

ASSESSMENT OF PHYSICOCHEMICAL PROPERTIES OF A LOAMY SAND
TREATED WITH BIOCHAR AND DAIRY MANURE UNDER SILAGE CORN
PRODUCTION IN WESTERN NEWFOUNDLAND

by

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Abstract

Soil physicochemical properties play an important role in crop growth and final harvest. Different agronomic practices can improve soil health through modulating these physicochemical properties. Dairy manure (DM) is a good and abundant source of nutrients in Newfoundland, and both biochar (BC) and DM are considered good soil amendments. A field experiment was conducted at Pynn's Brook Research Station, Pasadena NL, to evaluate the effect BC and DM has on various soil physicochemical properties, as well as to establish a relationship between soil electrical conductivity (EC) and these selected physicochemical properties. Experimental treatments with four replicates included control, inorganic nitrogen (IN), IN+BC, DM, and DM+BC. Dairy manure was applied at the rate of 30,000 L ha⁻¹, whereas BC was applied at 20 t ha⁻¹ (both were applied on May 23, 2016) and mixed within the top 10 cm of the loamy sand soil (82% sand + 6% silt + 12% clay). Disturbed soil samples were collected from treatment plots on four different days. Results showed no significant ($p > 0.05$) treatment effects on gravimetric and volumetric moisture contents, pH, ammonium nitrogen (NH₄⁺-N), cation exchange capacity (CEC), or EC of tested soils, within each field day. Soil organic carbon (SOC) had a significant ($P=0.042$) difference between DM and DM+BC treatments only on August 4. However, significant temporal effects were recorded for pH, NH₄⁺-N, and EC – both apparent electrical conductivity (ECa) and electrical conductivity of soil solution (ECw) – across four field days. The decrease of NH₄⁺-N could possibly be accounted to volatilization, uptake by plants, immobilization by microbes, or conversion to nitrate-nitrogen, while the decrease in EC may be due to a decrease in ion concentration from uptake by plants and leaching. No significant differences were observed between the 0-10 cm and 10-20 cm soil depths within each treatment for the tested soil properties. Positive correlations were recorded for EC with SOC, NH₄⁺-N, and CEC (ECa, ECw 0-10 cm, & ECw 10-20 cm, $p=0.000$). Once correlation data has been validated, the electromagnetic induction method can be used to map the ECa and respective spatio-temporal variability of important soil properties for similar soils in Newfoundland.

1.0 Introduction

1.1 Background

In order to attain food security in Newfoundland and Labrador (NL), local food production has to be increased. This will in turn cause food to be less expensive and fresher to buy for the people of NL. Farmers use different agronomic techniques to increase the productivity of the land, such as adding inorganic fertilizer, manure, or biochar – either as sole application, or in various combinations. It is important, however, to use techniques that not only increase the land productivity, but also minimize the negative impacts on soil, water, and environment. If too much fertilizer is applied to the soil, it can leach into groundwater, leading to the threat of contamination and eutrophication in water bodies. Efficient use of inorganic fertilizer, manure, and biochar will maintain healthy soils, as well as increase productivity. How do we determine the correct amounts of each to use? Will this vary with the different types of soil, climatic zones, cropping systems, or field location? How do these applications and different management techniques affect the spatial and temporal variability of soil properties and subsequent crop yield? Is it possible to both increase productivity as well as minimize the pollution of soil and water resources? What would be the best agronomic management system for both maintaining the soil health and increasing agricultural productivity? What would the variability of soil properties be under these different management systems? Field scale measurements of the spatial variability of soil properties are difficult, requiring hard labour, money, and various other resources. Thus, farmers, agricultural managers, and researchers often adopt only point scale measurements of soil properties and interpolate them to other areas. Can a relationship be seen for soil

productivity and health through various soil measurements such as ECa (apparent electrical conductivity), soil organic carbon (SOC), pH, cation exchange capacity (CEC), temperature, and nitrate and ammonium content? Not much research has yet been done to understand the variability of physicochemical properties of soils under intensive agriculture in NL, and so it is difficult to know which treatments would increase soil health and productivity while minimizing the harmful effects of these treatments on the environment.

1.2 Literature

1.2.1 The use of Biochar in Agricultural Fields

Biochar is a carbon-rich material produced through pyrolysis (**Figure 1.1**) of organic materials (Githinji, 2014; Joseph et al., 2015; Sorrenti et al., 2016). Studies have shown that it has many beneficial properties for agricultural soils, and has the potential to sequester carbon, increase plant yields, enhance and alter the physical and chemical properties of soils, alter emissions of nitrous oxide (N₂O), affect nutrient leaching, improve soil water properties, and impact the availability of contaminants in soils (Joseph et al., 2015; Sorrenti et al., 2016). When added to soils, biochar interacts with plant roots, root hairs, soil organic matter (SOM), proteins, microorganisms, and “the nutrient-rich soil solution to form organo-mineral-biochar complexes” (Joseph et al., 2015). Biochars are very effective at improving many aspects of soil physical and chemical properties, and these will be discussed below.

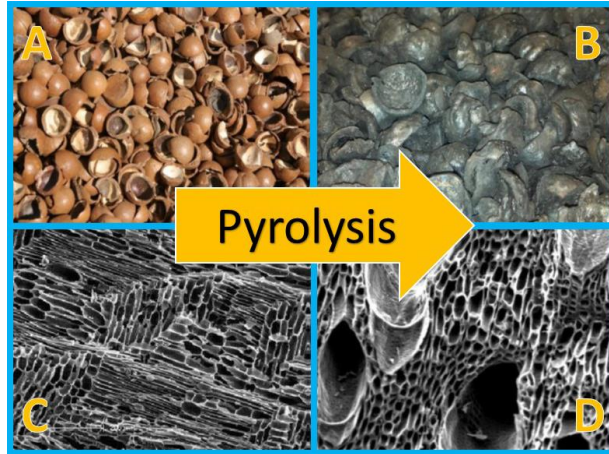


Figure 1.1 Production of Biochar Through Pyrolysis of Organic Materials (Illinois Sustainable Technology Center).

1.2.2 Soil Moisture Content

The moisture content of a soil is very important. The properties of water promote a wide variety of chemical, physical, and biological processes. These processes greatly influence most aspects of soil behaviour and development, such as weathering of minerals, decomposition of organic matter, pollution of groundwater, and growth of plants (Brady & Weil, 2008). Cohesion – the sticking of water molecules to each other – and adhesion – sticking of water molecules to solid soil particles – through hydrogen bonding, are the forces responsible for water retention and movement in soils (Brady & Weil, 2008). If there is not enough water in soils, plants may wilt permanently unless enough water is supplied through irrigation. This is especially a problem in sandy soils, as the main pore sizes are in the macropore range, where water drains quickly, not leaving much for plant uptake.

Biochar is a porous material, having the potential to absorb and retain large amounts of moisture (Liu et al., 2012). Thus, application of biochar to soil increases its capacity to retain water (Liu et al., 2012).

1.2.3 Soil pH

As Newfoundland receives a lot of rain, the soil is relatively acidic, and leaching of ions from the upper soil profile is high. This process is called podsolization and forms “Podzol,” which is a very common soil type in boreal regions. One of the disadvantages of acidic soils is that a decrease in pH influences, for example, potassium fixation of clay minerals by reducing its ability for fixation (Brady & Weil, 2008). The dominance of hydrogen (H^+) and aluminum (Al^{3+}) ions causes acidity of these soils, and limits crop utilization of many essential nutrients in the soil, thus limiting crop yield (Chintala et al., 2016). Due to this, there has been an increased interest in using liming agents. Some researchers have shown that addition of biochar to acidic soils also increases pH (Chintala et al., 2013; Molnár et al., 2016). This can in turn reduce the H^+ and Al^{3+} ion dominance, since Al^{3+} precipitates as hydroxy-Al polymers at higher pH, increasing essential nutrient availability and crop yield (Chintala et al., 2013; Haynes & Naidu, 1998).

1.2.4 Soil OC (SOC) and SOM

Soil organic carbon is known to be the largest carbon stock in terrestrial ecosystems (Lugato et al., 2014). Approximately 75% of the carbon stored in terrestrial ecosystems is in the form of SOC (Zhao et al., 2013). In Boreal ecosystems, the amount of carbon

stored in the soil is three times greater than that in the plants (Hakkinen et al., 2011). The soils of Canada's Boreal region contain large carbon reservoirs that have accumulated over thousands of years (Bhatti et al., 2001). It therefore plays an active role in source/sink relationships of terrestrial carbon (Bhatti et al., 2001). Due to this high storage of carbon, it is important to ensure that agricultural practices do not release this carbon as carbon dioxide (CO₂) into the atmosphere, increasing the risk of rising global temperatures. Decomposition of these large stocks of SOM in northern latitude ecosystems in response to rising temperatures may be one of the largest feedbacks with respect to climate change (He et al., 2014), and thus, it is important to know how using these soils for agriculture would affect its carbon storage. Conversion of forests to croplands, fertilizing the lands, and removing residue and tillage regimes decreases SOC very rapidly, eventually stabilizing at a much lower SOC values (Brady & Weil, 2008; Zhao et al., 2013). Environmental conditions that influence SOC levels are temperature, soil texture, and rainfall (Zhao et al., 2013). Quantifying SOC dynamics in response to various agricultural management practices may help to identify which practices sequester carbon while also sustaining agricultural productivity (Zhao et al., 2013). Protection from soil erosion, maintaining soil structure and SOM levels have been shown to be the minimum requirements needed to achieve good conditions and soil health for agricultural lands (Lugato et al., 2014). Biochar has been shown to increase SOC levels, in turn also increasing the SOM, and is a good fertilizer to apply for sequestering carbon (Li et al., 2015; Sorrenti et al., 2016).

Soil organic carbon is usually used to describe the carbon component of SOM (Brady & Weil, 2008; Schollenberger, 1945). To obtain SOM from SOC, SOC is usually multiplied by 1.724 to obtain the assumed SOM percentage (Schollenberger, 1945).

1.2.5 Ammonium and Nitrate

Nitrogen is an integral component for plant health. Nitrogen is vital for plant metabolism and is a major part of all amino acids – the building blocks of proteins – which includes enzymes, as well as nucleic acids and chlorophyll (Brady & Weil, 2008; Chibnall, 1953). If the plants can take up enough nitrogen, it enhances root growth, along with uptake of other nutrients, enhancing plant productivity, as well as carbon production (Brady & Weil, 2008; Zhu & Zhuang, 2013). When there is a nitrogen deficiency, the plant leaves will turn a yellowish to pale green colour. In soils, inorganic nitrogen (N) is predominantly found in the forms of nitrate (NO_3^-) and ammonium (NH_4^+), which are the main forms taken up by plants (Brady & Weil, 2008; Maynard & Crumbaugh, 2006; Rao & Puttanna, 2000). It is important, however, not to have too high concentrations of nitrogen in the soils, as this would cause losses through runoff and leaching. An increasing amount of N input into agricultural soils has been identified as a major issue that may potentially compromise human health (Rao & Puttanna, 2000; Sebilo et al., 2013). An increase in use of nitrogen fertilizer has increased leaching of these fertilizers into surface and groundwater, resulting in contamination of aquifers, as well as eutrophication of freshwater bodies, especially in areas close to agricultural fields (Rao & Puttanna, 2000; Riley et al., 2001; Sebilo et al., 2013). Ammonia loss specifically is governed by soil factors such as pH, CEC, moisture, and temperature (Kasim et al.,

2013). Organic substances, such as SOC, have a good effect on controlling the amount of nitrogen loss from soils (Kasim et al., 2009). Therefore, if SOC can be increased, the nitrogen-holding capacity of soils may potentially also be increased, causing nitrogen to be released at a slower rate into the soils for uptake by plants.

Biochar has been shown to have real potential in impacting the soil nitrogen cycle (Spokas et al., 2012). A study by Taghizadeh-Toosi et al. (2012) found that ammonium-N ($\text{NH}_4^+\text{-N}$) that has been adsorbed by biochar is stable in air. However, it is readily available once placed into soil, and is therefore not irreversibly bound, but available to the plants. Nitrogen recovery was especially high when acidic biochars were used for capturing ammonia (Taghizadeh-Toosi et al., 2012). It is therefore assumed that biochar application in soil will decrease the amount of leaching. As a result, less N would need to be applied to soil, as a lot more would be taken up by the plants instead of being leached into the environment.

1.2.6 Soil CEC

Cation exchange capacity can be defined as the total amount of exchangeable cations that a soil can adsorb, in an exchangeable fashion, onto the negative charge sites of soil (**Figure 1.2**) at a specific pH (van Erp et al., 2013; Hendershot et al., 2006). CEC is mainly due to inorganic constituents like hydrous oxides, primary and secondary minerals, clay minerals, and SOM (Schnitzer, 1965). The CEC is very important for determination of fertilizer retention capacity, intrinsic soil fertility, plant nutrition, etc. in agronomy (Pansu et al., 2006). Sandy soils are generally low in all colloidal materials, and therefore low CECs are expected compared to silt and clay loam soils (Brady &

Weil, 2008). At very low pH, CEC is generally low, and therefore an increase in pH would also be expected to increase CEC (Brady & Weil, 2008). Biochar, as was discussed with soil pH, would be expected to increase CEC by also increasing soil pH. Recent studies have shown that biochar increases CEC of soils (Githinji et al., 2014; Chintala et al., 2013). When biochar is exposed to oxygen (O_2) and water (i.e. when added to the soil) spontaneous oxidation reactions take place, most likely enhanced through microbial activity, resulting in high CEC (Lehman et al., 2009). As biochar ages, CEC increases even more due to an increase of the oxygenated functional groups attached to the biochar (Lehman et al., 2009).

As biochar is primarily composed of condensed and single ring aromatic carbons, it has a high charge density, as well as a high surface area per unit mass, therefore contributing to a higher adsorption capacity, and therefore can adsorb cations better than SOM (Githinji, 2014).

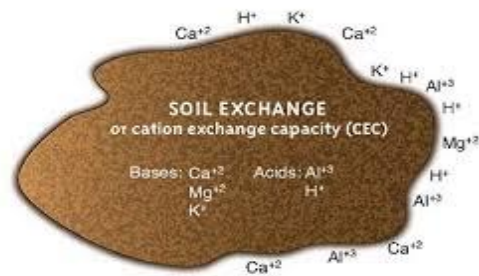


Figure 1.2 Adsorption of Cations onto the Negatively Charged Surface of a Soil Particle (Soil Science Society of America).

1.2.7 Electrical Conductivity (EC)

Electrical conductivity is a measurement that correlates with properties of the soil affecting crop productivity (Grisso et al., 2009). Physico-chemical properties, including CEC, soil salinity, subsoil characteristics, soil texture, clay content, bulk density (BD), and SOM levels influence EC (Corwin & Lesch, 2005; Grisso et al., 2009). Soil EC_a can be measured directly in the field by inserting probes into the soil (Brady & Weil, 2008). EC_w (EC of soil solution), on the other hand, is a measurement of EC that involves shaking of a soil sample in a 1:2 soil-distilled water mixture for 30 minutes (Brady & Weil, 2008; Miller & Curtin, 2006). EC_a is an easy measurement that often, but not always, relates to the yield of crops (Corwin & Lesch, 2005). Ristolainen et al. (2009) discussed how EC_a has mostly been correlated with soil salinity and water content in saline soils, while in nonsaline soils, EC_a depends mostly on SOM content, soil texture – especially clay content – and plant available nutrients. It is assumed that NL's soils are not saline, due to high leaching potentials with high amounts of well distributed rain received each year, and therefore it would be expected that the EC_a measurements obtained will depend on the non-saline soil properties discussed above.

Various studies have shown that biochar can also influence soil EC by increasing the EC values (Chintala et al., 2013; Molnár et al., 2016). One reason given for this is that biochar can hold more soluble salts due to higher proton consumption, and thus increase EC values (Chintala et al., 2013).

1.3 Objectives

The aim of this research is to employ various techniques for measuring chemical and some physical properties of soils to evaluate the effect of different soil amendments on the physicochemical properties. Special attention will be given to experimental plots containing biochar and manure. The chemical and physical properties will be compared to understand if biochar and manure application increases moisture content (MC), pH, SOC, $\text{NH}_4^+\text{-N}$, CEC, and EC of the soils compared to those without these treatments. Secondly, the control plots will be compared with the inorganic nitrogen (IN), dairy manure (DM), and biochar plots to see if there is a significant treatment effect within each field day. Inorganic nitrogen with biochar (IN+BC) and dairy manure with biochar (DM+BC) plots will be compared to see which would be the best to use for enhancing the physicochemical properties of soils, or if both will show similar results, as was shown in the study by Partey et al. (2014). Lastly, once all analysis of the soil samples has been done, data analysis will be performed to see if a relationship for ECa and ECw with other soil properties can be obtained. As most of these standard methods for obtaining soil properties only provide point scale measurements, a lot of resources and time can be saved if ECa or ECw measurements can be related to relevant soil properties. The long-term goal is to map the variability of soil properties over larger agricultural fields by measuring ECa using the electromagnetic induction (EMI) method.

2.0 Methodology

An agronomic experiment on increasing the silage corn productivity has been established at the Pynn's Brook Research Station (PBRS), in Pasadena, NL, using different nutrient management systems. The soil analyzed for this experiment was loamy sand (82% sand + 6% silt + 12% clay). Texture data for the soil can be seen in **Table 2.1**.

Table 2.1 Modeled Properties of Loamy Sand at Pynn's Brook Research Station.

Porosity	Field Capacity	Permanent Wilting Point	Bulk Density
45-51%	15.3%	9.1%	1.20-1.45 g cm ⁻³

Dairy manure was applied at a rate of 30,000 L ha⁻¹, while biochar was applied at a rate of 20 t ha⁻¹ and mixed within the top 10 cm of the soil (the properties of the biochar used in this experiment can be seen in **Appendix A**). Both were added on May 23, 2016 - approximately two months before the first sampling day. The silage corn was planted on May 24, 2016. The growth stages of the silage corn can be seen in **Table 2.2**.

Table 2.2 Growth Stages of Silage Corn.

July 8	August 13	August 28
Six leaf stage	Twelve leaf stage	Tasseling Stage

The treatments that will be analyzed are inorganic nitrogen (IN), IN with biochar, (IN+BC), dairy manure (DM) with high phosphorus and nitrogen, and DM with biochar, high phosphorus and high nitrogen (DM+BC), as well as a control field with no nitrogen (NO). The treatment numbering for this experiment was as follows:

T-1: Control (NO)

T-2: IN

T-3: IN + BC

T-4: DM

T-5: DM + BC

Composite soil samples representing three sampling locations will be taken at each treatment plot. These sampling locations are divided into two depths – D1 from 0-10 cm, and D2 from 10-20 cm. There are four replicates for each treatment and therefore, in total, 40 samples will be collected for each soil-sampling day. The field layout can be seen in **Figure 2.1**.

8	T4R1	T1R2	T4R3	
7		T3R2		
6		T2R2		T3R4
5		T5R2	T5R3	T1R4
4	T5R1		T2R3	
3	T2R1		T3R3	T5R4
2	T3R1	T4R2		T4R4
1	T1R1		T1R3	T2R4
	Row 1	Row 2	Row 3	Row 4

Figure 2.1 Layout of the Field where Sample Collection was done.

Along with soil sampling, portable TDR (time domain reflectometry) sensors will be used to measure EC_a, temperature, and moisture of the soil in the field. The soil samples will be brought to the lab in an insulated box and analyzed for moisture content, pH, and EC_w. Weighing out approximately 10 g of soil, placing it in the oven for 24 hours at

105°C, and then weighing it again will obtain gravimetric moisture content, θ_m of the soil by using the following equation:

$$\theta_m = \frac{m_w}{m_s}$$

where m_w is the mass of water that was in the soil, and m_s is the mass of the oven-dried soil (Brady & Weil, 2008; Sheppard & Addison, 2006). Electrical conductivity of soil solution will be measured using air-dried soil, diluted in a 1:1 and 1:2 soil to distilled water ratio (Brady & Weil, 2008; Miller & Curtin, 2006). Two probes, the HANNA ECw instrument (HI9813-6 Portable pH/EC/TDS/Temperature Meter with CAL Check) and the ECTester11+ (Fieldsout Direct Soil EC Meter, Spectrum Technologies, Inc.) will be used to measure ECw and temperature. Soil pH and total dissolved solids (TDS) will also be analyzed using the HANNA ECw instrument. Soil organic carbon content, CEC, and NH_4^+ -N and NO_3^- -N concentrations will also be measured. The SOC analysis will be performed using the Walkley-Black chromic acid wet oxidation method (Soil Survey Standard Test Method: Organic Carbon), and from this, SOM content can also be calculated. The CEC will be measured using the sodium acetate method (Method 9081: Cation-Exchange Capacity of Soils [Sodium Acetate]) and ion chromatography (IC) (Model: Dionex ICS-5000⁺ DC-5 Detector/Chromatography Module). NH_4^+ -N and NO_3^- -N will be measured through extraction with potassium chloride (Maynard et al., 2006) and using the SEAL Analytical instrument (Model: SEAL XY-2 Sampler; Serial Number: 5337A33563) for analyzing nitrate and ammonia.

Once all tests have been performed, these properties will be used to understand which method of fertilization or combination most enhances soil quality and productivity. These properties will also be compared to the ECa measurements to observe if there is a

specific relationship. If there is, it may suggest that all these various measurements may not be necessary, and that ECa could be used more extensively to map the spatial variability over the landscape, as it is much less time consuming and can cover a large area.

2.1 Sampling and Data Collection

On the 28th of July, the first field visit was conducted. The time frame was from approximately 10AM to 5:30PM. Forty composite disturbed soil samples were obtained from five different selected treatment plots. This was done by taking three soil samples within each of the 20 plots (4 plots/replicates for each treatment) and collecting the soil from the first depth (D1) to one sampling bag, and all the second depth (D2) soil samples to another labeled bag. Once each composite sample was obtained, the bags were shaken to ensure thorough mixing of the soil. The bags used were freezer zip-lock bags. The soil samples were placed in a Styrofoam box to keep the soil cool while being transported to the laboratory. Along with collecting soil samples, probe data was collected at each of the three soil sampling spots within each of the 20 plots. The instrument displayed the average volumetric moisture content (VMC), ECa, and temperature of the soil for the 0-5 cm probe. The instrument used for this was the ProCheck (ProCheck handheld reader, ICT International; GS3 sensor). The HD2 (IMKO GmbH, Germany) instrument was also used to obtain these values, and measured at a depth of 0-11 cm. Upon returning to the lab, the soil samples were placed in the refrigerator. The next morning, on the 29th of July, approximately 20 g of soil was weighed from each sample and placed in the oven over the weekend at 105°C, and weighed again to determine gravimetric moisture content

(GMC) three days later. The soil not used for these measurements was air dried over the weekend (29th July to 1st of August). The dried soil samples were sieved using a 2 mm sieve, discarding the rocks. These samples were then placed back into dry labeled bags for later measurement of SOC, CEC, and EC_w.

