001$, 0.001, 0.002, respectively). There were no significant differences of measured K under 1 and 0.1 cm suctions for all treatments.

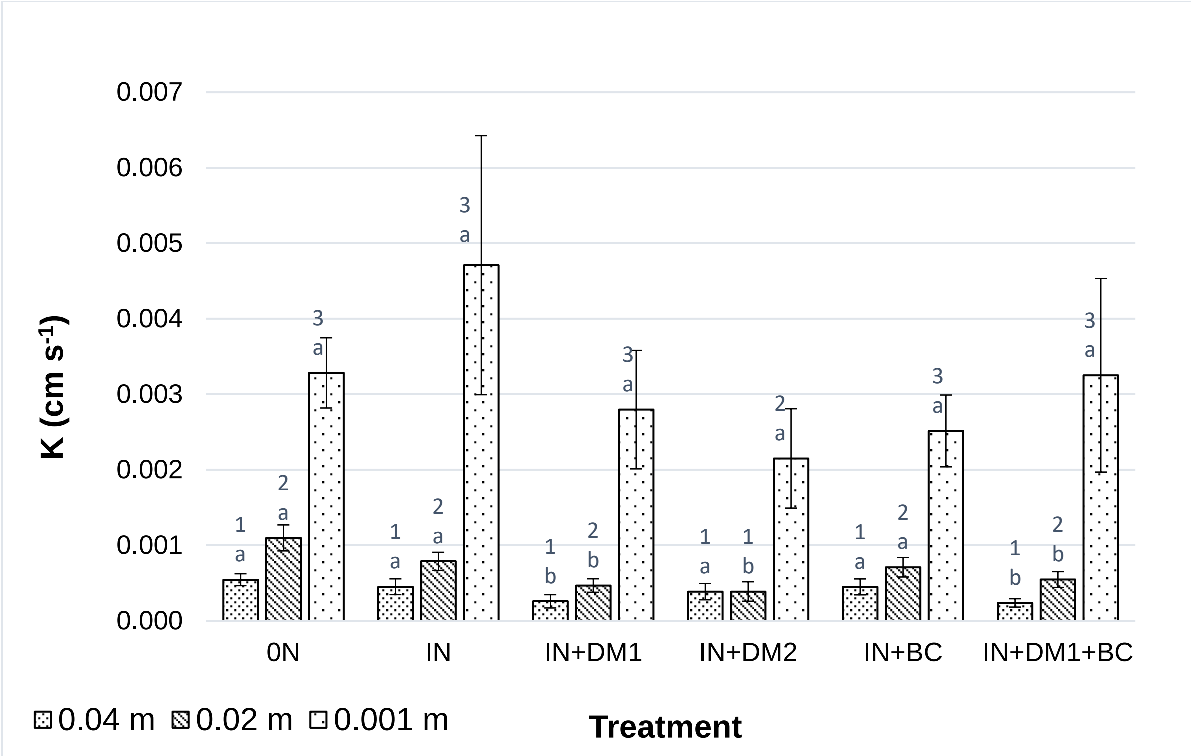


Figure 2.4: Average hydraulic conductivity (K) following the addition of biochar (BC) and dairy manure (DM) as soil amendments under field conditions. Error bars show the standard error of the mean (N=18, alpha=0.05). Letters represent significant differences between treatments for a given suction and numbers represent significant difference between suction levels in a given treatment.

Also, in all treatments, except IN+DM1 in laboratory experiment, K under 1 cm and 0.1 cm suction were significantly higher than the other suctions as expected for near saturated conditions. Except IN+DM2 in field experiment, all other treatments have significantly lower K under 4 cm suction than under 2 cm. However, this is more uniform under laboratory conditions. Also, the overall K values are lower in the laboratory experiment than the field measured K for each treatment. The near saturated K (at 0.1 cm suction) was significantly lower (around 2 ~ 5 times) in the laboratory experiment than the field experiment. This could be due to the packing effect and lack of macropore flows under laboratory conditions and more variabilities involve in field conditions (Anwar et al., 2017; Sandin et al., 2017). Macropore flows can be expected since infiltration test was done at near saturated (suction = -0.1 cm) conditions.

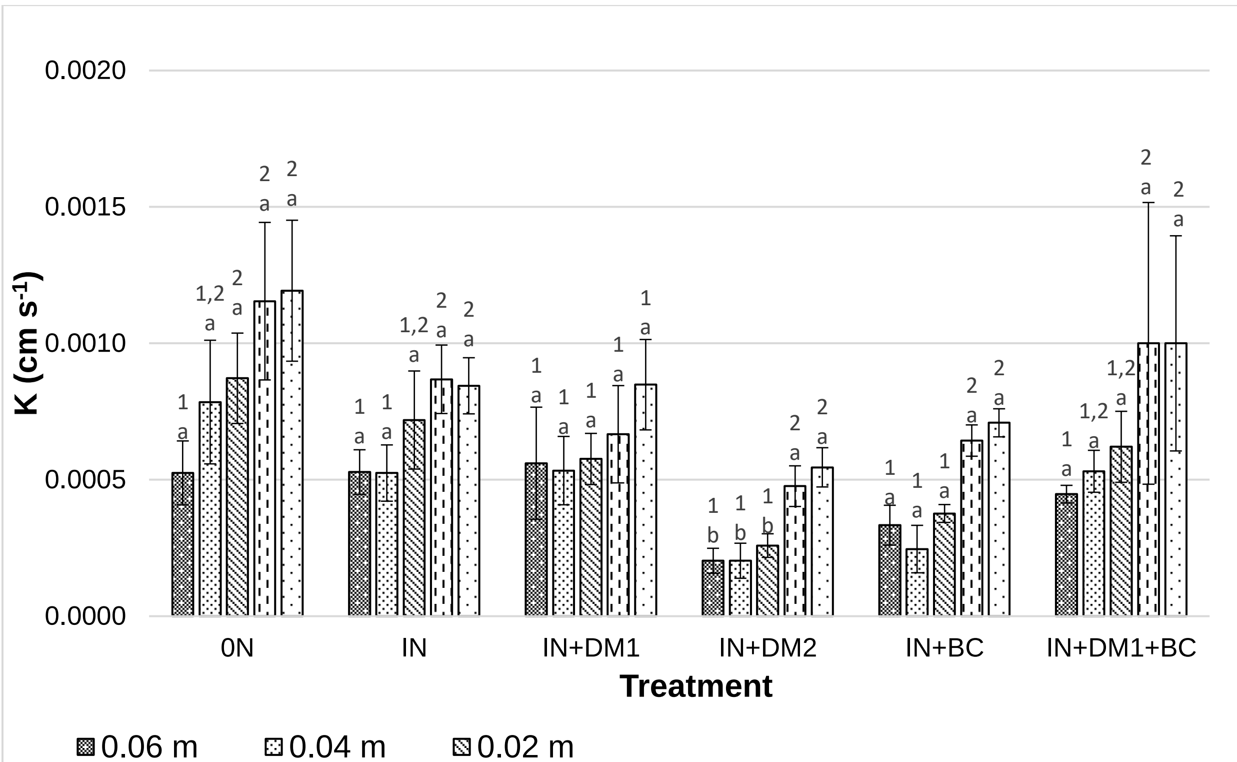


Figure 2.5: Average hydraulic conductivity (K) and standard error for studied treatments under laboratory conditions (N=18, alpha=0.05). Letters represent significant differences between treatments for a given suction and numbers represent significant difference between suction levels in a given treatment.

It is important to note that the flow through an unsaturated soil is more complex, non-linear and is strongly dependent on the detailed pore geometry, water content, and differences in matric potential (Matula et al., 2015; Brady & Weil, 1999; Rose, 1966). Studies reporting the effects of soil amendments on K_{unsat} are very few (Villagra-Mendoza, 2017; Miller et al., 2002). One study found that addition of BC enhances soil microporosity hence enhancing K_{unsat} at higher matric potentials and rapidly decreasing towards lower potentials of sandy and sandy loam soils (Villagra-Mendoza, 2017). Another study found that long-term manure application had little or no effect on K_{unsat} (−0.3, −0.5, −0.7, and −1.0 kPa) (Miller et al., 2002).

However, previously reported effects of BC on near K_{sat} vary in the literature. Some researchers reported that the addition of BC may significantly decrease (Barnes et al., 2014; Githinji, 2013; Deveraux et al. 2012; Uzoma et al., 2011a, b; Brockhoff et al., 2010) or has no effects (Rogovska et al., 2014; Hardie et al., 2013; Ouyang et al., 2013; Busscher et al., 2010; Laird et al., 2010) on the K_{sat} of sandy, loamy-sand and loamy soils. However, some studies have reported higher K_{sat} when BC is applied because of improvements in the structure and the porosity of the amended soil (Herath et al., 2013; Lei & Zhang 2013; Uzoma et al., 2011b; Major et al., 2010; Asai et al., 2009; Oguntunde et al., 2008). The variability found in these results should be strongly associated with the variability of soil texture and BC types, and rates added and their maturity.

On the other hand, solid cattle manure amendments significantly increased K_{sat} (Celik et al., 2004) or has no effect after one soil wetting; but increased after two to five wetting and drying cycles (Hafez, 1974). However, it has also been found that liquid cattle manure tends to block the soil pores with fine particles of manure (Fares et al., 2008; Roberts & Clanton, 2000; Barrington et al., 1987a, b; Rowsell et al., 1985; deTar, 1979; Chang et al., 1974), thus decrease the infiltration rates (Miller et al., 1985) and K_{sat} (Culley & Phillips, 1982). Also, the addition of high quantities (*i.e.*: 90 – 360 t ha^{-1} Tiarks et al., 1974) of organic manure resulted in surface crusting and decreased K_{sat} (Cherobim et al., 2018) (Roberts & Clanton, 2000).

According to the WDPT test (Table 2.3) used in this study, treatments containing DM (IN+DM1, IN+DM2 and IN+DM1+BC) were found to be slightly water repellent (in the range of 1 – 18 sec). Therefore, applied DM might not adversely affected our soil. However, long-term application of heavy rates might cause more unfavourable hydrological conditions, such as increasing water repellency resulting in reduced infiltration and increased surface runoff. Collectively, these findings indicate that amendments such as DM and BC need to be applied with caution.