For EC_w measurements, 15 g of each soil sample was weighed into a container. This was first done in a 1:1 ratio, and so 15 mL of distilled water was added to each sample. The samples were placed on the shaker and shaken for 30 minutes. Once this was done, the EC_w, pH, TDS, and temperature of each sample was obtained using the HANNA instrument, and EC_w and temperature were recorded using the ECTestr11+ as well. More water was added for the same measurements, this time at a 1:2 ratio of soil:distilled water. Once 15 mL more water was added, the samples were again shaken for 30 minutes, and the measurements were obtained for the same parameters with the instruments. These measurements were obtained on the 1st and 2nd of August.

The second field day was on the 4th of August, and sampling took place from about 10AM to 5PM. The same procedure as on July 28th was followed for soil sampling, data collection, probe measurements, bringing samples back to the laboratory, analysis (which took place on the 5th, 8th and 9th of August), and storage of samples for further analysis later. Approximately 10 g of wet soil was left in the bags, and placed in the freezer to later be analyzed for NH₄⁺-N and NO₃⁻-N.

On the 10th of August, the air-dried samples were again sieved, this time with a 500 μ m sieve. Approximately 2 grams of each sieved sample was placed into separate labeled containers, later to be used for SOC analysis. This was done for all 80 soil samples. These were then placed in the refrigerator.

SOC analysis was started on the 2nd of September. The automatic Titrator (Mettler Toledo G20 compact Titrator) was set up, and it was tested for accuracy. The method followed was the Walkley-Black chromic acid wet oxidation method (Soil Survey Standard Test Method: Organic Carbon). Approximately 1 g of soil was weighed into a 250 mL Erlenmeyer flask. 10 mL of potassium dichromate ($K_2Cr_2O_7$) was added to the beaker containing the soil. 20 mL of 95 – 98% sulfuric acid (H_2SO_4) was then also added to the solution. Once this was done, a vigorous reaction took place, and the solution changed from an orange to a blue colour (the reactions that occur during this reaction can be seen in **Appendix B**). The temperature of the solution was measured and found to go up to approximately 135°C within seconds. The titrant used was an iron (II) sulfate solution ($FeSO_4$). Upon determination, however, it was noted that too much soil was added, as the solution was already past the endpoint of the titration. A soil sample of 0.5 g was then used, and it was shown to work well. The titration was done manually to see if the instrument worked properly.

On the 6th of September, two more manual titrations using 0.5 g of soil were done to test whether the instrument was accurate. This was a long but successful procedure.

On the 8th of September, the automatic titrator was used. Three soil samples with known SOC content were measured using the instrument to confirm its accuracy. The values obtained by the instrument for these known SOC contents were very close to that of the previously measured values, and thus, it was confirmed that the instrument measures SOC accurately.

On the 9th of September, determination of SOC for the 28th of July's samples began. On this day, 10 samples were completed. It is important to note that once the

potassium dichromate and sulfuric acid were added to the soil samples, the colour changed from orange to yellow or green, which is the colour just before the endpoint. The samples were allowed to cool for 20 minutes. After this time, the Erlenmeyer flasks containing the solutions were filled to the 200 mL mark with deionized water and placed in plastic containers for analysis. For each run of 8 to 9 samples, two were control samples, containing only H₂SO₄, K₂Cr₂O₇, and deionized water. The equation used to calculate the SOC for these samples was as follows:

$$\text{SOC}(\%) = \frac{3(1-T/S)}{W}$$

where T is the volume of the FeSO₄ used in titrating the sample (mL), S is the volume of FeSO₄ used in the blank titration (mL), and W is the weight of the sample that was used for titration, corrected for oven-dried weight (g) (Soil Survey Standard Test Method: Organic Carbon). The rest of the 28th of July's soil samples were analyzed on the 12th and 13th of September. The samples from the 4th of August's field trip were analysed on the 15th, 19th and 20th of September.

The third field trip was on the 30th of September. Soil sampling took place from approximately 12PM to 4PM. Once at the lab, approximately 10 g of wet soil from each sample was weighed and placed in the oven to obtain GMC. On the 1st of October, the soil samples were again weighed after the 24-hour drying period. Most of the soil was placed out to dry over the weekend on this day as well, while the remaining wet soil was placed in the freezer. On the 4th of October, the air-dried soils were placed in dry bags and placed in the fridge for later analysis.

The last field trip was on the 6th of October. Soil sampling took place from 12PM to 3PM. The same procedure as on September 30th was followed for soil sampling, data

collection, probe measurements, bringing samples back to the laboratory, analysis (which took place on the 6th, 7th, and 11th of October), and storage of samples for further analysis later.

In the following weeks from the 20th of October to the 4th of November, the air-dried soils were sieved with both the 2 mm and 500 μm sieves to prepare soil samples for CEC, ECw, and SOC analysis.

On the 12th, 15th and 16th of December, the last two field day samples were analyzed for SOC, and for ECw on the 13th and 14th.

Early in January, approximately 4 g of the 100 samples chosen for CEC analysis were weighed out and placed in appropriate containers. The oven-dried weights from the air-dried samples were also obtained and corrected for the SOC values. All solutions needed for analysis of $\text{NH}_4^+\text{-N}$, $\text{NO}_3^-\text{-N}$, and CEC that are stable for a long time were prepared and stored.

On the 26th of January, the 120 frozen samples were removed from the freezer and extracted using the KCl solution. Approximately 2 g of field-moist soil was weighed out in Erlenmeyer flasks, and 20 mL of the KCl solution was added. These samples were then shaken for 30 minutes on the mechanical shaker, and the solution extracted, using filter paper, into labeled containers. The next day, the 27th of January, all samples were analyzed for $\text{NH}_4^+\text{-N}$ using the SEAL Analytical Instrument. The remainder of the samples not used for analysis were placed in the freezer, later to be analyzed for $\text{NO}_3^-\text{-N}$.

On the 30th of January, more sodium acetate (14 L in total) and ammonium acetate (11 L) for CEC were prepared for analysis the next day. CEC analysis took place from the 31st of January to the 9th of February. The soil samples were washed with 33 mL

of sodium acetate, shaken for 5 minutes on a shaker, and then centrifuged and the liquid decanted. This was done four times for each sample. The soil samples were then washed with 33 mL of isopropyl alcohol 3 times, following the same procedure. When washed with ammonium acetate, the 33 mL (three times) solution was poured into a 100 mL plastic volumetric flask, and filled to volume with ammonium acetate. These finished, samples were then placed in clean, labeled containers and placed in the fridge for later analysis.

On the 9th of March, CEC samples were diluted to a 1:100 mL solution, and the first 50 samples were analyzed for sodium concentration through with IC. No interference was observed between the sodium and ammonium peaks, as can be seen in **Figure 2.2**. On the 10th of March, the last 50 were analyzed. Values were corrected for actual concentration, through multiplying by 100, and converted from ppm to cmol kg^{-1} by using the following equation:

$$\text{Exchangeable Na} = \frac{a \cdot (33) \cdot \text{mcf}}{(10) \cdot (23.00) \cdot s}$$

where a is ppm of Na, mcf is the moisture factor to correct for oven-dried weight, s is the air-dried sample weight (g), 33 is for the 33 mL of ammonium acetate added, and 10 is the conversion factor to cmol/kg (derived from an equation used in van Reeuwijk, L.P., 2002).

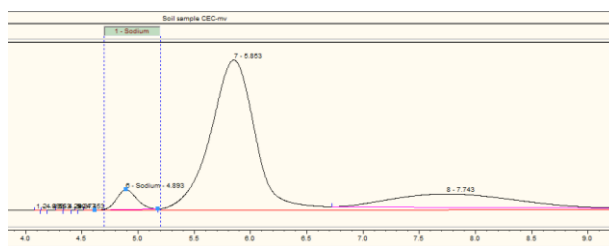


Figure 2.2 IC Peaks showing Sodium and Ammonium do not interfere.

It is important to note that analysis of NO_3^- -N could not be performed because a certain part of the SEAL Analytical instrument needed for analyzing this has not arrived as of yet.

3.0 Results

3.1 Temporal Variability of Measured Soil Properties

Treatment effects were analyzed by grouping all five treatments into two main groups and comparing. Group 1 had treatments T1, T2, and T3, while group 2 had treatments T4, T5. The analysis was mainly done within each field day, and statistical analyses were done using ANOVA and Tukey's test in Minitab. The capital letters for the bar graphs show comparisons among treatments within each field day, and so are independent of each other. Error bars are indicated as standard error. Statistics for temporal variation within each treatment can be seen in **Appendix C**.

3.1.1 Temporal Variability of GMC and VMC

There was no significant difference ($p > 0.05$) among treatments (T1-T2, T1-T3, and T2-T3) for both 0-10 cm and 10-20 cm depths for GMC. When referring to **Figure 3.1**, T1 had the highest GMC for the first three field days, while T3 had the highest GMC on the last day (October 6). GMC did increase on the last two field days (September 30 and October 6) due to large amounts of rainfall from the 2nd to the 9th of September (precipitation graph can be seen in **Appendix D**). The 10-20 cm depth had slightly higher GMC than the 0-10 cm depth for most days, although it was not statistically significant within each treatment. For the 10-20 cm depth (**Figure 3.2**), T1 had the highest GMC on the first, second, and last field days (July 28, August 4, and October 6),

while T3 had the highest on the third field day (September 30). T2 and T3 did not have a significant effect on GMC.

There was no significant difference ($p>0.05$) among treatments (T1-T4, T1-T5, and T4-T5) for both 0-10 cm and 10-20 cm depths for GMC. For the 0-10 cm depth (**Figure 3.3**), T4 had the highest GMC on the first and last field days (July 28 and October 6), while T1 had the highest GMC on the second and third field days (August 4 and September 30). It can again be seen that GMC was higher on the last two field days than the first. Per statistical analyses, no significant difference in GMC was observed between the two depths for each treatment. For the 10-20 cm depth (**Figure 3.4**), T1 had the highest GMC on the first two field days (July 28 and August 4). T5 had the highest GMC on the third day (September 30), while T4 had the highest on the last day (October 6). T4 and T5 did not significantly increase GMC.

Soil ECa, VMC, and temperature data were collected at the 0-11 cm depth for three of the four field days using a portable HD2 probe (IMKO GmbH, Germany). For VMC, the values shown were obtained for three field days with the HD2 probe. No significant difference ($p>0.05$) among treatments (T1-T2, T1-T3, and T2-T3) was observed. When referring to **Figure 3.5**, T1 had the highest VMC on the first and third days (July 28 and September 30), while T3 had the highest value on the last day (October 6). T2 and T3 had no significant impact on VMC. When referring to **Figure 3.6**, no significant difference ($p>0.05$) among treatments was observed. T1 had the highest VMC on the first day (July 28), T4 the highest on the third day (September 30), and T5 on the last day (October 6). T4 and T5 had no significant impact on VMC.

The measured GMC from the soil samples and VMC using the portable TDR probe show very consistent behaviour during sampling days. Slightly higher, but not significant MC was observed for the third field day (September 30). There was a continuous rainfall event from September 2 to 9, which could have potentially increased the MC of the soil. However, weather data from the site showed low daily temperatures with an average value of 10.6°C (maximum =10.9°C, and minimum = 10.4°C) from September 10 to 29. This low temperature, as well as the matured silage corn, must have reduced the evapotranspiration rates, keeping soil moisture levels high even in a relatively non-rainy period.

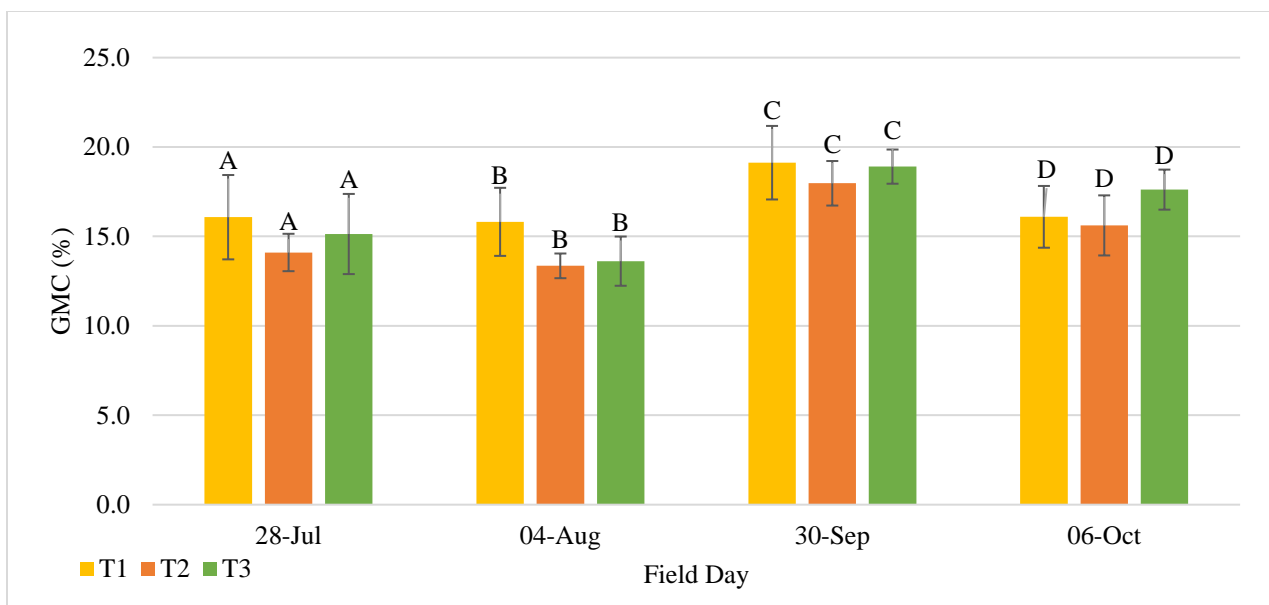


Figure 3.1 Temporal Variation of Gravimetric Moisture Content for T1, T2, and T3 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

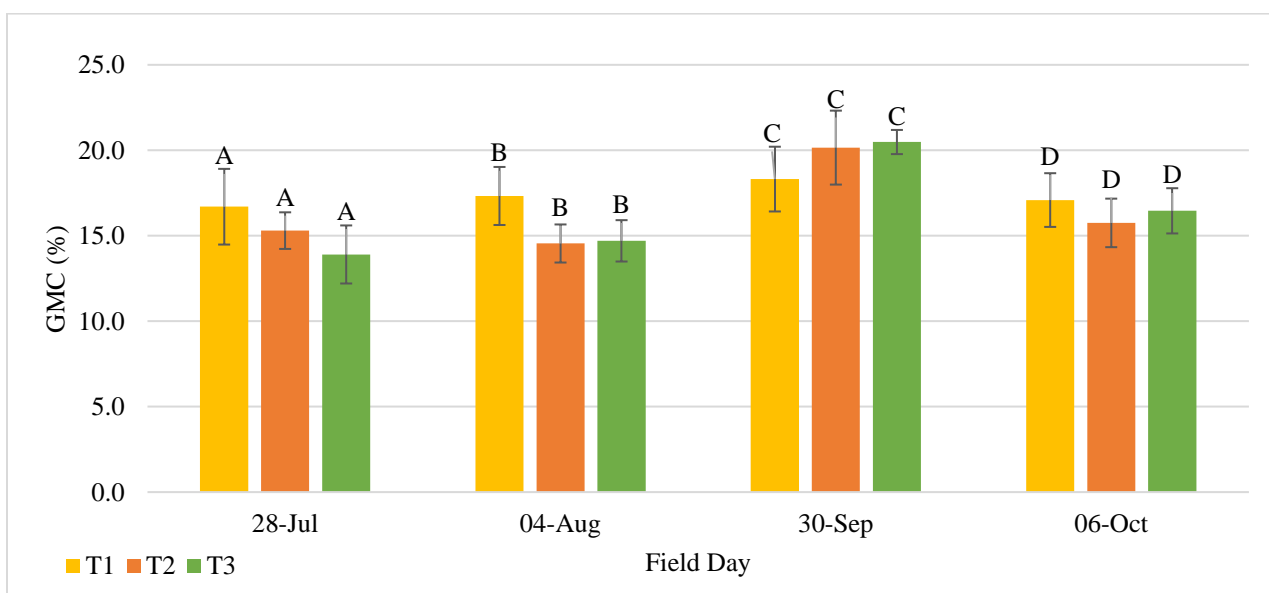


Figure 3.2 Temporal Variation of Gravimetric Moisture Content for T1, T2, and T3 at 10-20 cm Depth. (Note: A, B, C, and D are independent of each other).

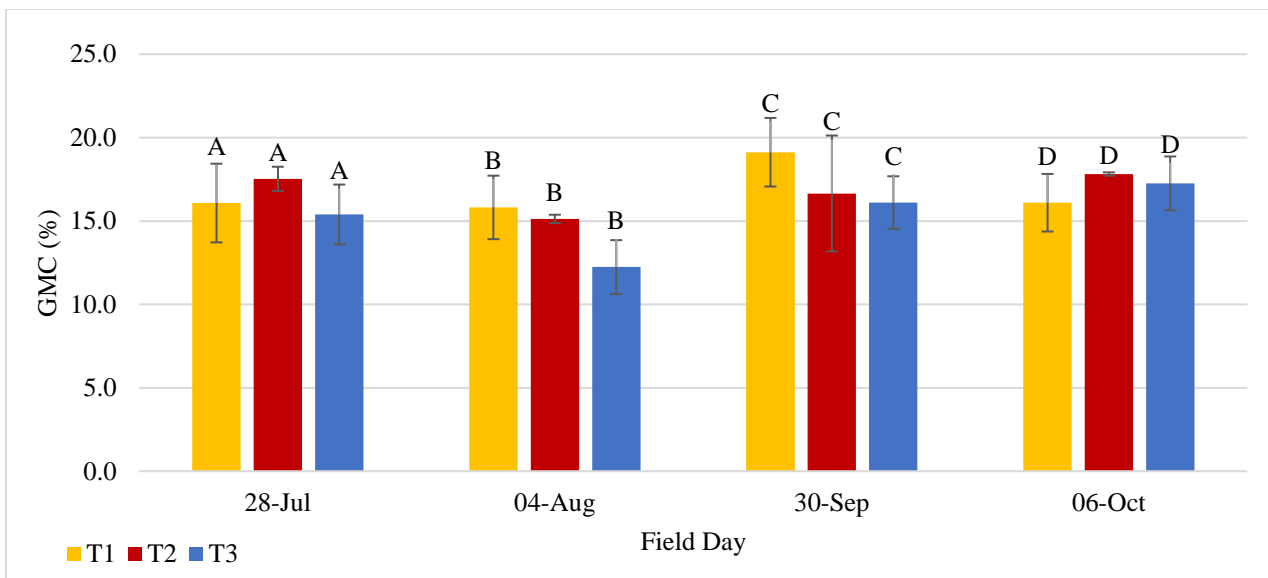


Figure 3.3 Temporal Variation of Gravimetric Moisture Content for T1, T4, and T5 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

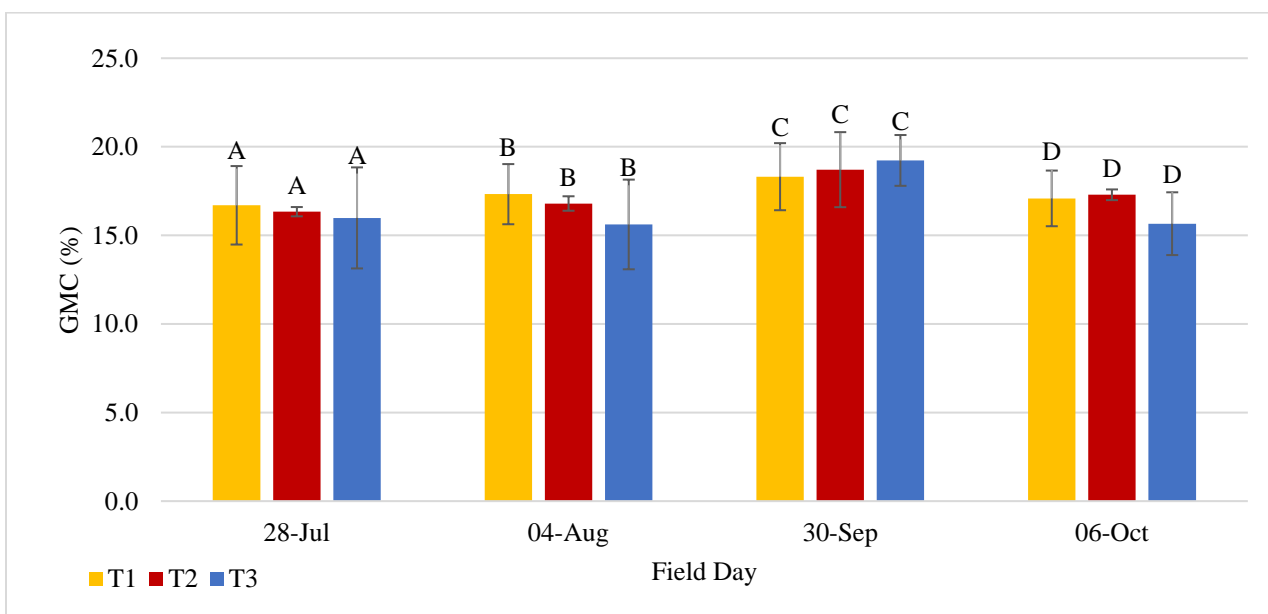


Figure 3.4 Temporal Variation of Gravimetric Moisture Content for T1, T4, and T5 at 10-20 cm Depth. (Note: A, B, C, and D are independent of each other).

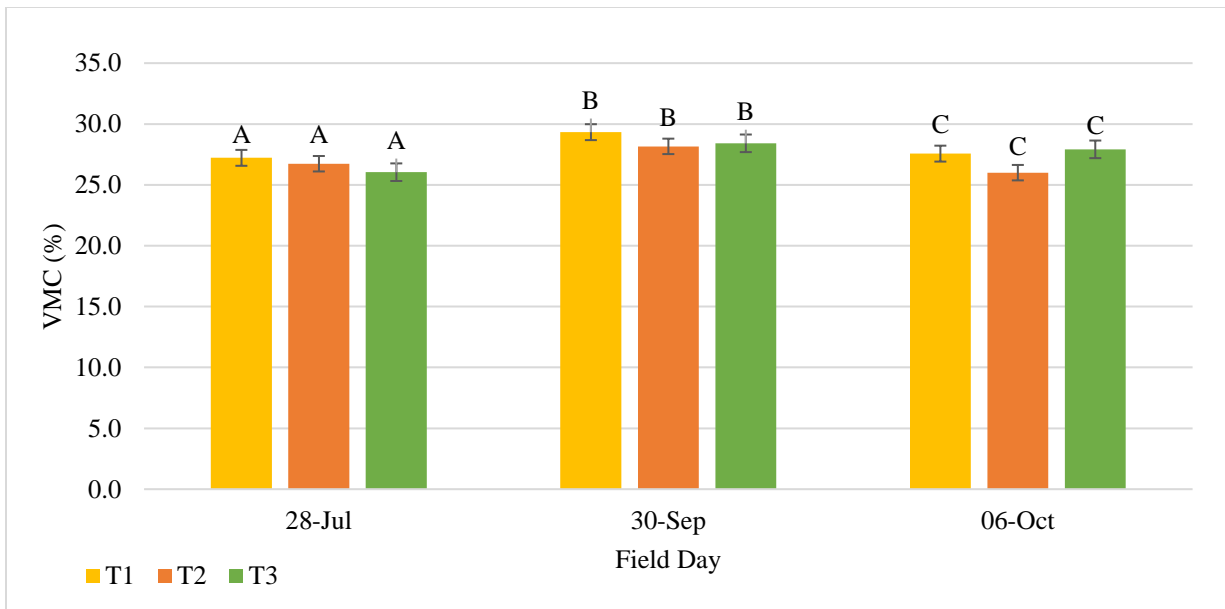


Figure 3.5 Temporal Variation of Volumetric Moisture Content for T1, T2, and T3.

(Note: A, B, and C are independent of each other).

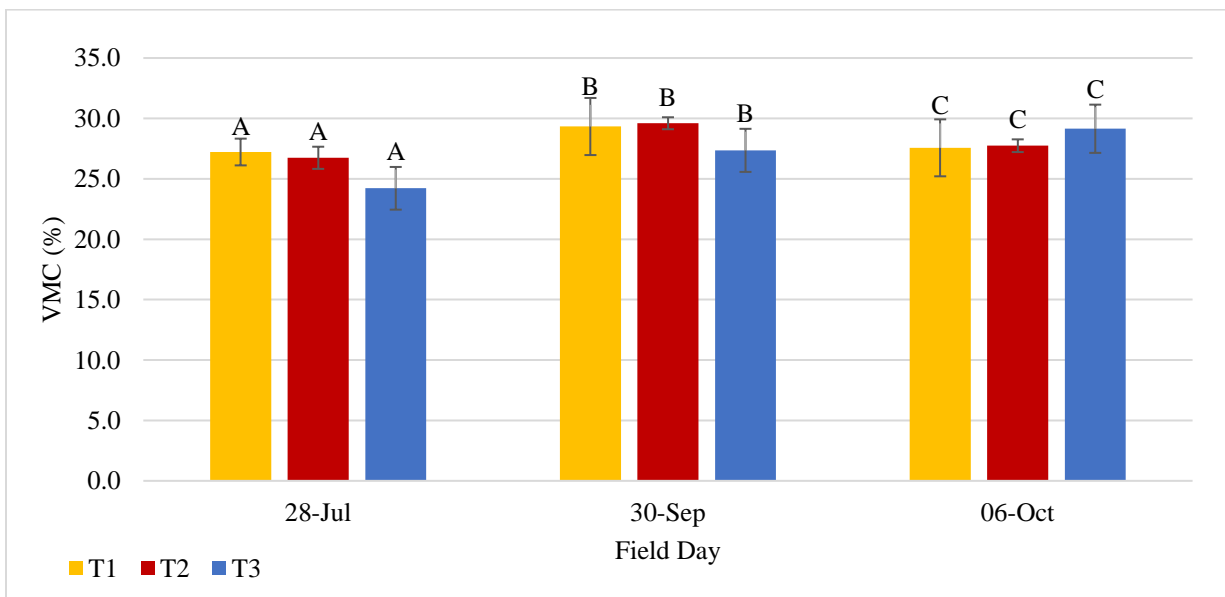


Figure 3.6 Temporal Variation of Volumetric Moisture Content for T1, T4, and T5.

(Note: A, B, and C are independent of each other).

3.1.2 Temporal Variability of pH

When referring to **Figure 3.7**, there was no significant difference ($p>0.05$) for pH among treatments within each field day for the 0-10 cm depth. T3 had the highest pH on the first and last field days (July 28 and October 6). T1 had the highest pH on the second field day (August 4), while T2 did on the third field day (September 30). It is also important to note that the pH first decreased from the first to the second field day, then increased with time irrespective of treatment, reaching more favourable conditions for crops on the last two field days. For the 10-20 cm depth (**Figure 3.8**), no significant difference among treatments within each field day was observed ($p>0.05$). T2 had the highest pH on the first field day (July 28); T3 had the highest pH on the second field day (August 4), and T1 the highest on the third field day (September 30). Both T1 and T3 had the highest pH on the last field day (October 6). T2 and T3 did not significantly affect the pH values.

No significant difference was observed among treatments within each field day for T1, T4 and T5 ($p>0.05$) for both 0-10 cm and 10-20 cm depths. When referring to **Figure 3.9**, T4 had the highest pH on the first day (July 28). T1 had a slightly higher pH than T4 and T5 on the second day (August 4). T5 had the highest pH on the third day (September 30), and T4 on the last day (October 6), closely followed by T1. pH again increased on the last two field days. When referring to **Figure 3.10**, T4 had the highest pH on the first and second days (July 28 and August 4), while T1 had the highest pH among the three treatments for the third and fourth days (September 30 and October 6). pH was again higher for the last two field days than the first two. As shown per statistical analyses, there was no significant difference between values obtained for the two depths

for each day over the entire test period. T4 and T5 did not significantly increase soil pH. pH increased significantly on the last two field days for all treatments at both depths.

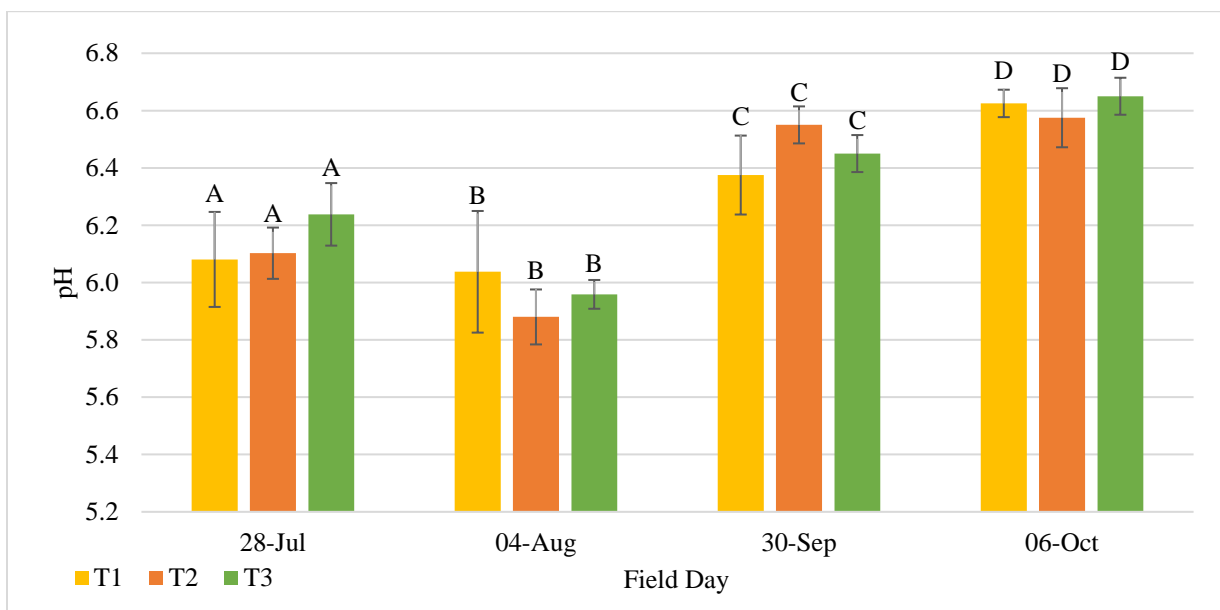


Figure 3.7 Temporal Variation of pH for T1, T2, and T3 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

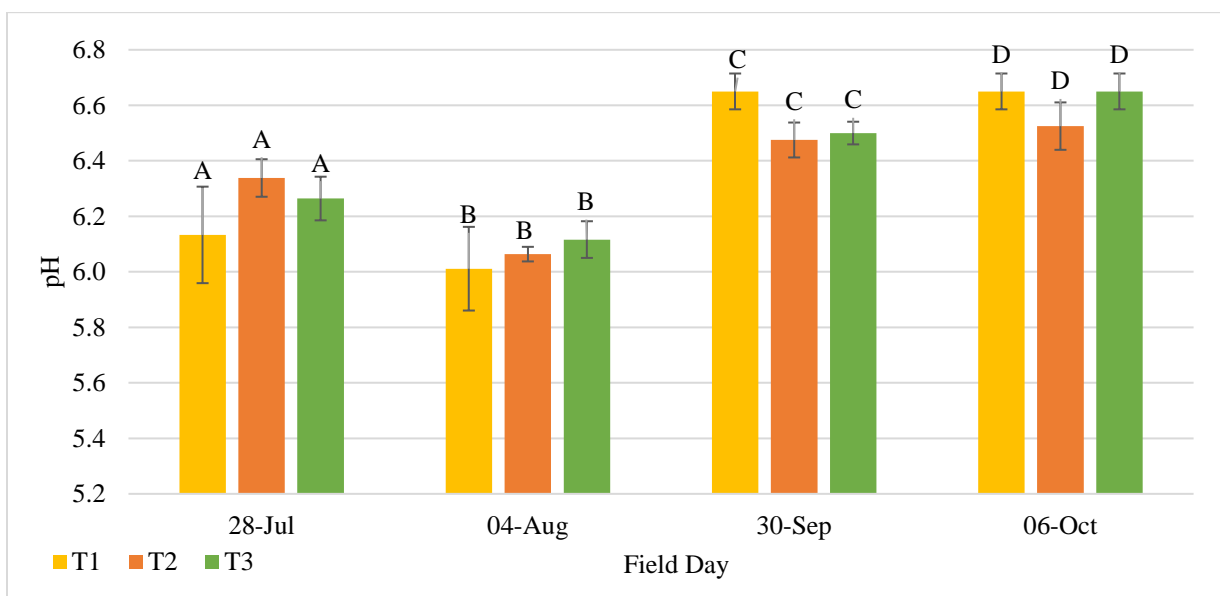


Figure 3.8 Temporal Variation of pH for T1, T2, and T3 at 10-20 cm Depth. (Note: A, B, C, and D are independent of each other).

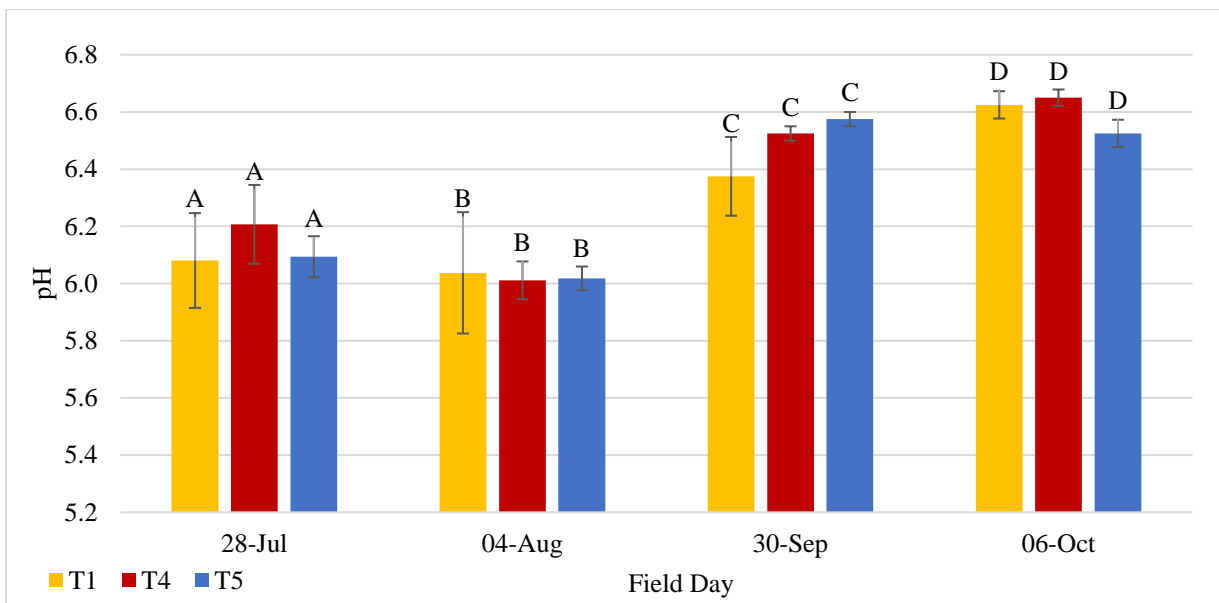


Figure 3.9 Temporal Variation of pH for T1, T4, and T5 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

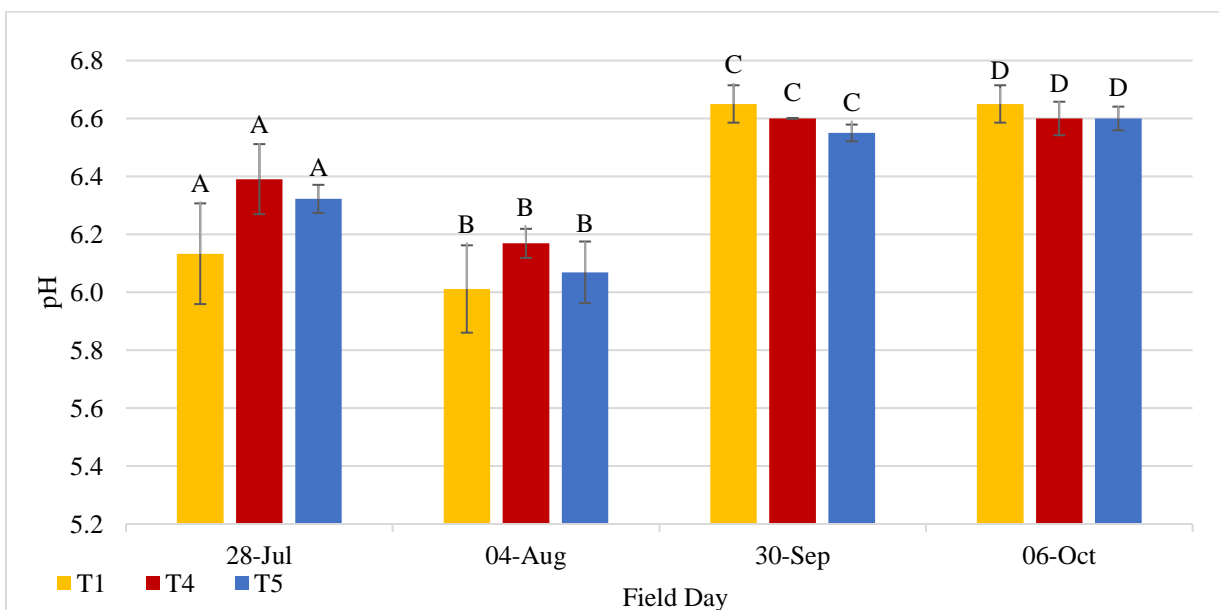


Figure 3.10 Temporal Variation of pH for T1, T4, and T5 at 10-20 cm Depth. (Note: A, B, C, and D are independent of each other).

3.1.3 Temporal Variability of SOC

When referring to **Figure 3.11**, no significant change ($p>0.05$) was observed for SOC among treatments (T1-T2, T1-T3, and T2-T3) within each field day for the 0-10 cm depth. However, T3 did have a comparatively higher amount of SOC on the first field day than T2 and T3, and significantly decreased across the first three field days. T1 and T2 also decreased, but not significantly. On the fourth field day (October 6), the amount of SOC in T2 and T3 again slightly increased from what it was on the third field day (September 30). With respect to the 10-20 cm depth, no significant change among treatments ($p>0.05$) within each field day was observed (**Figure 3.12**). Like the shallow depth (**Figure 3.11**), T3 again had the highest SOC percentage, this time remaining highest for the second (August 4) and the fourth (October 6) field days, while T2 had the highest SOC on the third field day (September 30). There was also no significant difference between values obtained for the two depths for each day for the entire test period. The data shows that SOC variation with time and the tested two depths do not show significant variation. These results follow the normal behavior that SOC does not change within short time periods, as tested in this study. T2 and T3 also did not significantly increase the SOC in the field test either.

When referring to **Figure 3.13**, no significant change ($p>0.05$) was observed among treatments (T1-T4, T1-T5, and T4-T5) for SOC at the 0-10 cm depth within each field day. SOC was highest for T4 on the first field day (July 28). T1 had the highest SOC on the second field day (August 4), and T4 again on the last two field days (September 30 and October 6). T5 remained lowest throughout all field days, indicating that the combination of DM+BC does not increase SOC over a short period of time. For the 10-

20 cm depth (**Figure 3.14**), there was a significant ($p=0.042$) difference between T4 and T5 on the second field day (August 4). No significant difference ($p>0.05$) was found among treatments on the other field days, however. T1 initially had the highest SOC percentage on the first field day (July 28). On the other field days, however, T4 had the highest amount of SOC, while T5 remained the lowest for all days except the third field day (September 30). T4 and T5 did not significantly increase the SOC. SOC content varied from 1.27 to 4.58, with an average value of 2.85 irrespective of treatment or field day in the experimental field. These values are typical of most agricultural soils in NL.

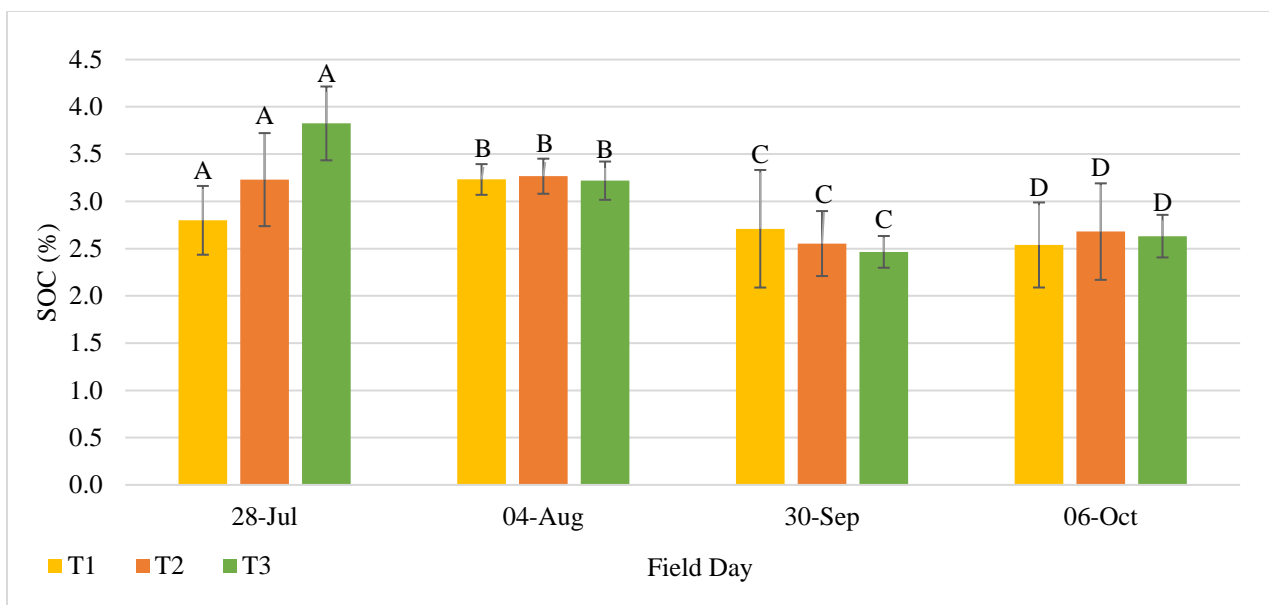


Figure 3.11 Temporal Variation of Soil Organic Carbon for T1, T2, and T3 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

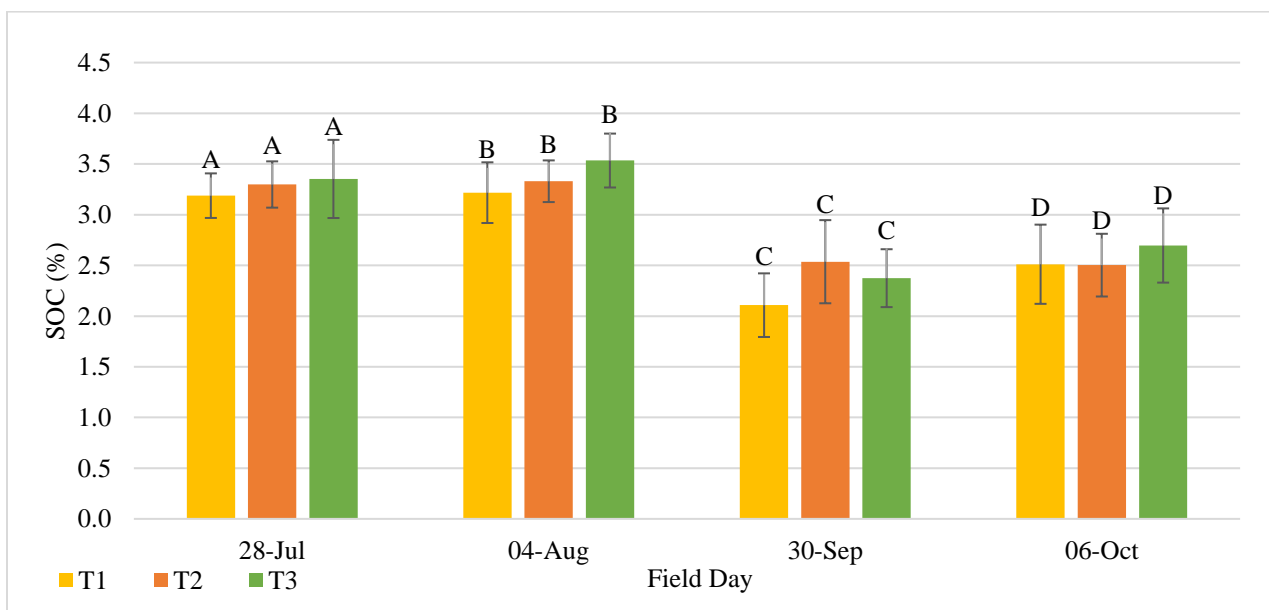


Figure 3.12 Temporal Variation of Soil Organic Carbon for T1, T2, and T3 at 10-20 cm Depth. (Note: A, B, C, and D are independent of each other).