Table 2.3: Water drop penetration time (WDPT) and repellency rating for studied treatments.

Treatment	0N	IN	IN+DM1	IN+DM2	IN+BC	IN+DM1+BC
WDPT (s)	0.5	0.5	12	18	0.6	10
Repellency Rating	Non-Repellent	Non-Repellent	Slightly Repellent	Slightly Repellent	Non-Repellent	Slightly Repellent

On the other hand, both BC and DM facilitate or increase SOM (Jiao et al., 2006), which might have helped the formation and stabilization of soil aggregates, thereby enhancing the soil structure, strength, porosity and bulk density providing better water and nutrient movement and retention; thus, improving the number and activity of beneficial soil microorganisms, which can ultimately increase crop yield (Abdallah et al., 1998).

2.4 Conclusions

The study evaluated the effect of DM and BC incorporation on K of agricultural podzolic soils under both field and laboratory conditions. According to the field study, the treatments containing IN+DM1 showed significantly reduced K_{unsat} values compared to the control under 4 and 2 cm suctions; while IN+DM2 significantly reduced K_{unsat} under 2 cm suction. There were no significant changes in near K_{sat} under 0.1 cm suction, which was considered as the near-saturated hydraulic conductivity. According to the laboratory study, only IN+DM2 showed significant reductions of K_{unsat} under 6, 4 and 2 cm suctions. There were no significant changes in K_{unsat} and near K_{sat} under 1 and 0.1 cm suctions, respectively. According to the WDPT test, IN+DM1, IN+DM2 and IN+DM1+BC were slightly water repellent. Therefore, applied DM might have not affected adversely in our soil. However, long-term application of heavy rates might cause more unfavourable conditions on soil hydraulic properties, hence need to apply with caution. Further studies are recommended to identify the differences in hydrophobicity and particulate matter in different types and rates of DM and BC amendments and their effects on soil hydrology.

Acknowledgement: Authors wish to acknowledge the financial support from the Research and Development Corporation (RDC), of the Government of Newfoundland and Labrador (Ignite R&D – 5404-1962-101) and the Memorial University of Newfoundland for research support and graduate research assistantship

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CHAPTER 3

EFFECT OF BIOCHAR ON TDR BASED VOLUMETRIC SOIL MOISTURE MEASUREMENTS IN A LOAMY SAND PODZOLIC SOIL

Dinushika Wanniarachchi, Adrian Unc, Mumtaz Cheema, Lakshman Galagedara (2018). EFFECT OF BIOCHAR ON TDR BASED VOLUMETRIC SOIL MOISTURE MEASUREMENTS IN A LOAMY SAND PODZOLIC SOIL (Submitted to Transactions of the ASABE).

Abstract

Time Domain Reflectometry (TDR) is a well-established method for measuring volumetric soil moisture content (VSMC) at point scales using soil's dielectric properties. The method assumes that soil properties are uniformly distributed along the probe length and has negligible influence from other physical properties, which allows it to be used in a wide range of soils. With the increasing interest in biochar (BC) as a soil amendment and growing media substrate in agriculture, management practices also need to be adjusted for changing soil/media properties. The study evaluated the effect of BC incorporation on TDR based VSMC estimations in a loamy sand podzolic soil. Two commercial BCs having two distinct particle sizes, *i.e.* powdered, BC_P (<0.5 mm) and granular, BC_G (1–6 mm) were used. Eight different BC:soil mixtures, including non-BC (0%) and BC only (100%), were packed in plastic containers (volume 750 mL). The dielectric constants measured using a TDR cable tester (MOHR CT 100) were converted to VSMC using three models: i) Topp's equation, M−1; ii) mixing model, M−2; and iii) the forest soil model, M−3. The accuracy of the estimated VSMC using these models was compared via regression analyses against a standard (M0) VSMC calculated using gravimetric moisture and bulk density. For the lowest BC_P rates, M-1 and M-2 produced very similar results to the actual VSMC. However, the estimated VSMC was gradually underestimated with increasing BC_P rates. The VSMC estimated with the M−3 non-linearly related to BC_P rates, shifting from overestimation to underestimation as the BC_p rate increased. In BC_G treatments, all models overestimated the VSMC. However, BC_G rates higher than 15% resulted in highly attenuated TDR waveforms and completely dissipated (i.e. no reflection) in >50% BC_G

treatments. These results show that predictions of the soil moisture content based on the soil dielectric constant might not be feasible for soils amended with higher BC rates.

Keywords: Biochar, Dielectric constant, Time Domain Reflectometry, Volumetric soil moisture content

3.1 Introduction

Among the techniques used in measuring volumetric soil moisture content (VSMC), Time Domain Reflectometry (TDR) is the most widely used method in agriculture, forestry, soil science, hydrology etc. TDR has rapidly become a method of choice for field research as it offers accurate results (usually within 2% of VSMC), the ability to obtain real-time repeated measurements with minimal efforts and does not pose the radiation hazards associated with neutron probe or gamma-ray attenuation methods (Nagare et al., 2011). By automating TDR measurements using multiplexers and data storage devices (Baker & Allmaras, 1990), continuous spatial and temporal monitoring of VSMC is possible, for instance, studies on water movement and solute transport (Topp et al., 1982) or plant water availability (Jackson & Wallace, 1999). In addition, TDR can be used to monitor water levels (Moret & Lopez, 2004), the position of the advancing frost levels in freezing soils (Overduin & Kane, 2006), and the water table in the subsurface of contaminated lands (Gaur et al., 2003).

The TDR technique is based on the travel time analysis of electromagnetic (EM) wave propagation in a metallic waveguide of a known length inserted into the soil. The EM frequencies range from 1 MHz to 1 GHz at a speed of 30 cm ns^{-1}. The soil is a complex system having solid, liquid and air components. However, water has a much higher dielectric constant, K_a (81 at 20 ^{o}C) than both the solid (2–7) fraction, which composed of mineral and organic soil particles, and the air (1) fraction. Therefore, water has the highest influence on the K_a of the soil and it is possible to estimate the VSMC of a given soil sample from estimates of its K_a (Topp et al., 1980; Cassel et al., 1994; Jones et al., 2002; Yu & Yu, 2006). The EM wave propagation time increases (low EM velocity) with increasing soil moisture content (Gasvoda, 1998; Gao & Yu, 2015). Moreover, the VSMC obtained by TDR is calculated as the average over the probe length and for a certain radius immediately around the probe (Ferre et al., 1998; Yu & Yu, 2006).

TDR measurements can be converted to VSMC using different calibration models including empirical calibrations and multiphase mixing models (Topp et al., 1980; Dobson et al., 1985; Roth et al., 1990; Schaap et al., 1996; Drnevich et al., 2001; Jones et al., 2002). Empirical models that relate VSMC and soil K_a were found to be useful for the determination of TDR calibration curves (Kaiser et al., 2010; Teixeira et al., 2003). Several adjusted models for specific conditions such as variable soil electrical conductivity (EC), amount of clay, quartz, or organic matter content and peat have also been evaluated (Pepin et al., 1992; Kellner & Lundin, 2001; Yoshikawa et al., 2004; Pumpanen & Ilvesniemi, 2005). Nevertheless, while the TDR technology is extensively employed, research on the influence of non-uniform soil properties on its measurement accuracy is very limited (Yu & Yu, 2006; Dettmann & Bechtold, 2018).

Biochar (BC) is a carbonaceous porous material obtained by pyrolysis using various biomass sources. It shows immense potential in improving soil properties and fertility. Amending soil with BC has also been recognized as a possible method to address issues related to atmospheric carbon increase and global warming, and food insecurity (Lopez, 2014). Extensive research studies have been carried out to investigate the utilization of BC as a soil amendment to improve soil physical properties (Chan et al., 2007; Novak et al., 2012; Abel et al., 2013; Mukherjee & Lal, 2013). Despite the number of studies dealing with BC's role in soil fertility, the mechanisms of its action in the environment are still poorly understood. Due to the porous structure of BC, its incorporation into agricultural soil can change the soil's physical properties such as porosity, pore size distribution and bulk density (Lehmann et al., 2009), consequently altering the soil's hydraulic properties, including soil water retention and permeability (Kameyama et al., 2012). It is also important to note that the physicochemical properties, EC and electrostatic properties (Ishihara, 1996), and thus water repellency (Kinney et al., 2012) of BC vary with the feedstock used, and pyrolysis temperature and duration (Lehmann et al., 2009).

Application of BC to agricultural soils or to soil based or non-soil based growing media requires further understanding of its effects on the physiochemical properties of the soil or the media. One important step towards understanding these effects would be to develop accurate methods for the measurement of VSMC in BC-amended soils. Even though the TDR technique is widely used to measure VSMC of mineral soils, based on our understanding, the effect of BC amendments on the accuracy of VSMC estimation models using the TDR method has not been evaluated extensively. A study by Dettmann and Bechtold (2018) reported that a commercially available TDR probe (TRIME – PICO64) increased the accuracy of K_a estimation with a new calibration (RMSE = 3.55) from the manufacture's calibration (RMSE = 18.73) under mineral to peat soil conditions. Although Kameyama et al. (2014) has used TDR for monitoring VSMC and water availability for plants in BC amended soils, they have not necessarily offered any evidence for thorough evaluation of dielectric based calibration equations for BC amended soils.

Therefore, the objective of this study was to determine the effects of BC incorporation on the uncertainty of TDR based VSMC estimations. It was hypothesized that the incorporation of BC affects the measurements of K_a as obtained with TDR. The study evaluated three existing calibration models that link VSMC and the K_a obtained from TDR, and the applicability of these models for agriculturally important BC amended soils. Such clarification is paramount to the further applicability of these models, which in turn can save time and efforts required to determine a complete TDR calibration curve.

While the principles of TDR measurements have been widely explored and reported, a few fundamentals relevant to this study are briefly summarized in Chapter 1 of this thesis.