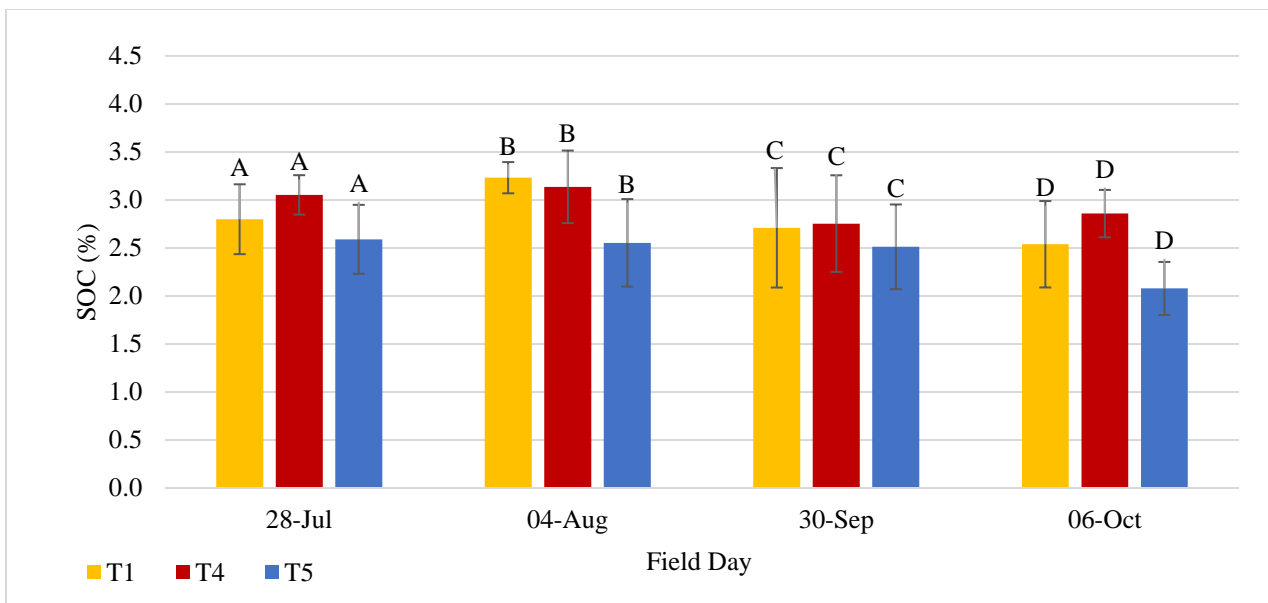


Figure 3.13 Temporal Variation of Soil Organic Carbon for T1, T4, and T5 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

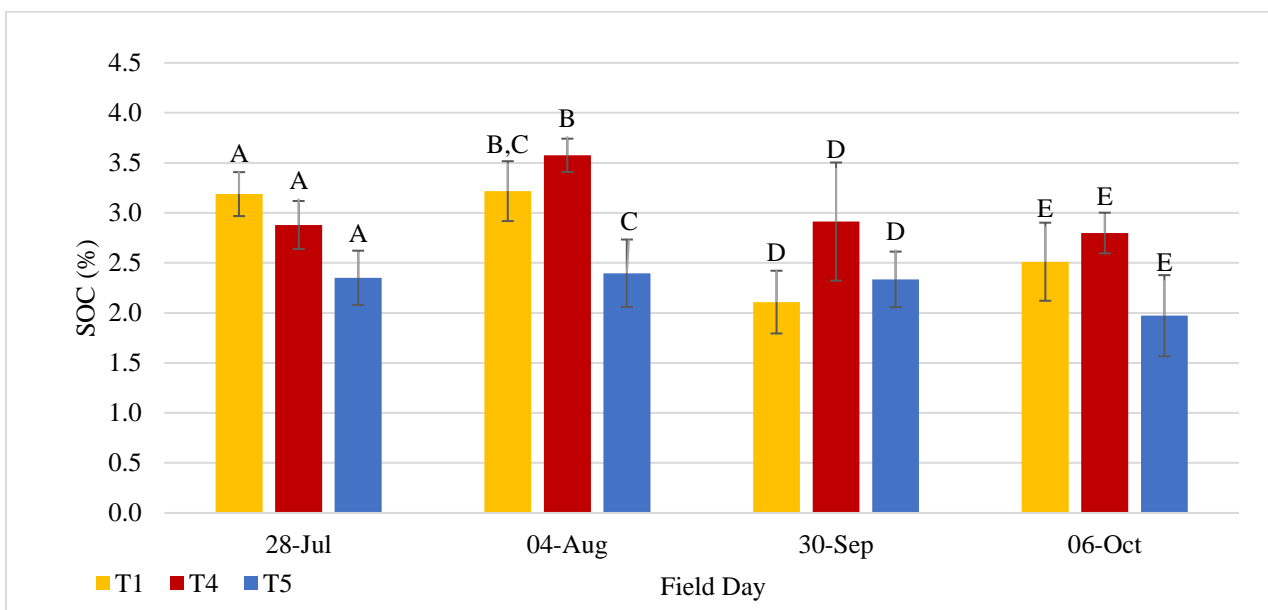


Figure 3.14 Temporal Variation of SOC for T1, T4, and T5 at 10-20 cm Depth. (Note: A, B, D, and E are independent of each other).

3.1.4 Temporal Variability of $\text{NH}_4^+\text{-N}$

Data for $\text{NH}_4^+\text{-N}$ was only obtained for the last three field days. There was no significant difference among treatments (T1-T2, T1-T3, and T2-T3) for both 0-10 cm and 10-20 cm depths ($p>0.05$). When referring to **Figure 3.15**, T3 had the highest $\text{NH}_4^+\text{-N}$ on the second and third days (August 4 and September 30) for the 0-10 cm depth. T3 was not significantly higher on the second day (August 4) due to the large variation of the values obtained from the four plots for this treatment. $\text{NH}_4^+\text{-N}$ concentrations were the same for all three treatments on the last day (October 6). It is also important to note that the concentration in $\text{NH}_4^+\text{-N}$ for the three treatments decreased temporally. This is a general trend normally found with $\text{NH}_4^+\text{-N}$ concentrations if additional fertilizer is not added within the growing season. SOM decomposition can increase $\text{NH}_4^+\text{-N}$ concentrations temporally (Sadej & Przekwas, 2008). However, it is highly unlikely to observe significant increases within the testing period. When referring to **Figure 3.16**, all three treatments showed similar values for all three days, and decreased with time similar to the 0-10 cm depth range, although the values were relatively lower. T2 and T3 did not significantly increase $\text{NH}_4^+\text{-N}$ concentrations.

There was no significant difference ($p>0.05$) among T1, T4, and T5 for both 0-10 cm and 10-20 cm depths for $\text{NH}_4^+\text{-N}$. When referring to **Figure 3.17**, T5 had the highest concentration of $\text{NH}_4^+\text{-N}$ on the second day (August 4) for the 0-10 cm depth, although lower than that of T3 discussed above. T1 had the highest $\text{NH}_4^+\text{-N}$ on the third day (September 30), although only slightly higher than T4 and T5, and T4 had the highest concentration on the last day (October 6). $\text{NH}_4^+\text{-N}$ concentrations decreased temporally. However, T4 seemed to again increase on the last day (October 6). When referring to

Figure 3.18, T5 again had the highest $\text{NH}_4^+\text{-N}$ concentration for 10-20 cm, on the second day (August 4), although lower than in the 0-10 cm depth. T1 had the highest on the third day (September 30), and T1 and T4 both had the highest $\text{NH}_4^+\text{-N}$ concentration on the last day (October 6). There was no significant difference between values obtained for the two depths for each day over the entire test period. T4 and T5 did not significantly increase $\text{NH}_4^+\text{-N}$ concentration.

$\text{NH}_4^+\text{-N}$ generally decreases over time due to volatilization, uptake by crops, immobilization by microbes, or loss as nitrous oxide (N_2O) gas (Sadej & Przekwas, 2008). If plants do not utilize $\text{NH}_4^+\text{-N}$, it undergoes nitrification to $\text{NO}_3^-\text{-N}$ (Sadej & Przekwas, 2008). This is a general trend for $\text{NH}_4^+\text{-N}$ concentrations in soils.

Comparatively higher $\text{NH}_4^+\text{-N}$ values at shallow depths could be due to mineralization of organic matter and the effect of manure and biochar application (Sadej & Przekwas, 2008). In addition, plant uptake of nitrogen generally occurs in deeper soil layers, as the root zone of matured silage corn is within the 30-50 cm depth range at the Pynn's Brook Research Station.

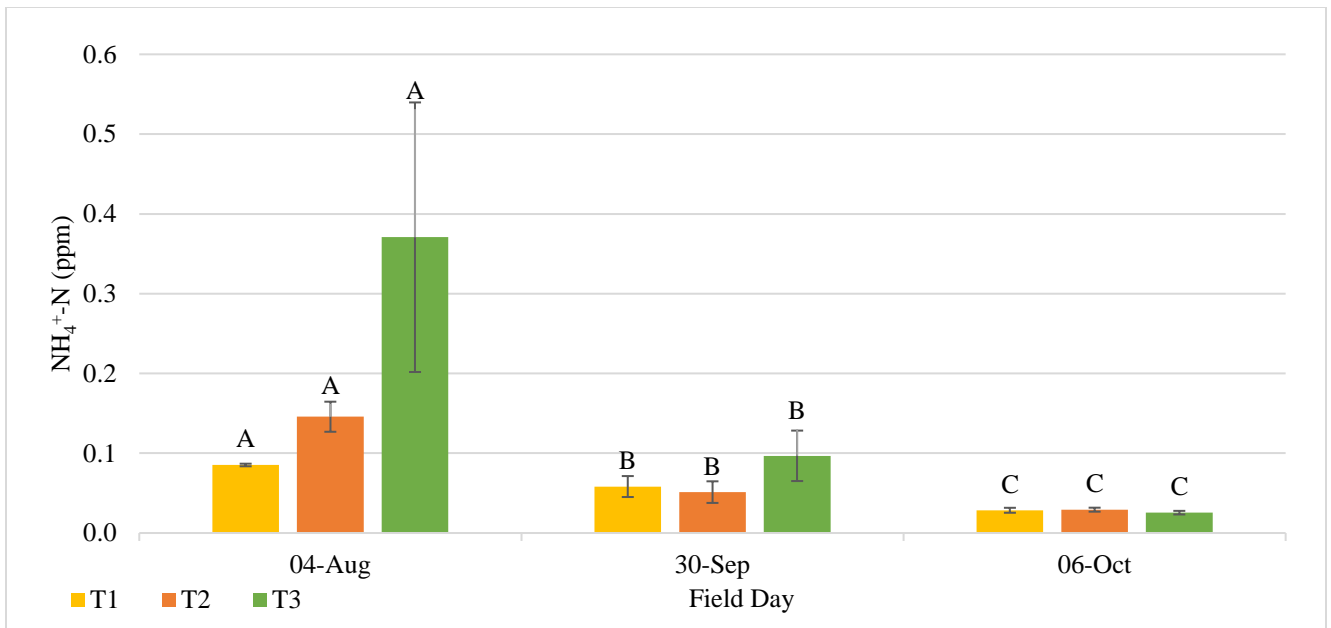


Figure 3.15 Temporal Variation of $\text{NH}_4^+\text{-N}$ for T1, T2, and T3 at 0-10 cm Depth.

(Note: A, B, and C are independent of each other).

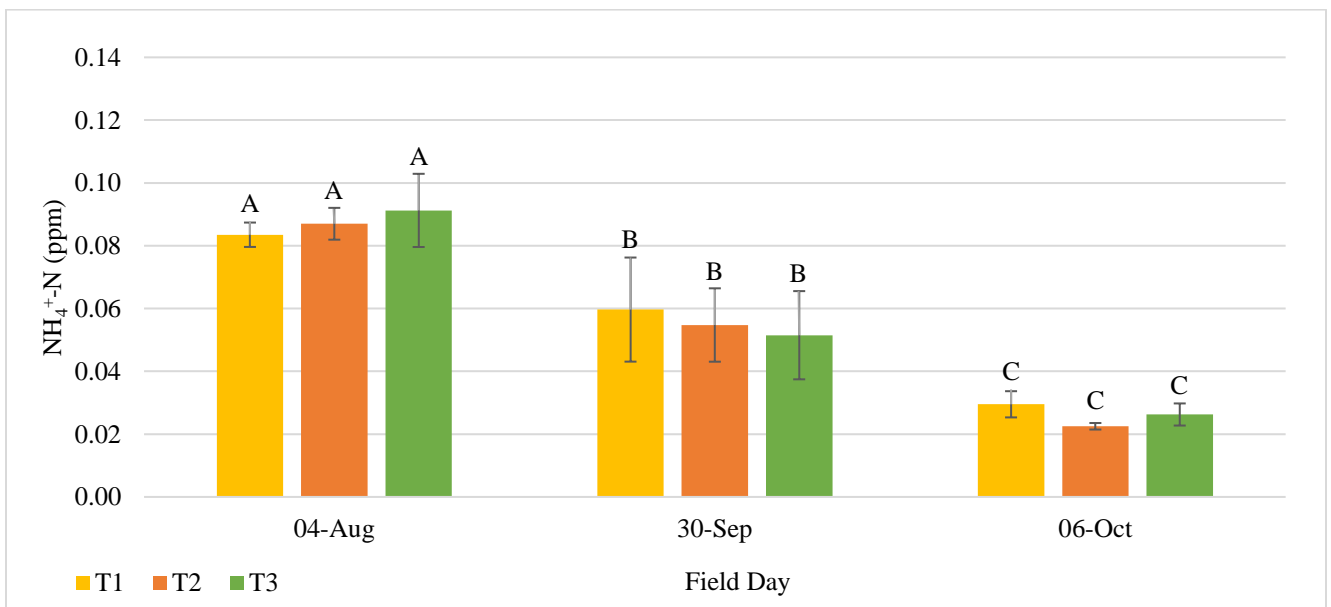


Figure 3.16 Temporal Variation of $\text{NH}_4^+\text{-N}$ for T1, T2, and T3 at 10-20 cm Depth.

(Note: A, B, and C are independent of each other).

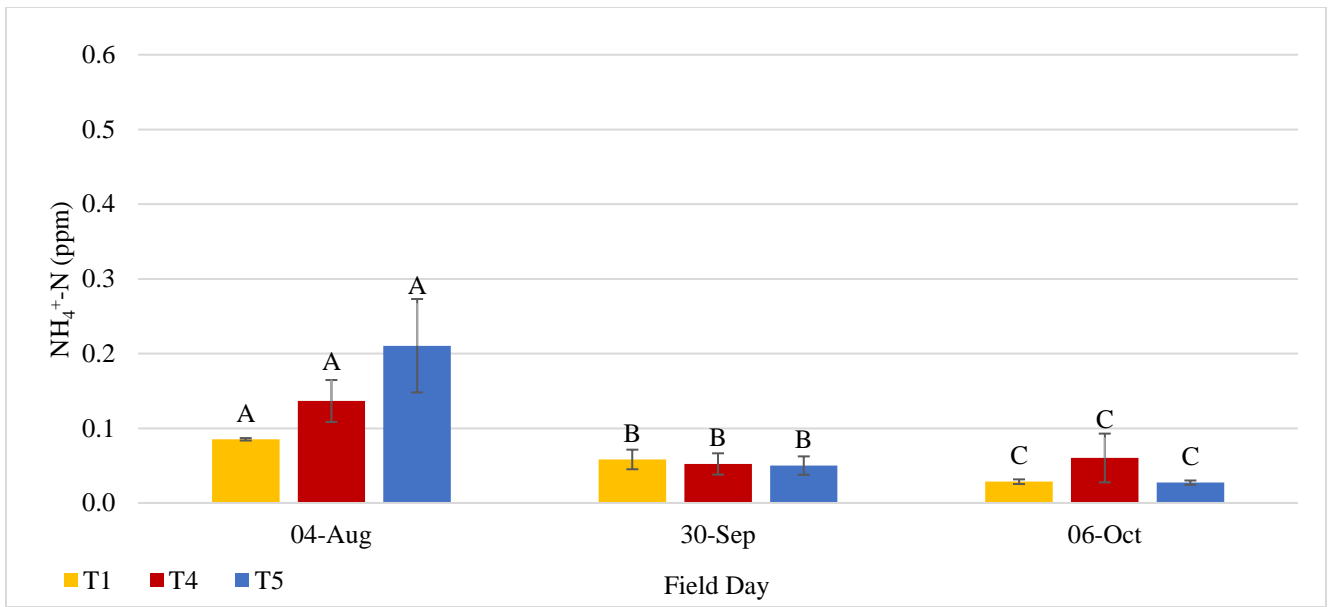


Figure 3.17 Temporal Variation of $\text{NH}_4^+\text{-N}$ for T1, T4, and T5 at 0-10 cm Depth.

(Note: A, B, and C are independent of each other).

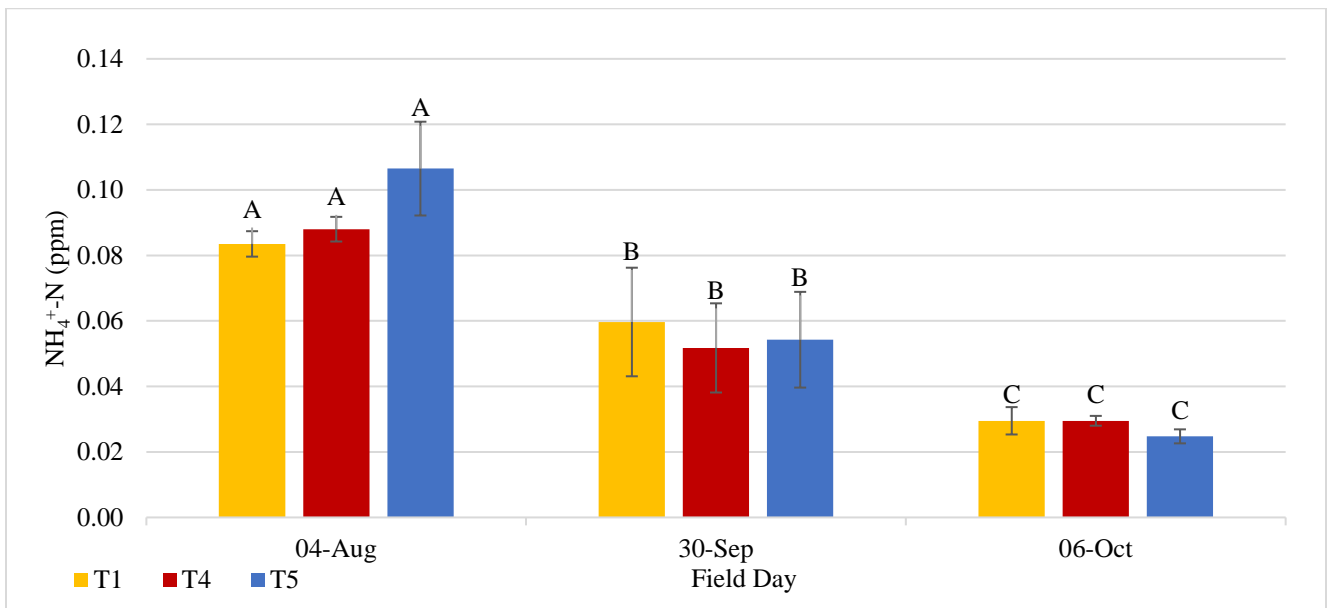


Figure 3.18 Temporal Variation of $\text{NH}_4^+\text{-N}$ for T1, T4, and T5 at 10-20 cm Depth.

(Note: A, B, and C are independent of each other).

3.1.5 Temporal Variability of CEC

Cation exchange capacity data was obtained for 100 samples: all from the first and last field days (July 28 and October 6), and the first depth from the third field day (September 30). The average CEC value based on a soil report at the beginning of 2016 was 12.5 cmol kg^{-1} (Soil report from the Department of Natural Resources, NL Government). The CEC values obtained in this study ranged from approximately 8.2 to 15.7 cmol kg^{-1} for all treatments tested, with an average CEC of 12.6 cmol kg^{-1} . This value falls within the range of CEC values expected for agricultural soils.

There was no significant difference ($p>0.05$) observed among treatments (T1-T2, T1-T3, and T2-T3) for CEC at the 0-10 cm and 10-20 cm depths. For the 0-10 cm depth (**Figure 3.19**), T1 had the highest CEC on the first day (July 28), and T3 on the third and last days (September 30 and October 6). The values for CEC remained fairly stable across field days. T3 also slightly increased across the field days, indicating that biochar does potentially have an effect on increasing CEC over time (Githinji et al., 2014; Chintala et al., 2013). However, to test if this is true, the study should be conducted over a longer time frame. For the 10-20 cm depth (**Figure 3.20**), T1 had the highest CEC on the first day (July 28), while T3 did on the last day (October 6). Of the three treatments in this depth, T2 and T3 increased in CEC from the first to last day. T2 and T3 had no significant effect on increasing CEC during this 3.5 month time period.

No significant ($p>0.05$) treatment effect was observed for the 0-10 cm and 10-20 cm depths among treatments (T1-T4, T1-T5, and T4-T5). For the 0-10 cm depth (**Figure 3.21**), T1 had the highest CEC on the first day (July 28), while T5 had the highest on the third and fourth days (September 30 and October 6). T5 also slightly increased CEC

temporally. For the 10-20 cm depth (**Figure 3.22**), T1 had the highest CEC on the first day (July 28), while T4 was highest on the last day (October 6). T4 and T5 had no significant effect on CEC. There was also no statistically significant difference between the two depths for all treatments.

In general, these results show the increasing trend of CEC values over time for the experimental plots treated with biochar (T3 and T5), as expected (Githinji et al., 2014; Chintala et al., 2013). However, it is important to note that the testing period was too short for this study to observe a significant increase in CEC for the biochar treatments, as CEC increases as biochar ages (Lehman et al., 2009). On the other hand, a general trend of slightly increasing CEC values can be observed for all treatments (except T2, which decreased on October 6) for the 0-10 cm depth. This could be attributable to the gradual increase in soil pH (**Figures 3.7** and **3.9**), as an increase in soil pH has been shown to increase CEC (Brady & Weil, 2008).

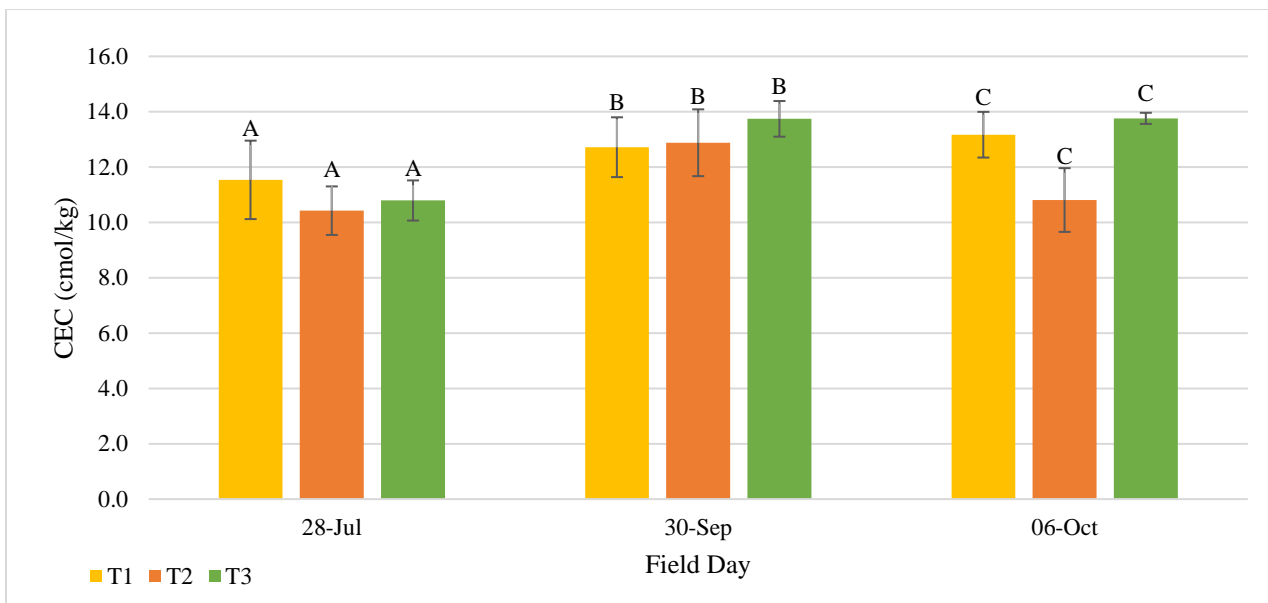


Figure 3.19 Temporal Variation of Cation Exchange Capacity for T1, T2, and T3 at 0-10 cm Depth. (Note: A, B, and C are independent of each other).

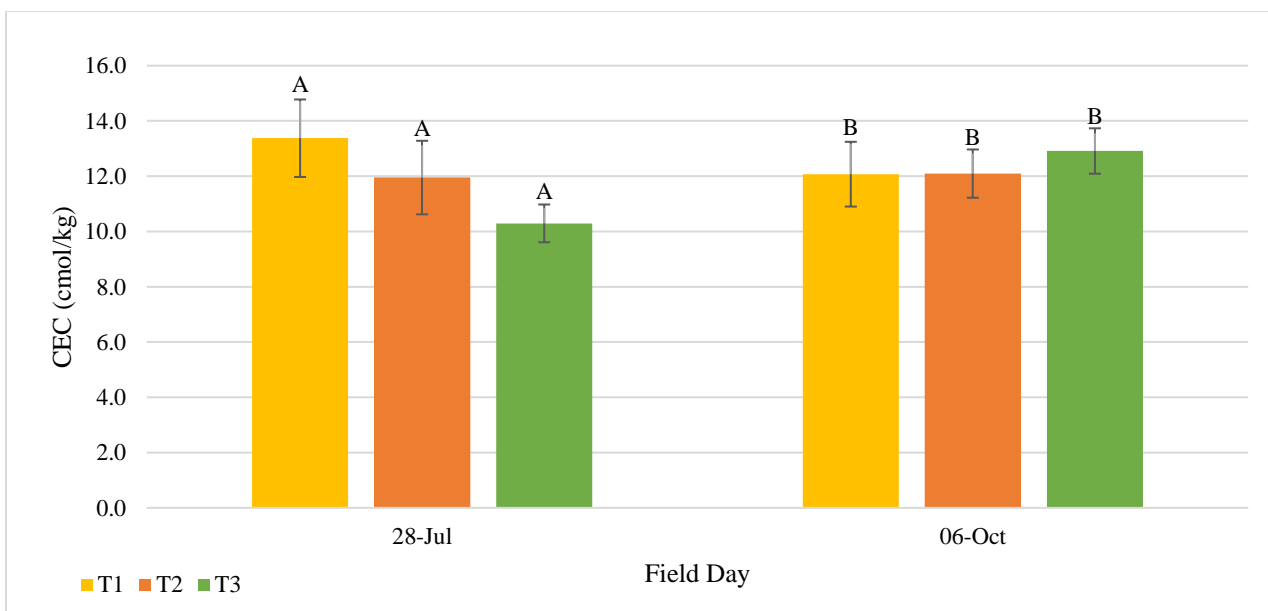


Figure 3.20 Temporal Variation of Cation Exchange Capacity for T1, T2, and T3 at 10-20 cm Depth. (Note: A and B are independent of each other).

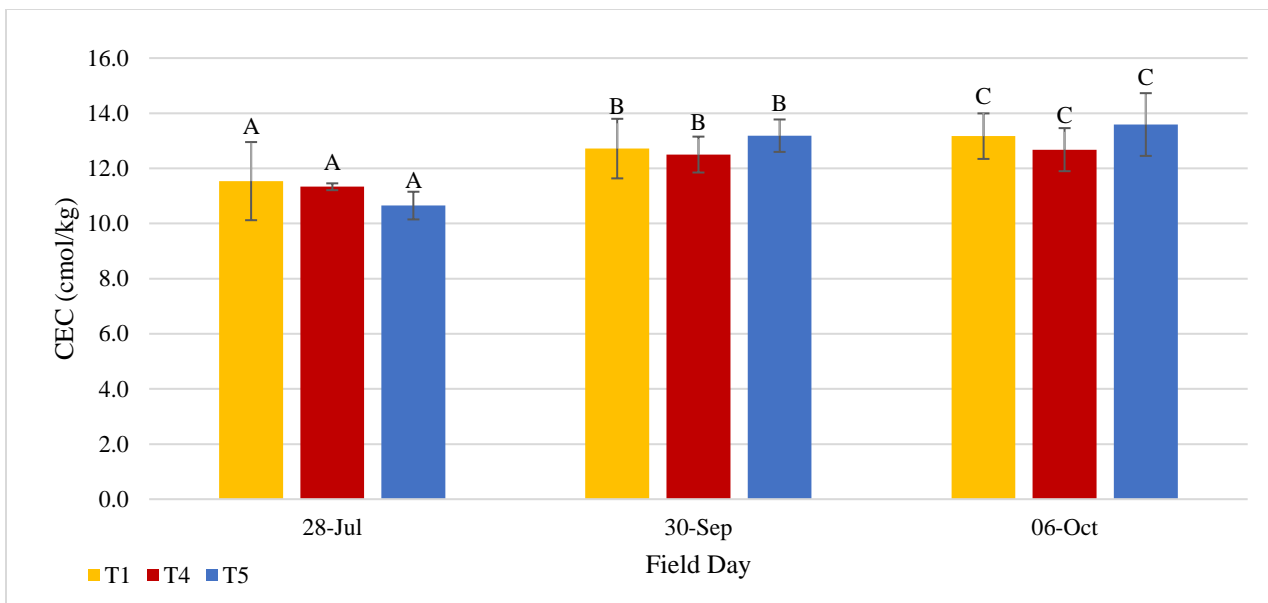


Figure 3.21 Temporal Variation of Cation Exchange Capacity for T1, T4, and T5 at 0-10 cm Depth. (Note: A, B, and C are independent of each other).

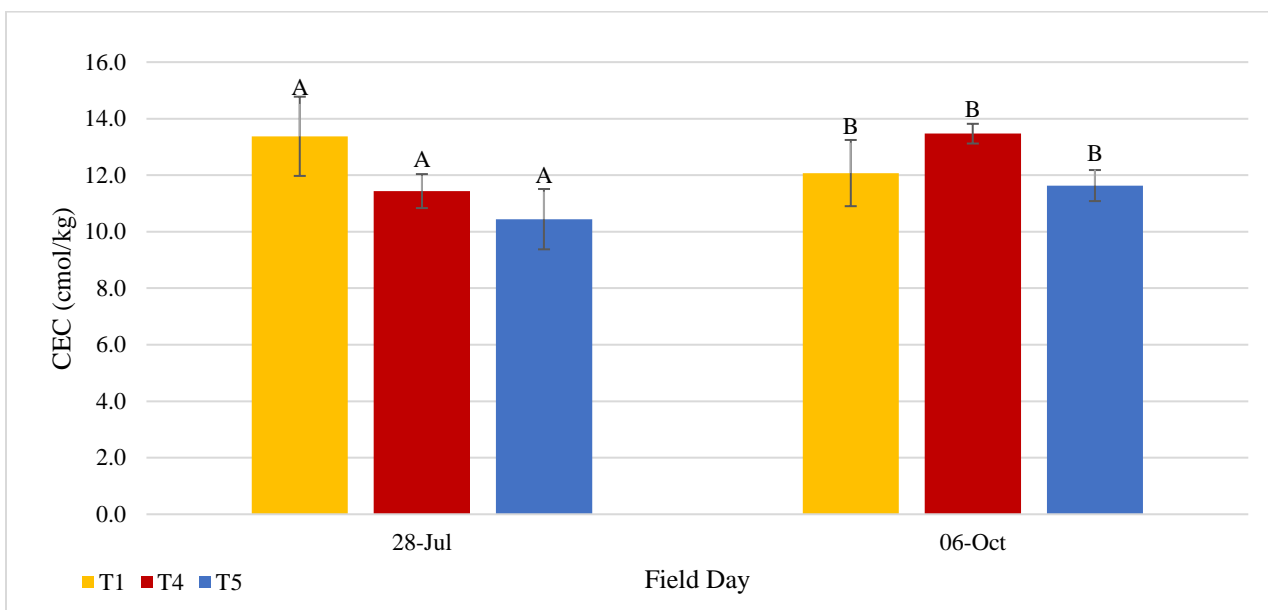


Figure 3.22 Temporal Variation of Cation Exchange Capacity for T1, T4, and T5 at 10-20 cm Depth. (Note: A and B are independent of each other).

3.1.6 Temporal Variability of EC_w and EC_a

There was no significant difference ($p>0.05$) among treatments (T1-T2, T1-T3, and T2-T3) for both 0-10 cm and 10-20 cm depths for EC_w. When referring to **Figure 3.23**, T2 had a comparatively higher EC_w on the first and second days (July 28 and August 4), which could be due to an increase in ionic concentration with application of inorganic fertilizer. A significant reduction in EC_w for T2 was observed in the third and fourth days (September 30 and October 6), potentially due to absorption of nutrients by plants, or due to gaseous or leaching losses. EC_w values obtained for the 10-20 cm depth were lower, although there was no significant difference between the values across the two depths. For the 10-20 cm depth (**Figure 3.24**), T3 had the highest EC_w on the first and third day (July 28 and September 30), while T2 had the highest on the second and last field days (August 4 and October 6). T2 (and T1, very slightly) increased on the last field day, more than likely due to slow release properties of the fertilizer, or leaching of the fertilizer into the deeper layers of the soil. Overall, T2 and T3 did not significantly increase EC_w.

There was no significant difference ($p>0.05$) among treatments (T1-T4, T1-T5, and T4-T5) for the 0-10 cm and 10-20 cm depths for EC_w. For the first depth (**Figure 3.25**), T5 had the highest values on the first day (July 28). T4 had the highest value on the second day (August 4), and T1 on the third day (September 30). On the last day (October 6), T1 and T5 had the highest values. T4 had the highest EC_w values on the first two field days (July 28 and August 4) for the second depth (**Figure 3.26**), while T1 had the highest on the third day (September 30). On the last field day (October 6), T5 had the highest value. All three treatments increased (T1 only slightly) on the last field day, again more

than likely due to slow release properties or leaching of ions from the upper layer. T4 and T5 did not significantly increase EC_w.

In general, EC_w decreased over time for all treatments, which can be attributed to absorption of nutrients by plants, immobilization, or losses due to leaching and volatilization. Increases in EC_w of soils can be expected when ion concentrations are high. In dairy manure and inorganic nitrogen plots, it would be expected that ammonium concentration would be high initially. As NH₄⁺-N is water-soluble, EC_w will primarily measure these and other water-soluble ions. This can be seen from the initial high EC_w values for T2 and T4 especially (Manitoba Agriculture, 2015). Adding biochar to these plots may increase the concentration of these water-soluble ions more in the long run, decreasing losses of these ions, and causing EC_w to decrease at a slower rate (Chintala et al., 2013; Molnár et al., 2016).

For the EC_a values obtained using the HD2 probe (IMKO GmbH, Germany), there was no significant difference among treatments ($p>0.05$) for T1, T2, and T3. When referring to **Figure 3.27**, T2 had the highest value on the first field day (July 28). On the third field day (September 30), T2 and T3 had the highest value, while on the last field day (October 6), T1 was slightly higher than T2 and T3. EC_a values decreased across the three field days shown, as is expected for most soils. T2 and T3 did not significantly increase EC_a.

There was no significant difference among treatments ($p>0.05$) for T1, T4, and T5 (**Figure 3.28**). On the first field day (July 28), T4 had the highest EC_a value, while T5 had the highest on the third and fourth field days (September 30 and October 6). T4 and T5 did not significantly increase EC_a. Like the variation of EC_w, EC_a values also

decreased with time irrespective of treatment. This behavior clearly shows the reduction in ionic concentration of soil across the growing season due to nutrient uptake by plants, or losses due to leaching and volatilization (Chintala et al., 2013).

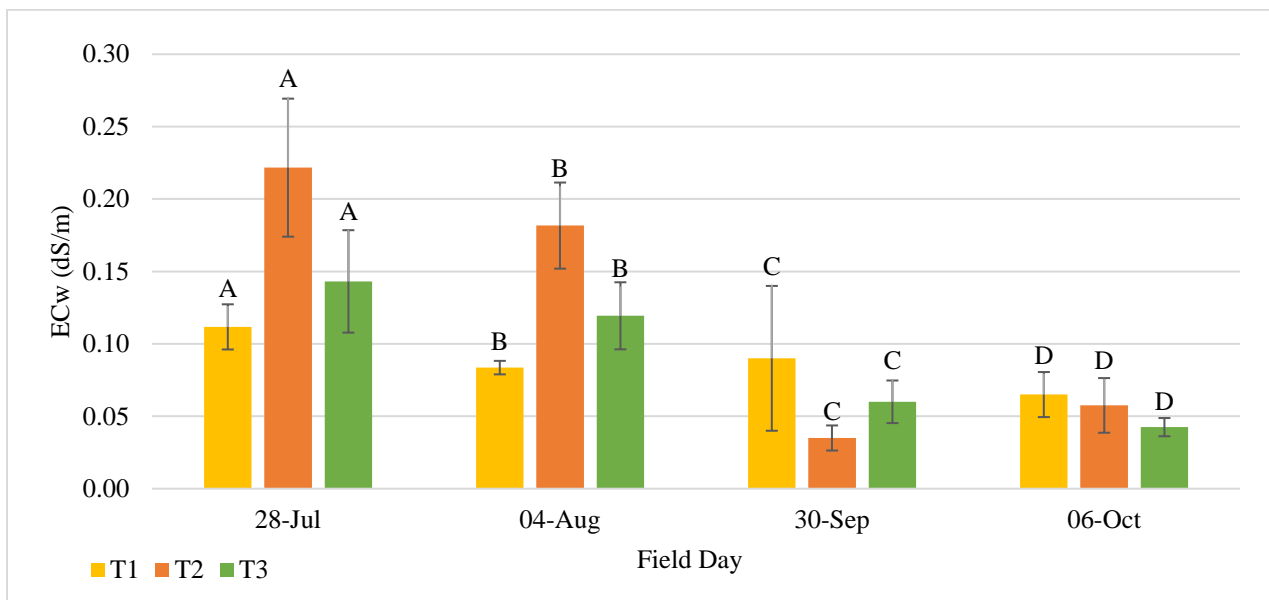


Figure 3.23 Temporal Variation of ECw for T1, T2, and T3 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

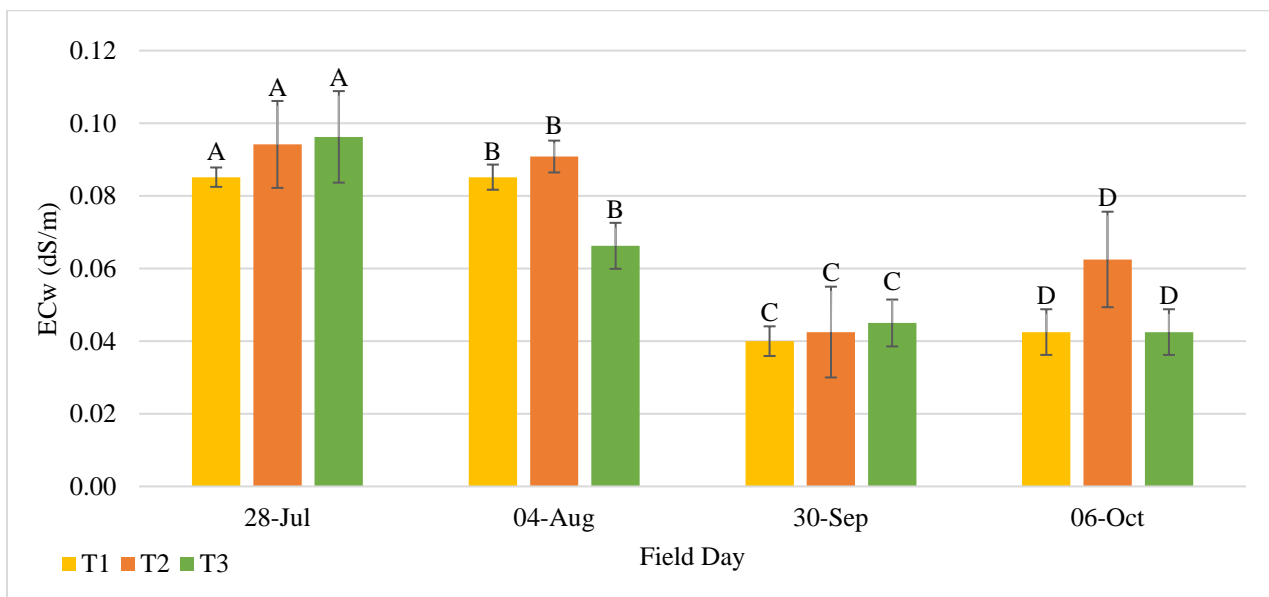


Figure 3.24 Temporal Variation of ECw for T1, T2, and T3 at 10-20 cm Depth.

(Note: A, B, C, and D are independent of each other).

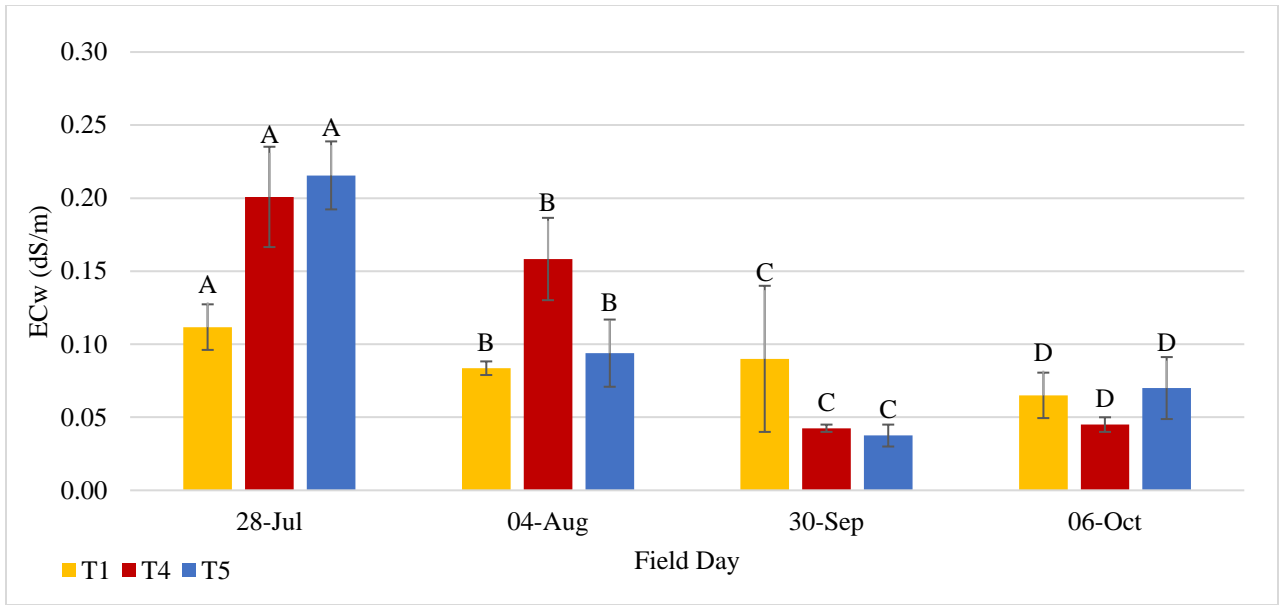


Figure 3.25 Temporal Variation of ECw for T1, T4, and T5 at 0-10 cm Depth. (Note: A, B, C, and D are independent of each other).