Soils with higher OM content and many horticultural growth media have distinct properties from typical mineral soils such as higher porosity, low BD, substantial amounts of bound water fraction, and limited clay amounts, all of which can affect the dielectric signatures.

By considering these factors, Schaap et al. (1996) obtained an empirical expression for organic forest soil horizons. Therefore, tested this model was tested given that: 1) BC has been proposed as an amendment for improving horticultural media, particularly under greenhouse conditions, and 2) TDR is the most commonly used method to measure VSMC in this industry. Also, the estimation of VSMC and EC with very high accuracy is essential for the efficiency level expected in the greenhouse industry compared to field crop production.

Although, consideration of the soil bulk density should improve calibration models, the influences of bound water fraction, which mostly correlated to the surface area of the soil / media particles, may not be easily resolved due to large variations in soil type and local conditions including origin of the OM and degree of decomposition (Oleszczuk et al., 2007).

3.2 Materials and methods

3.2.1 Soil

Soil samples were obtained randomly from 0 – 20 cm soil depth of an experimental silage corn (*Zea mays*) field at Pynn’s Brook Research Station (PBRS), Department of Fisheries and Land Resources, Government of Newfoundland and Labrador, Pasadena (49°04'22.6"N; 57°33'38.9"W), Canada. The samples were air-dried for 72 h, sieved (<2 mm), pulverized, and homogenized. The texture of the soil was loamy sand (73.7±4.1 % sand, 23.0±3.8 % silt, 3.3±0.3 % clay) (Badewa, 2017).

3.2.2 Preparation of BC:soil mixtures

Two different BC types *i.e.* powdered (BC_P) and granular (BC_G) obtained from commercial suppliers were used, and their basic properties are given in Table 3.1. The two different BC were mixed with soils separately at ratios of 0%, 5%, 10%, 15%, 20%, 40%, 50% and 100% w/w by air dry mass. Soils without BC (0%) acted as the control, resulting in a total of 16 (2 BC types × 8 rates) treatments. The incorporation rates were equivalent to approximately 0, 125, 250, 375, 500, 1000, 1250 and 2500 BC t ha^{-1}, respectively, at a depth of 0 – 20 cm and for a bulk density of 1.25 g cm^{-3}. Often, BC application experiments have been conducted for a wide range of application rates; for instance, 1 – 135 t ha^{-1} in pot and field experiments as a soil amendment (Jeffery et al., 2011), 0 – 50% w/w in greenhouse experiments (Schulz et al., 2013) and 10 – 100% v/v in growing media experiments as a substrate for soil-free nursery plants and a substitute for peat (Kaudal et al., 2016; Margenot et al., 2018). Even though the studied rates were comparatively higher than the actual field application of BC rates, I aimed to evaluate the full range of BC mixing rates in this laboratory experiment by giving special attention to the use of BC in the horticultural industry. Also, to observe any differences arising from properties of BC, the present study used two types of BC that were available at the time.

Table 3.1: Basic properties of two types of biochar (BC) used for the study.

	Powdered	**Granular**
Feedstock	Mix softwood	Yellow pine wood (*Pinus taeda*)
Particle size	<0.5 mm	1–6 mm
Bulk Density (g cm^{-3})	0.75	0.20
Moisture	5 %	15.2 %
pH (1: 10 BC: Water)	8.9	9.0
EC_a at 21 – 22 °C (dS m^{-1})	1.3	5.2
Fixed carbon	69 %	87.3 %
Volatile Carbon (600 °C)	22 %	12.7 %
Ash	4 %	6 %

The bulk density of the mixtures of soil and BC was calculated as in Eq.15:

$$\rho_b = \frac{100}{[\left(\frac{x}{\rho_1}\right)+\left(\frac{100-x}{\rho_2}\right)]} \qquad \text{Eq. 115}$$

Where $\rho_{b,}$ ρ_1 and ρ_2 are the bulk densities (g cm^{-3}) of soil:BC mixtures, BC only and soil only, respectively and x is the BC rate (%) by weight (Adams, 1973).

The bulk densities of 0.75 g cm^{-3} for powdered BC, 0.20 g cm^{-3} for granular BC, and 1.25 g cm^{-3} for the tested mineral soil were used for calculation.

Each of the BC:soil mixture and the soil was packed (based on the bulk densities above) into 750 mL plastic containers with drainage holes in the bottom. Packing process was consistent and added approximately 25 – 35 g of BC:soil mixture for every packing segment. After each addition of the mixture, the container was tapped down four times to achieve the desired BD. Following the packing, the samples were saturated by keeping the containers in a water tub and gradually raising water levels through the bottom holes via capillarity. This process helped to reach the full saturation with minimum air trapped in each sample.

3.2.3 Measurements of volumetric soil moisture content

The measurements began with the saturated samples and afterwards, the TDR measurements were obtained every 6 h, while keeping the samples in a laboratory drying oven (forced air) at 30 °C to maintain constant drying conditions, equally for each sample. The containers were weighed soon after obtaining TDR measurements to calculate the gravimetric moisture contents. Three replicates were used for each measurement.

VSMC was estimated using the measured K_a obtained from a TDR cable tester (MOHR CT 100) with a three-rod TDR waveguide. The three commonly used models; (i) Topp's equation – M1 (Topp et al., 1980), (ii) mixing model – M2 (Roth et al., 1990) and (iii) the forest soil model – M3 (Schaap et al., 1996) were used to estimate VSMC. The models were compared with a standard (M0) VSMC calculated using gravimetric moisture content and bulk density for each treatment.

3.2.4 Statistical analysis

Data were first checked for normality (Anderson-Darling test) and possible outliers (Grubb's test). Performance of the models was evaluated by coefficients of determination (R^2), root mean square error (RMSE), and comparison between the observed (measured) and model estimated data using 1:1 line and statistical testing of the significant changes in slope and the intercept of each relationship compared to the slope (1) and intercept (0) of the 1:1 line. The significance level was $p=0.05$. All statistical analyses were performed with Minitab 17® statistical software (©Minitab Inc. at http://www.minitab.com/en-us/).

3.3 Results and discussion

The decrease in bulk density with increasing BC rate was observed for both BC_P and BC_G, as expected. Also, BC_G had lower bulk density than BC_P, hence BC_G amended soils were also observed to have lower bulk density compared to both non-amended soil and BC_P amended soils (Table 3.2).

Table 3.2: Bulk densities (BD) for each biochar:soil mixtures of both powdered and granular biochar

BC rate (%)	0	5	10	15	20	40	50	100
BD_P (g cm^{-3})	1.25	1.21	1.17	1.14	1.10	0.99	0.94	0.75
BD_G (g cm^{-3})	1.25	0.99	0.82	0.70	0.61	0.40	0.34	0.20

For the studied BC rates, K_a varied over a range from 8 to 35 for the change in the measured VSMC (gravimetrically measured) from 25 to 65 % for BC_p amended soil, while it was from 10 to 80 and from 28 to 43 % for BC_G amended soils.

Of the two BC types, the TDR measurements could be obtained only up to the rate of 10% BC for soils amended with BC_G. BC rates >15% led to highly attenuated waves or did not show any reflection (Figure 3.1). However, this behaviour was not observed for BC_P.

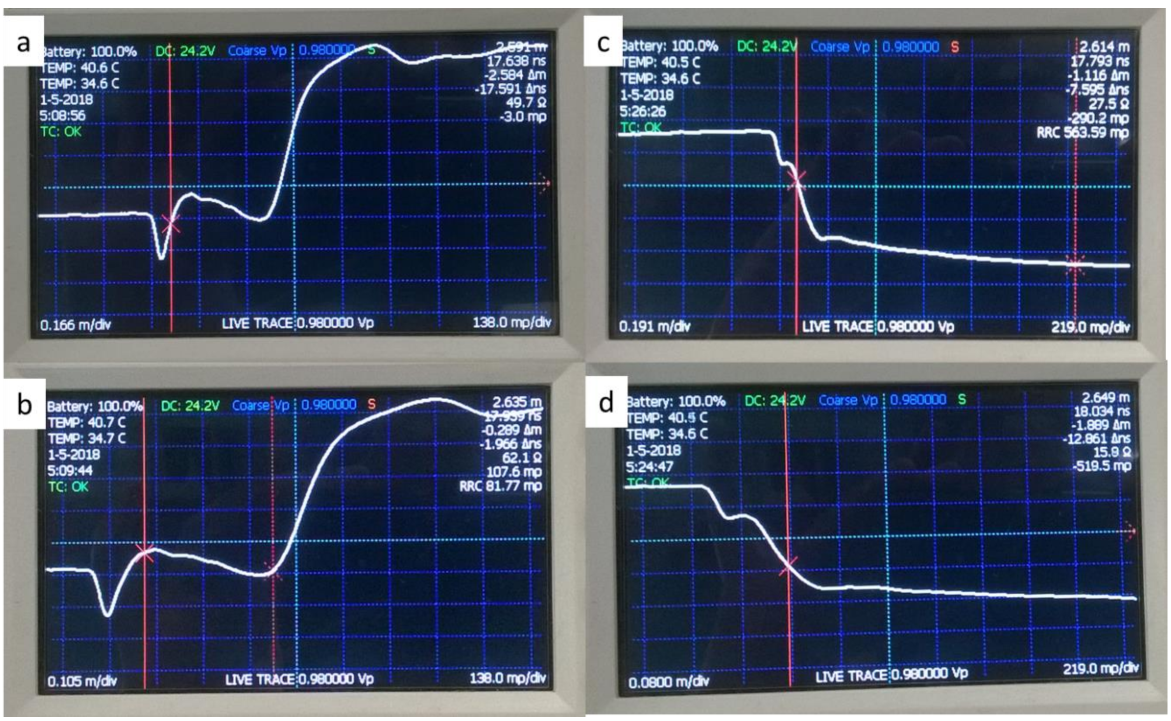

Figure 3.1: Time Domain Reflectometry (TDR) waves obtained from samples containing granular biochar (BC_G). (a) 0%, (b) 5%, (c) 50% and (d) 100% rates of BC_G.

Moreover, at the dry end in BC_G samples, the travel time of EM wave increased with increasing BC rate with gradual reduction of the strength of the reflected signal. On the other hand, the travel time decreased with increasing BC rate at the dry end for BC_p amended soil. A similar observation of decreasing travel time was previously reported when soil was incorporated with less conductive sugarcane bagasse derived BC (Kameyama et al., 2014).

Models selected in this study and other studies showed that the K_a of soils (with respect to vacuum) increased with increasing moisture content. The other variables that affect the electrical response in soils are texture, structure, soluble salts, temperature, density, and measurement frequency (Topp et al., 1980; Bridge et al., 1996; Wyseure et al., 1997; Jones et al., 2002; Yu & Yu, 2006; Bittelli et al., 2008).

Hook et al. (2004) reported that the travel time of an EM wave increases with increasing EC of pore water resulting in the overestimation of VSMC. Robinson et al. (1994) reported that the reflected signals stretch to the right when soils contain iron minerals, as they increase the conductivity and magnetism of the medium. EC_a values of two BC types tested were different, BC_P = 1.3 dS m^{-1} and BC_G = 5.2 dS m^{-1}. The higher EC and the increased travel time observed in BC_G also agree with previous findings demonstrating high EC values caused an increase of travel time and overestimation of VSMC (Hook et al., 2004; Chen et al., 2012). Therefore, apart from the high moisture content, the highly conductive solid phase and the high EC in soil solution might have played a role in the increased travel time of EM waves.

Also, the reflection of EM waves only takes place when the pulse signal enters the soil and arrives at the end of the waveguide. Therefore, the reflection of the EM wave might be attenuated rapidly in BC_G amended soils, as the sample's porosity and EC increase, which prevented the measurements (Table 3.1). This inability to interpret TDR waveforms by travel time analysis was also reported by Chen et al. (2012) who applied the TDR to measure moisture content in municipal solid wastes. Dettmann and Bechtold (2017) evaluated two commercially available moisture probes covering from mineral to peat soils and found that uncertainty of measurements increases with increasing K_a.

Previous studies (Miyamoto & Chikushi, 2006; Kameyama et al., 2014) reported the decrease in K_a with decreasing bulk density, and hence increasing porosity, in low moisture soils. The waveforms at the dry end of the soils amended with BC_P in our experiment can be explained by the decrease in bulk density (Miyamoto & Chikushi, 2006), and thus low dielectric values resulting from a greater underestimation of the VSMC at the dry end. Though soils with BC_G also have lower bulk density, this reduction in K_a might be masked by the high EC of BC_G as explained above.

Evaluation of models

The 1:1 comparison of estimated and measured VSMC of 0% BC is shown in Figure 3.2. While the results for all three models are almost parallel to the 1:1 line, the Topp's equation (M–1) and the mixing model (M–2) data aligned very close to the 1:1 line. This shows a lower dispersion and better accuracy in the estimation of VSMC using M–1 and M–2 for the tested loamy sand soil. Even though the model M–1 slightly underestimated the VSMC, it was not statistically significant (p=0.497). However, the forest soil model (M–3) significantly (p=0.032) overestimated the VSMC for the control (0% BC). This observation confirmed that the empirical polynomial equation (M–1) proposed by Topp et al. (1980) and the mixing model theory by Roth et al. (1990) and Birchak et al. (1974) to correlate K_a with VSMC is applicable for the tested loamy sand podzolic soil. These two models have been confirmed by several researchers and are quite broadly applicable for different soils (Wesenbeeck & Kachanoski, 1988; Kameyama et al., 2014).

When it comes to 100% BC_P (Figure 3.2), slopes of all three models had significantly increased (p=0.00) and away from the 1:1 line (slope $\neq$ 1). Both M–1 and M–2 model significantly underestimated VSMC (p=0.000). The model M–3 underestimated the VMSC as BC was drying (less than 60% moisture) but showed less dispersion and more similarity to the 1: 1 line as BC was wetted. The increase in slopes was directly related to the increase in BC rates for both BC_P and BC_G. Figures 3.3 and 3.4, Tables 3.3 and 3.4 show the relationships between measured and model estimated values for VSMC for each studied BC rates.

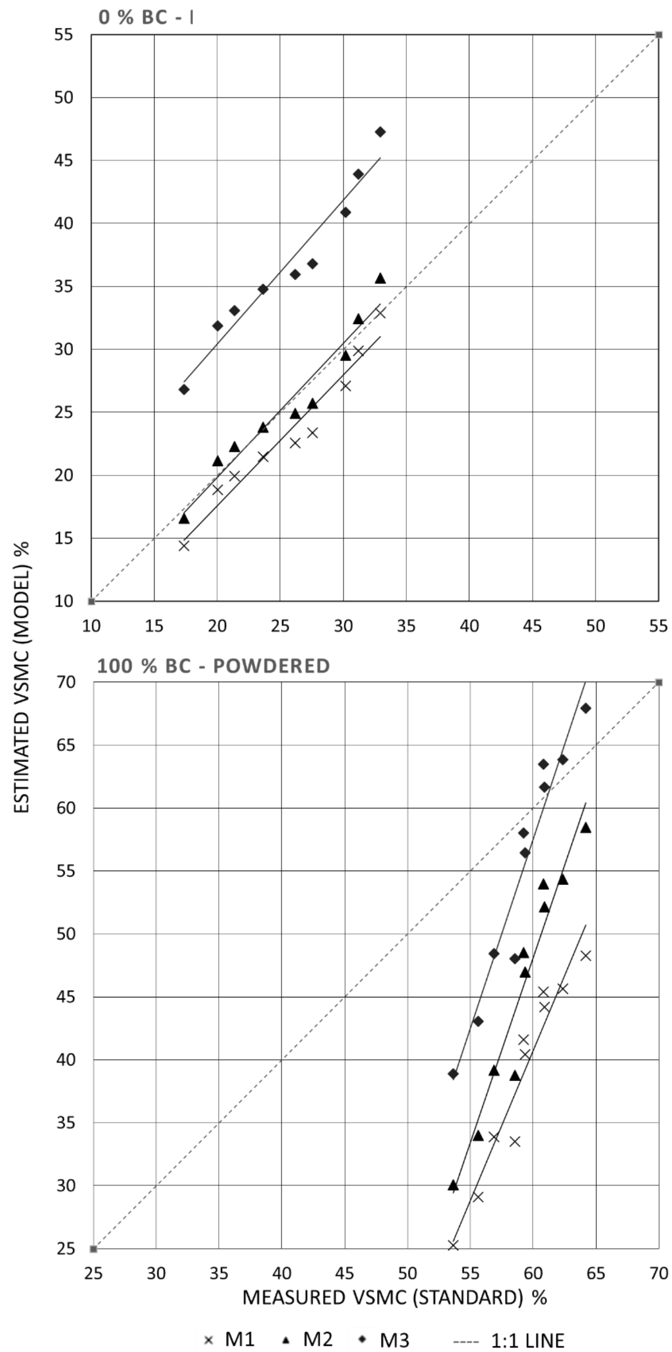

Figure 3.2: Relationship (1:1 graph) between the measured and estimated volumetric soil moisture content (VSMC) with three models; M1, M2 and M3 for 0% and 100% biochar (N=10, alpha=0.05).

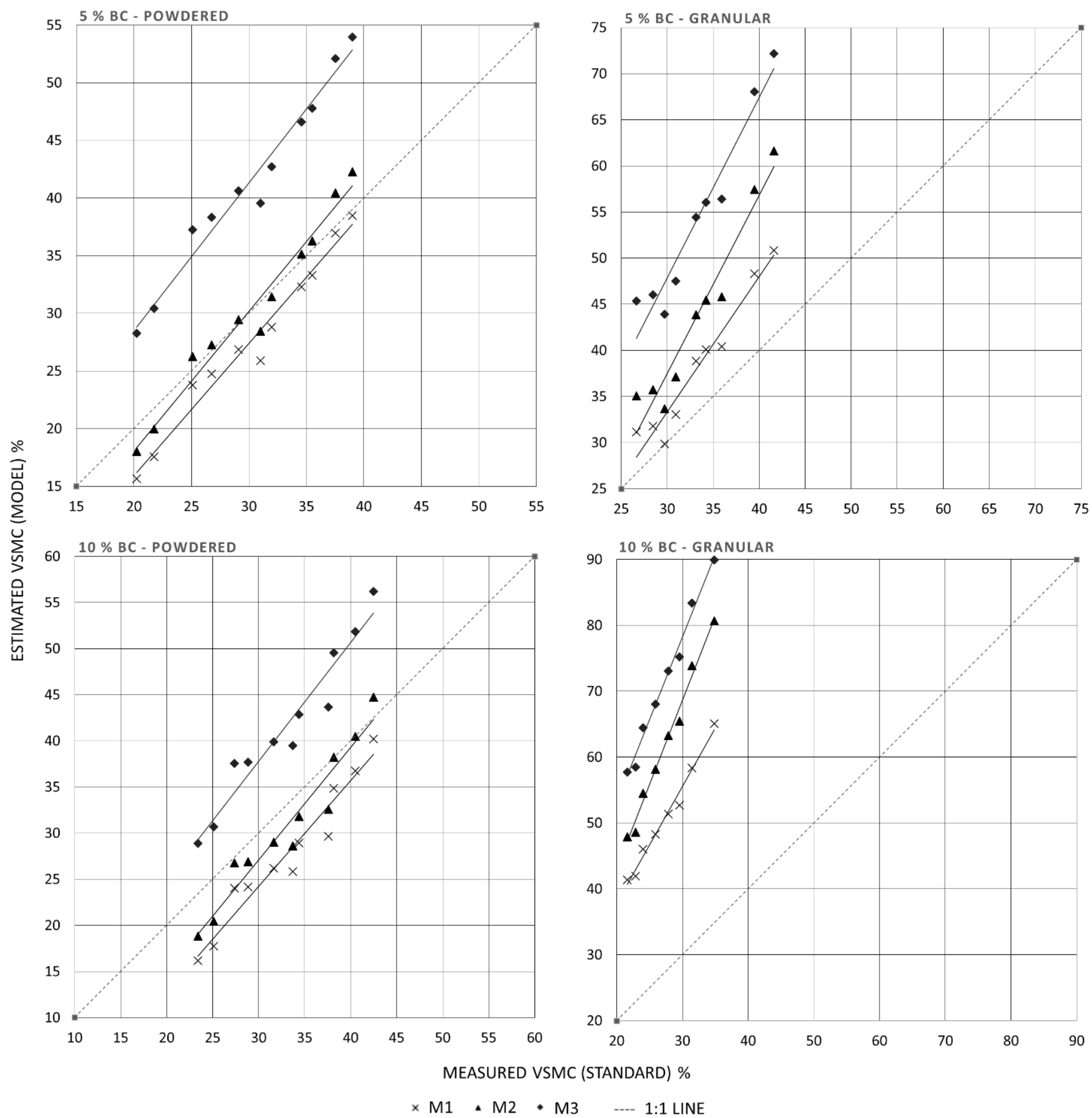

Figure 3.3: Relationship (1:1 graph) between the measured and estimated volumetric soil moisture content (VSMC) with three models; M1, M2, and M3, for 5% and 10% powdered and granular biochar (N=10, alpha=0.05).