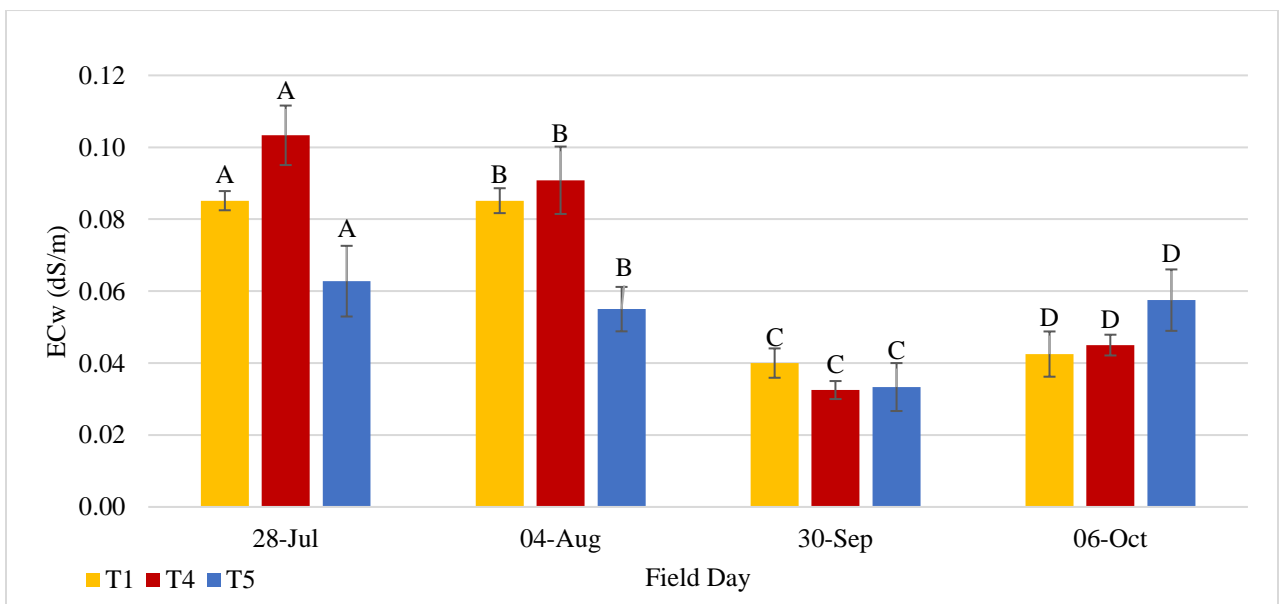


Figure 3.26 Temporal Variation of ECw for T1, T4, and T5 at 10-20 cm Depth.

(Note: A, B, C, and D are independent of each other).

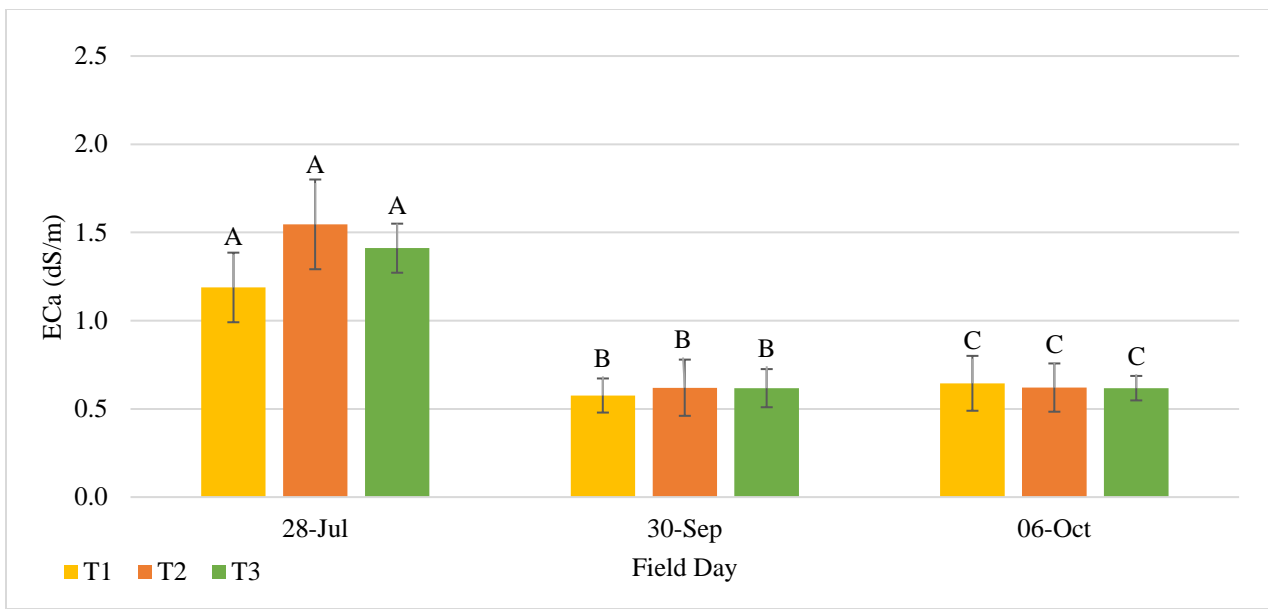


Figure 3.27 Temporal Variation of ECa for T1, T2, and T3. (Note: A, B, and C are independent of each other).

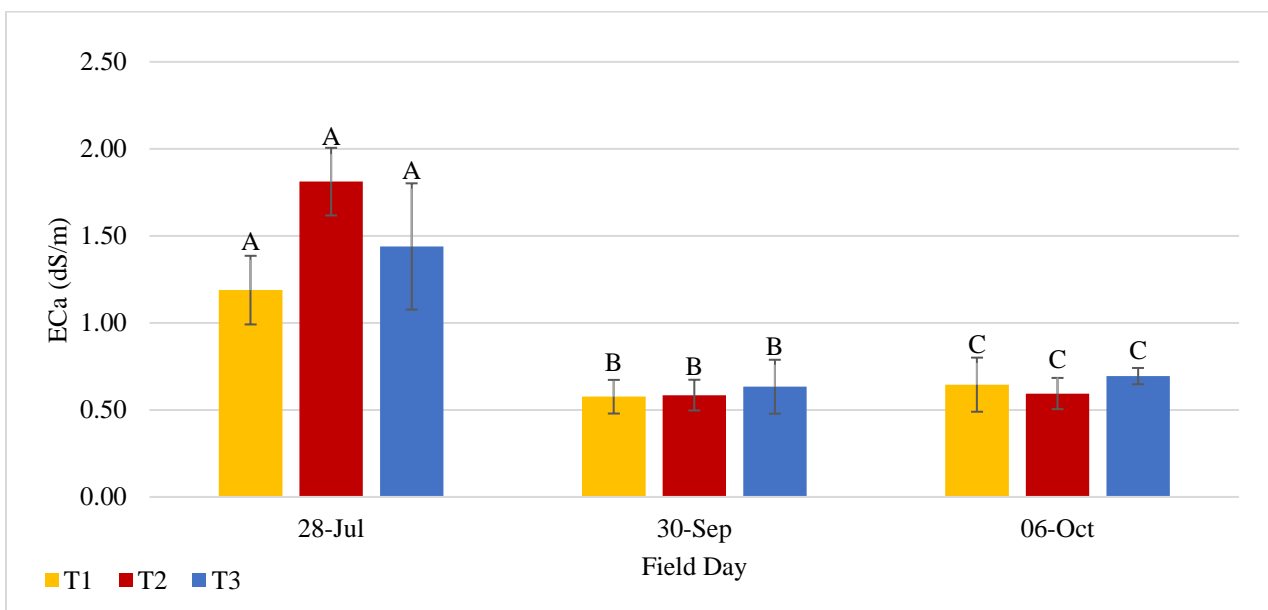


Figure 3.28 Temporal Variation of ECa for T1, T4, and T5. (Note: A, B, and C are independent of each other).

3.2 Correlation Analysis of EC with Measured Soil Properties

As stated in the final objective, analysis was done to see if a relationship could be obtained with EC and the measured soil properties. Those analyzed were SOC, $\text{NH}_4^+\text{-N}$, and CEC. Relationships were also tested statistically through regression analysis to ensure if it was significant or not.

3.2.1 Correlation Analysis of EC with SOC

Soil organic carbon was compared to ECa and ECw irrespective of treatment and field day. When referring to **Figures 3.29, 3.30, and 3.31**, it can be seen that significant correlations were obtained for SOC with ECa (Martinez et al., 2009) and ECw at both depths (ECa, ECw 0-10 cm, & ECw 10-20 cm, $p=0.000$). It would be assumed that SOC is more closely correlated with ECa than ECw, as ECa measures the bulk properties of the soil, such as SOC. When referring to the R^2 values, SOC has a higher value in its correlation with ECa than ECw, which may indicate that ECa is more closely correlated with SOC.

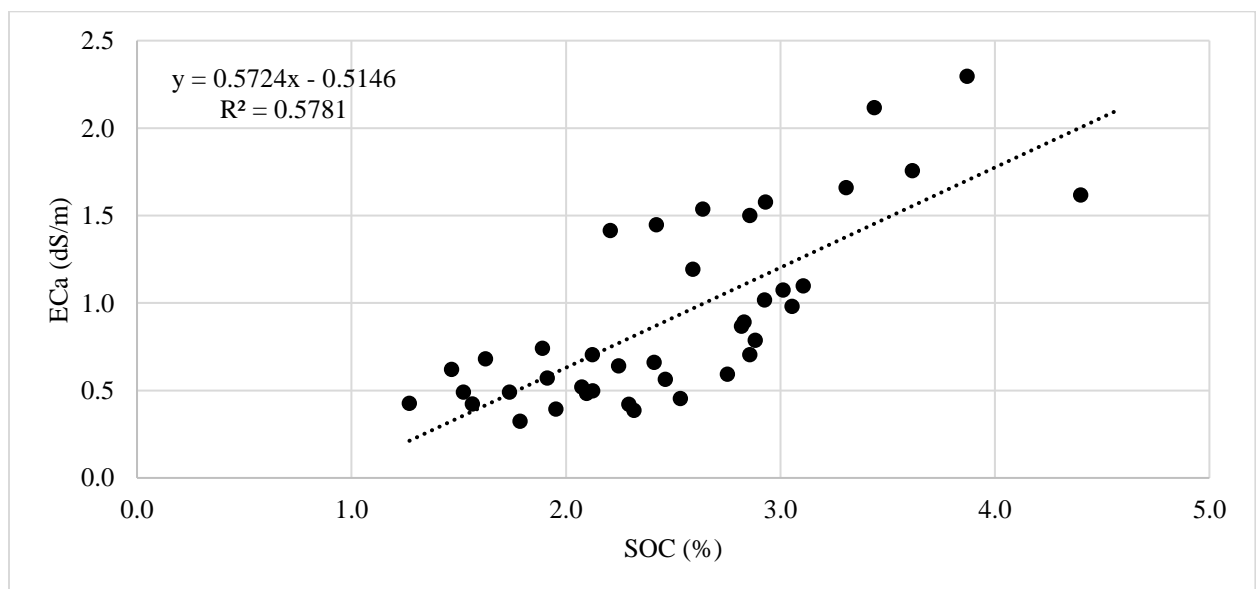


Figure 3.29 ECa vs SOC ($p=0.000$).

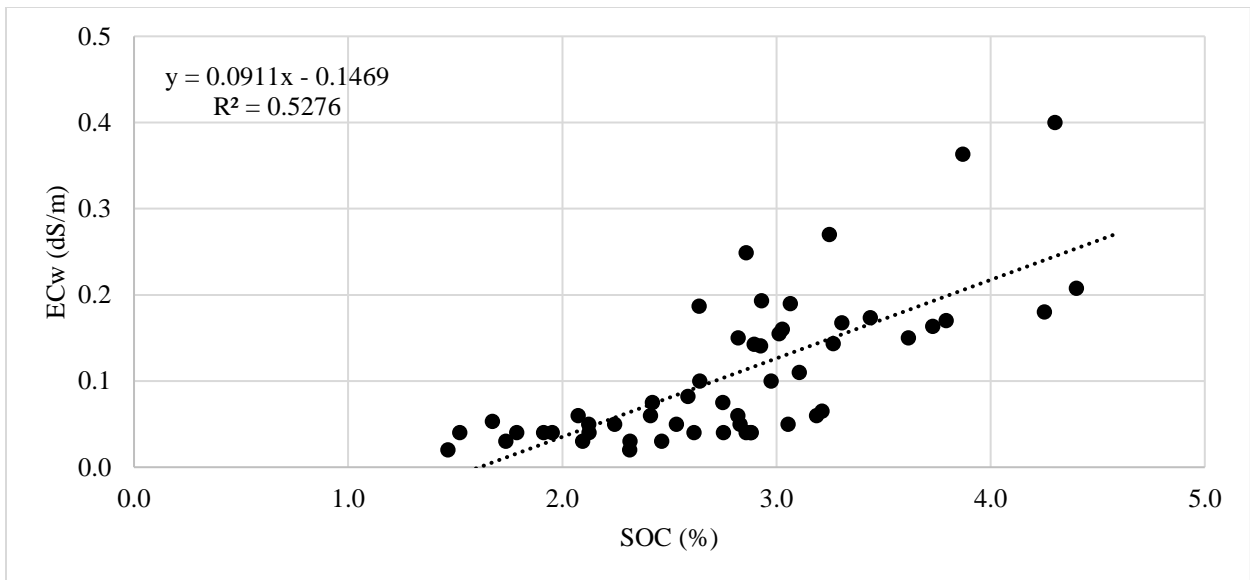


Figure 3.30 ECw vs SOC at 0-10 cm Depth (p=0.000).

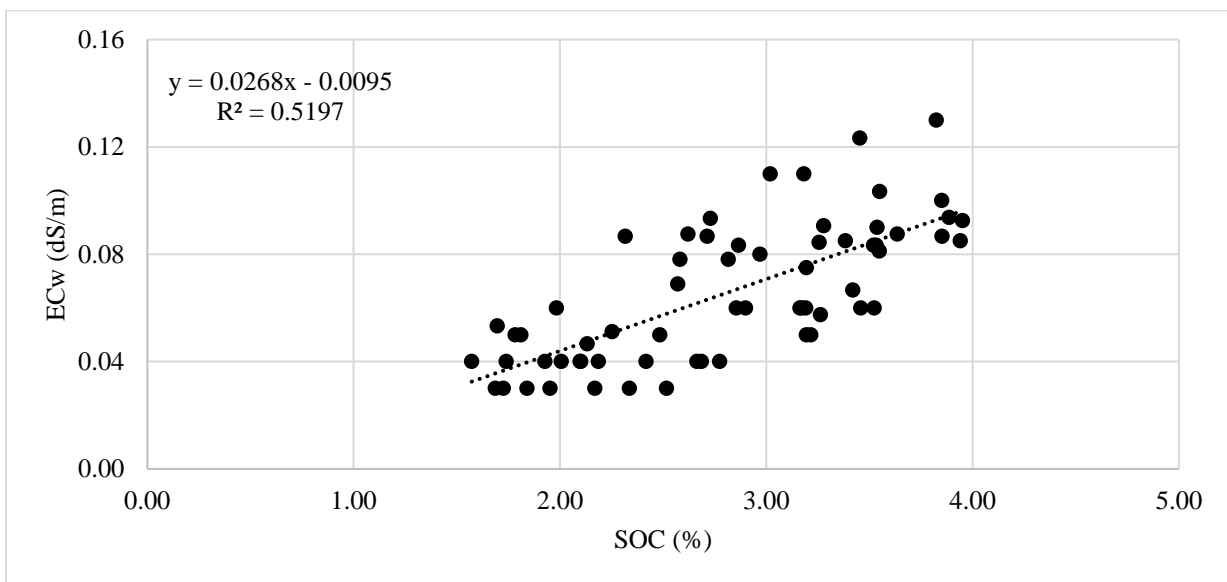


Figure 3.31 ECw vs SOC at 10-20 cm Depth (p=0.000).

3.2.2 Correlation Analysis of EC with $\text{NH}_4^+\text{-N}$

$\text{NH}_4^+\text{-N}$ was compared with ECw and ECa across all field days, irrespective of treatment. When referring to **Figure 3.32, 3.33** and **3.34**, it can be seen that $\text{NH}_4^+\text{-N}$ had significant correlation with ECa (Chaudhari & Ahire, 2013) as well as ECw for both depths (ECa, ECw 0-10 cm, & ECw 10-20 cm, $p=0.000$). It can be seen that ECw had a more significant correlation with $\text{NH}_4^+\text{-N}$ than that of ECa. The reason for this is that ECw mostly measures the amount of ions present in solution, while ECa measures the bulk properties of the soil. Due to the significant correlation, a relationship can be developed, using only EC to obtain these properties in the long run.

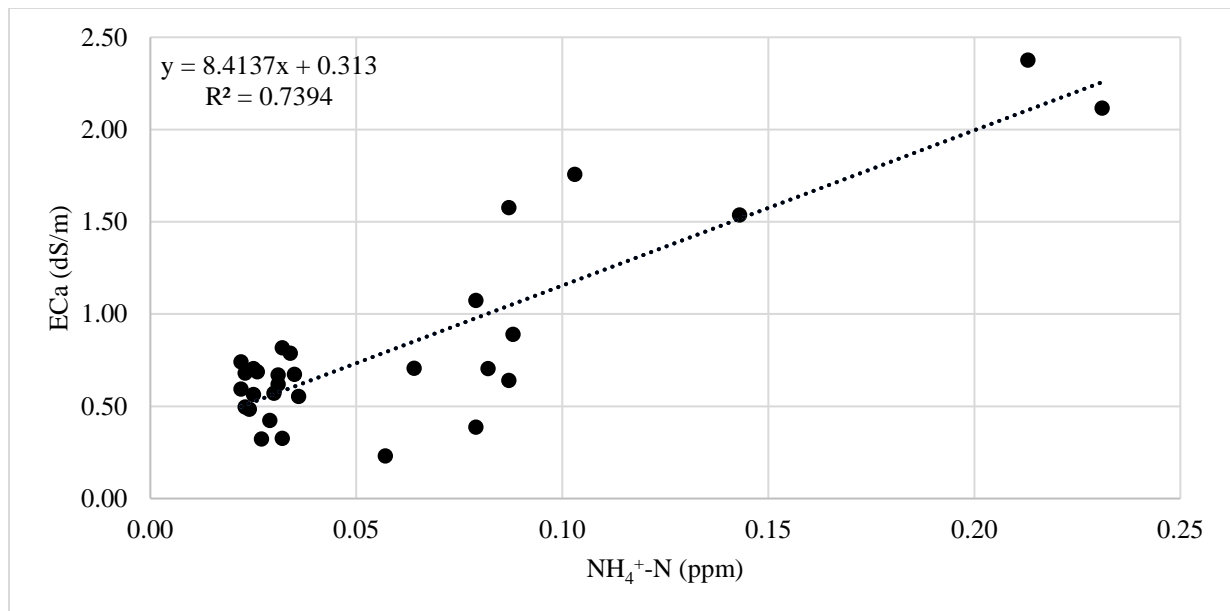


Figure 3.32 ECa vs $\text{NH}_4^+\text{-N}$ ($p=0.000$).

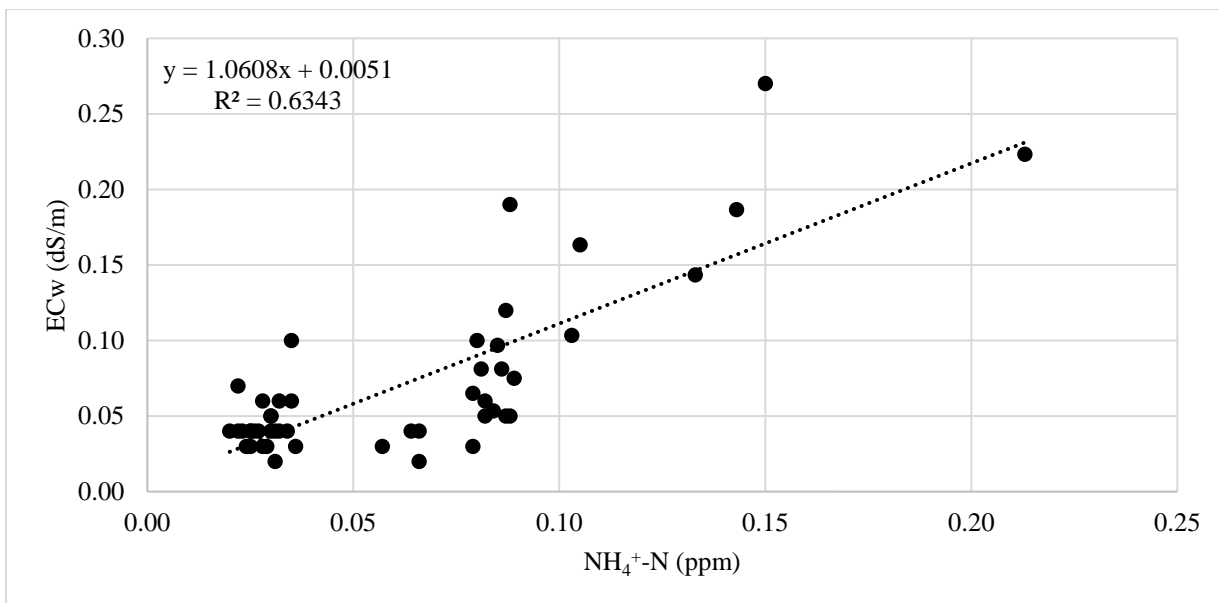


Figure 3.33 ECw vs NH₄⁺-N at 0-10 cm Depth (p=0.000).