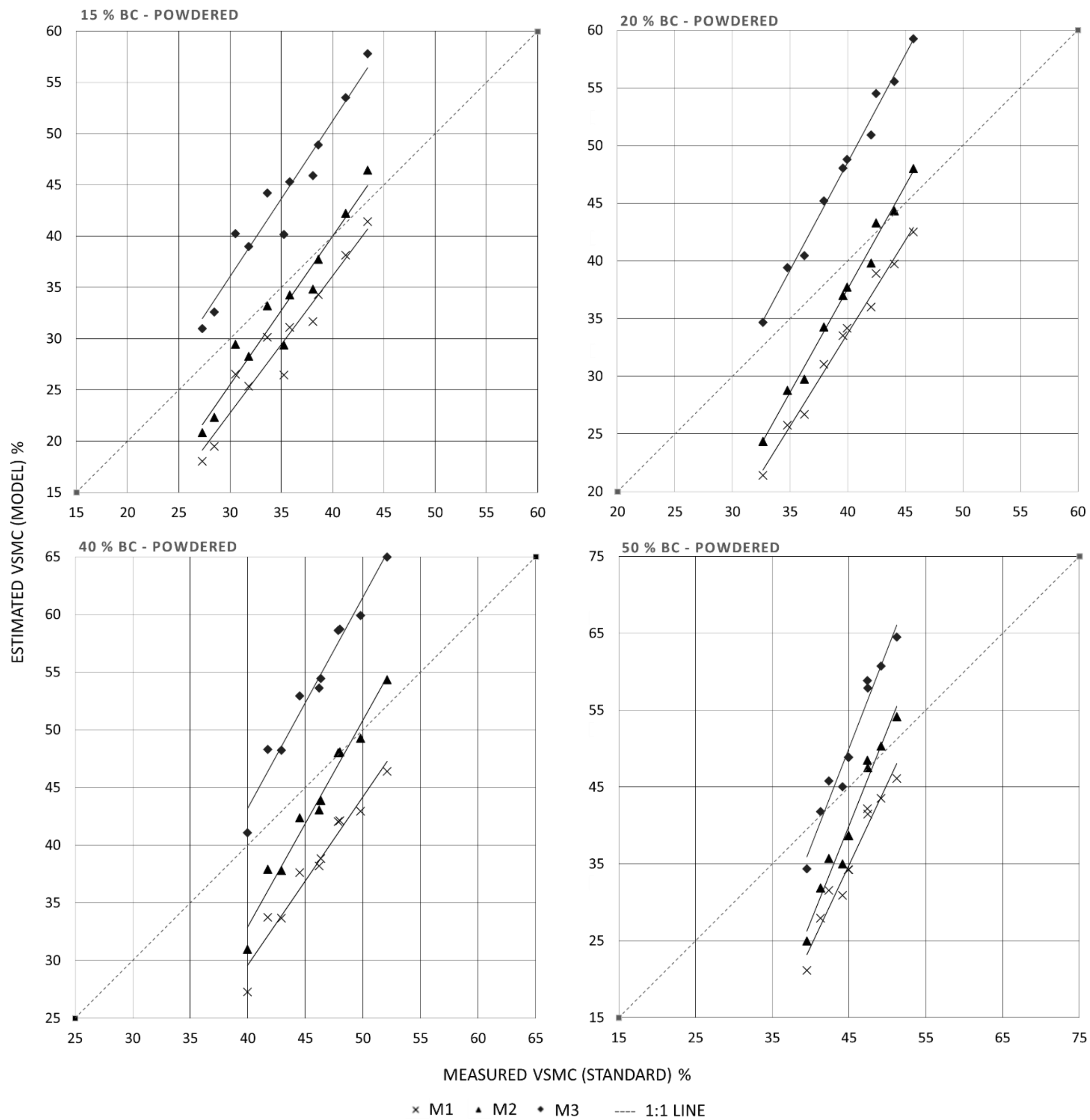

Figure 3.4: Relationship (1:1 graph) between the measured and estimated volumetric soil moisture content (VSMC) with three models; M1, M2, and M3, for 15, 20, 40 and 50% powdered biochar (BC_P) (N=10, alpha=0.05). NOTE: No data for granular biochar (BC_G)with these corresponding rates.

Table 3.3: Regression equations (Reg. Eq.), coefficients of determination (R2), root mean square error (RMSE %) and p-values (P) between measured and modelled volumetric soil moisture content (VSMC) for studied powdered biochar (BC_P) rates (corresponding graphical representations are given in figures 3,4 and 5). N=10, alpha= 0.05

BC (%)	**Model 1 (M-1)**				**Model 2 (M-2)**				**Model 3 (M-3)**			
	Reg. Eq.	**R^2**	**RMSE**	**P**	**Reg. Eq.**	**R^2**	**RMSE**	**P**	**Reg. Eq.**	**R^2**	**RMSE**	**P**
0	$Y = 1.001 \times x - 2.54$	0.91	1.8	0.000	$Y = 1.026 \times x - 0.78$	0.91	1.9	0.000	$Y = 1.100 \times x - 8.32^{\#}$	0.91	2.0	0.000
5	$Y = 1.152 \times x - 7.50^{\#}$	0.96	1.5	0.000	$Y = 1.216^{\#} \times x - 6.67^{\#}$	0.96	1.7	0.000	$Y = 1.287^{\#} \times x - 2.30$	0.96	1.8	0.000
10	$Y = 1.198 \times x - 11.13^{\#}$	0.96	1.6	0.000	$Y = 1.281^{\#} \times x - 10.73^{\#}$	0.96	1.7	0.000	$Y = 1.351^{\#} \times x - 2.08$	0.96	1.8	0.000
15	$Y = 1.297 \times x - 16.13^{\#}$	0.92	2.1	0.000	$Y = 1.397^{\#} \times x - 16.38^{\#}$	0.92	2.3	0.000	$Y = 1.467^{\#} \times x - 7.93$	0.92	2.4	0.000
20	$Y = 1.537^{\#} \times x - 27.85^{\#}$	0.99	0.8	0.000	$Y = 1.720^{\#} \times x - 31.31^{\#}$	0.98	1.1	0.000	$Y = 1.786^{\#} \times x - 23.00^{\#}$	0.98	1.1	0.000
40	$Y = 1.676^{\#} \times x - 39.51^{\#}$	0.96	1.3	0.000	$Y = 2.033^{\#} \times x - 50.73^{\#}$	0.97	1.4	0.000	$Y = 2.082^{\#} \times x - 42.51^{\#}$	0.97	1.4	0.000
50	$Y = 2.039^{\#} \times x - 56.88^{\#}$	0.96	1.8	0.000	$Y = 2.366^{\#} \times x - 66.37^{\#}$	0.96	1.9	0.000	$Y = 2.444^{\#} \times x - 59.78^{\#}$	0.96	2.0	0.000
100	$Y = 2.326^{\#} \times x - 99.00^{\#}$	0.95	2.1	0.000	$Y = 2.796^{\#} \times x - 120.00^{\#}$	0.95	2.5	0.000	$Y = 2.871^{\#} \times x - 115.00^{\#}$	0.95	2.5	0.000

$^{\#}$Significant changes in slope (slope ≠ 1) and intercept (intercept ≠ 0) compared to the slope and the intercept of the 1:1 line

Table 3.4: Regression equations (Reg. Eq.), coefficients of determination (R2), root mean square error (RMSE %) and p-values (P) between measured and modelled volumetric soil moisture content (VSMC) for studied granular biochar (BC_G) rates (corresponding graphical representations are given in figures 3,4 and 5). N=10, alpha=0.05. No data for BC_G rates > 15%

BC (%)	**Model 1 (M-1)**				**Model 2 (M-2)**				**Model 3 (M-3)**			
	Reg. Eq.	**R2**	**RMSE**	**P**	**Reg. Eq.**	**R2**	**RMSE**	**P**	**Reg. Eq.**	**R2**	**RMSE**	**P**
0	$Y = 1.001 \times x - 2.54$	**0.91**	**1.8**	**0.000**	$Y = 1.026 \times x - 0.78$	**0.91**	**1.9**	**0.000**	$Y = 1.100 \times x - 8.32^{\#}$	**0.91**	**2.0**	**0.000**
5	$Y = 1.333 \times x - 6.88$	**0.93**	**2.1**	**0.000**	$Y = 1.691^{\#} \times x - 13.27^{\#}$	**0.92**	**2.9**	**0.000**	$Y = 1.722^{\#} \times x - 3.86$	**0.92**	**2.9**	**0.000**
10	$Y = 2.400^{\#} \times x - 12.22^{\#}$	**0.88**	**5.5**	**0.000**	$Y = 2.501^{\#} \times x - 5.21$	**0.99**	**1.7**	**0.000**	$Y = 2.432^{\#} \times x - 6.32$	**0.99**	**1.6**	**0.000**

[#]Significant changes in slope (slope ≠ 1) and intercept (intercept ≠ 0) compared to the slope and the intercept of the 1:1 line

The model (M–1) proposed by Topp et al. (1980) underestimated VSMC for all BC_P:soil mixtures throughout the range observed, but estimated VSMC values were better assessed at the wet end (from 30 to 60 %). The same behaviour was observed for M–2 (Roth et al., 1990). On the other hand, in BC_P:soil mixtures, the results obtained with the forest soil model (M–3; Schaap et al., 1996) gradually migrated from overestimation towards underestimation as BC_P rates increased. This was opposite for BC_G:soil mixtures (0 to 10% BC), as all the models overestimated the VSMC when increasing the moisture content (Figures 3.2, 3.3 and 3.4).

Although the change in intercept values was significant ($p<0.05$) for all BC amended samples, the slope for the M-1 model was not significant (slope = 1) up to 20% of BC_P and up to 5% of BC_G amended samples. On the other hand, both M–2 and M–3 models showed significant changes in slopes (Tables 3.3 and 3.4). Therefore, this analysis shows that the commonly used Topp's equation (M-1) performed slightly better for the BC_P: soil mixtures up to 20% and BC_G:soil mixtures up to 5% of w/w incorporation rates used in this study using a sandy loam podzolic soil. Though the M–1 model underestimates the VSMC, the model seemed to be fairly stable up to these rates.

This may suggest that BC amended soils tend to behave differently from both typical mineral soils and soils with high clay or OM contents. For instance, unlike soils having higher OM content with higher soil porosity, low bulk density and substantial amounts of bound water fraction, BC amended soils with high porosity and low bulk density did not perform well when porosity was considered and neither when OM content hence bound water fractions were considered (models M–2 and M–3).

Since BC is known to absorb water apart from the pore water and hygroscopic water, BC amended soils may have absorbed water by BC particles. Soil K_a is affected mostly by the loosely held water (Cihlar & Ulaby, 1974). However, calculation of VSMC from the measured gravimetric moisture does include the weight of absorbed water, causing the original models applied to mineral soil to shift. Also, increased porosity, surface area hence bound water fraction and decreased bulk density may have affected the estimated values using M–2 and M–3 models. These results can suggest that most of the water held in BC amended soils might be absorbed via capillarity into BC particles and less of it can be found as pore water and bound water.

3.4 Conclusions

This study carried out a preliminary evaluation on the influence of non-uniform soil conditions resulting from the application of BC on the accuracy of TDR based VSMC estimation using three commonly used models originally developed for uniform (M-1), non-uniform (M-2), and forest (M-3) soils. The analysis clearly confirms the importance of bulk density and EC as significant factors in the estimation of VSMC using the TDR measured K_a values. Analyses of the experimental data indicated that the Topp's model (M–1) and the mixing model (M–2) provided a reasonably accurate estimation of the VSMC of loamy sand podzolic soil. The model M–1 showed slightly better performances and resolutions for BC:soil mixtures up to 20% of BC_P and 5% of BC_G of w/w incorporation rates used in this study. Therefore, these results indicate that for agricultural purposes (less than 5% BC rates), the measured VSMC of studied BC amended soils are reasonably close to the values obtained from the Topp's equation. Therefore, the commonly used Topp's model can thus be used to determine the VSMC of BC amended soils in the rates not exceeding 5% without significantly compromising the accuracy. However, it needs to be pointed out that due to the limited scope of this experimental study, the conclusion might not be inclusive of different BC types and for soils showing high heterogeneities including the influence of BC on water repellency of soils. Therefore, further investigations along with the underlying mechanisms are needed.

Acknowledgement: Authors wish to acknowledge the financial support from the Research and Development Corporation (RDC), Government of Newfoundland and Labrador (Ignite R&D – 5404-1962-101) and the Memorial University of Newfoundland for research support and graduate research assistantship.

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CHAPTER 4

GENERAL DISCUSSION, CONCLUSIONS AND RECOMMENDATIONS

4.1 General Discussion

Soil amendments are the materials added to soil to improve it's physical, chemical and biological properties. Dairy manure (DM) and biochar (BC) are two such organic amendments used by most farmers to improve soil properties. Soils that benefit the most from organic soil amendments are those lacking in organic matter. These often include sandy soils, urban soils that have been disturbed during construction, landscaping, and utility works. Addition of soil amendments has been known to improve soil physical and hydraulic properties, such as increase soil organic matter (SOM) and soil aggregation, improve soil tilth and porosity, decrease soil bulk density hence increase soil infiltration rates, moisture content, soil hydraulic properties, improve soil aeration, soil strength. Also, they improve soil chemical properties such as pH, cation exchange capacity (CEC) and enhance crop yields. Furthermore, soil amendments may provide suitable habitats to microorganisms. Therefore, stimulating microbe and fungal activity, increasing their exudate production, and providing greater binding agents between soil particles.

However, if applied in high rates, more organic matter/nutrient are added into the soil and when released can cause an outflow of nutrients into the groundwater and surrounding rivers and lakes, which can result in water pollution. Also, a very important and indirect effect due to high rates of manure amendment is that the soil can become water-repellent due to production of water-repellent organic substances. This cause reduction in infiltration rates or uneven patterns of infiltration, influences water flow in the soils through phenomena such as bio-crust formation, and therefore increases surface runoff and overland flow intensifying soil erosion.

The present study evaluated the effect of DM and BC incorporation on VSMC, unsaturated (K_{unsat}) and near-saturated (near K_{sat}) hydraulic conductivity of agricultural podzolic soils. Six different treatment combinations of BC or DM and inorganic fertilizer were tested. Infiltration data were collected using a mini disk infiltrometer under three suction levels; 4, 2 cm as unsaturated and 0.1 cm as the near-saturated condition in the field study. Five suction levels (6, 4, 2, 1, 0.1 cm) were used in the laboratory study. Based on the measured infiltration rates, K_{unsat} and near K_{sat} hydraulic conductivities were estimated. Also, the accuracy of TDR based VSMC estimation in BC amended soils were evaluated using three commonly used models originally developed for uniform (M-1), non-uniform (M-2), and forest (M-3) soils. Two BC types; granular (BC_G) and powdered (BC_P) were used in this study. The accuracy of estimated VSMC using these three models was compared via regression analyses against a standard (M0) VSMC calculated using gravimetric moisture and bulk density (BD).

4.2 General Conclusions

According to the field study, the treatments containing IN+DM1 showed significantly reduced K_{unsat} values compared to the control under 4 and 2 cm suctions, and IN+DM2 significantly reduced K_{unsat} under 2 cm suction. There were no significant changes in near K_{sat} under 0.1 cm suction which was considered as the near-saturated hydraulic conductivity. According to the laboratory study, only IN+DM2 showed significant reductions of K_{unsat} under 6, 4 and 2 cm suctions. There were no significant changes in K_{unsat} and near K_{sat} under 1 and 0.1 cm suctions, respectively. According to the WDPT test, IN+DM1, IN+DM2 and IN+DM1+BC were slightly water repellent.

The tested soil being a loamy sand with higher sand content (73.7±4.1%) relatively has more macropores. The reduction in K in DM treatments might be due to liquid dairy manure clogging the soil pores or changes in soil porosity. However, amended BC did not reduce K significantly and this might be due to its granular nature (1 – 6 mm particle size). Hence, the soil porosity may be less affected by BC amendment compared to when DM was applied as a soil amendment. However, the effect of SMC on K_{unsat} is not clear in this experiment, since the SMC variation was not measured. Also, the near saturated K (at 0.1 cm suction) was significantly lower (around 2 ~ 5 times) in the laboratory experiment than the field experiment. This could be due to the packing effect and lack of macropore flows under laboratory condition. Macropore flows can be expected since infiltration test was done at near saturated (suction = -0.1 cm) conditions. Though, applied DM might have not affected adversely in our soil, long-term application of heavy rates might cause more unfavourable conditions on soil hydraulic properties hence need to apply with caution.

When accuracy of TDR based VSMC estimation was tested, M-1 and M-2 produced very similar results to the actual VSMC in the lowest BC_P rates, but the estimated VSMC was gradually underestimated with increasing BC_P rates. The VSMC estimated with the M–3 non-linearly related to BC_P rates, shifting from overestimation to underestimation as the BC_p rate increased. In BC_G treatments, all models overestimated the VSMC. However, BC_G rates higher than 15% resulted in highly attenuated TDR waveforms and completely dissipated (i.e. no reflection) when >50% BC_G treatments. The model (M–1) underestimated VSMC for all BC_P:soil mixtures throughout the range observed, but estimated VSMC values were better assessed at the wet end (from 30 to 60 %). The same behaviour was observed for M–2. On the other hand, in BC_P:soil mixtures, the results obtained with the forest soil model (M–3) gradually migrated from overestimation towards underestimation as BC_P rates increased. This was opposite for BC_G:soil mixtures, as all the models overestimated the VSMC when increasing the moisture content. This may suggest that BC amended soils tend to behave differently from both typical mineral soils, and soils with high clay or OM contents and the results show that predictions of the soil moisture content based on the soil dielectric constant might not be feasible for soils amended with higher BC rates. These results suggest that new calibrations between dielectric constant measured using TDR and VSMC at higher BC rates and different BC types should be evaluated.

4.3 Recommendations

Further studies are recommended to identify differences in hydrophobicity and particulate matter in different types and rates of DM and BC amendments and their effects on soil hydrology. Also, it needs to be pointed out that due to the limited scope of this experimental study, the conclusion might not be inclusive of different DM and BC types and for soils showing high heterogeneities including the influence of DM and BC on water repellency of soils. Therefore, further investigations along with the underlying mechanisms are needed. Since these soil amendments (DM and BC) can influence soil hydrology such as reduced infiltration and increased surface runoff, carefully monitored application of soil amendments are recommended.