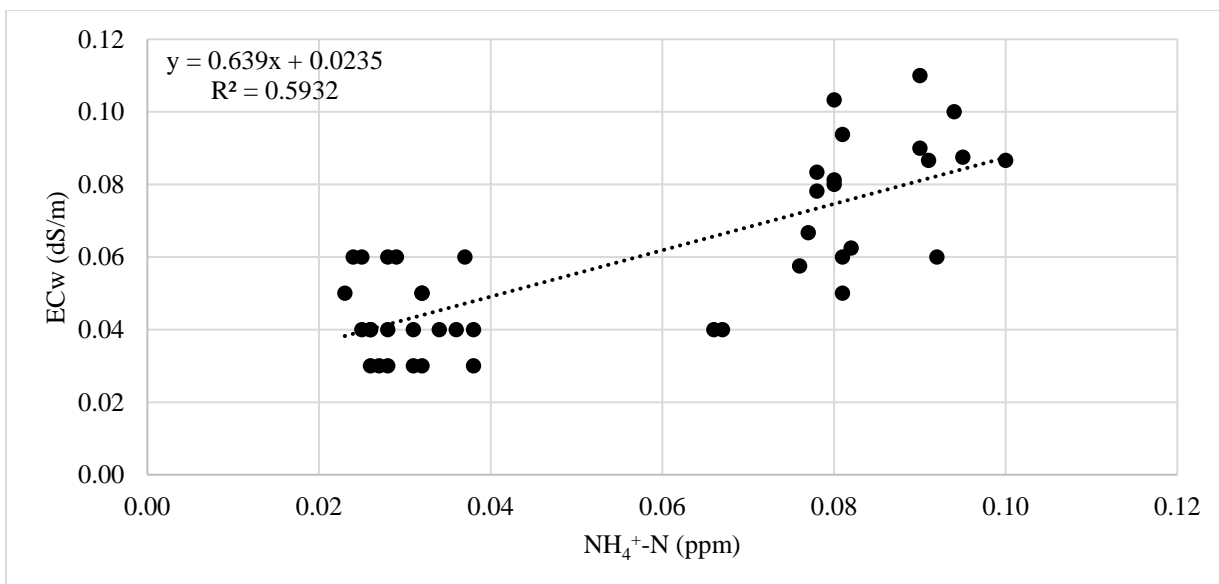


Figure 3.34 ECw vs NH₄⁺-N at 10-20 cm Depth (p=0.000).

3.2.3 Correlation Analysis of EC with CEC

Cation exchange capacity was compared with ECa and ECw across all field days irrespective of treatment. When referring to **Figure 3.35, 3.36, and 3.37**, it can be seen that CEC had a significant correlation with ECa (Kweon et al., 2013) and ECw at both depths (ECa, ECw 0-10 cm, & ECw 10-20 cm, $p=0.000$). The correlations obtained were negative. This can be accounted for by the increase in CEC temporally while ECa and ECw decreased temporally, as discussed earlier.

Significant correlation data for individual treatments of EC with SOC, $\text{NH}_4^+\text{-N}$, and CEC can be seen in **Appendix E**.

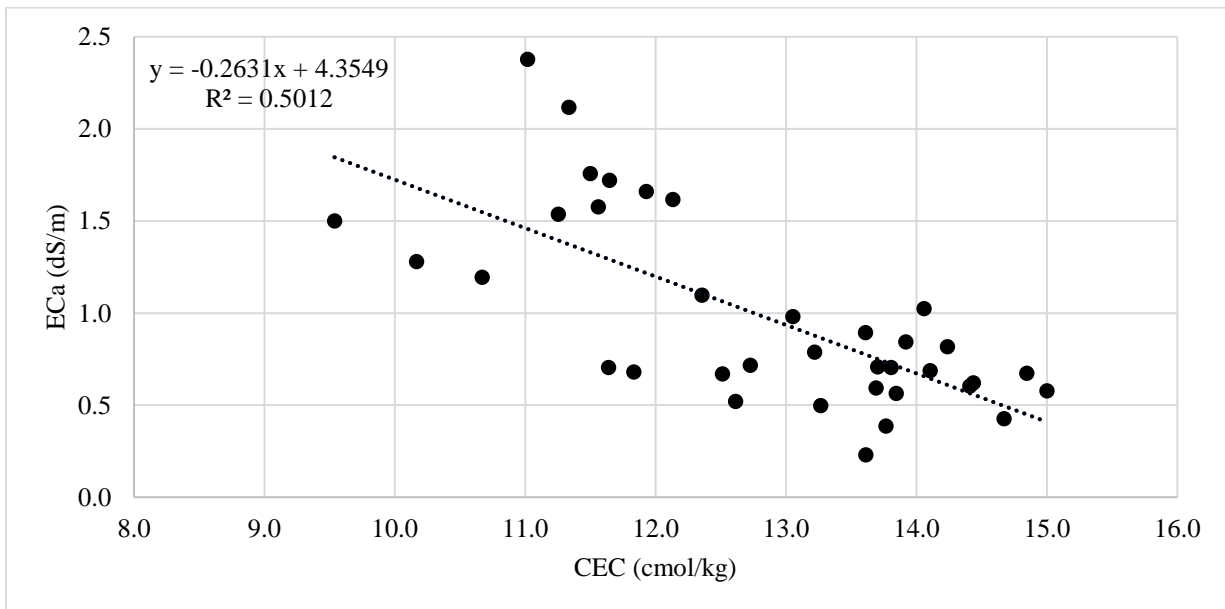


Figure 3.35 ECa vs CEC ($p=0.000$).

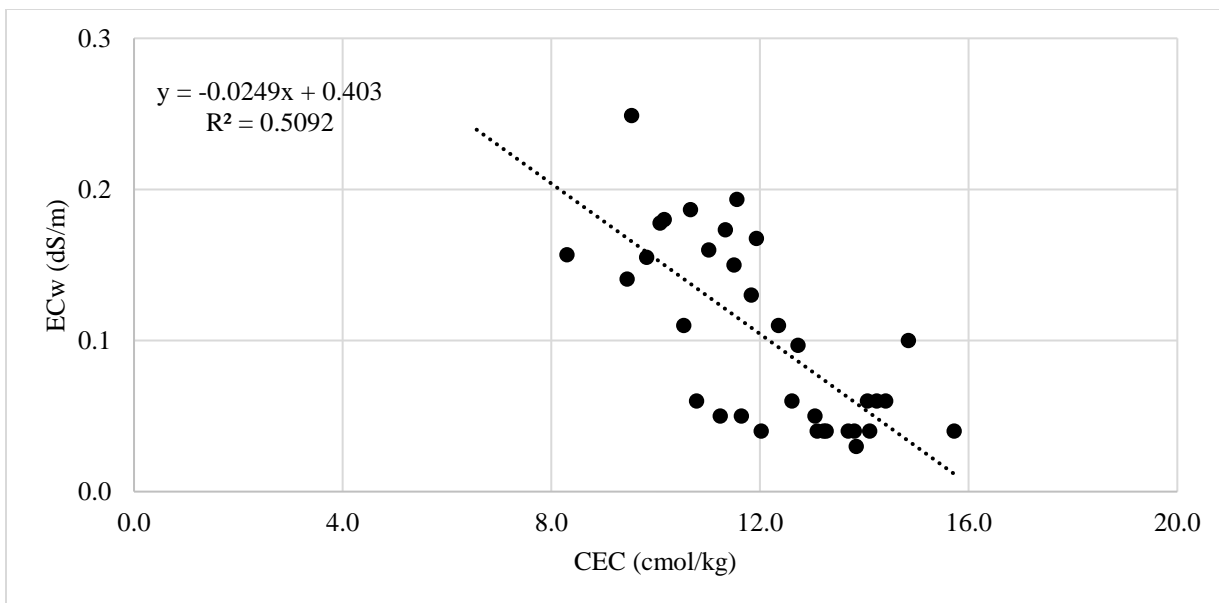


Figure 3.36 ECw vs CEC at 0-10 cm Depth (p=0.000).

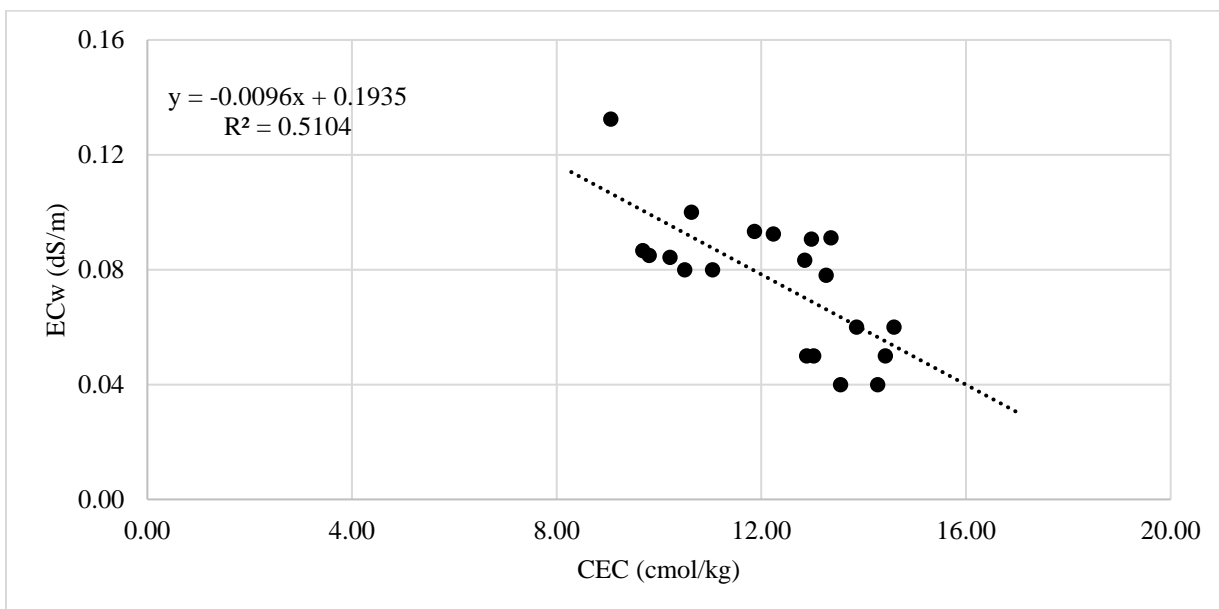


Figure 3.37 ECw vs CEC at 10-20 cm Depth (p=0.000).

4.0 Discussion

4.1 Temporal Variability of Measured Soil Properties

When referring back to the results, no significant change in VMC and GMC was observed among treatments, or temporally for each treatment. This was more than likely due to rainfall and low temperatures, which reduced evapotranspiration of water for the last two field days (September 30 and October 6), as was discussed earlier. The effect of biochar in T3 could be seen, as MC was slightly higher for this treatment, although not significant (Liu et al., 2012).

For pH values, all treatments increased temporally. This may be accounted to the period of no rainfall from September 10 to 29. However, the VMC and GMC indicate that the moisture content of the soils were higher for the last two days than the first, and therefore pH would not necessarily be expected to increase if the soil has high moisture content. Liming agents, like biochar, do increase pH, as could be seen for T3 plots, although not significantly (Chintala et al., 2013; Molnár et al., 2016). As mentioned earlier, when soil pH increases, Al^{3+} precipitates as hydroxy-Al polymers, allowing more space for adsorption of other cations to the clay particle surfaces (Haynes & Naidu, 1998).

SOC remained fairly constant for all treatments temporally, as was expected over the time frame of the experiment. The effect of biochar could be seen slightly for T3, as it initially had the highest SOC in the 0-10 cm depth, and the highest SOC for most days in the 10-20 cm depth. This is expected, as biochar has been shown to increase SOC (Li et al., 2015; Sorrenti et al., 2016). T5, however, did not seem to increase SOC, but rather decrease it, as it was lower than the control for most days, and even significantly lower

than T4 in the 10-20 cm depth on the second field day (August 4), indicating that the combination of DM+BC, over a short period of time, decreases SOC. If the experiment was conducted over a longer time frame, this might be different, however. DM seemed to increase SOC, as it had the highest SOC content for the 0-10 cm and 10-20 cm depth for a few days, as was discussed in the results. This is expected, as Haynes & Naidu (1998) have also shown that adding DM to soils does increase SOC.

For NH_4^+ -N data, it is important to note that the IN and DM were added a few months prior to the first day of NH_4^+ -N analysis, and, as a result, concentrations were lower than they would have been in the soil initially. However, it could still be seen that T3 and T5 had the highest NH_4^+ -N concentrations. This was expected, as biochar adsorbs cations, such as NH_4^+ -N, onto its negatively charged surface, decreasing losses (Taghizadeh-Toosi et al., 2012). It is also expected for NH_4^+ -N concentrations to be lower in the 10-20 cm depth due to the fertilizer only being added in the top part of the soil, and so the fertilizer would have to leach down to the lower depths. T4 increased in NH_4^+ -N concentration on the last day (October 6). This can be accounted for by potential slow-release properties of the DM plots. DM has been shown to cause an increase in NH_4^+ -N concentration with respect to other treatments (Sadej & Przekwas, 2008). As mentioned earlier, losses of NH_4^+ -N could be accounted for by volatilization, uptake by plants or immobilization by microbes, and conversion to NO_3^- -N (Sadej & Przekwas, 2008). If NH_4^+ -N losses are attributed to gaseous loss as N_2O , this could be accounted for by the increase in pH of the soils (Russenes et al., 2016) on the last two field days (September 30 and October 6).

Taghizadeh-Toosi et al. (2012) suggested that soaking biochar in the IN fertilizer and then applying it to the soils could potentially cause $\text{NH}_4^+\text{-N}$ concentrations to be higher initially, or cause less volatilization, as the fertilizer would already be adsorbed by the biochar (Taghizadeh-Toosi et al., 2012). This may, however, limit the adsorption of other cations to biochar. This was not done for this field experiment, however, as biochar was added separately from DM and IN on the same day.

CEC remained fairly constant for all treatments temporally, as would be expected over a short period of time. The effect of biochar could also be seen in T3 and T5, as CEC increased, although not significantly, across the field days (Githinji et al., 2014; Chintala et al., 2013), and had the highest CEC values in their groups (**Figures 3.19** and **3.21**). As biochar ages, the spontaneous oxidation reactions that occur will cause CEC to increase through addition of functional oxygenated groups, so causing its surface charge to be more negative, adsorbing more cations onto its surface (Lehman et al., 2009). As a result, it is recommended to test CEC over long-term experiments (Lehman et al., 2009). CEC increased for all other treatments in the 0-10 cm depth (except for T2, which decreased on October 6). This could be accounted for by soil pH, as an increase in soil pH has been shown to increase CEC (Brady & Weil, 2008).

For EC_w and EC_a measurements, values decreased temporally. EC_w values were mostly lower in the 10-20 cm depth than the 0-10 cm depth as a result of ion presence being higher on the top layers of the soil initially, where the fertilizer and biochar were applied. The effect of biochar on EC could be seen for T5 (Chintala et al., 2013), as the values again increased on the last field day for EC_a and both depths for EC_w (October 6).

The effect could not be seen for T3. However, values for T3 would more than likely also increase over a longer time frame. T2 and T4 had high EC_w and EC_a values initially, which was also expected, as EC has been shown to increase when there are high ion concentrations in the soil (Chintala et al., 2013). Hence, the temporal decrease for most treatments can be attributed to the decrease in ion concentrations due to leaching or uptake by plants.

Another potential cause for different variability could be due to the sampling locations and number of samples collected from the plots. Each plot as an area of 1 m x 6 m, and stratified samples were collected only from 3 locations within each plot. Biochar mixing was done manually in its treated plots, and it is possible that mixing was not uniform throughout the plot. Thus, some sampling locations may not have represented fully mixed biochar areas, leading to uncertainties in effect of biochar on soil properties. On the other hand, DM was applied as a liquid, and so it is possible for it to be mixed better within the entire treatment plot compared to that of biochar.

4.2 Correlation of EC with Measured Soil Properties

When referring back to the correlation analysis, it is seen that EC_w and EC_a can be correlated with SOC, NH₄⁺-N, and CEC with reasonably acceptable R² values regardless of field day and treatments. As significant results were obtained, EC_a measurements alone can be made and related back to the SOC, NH₄⁺-N, and CEC measurements without these having to be measured directly, saving money, time, and labour.

5.0 Conclusion

Biochar was shown to have positive effects on the measured physicochemical properties of the soils obtained in the silage corn field at Pynn's Brook Research Station. Over the 3.5 month time frame, biochar plots had the highest GMC for T3, and VMC for both T3 and T5. SOC was highest initially for T3, while T5 had fairly low SOC for all field days compared to those in its group (T1, T4 and T5). The effect of biochar could also be seen for T3 and T5 with initially high $\text{NH}_4^+\text{-N}$ concentrations. T3 and T5 did increase CEC the most out of all treatments in the 0-10 cm depth. For ECw and ECa, T5 increased on the last day, indicating that the combination of DM+BC is effective in increasing ECw. If these tests were to be done over a longer time frame, it is assumed that the positive effects of biochar will be amplified, showing significant increases of these physicochemical properties. Enhancing these soil properties may potentially enhance soil health, and as a result, soil fertility and crop productivity.

As significant correlations were found for ECw and ECa with SOC, $\text{NH}_4^+\text{-N}$, and CEC data, this will be used to map large areas of soil with ECa, and hopefully also using EMI. However, further investigation between ECa data and soil properties should be carried out to address uncertainties associated in predicting these properties. If EMI can be used, continuous, large, and non-point scale measurements can be made without having to disturb the soil, saving a lot of labour, time, and money. This will be used to map the spatio-temporal variability of important soil properties of Newfoundland.

From this study, it can be concluded that biochar is a great soil amendment that can be used to increase cation exchange capacity and pH of acidic Boreal soils, reducing the liming requirement.

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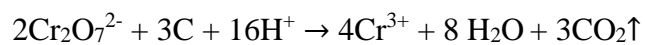
Appendix A - Biochar Properties

Bulk Density	0.232 g cm ⁻³
Electrical Conductivity	0.430 mmho cm ⁻¹
Void Space	87.5% (dry weight basis)
Solid Space	12.5% (dry weight basis)
pH	9.00
Water Holding Capacity	74.9 mL per 100 g dry biochar
Moisture	15.2%
Total Volatile Fraction	78.7%
Total Ash	6.0%

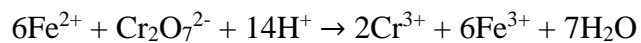
Table 1 Properties of Biochar used at the Pynn's Brook Research Station (Keith Day Company, Inc.).

Appendix B - SOC Reactions (Soil Survey Standard Test Method: Organic Carbon)

When $\text{K}_2\text{Cr}_2\text{O}_7$ and H_2SO_4 are added to the soil, the following reaction occurs:



The excess $\text{Cr}_2\text{O}_7^{2-}$ is then back-titrated with Fe^{2+} :



Appendix C - Temporal Variation Statistics within Each Treatment

Statistical analysis was done temporally within each treatment for each of the measured soil properties using ANOVA and Tukey's test in Minitab.

Table 1 Temporal Variation of Gravimetric Moisture Content at 0-10 cm Depth.

GMC (%) 0-10 cm	28 July	4 August	30 September	6 October
T1	16.10 ^a	15.82 ^a	19.12 ^a	16.10 ^a
T2	14.10 ^a	13.35 ^a	17.97 ^a	15.62 ^a
T3	15.14 ^a	13.55 ^a	18.90 ^a	17.26 ^a
T4	17.53 ^a	15.13 ^a	16.65 ^a	17.82 ^a
T5	15.39 ^a	12.24 ^a	16.11 ^a	17.26 ^a

Table 2 Temporal Variation of Gravimetric Moisture Content at 10-20 cm Depth.

GMC (%) 10-20 cm	28 July	4 August	30 September	6 October
T1	16.70 ^a	17.33 ^a	18.31 ^a	17.09 ^a
T2	15.23 ^a	14.55 ^a	20.16 ^a	15.75 ^a
T3	13.90 ^b	14.79 ^b	20.48 ^a	15.66 ^{ab}
T4	16.34 ^a	16.80 ^a	18.71 ^a	17.29 ^a
T5	15.99 ^a	15.62 ^a	19.23 ^a	15.66 ^a

Table 3 Temporal Variation of Volumetric Moisture Content.

VMC (%)	28 July	4 August	30 September	6 October
T1	27.22 ^a	-	29.33 ^a	27.57 ^a
T2	26.74 ^a	-	28.16 ^a	26.00 ^a
T3	26.04 ^a	-	28.41 ^a	27.92 ^a
T4	26.74 ^b	-	29.60 ^a	27.74 ^{ab}
T5	24.22 ^a	-	27.36 ^a	29.14 ^a

Table 4 Temporal Variation of pH at 0-10 cm Depth.

pH 0-10 cm	28 July	4 August	30 September	6 October
T1	6.08 ^a	6.04 ^a	6.38 ^a	6.63 ^a
T2	6.10 ^b	5.88 ^b	6.55 ^a	6.58 ^a
T3	6.24 ^{ab}	5.96 ^b	6.45 ^a	6.53 ^a
T4	6.21 ^{bc}	6.01 ^c	6.53 ^{ab}	6.65 ^a
T5	6.09 ^b	6.02 ^b	6.58 ^a	6.53 ^a

Table 5 Temporal Variation of pH at 10-20 cm Depth.

pH 10-20 cm	28 July	4 August	30 September	6 October
T1	6.13 ^{ab}	6.01 ^b	6.65 ^a	6.65 ^a
T2	6.34 ^a	6.06 ^b	6.48 ^a	6.53 ^a
T3	6.26 ^{bc}	6.11 ^c	6.50 ^{ab}	6.60 ^a
T4	6.39 ^{ab}	6.17 ^b	6.60 ^b	6.60 ^a
T5	6.32 ^{bc}	6.07 ^c	6.55 ^{ab}	6.60 ^a

Table 6 Temporal Variation of Soil Organic Carbon at 0-10 cm Depth.

SOC (%) 0-10 cm	28 July	4 August	30 September	6 October
T1	2.80 ^a	3.23 ^a	2.71 ^a	2.54 ^a
T2	3.22 ^a	3.23 ^a	2.55 ^a	2.68 ^a
T3	3.82 ^a	3.22 ^{ab}	2.47 ^b	2.63 ^b
T4	3.05 ^a	3.14 ^a	2.75 ^a	2.86 ^a
T5	2.59 ^a	2.55 ^a	2.51 ^a	2.63 ^a

Table 7 Temporal Variation of Soil Organic Carbon at 10-20 cm Depth.