APPENDIX

0% BC

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	230.74	230.735	71.88	0.000
Standard	1	230.74	230.735	71.88	0.000
Error	7	22.47	3.210		
Total	8	253.21			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.79165	91.13%	89.86%	85.89%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-2.54	3.08	-0.83	0.436	
Standard	1.001	0.118	8.48	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	242.54	242.542	69.30	0.000
Standard	1	242.54	242.542	69.30	0.000
Error	7	24.50	3.500		
Total	8	267.04			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.87083	90.83%	89.51%	85.26%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-0.78	3.22	-0.24	0.815	
Standard	1.026	0.123	8.32	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	279.02	279.018	72.20	0.000
Standard	1	279.02	279.018	72.20	0.000
Error	7	27.05	3.865		
Total	8	306.07			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.96589	91.16%	89.90%	85.93%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	8.32	3.38	2.46	0.043	
Standard	1.100	0.129	8.50	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	304.98	304.976	119.48	0.000
Model	3	23.81	7.937	3.11	0.042
Measured*Model	3	1.62	0.538	0.21	0.888
Error	29	74.02	2.553		
Total	36	2076.21			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.59766	96.43%	95.57%	94.31%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-0.00	2.48	-0.00	1.000	
Measured	1.0000	0.0915	10.93	0.000	3.29
Model					
Eq - 1	-2.54	3.70	-0.69	0.497	36.50
Eq - 2	-0.78	3.70	-0.21	0.834	36.50
Eq - 3	8.32	3.70	2.25	0.032	36.50

Measured*Model

Eq - 1	0.001	0.139	0.00	0.997	35.76
Eq - 2	0.026	0.139	0.18	0.855	35.76
Eq - 3	0.100	0.139	0.72	0.478	35.76

Regression Equations

1 : 1 Predicted = -0.00 + 1.0000 Measured

Eq - 1 Predicted = -2.54 + 1.001 Measured

Eq - 2 Predicted = -0.78 + 1.026 Measured

Eq - 3 Predicted = 8.32 + 1.100 Measured

5% BC _Powder

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	520.07	520.074	227.10	0.000
Standard	1	520.07	520.074	227.10	0.000
Error	9	20.61	2.290		

Total	10	540.68

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.51330	96.19%	95.76%	94.54%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-7.50	2.35	-3.19	0.011	
Standard	1.1516	0.0764	15.07	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	579.85	579.849	193.70	0.000
Standard	1	579.85	579.849	193.70	0.000
Error	9	26.94	2.994		
Total	10	606.79			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.73019	95.56%	95.07%	93.56%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-6.67	2.69	-2.48	0.035	
Standard	1.2160	0.0874	13.92	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	650.00	649.996	204.16	0.000
Standard	1	650.00	649.996	204.16	0.000
Error	9	28.65	3.184		
Total	10	678.65			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.78431	95.78%	95.31%	93.88%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	2.30	2.78	0.83	0.428	
Standard	1.2874	0.0901	14.29	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	392.17	392.167	185.26	0.000
Model	3	29.24	9.748	4.61	0.008
Measured*Model	3	17.64	5.880	2.78	0.055
Error	36	76.21	2.117		
Total	43	3467.98			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.45494	97.80%	97.38%	96.83%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-0.00	2.26	-0.00	1.000	
Measured	1.0000	0.0735	13.61	0.000	4.00
Model					
Eq - 1	-7.50	3.20	-2.34	0.025	39.96
Eq - 2	-6.67	3.20	-2.08	0.044	39.96
Eq - 3	2.30	3.20	0.72	0.477	39.96

Measured*Model

Eq - 1	0.152	0.104	1.46	0.153	40.46
Eq - 2	0.216	0.104	2.08	0.045	40.46
Eq - 3	0.287	0.104	2.77	0.009	40.46

Regression Equation

1 : 1 Predicted = -0.00 + 1.0000 Measured

Eq - 1 Predicted = -7.50 + 1.1516 Measured

Eq - 2 Predicted = -6.67 + 1.2160 Measured

Eq - 3 Predicted = 2.30 + 1.2874 Measured

10% BC _Powder

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	577.35	577.352	230.25	0.000
Standard	1	577.35	577.352	230.25	0.000
Error	9	22.57	2.507		
Total	10	599.92			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.58350	96.24%	95.82%	94.39%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-11.13	2.65	-4.20	0.002	
Standard	1.1983	0.0790	15.17	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	660.17	660.166	216.01	0.000
Standard	1	660.17	660.166	216.01	0.000
Error	9	27.51	3.056		
Total	10	687.67			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.74821	96.00%	95.56%	93.96%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-10.73	2.93	-3.67	0.005	

Standard	1.2814	0.0872	14.70	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	733.43	733.434	218.02	0.000
Standard	1	733.43	733.434	218.02	0.000
Error	9	30.28	3.364		
Total	10	763.71			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.83415	96.04%	95.60%	94.04%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-2.08	3.07	-0.68	0.515	
Standard	1.3506	0.0915	14.77	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	402.06	402.055	180.14	0.000
Model	3	35.69	11.896	5.33	0.004
Measured*Model	3	27.78	9.259	4.15	0.013
Error	36	80.35	2.232		
Total	43	3665.50			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.49397	97.81%	97.38%	96.71%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	0.00	2.50	0.00	1.000	
Measured	1.0000	0.0745	13.42	0.000	4.00
Model					
Eq - 1	-11.13	3.54	-3.15	0.003	46.24
Eq - 2	-10.73	3.54	-3.03	0.004	46.24
Eq - 3	-2.08	3.54	-0.59	0.560	46.24
Measured*Model					
Eq - 1	0.198	0.105	1.88	0.068	46.74
Eq - 2	0.281	0.105	2.67	0.011	46.74
Eq - 3	0.351	0.105	3.33	0.002	46.74

Regression Equation

1 : 1   Predicted = 0.0 + 1.0000 Measured

Eq - 1 Predicted = -11.13 + 1.1983 Measured

Eq - 2 Predicted = -10.73 + 1.2814 Measured

Eq - 3 Predicted = -2.08 + 1.3506 Measured

15% BC _Powder

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	450.79	450.787	97.70	0.000
Standard	1	450.79	450.787	97.70	0.000
Error	9	41.52	4.614		
Total	10	492.31			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.14799	91.57%	90.63%	88.59%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-16.13	4.63	-3.49	0.007	
Standard	1.297	0.131	9.88	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	523.72	523.718	100.92	0.000
Standard	1	523.72	523.718	100.92	0.000
Error	9	46.71	5.190		
Total	10	570.42			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.27806	91.81%	90.90%	88.94%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-16.38	4.91	-3.34	0.009	
Standard	1.397	0.139	10.05	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	577.14	577.140	100.21	0.000
Standard	1	577.14	577.140	100.21	0.000
Error	9	51.83	5.759		
Total	10	628.97			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.39986	91.76%	90.84%	88.86%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-7.93	5.17	-1.54	0.159	
Standard	1.467	0.147	10.01	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	268.17	268.170	68.93	0.000
Model	3	39.33	13.110	3.37	0.029
Measured*Model	3	34.07	11.355	2.92	0.047
Error	36	140.06	3.891		
Total	43	3166.10			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.97248	95.58%	94.72%	94.02%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-0.00	4.25	-0.00	1.000	
Measured	1.000	0.120	8.30	0.000	4.00
Model					
Eq - 1	-16.13	6.01	-2.68	0.011	76.52
Eq - 2	-16.38	6.01	-2.73	0.010	76.52
Eq - 3	-7.93	6.01	-1.32	0.195	76.52
Measured*Model					
Eq - 1	0.297	0.170	1.74	0.090	77.02
Eq - 2	0.397	0.170	2.33	0.025	77.02
Eq - 3	0.467	0.170	2.74	0.009	77.02

Regression Equation

1 : 1   Predicted = -0.00 + 1.000 Measured

Eq - 1 Predicted = -16.13 + 1.297 Measured

Eq - 2 Predicted = -16.38 + 1.397 Measured

Eq - 3 Predicted = -7.93 + 1.467 Measured

20% BC _Powder

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	350.534	350.534	525.76	0.000
Standard	1	350.534	350.534	525.76	0.000
Error	7	4.667	0.667		
Total	8	355.201			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
0.816528	98.69%	98.50%	98.22%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-31.85	2.86	-11.13	0.000	
Standard	1.6303	0.0711	22.93	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	437.519	437.519	383.45	0.000
Standard	1	437.519	437.519	383.45	0.000
Error	7	7.987	1.141		
Total	8	445.506			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.06818	98.21%	97.95%	97.15%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-35.68	3.74	-9.53	0.000	
Standard	1.8214	0.0930	19.58	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	472.659	472.659	418.89	0.000
Standard	1	472.659	472.659	418.89	0.000
Error	7	7.899	1.128		
Total	8	480.557			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)

1.06224	98.36%	98.12%	97.51%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-27.59	3.72	-7.41	0.000	
Standard	1.8932	0.0925	20.47	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	157.03	157.031	109.84	0.000
Model	3	59.43	19.809	13.86	0.000
Measured*Model	3	59.81	19.937	13.94	0.000
Error	32	45.75	1.430		
Total	39	2705.72			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.19569	98.31%	97.94%	97.29%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	0.00	3.79	0.00	1.000	
Measured	1.0000	0.0954	10.48	0.000	4.00
Model					
Eq - 1	-27.85	5.36	-5.19	0.000	150.79
Eq - 2	-31.31	5.36	-5.84	0.000	150.79
Eq - 3	-23.00	5.36	-4.29	0.000	150.79
Measured*Model					
Eq - 1	0.537	0.135	3.98	0.000	151.29
Eq - 2	0.720	0.135	5.33	0.000	151.29
Eq - 3	0.786	0.135	5.82	0.000	151.29

Regression Equation

1 : 1 Predicted = 0.00 + 1.0000 Measured

Eq - 1 Predicted = -27.85 + 1.5369 Measured

Eq - 2 Predicted = -31.31 + 1.7195 Measured

Eq - 3 Predicted = -23.00 + 1.7860 Measured

40% BC _Powder

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	353.10	353.102	214.43	0.000
Standard	1	353.10	353.102	214.43	0.000
Error	8	13.17	1.647		
Total	9	366.28			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.28323	96.40%	95.95%	93.51%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-39.51	5.27	-7.49	0.000	
Standard	1.676	0.114	14.64	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	519.93	519.932	277.80	0.000
Standard	1	519.93	519.932	277.80	0.000
Error	8	14.97	1.872		
Total	9	534.91			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.36807	97.20%	96.85%	95.73%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-50.73	5.62	-9.02	0.000	
Standard	2.033	0.122	16.67	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	545.29	545.291	271.58	0.000
Standard	1	545.29	545.291	271.58	0.000
Error	8	16.06	2.008		
Total	9	561.35			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.41697	97.14%	96.78%	95.52%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF

Constant	-42.51	5.82	-7.30	0.000	
Standard	2.082	0.126	16.48	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	125.76	125.759	91.03	0.000
Model	3	90.98	30.328	21.95	0.000
Measured*Model	3	94.05	31.349	22.69	0.000
Error	32	44.21	1.382		
Total	39	2880.45			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.17539	98.47%	98.13%	97.51%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	0.00	4.83	0.00	1.000	
Measured	1.000	0.105	9.54	0.000	4.00
Model					

Eq - 1	-39.51	6.83	-5.78	0.000	253.28
Eq - 2	-50.73	6.83	-7.43	0.000	253.28
Eq - 3	-42.51	6.83	-6.22	0.000	253.28
Measured*Model					
Eq - 1	0.676	0.148	4.56	0.000	253.78
Eq - 2	1.033	0.148	6.97	0.000	253.78
Eq - 3	1.082	0.148	7.30	0.000	253.78

Regression Equation

1 : 1   Predicted = 0.00 + 1.000 Measured

Eq - 1 Predicted = -39.51 + 1.676 Measured

Eq - 2 Predicted = -50.73 + 2.033 Measured

Eq - 3 Predicted = -42.51 + 2.082 Measured

50% BC _Powder

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value

Regression	1	655.84	655.836	211.98	0.000
Standard	1	655.84	655.836	211.98	0.000
Error	9	27.84	3.094		
Total	10	683.68			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.75892	95.93%	95.47%	93.70%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-56.88	6.27	-9.06	0.000	
Standard	2.039	0.140	14.56	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	882.36	882.357	237.05	0.000
Standard	1	882.36	882.357	237.05	0.000
Error	9	33.50	3.722		
Total	10	915.86			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.92933	96.34%	95.94%	94.69%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-66.37	6.88	-9.64	0.000	
Standard	2.366	0.154	15.40	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	941.64	941.644	235.95	0.000
Standard	1	941.64	941.644	235.95	0.000
Error	9	35.92	3.991		
Total	10	977.56			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.99770	96.33%	95.92%	94.61%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-59.78	7.13	-8.39	0.000	
Standard	2.444	0.159	15.36	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	157.68	157.681	58.36	0.000
Model	3	223.10	74.368	27.53	0.000
Measured*Model	3	209.14	69.714	25.80	0.000
Error	36	97.26	2.702		
Total	43	4157.12			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.64369	97.66%	97.21%	96.53%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	0.00	5.86	0.00	1.000	
Measured	1.000	0.131	7.64	0.000	4.00
Model					
Eq - 1	-56.88	8.29	-6.86	0.000	209.96
Eq - 2	-66.37	8.29	-8.00	0.000	209.96
Eq - 3	-59.78	8.29	-7.21	0.000	209.96
Measured*Model					

Eq - 1	1.039	0.185	5.61	0.000	210.46
Eq - 2	1.366	0.185	7.38	0.000	210.46
Eq - 3	1.444	0.185	7.80	0.000	210.46

Regression Equation

1 : 1   Predicted = 0.00 + 1.000 Measured

Eq - 1 Predicted = -56.88 + 2.039 Measured

Eq - 2 Predicted = -66.37 + 2.366 Measured

Eq - 3 Predicted = -59.78 + 2.444 Measured

100% BC _Powder

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	690.04	690.041	155.44	0.000
Standard	1	690.04	690.041	155.44	0.000
Error	9	39.95	4.439		
Total	10	729.99			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.10696	94.53%	93.92%	92.32%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-99.0	10.9	-9.05	0.000	
Standard	2.326	0.187	12.47	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	997.70	997.701	161.00	0.000
Standard	1	997.70	997.701	161.00	0.000
Error	9	55.77	6.197		
Total	10	1053.47			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.48934	94.71%	94.12%	92.96%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-120.0	12.9	-9.28	0.000	

Standard	2.796	0.220	12.69	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	1051.63	1051.63	162.98	0.000
Standard	1	1051.63	1051.63	162.98	0.000
Error	9	58.07	6.45		
Total	10	1109.71			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.54020	94.77%	94.19%	93.01%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-115.0	13.2	-8.72	0.000	
Standard	2.871	0.225	12.77	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	127.6	127.583	29.86	0.000
Model	3	353.5	117.843	27.58	0.000
Measured*Model	3	287.4	95.792	22.42	0.000
Error	36	153.8	4.272		
Total	43	6012.9			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.06692	97.44%	96.94%	96.55%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-0.0	10.7	-0.00	1.000	
Measured	1.000	0.183	5.46	0.000	4.00
Model					
Eq - 1	-99.0	15.2	-6.52	0.000	445.10
Eq - 2	-120.0	15.2	-7.90	0.000	445.10
Eq - 3	-115.0	15.2	-7.58	0.000	445.10
Measured*Model					
Eq - 1	1.326	0.259	5.12	0.000	445.60
Eq - 2	1.796	0.259	6.94	0.000	445.60
Eq - 3	1.871	0.259	7.23	0.000	445.60

Regression Equation

1 : 1   Predicted = -0.0 + 1.000 Measured

Eq - 1 Predicted = -99.0 + 2.326 Measured

Eq - 2 Predicted = -120.0 + 2.796 Measured

Eq - 3 Predicted = -115.0 + 2.871 Measured

5% BC _Granular

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	459.09	459.090	108.06	0.000
Standard	1	459.09	459.090	108.06	0.000
Error	8	33.99	4.248		
Total	9	493.08			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.06117	93.11%	92.25%	89.75%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-6.88	4.22	-1.63	0.142	
Standard	1.333	0.128	10.40	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	738.29	738.294	89.49	0.000
Standard	1	738.29	738.294	89.49	0.000
Error	8	66.00	8.250		
Total	9	804.29			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.87221	91.79%	90.77%	86.67%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-13.27	5.89	-2.25	0.054	
Standard	1.691	0.179	9.46	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	765.54	765.543	92.36	0.000
Standard	1	765.54	765.543	92.36	0.000
Error	8	66.31	8.288		
Total	9	831.85			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.87896	92.03%	91.03%	87.20%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-3.86	5.90	-0.65	0.532	
Standard	1.722	0.179	9.61	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	258.26	258.265	49.70	0.000
Model	3	22.43	7.477	1.44	0.250

Measured*Model	3	89.66	29.887	5.75	0.003
Error	32	166.29	5.197		
Total	39	4555.49			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.27961	96.35%	95.55%	94.20%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	0.00	4.67	0.00	1.000	
Measured	1.000	0.142	7.05	0.000	4.00
Model					
Eq - 1	-6.88	6.61	-1.04	0.305	62.99
Eq - 2	-13.27	6.61	-2.01	0.053	62.99
Eq - 3	-3.86	6.61	-0.58	0.564	62.99
Measured*Model					
Eq - 1	0.333	0.201	1.66	0.106	63.49
Eq - 2	0.691	0.201	3.44	0.002	63.49
Eq - 3	0.722	0.201	3.60	0.001	63.49

Regression Equation

1 : 1   Predicted = 0.00 + 1.000 Measured

Eq - 1 Predicted = -6.88 + 1.333 Measured

Eq - 2 Predicted = -13.27 + 1.691 Measured

Eq - 3 Predicted = -3.86 + 1.722 Measured

10% BC _Granular

Regression Analysis: Eq 1 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	1806.5	1806.46	60.11	0.000
Standard	1	1806.5	1806.46	60.11	0.000
Error	8	240.4	30.05		
Total	9	2046.9			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
5.48202	88.25%	86.79%	69.76%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-12.22	8.78	-1.39	0.202	

Standard	2.400	0.310	7.75	0.000	1.00

Regression Analysis: Eq 2 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	1961.60	1961.60	666.88	0.000
Standard	1	1961.60	1961.60	666.88	0.000
Error	8	23.53	2.94		
Total	9	1985.14			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.71507	98.81%	98.67%	97.94%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	-5.21	2.75	-1.90	0.094	
Standard	2.5009	0.0968	25.82	0.000	1.00

Regression Analysis: Eq 3 versus Standard

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	1	1854.49	1854.49	758.65	0.000
Standard	1	1854.49	1854.49	758.65	0.000
Error	8	19.56	2.44		

Total	9	1874.04

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
1.56348	98.96%	98.83%	98.26%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	6.32	2.50	2.53	0.036	
Standard	2.4317	0.0883	27.54	0.000	1.00

General Linear Model: Predicted versus Measured, Model

Factor Information

Factor	Type	Levels	Values
Model	Fixed	4	1 : 1, Eq - 1, Eq - 2, Eq - 3

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Measured	1	313.6	313.628	35.40	0.000
Model	3	72.3	24.109	2.72	0.061
Measured*Model	3	492.3	164.088	18.52	0.000
Error	32	283.5	8.860		
Total	39	18074.3			

Model Summary

S	R-sq	R-sq(adj)	R-sq(pred)
2.97651	98.43%	98.09%	96.17%

Coefficients

Term	Coef	SE Coef	T-Value	P-Value	VIF
Constant	0.00	4.77	0.00	1.000	
Measured	1.000	0.168	5.95	0.000	4.00
Model					
Eq - 1	-12.22	6.74	-1.81	0.079	38.48
Eq - 2	-5.21	6.74	-0.77	0.445	38.48
Eq - 3	6.32	6.74	0.94	0.355	38.48
Measured*Model					
Eq - 1	1.400	0.238	5.89	0.000	38.98
Eq - 2	1.501	0.238	6.31	0.000	38.98
Eq - 3	1.432	0.238	6.02	0.000	38.98

Regression Equation

1 : 1   Predicted = 0.00 + 1.000 Measured

Eq - 1 Predicted = -12.22 + 2.400 Measured

Eq - 2 Predicted = -5.21 + 2.501 Measured

Eq - 3 Predicted = 6.32 + 2.432 Measured