SOC (%) 10-20 cm	28 July	4 August	30 September	6 October
T1	3.18 ^a	3.22 ^a	2.10 ^a	2.51 ^a
T2	3.30 ^a	3.33 ^a	2.54 ^a	2.50 ^a
T3	3.35 ^a	3.53 ^a	2.37 ^a	2.70 ^a
T4	2.88 ^a	2.58 ^a	2.91 ^a	2.80 ^a
T5	2.35 ^a	2.40 ^a	2.33 ^a	2.70 ^a

Table 8 Temporal Variation of NH₄⁺-N at 0-10 cm Depth.

NH ₄ ⁺ -N (ppm) 0-10 cm	28 July	4 August	30 September	6 October
T1	-	0.09 ^a	0.06 ^{ab}	0.03 ^b
T2	-	0.15 ^a	0.05 ^b	0.03 ^b
T3	-	0.37 ^a	0.10 ^a	0.03 ^a
T4	-	0.14 ^a	0.06 ^a	0.05 ^a
T5	-	0.21 ^a	0.05 ^b	0.03 ^b

Table 9 Temporal Variation of NH₄⁺-N at 10-20 cm Depth.

NH ₄ ⁺ -N (ppm) 10-20 cm	28 July	4 August	30 September	6 October
T1	-	0.08 ^a	0.09 ^a	0.03 ^a
T2	-	0.09 ^a	0.05 ^b	0.02 ^c
T3	-	0.09 ^a	0.05 ^{ab}	0.03 ^b
T4	-	0.09 ^a	0.05 ^b	0.03 ^b
T5	-	0.11 ^a	0.05 ^b	0.02 ^b

Table 10 Temporal Variation of Cation Exchange Capacity at 0-10 cm Depth.

CEC (cmol/kg) 0-10 cm	28 July	4 August	30 September	6 October
T1	11.53 ^a	-	12.71 ^b	13.17 ^a
T2	10.42 ^a	-	12.88 ^a	10.81 ^a
T3	10.79 ^a	-	11.95 ^a	13.76 ^a
T4	11.33 ^a	-	12.50 ^a	12.68 ^a
T5	10.65 ^a	-	13.19 ^a	12.24 ^a

Table 11 Temporal Variation of Cation Exchange Capacity at 10-20 cm Depth.

CEC (cmol/kg) 10-20 cm	28 July	4 August	30 September	6 October
T1	13.38 ^a	-	-	12.07 ^a
T2	11.95 ^a	-	-	12.09 ^a
T3	10.29 ^b	-	-	12.91 ^a
T4	11.44 ^b	-	-	13.47 ^a
T5	10.44 ^a	-	-	11.63 ^a

Table 12 Temporal Variation of ECw at 0-10 cm Depth.

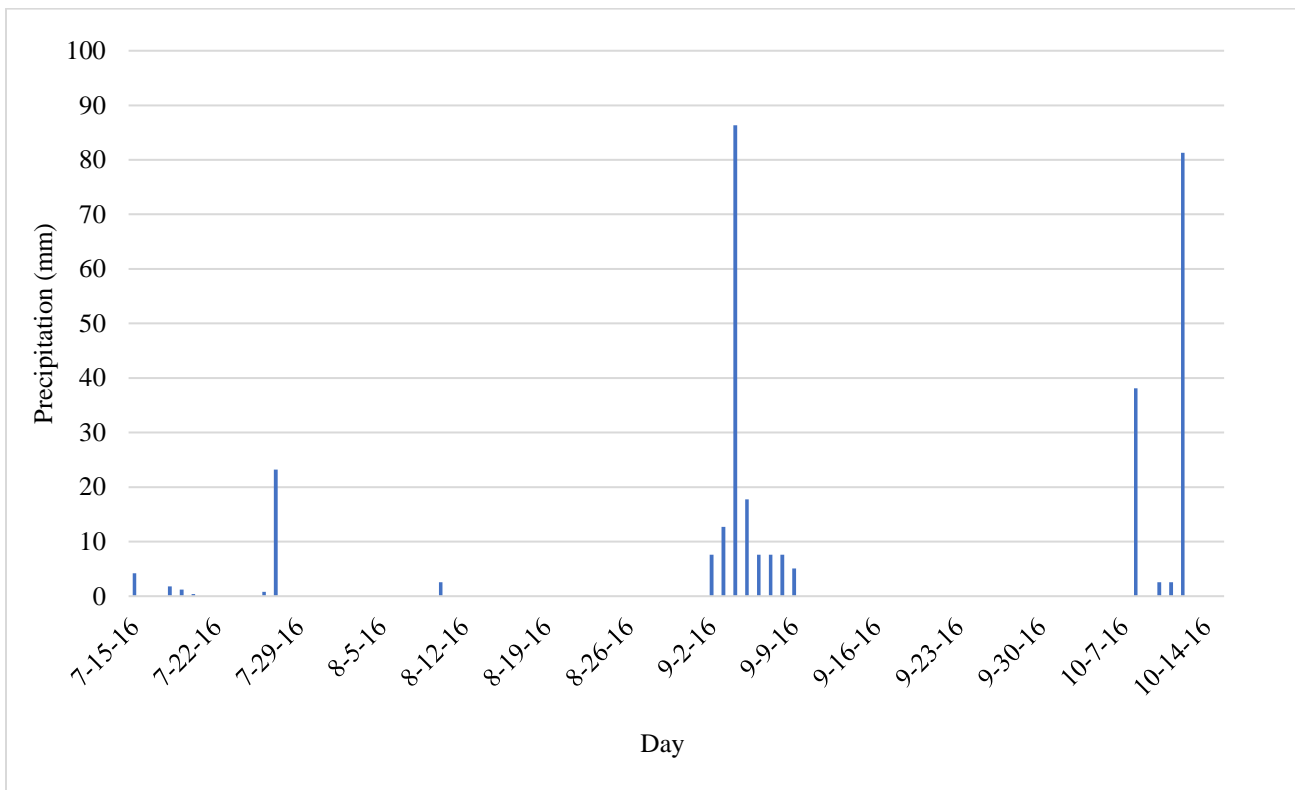
ECw (dS/m) 0-10 cm	28 July	4 August	30 September	6 October
T1	0.11 ^a	0.08 ^a	0.17 ^a	0.07 ^a
T2	0.22 ^a	0.18 ^{ab}	0.04 ^c	0.06 ^{bc}
T3	0.14 ^a	0.12 ^{ab}	0.06 ^{ab}	0.04 ^b
T4	0.20 ^a	0.16 ^a	0.04 ^b	0.05 ^b
T5	0.22 ^a	0.09 ^b	0.04 ^b	0.07 ^b

Table 13 Temporal Variation of ECw at 10-20 cm Depth.

ECw (dS/m) 10-20 cm	28 July	4 August	30 September	6 October
T1	0.09 ^a	0.09 ^a	0.04 ^b	0.04 ^b
T2	0.09 ^a	0.09 ^a	0.04 ^b	0.06 ^{ab}
T3	0.10 ^a	0.07 ^{ab}	0.05 ^b	0.04 ^b
T4	0.10 ^a	0.09 ^a	0.03 ^b	0.05 ^b
T5	0.06 ^a	0.06 ^a	0.13 ^a	0.06 ^a

Table 14 Temporal Variation of ECa.

ECa (dS/m)	28 July	4 August	30 September	6 October
T1	1.19 ^a	-	0.58 ^a	0.65 ^a
T2	1.55 ^a	-	0.62 ^b	0.62 ^b
T3	1.41 ^a	-	0.62 ^b	0.62 ^b
T4	1.81 ^a	-	0.59 ^b	0.59 ^b
T5	1.44 ^a	-	0.63 ^a	0.69 ^a

Appendix D - Precipitation Data**Figure 1 Precipitation Data obtained at Pynn's Brook Research Station.**

Appendix E - Correlation for Individual treatments of EC with SOC, $\text{NH}_4^+\text{-N}$, and CEC

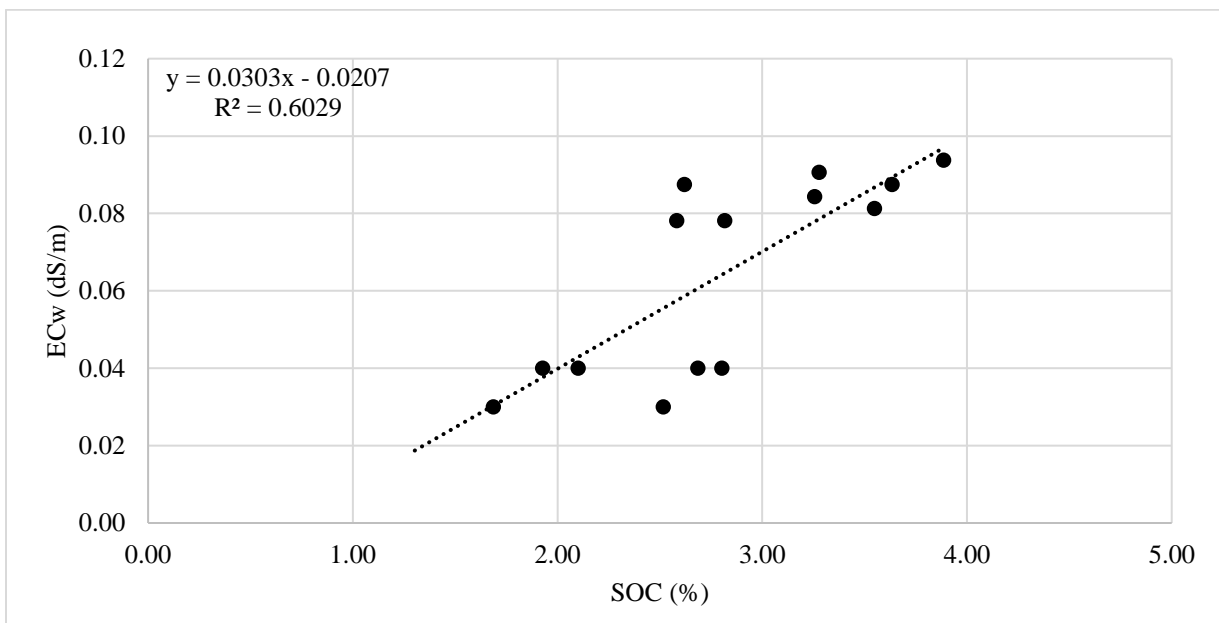


Figure 1 ECw vs SOC at 10-20 cm Depth for T1 ($p=0.001$).

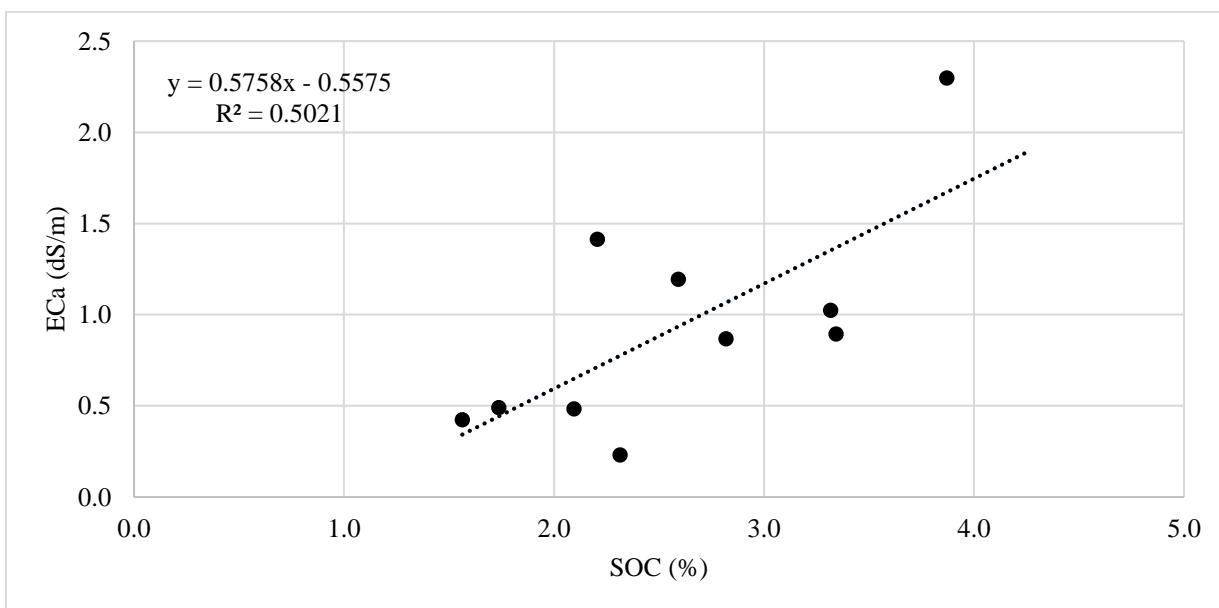


Figure 2 ECa vs SOC for T2 ($p=0.02$).

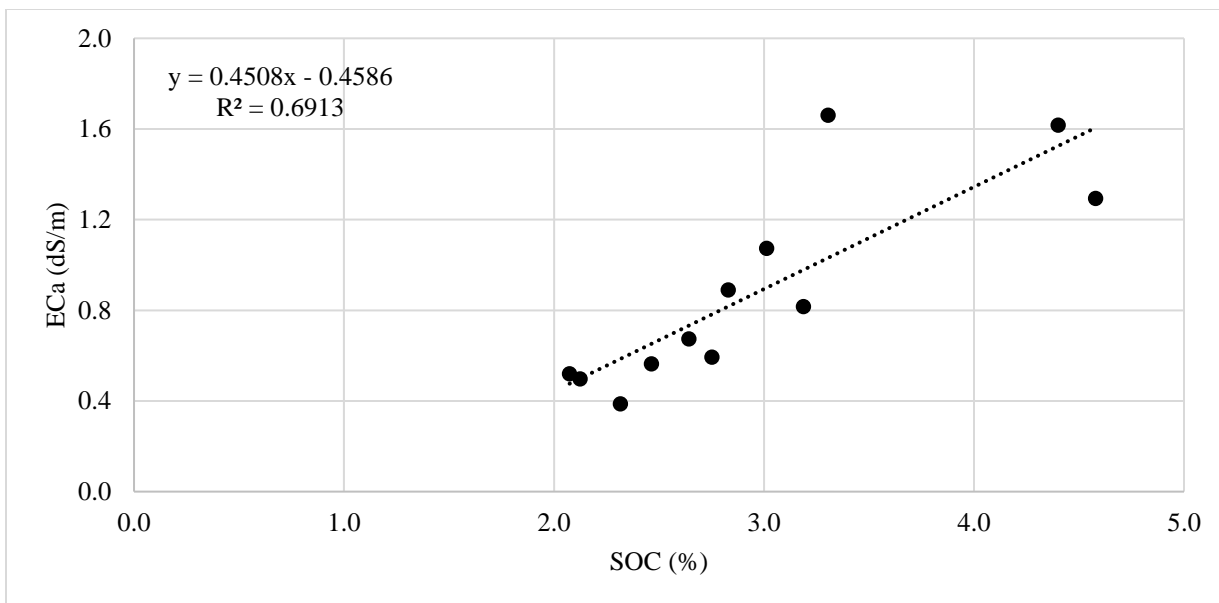


Figure 3 ECa vs SOC at 0-10 cm depth for T3 (p=0.02).

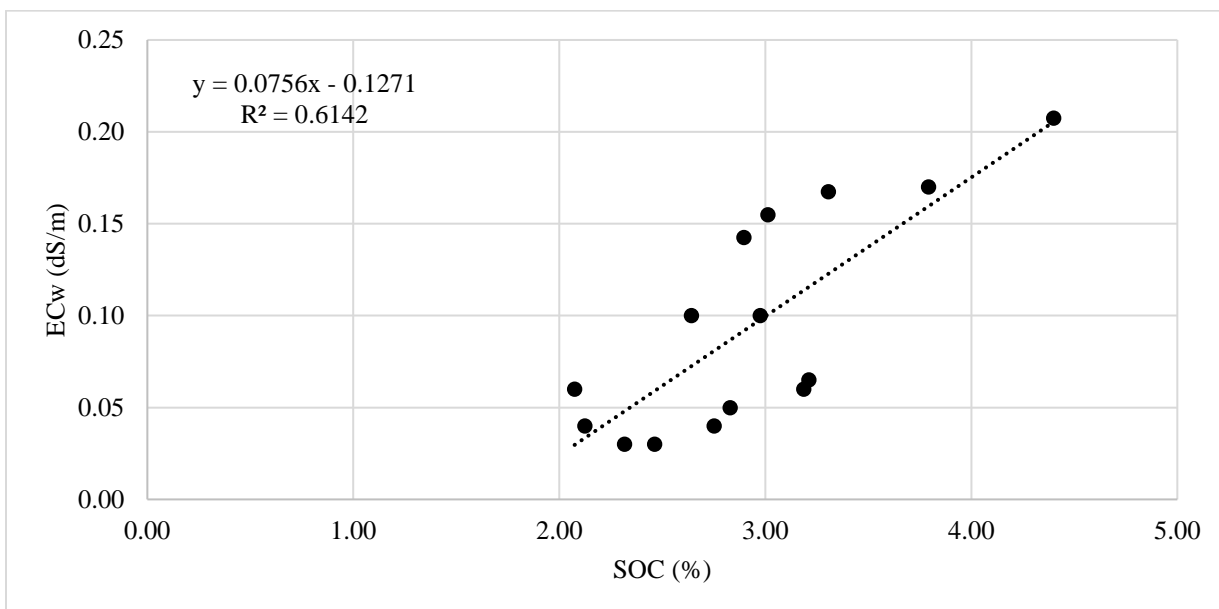


Figure 4 ECw vs SOC at 0-10 cm depth for T3 (p=0.0005).

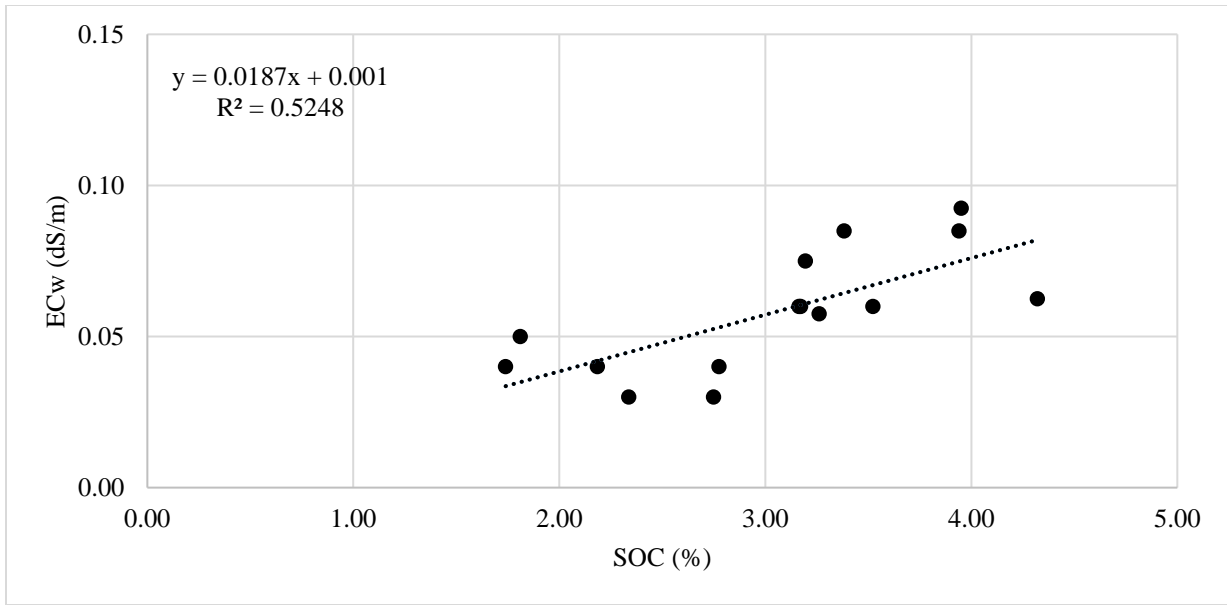


Figure 5 ECw vs SOC at 10-20 cm depth for T3 (p=0.002).

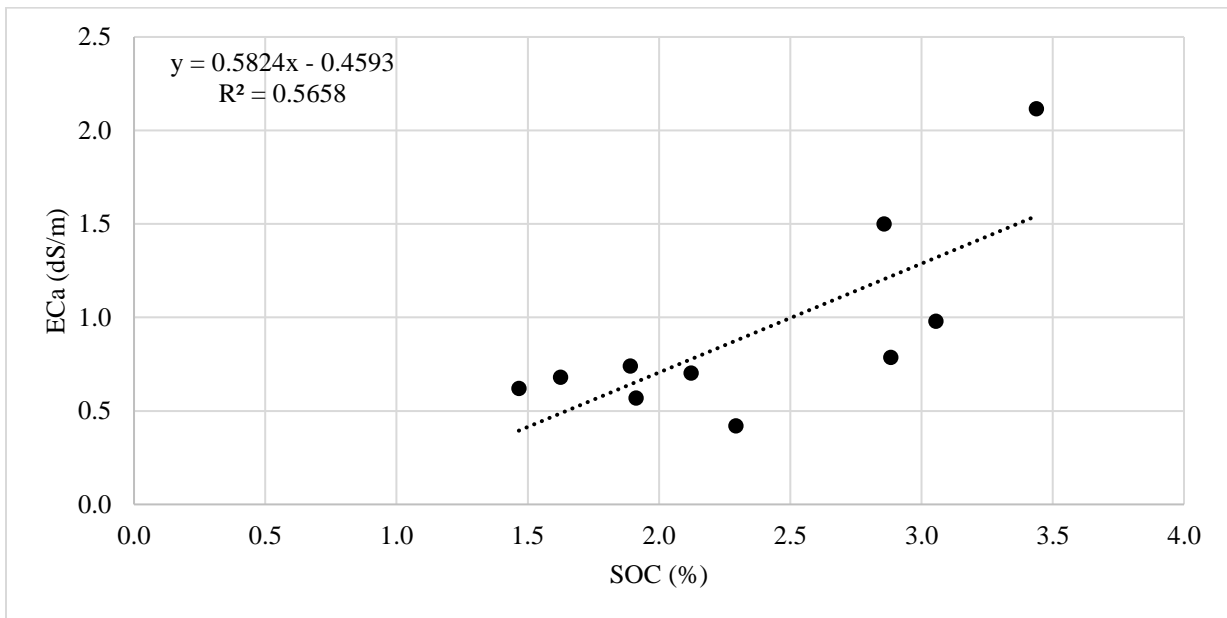


Figure 6 ECa vs SOC at 0-10 cm Depth for T5 (p=0.01).

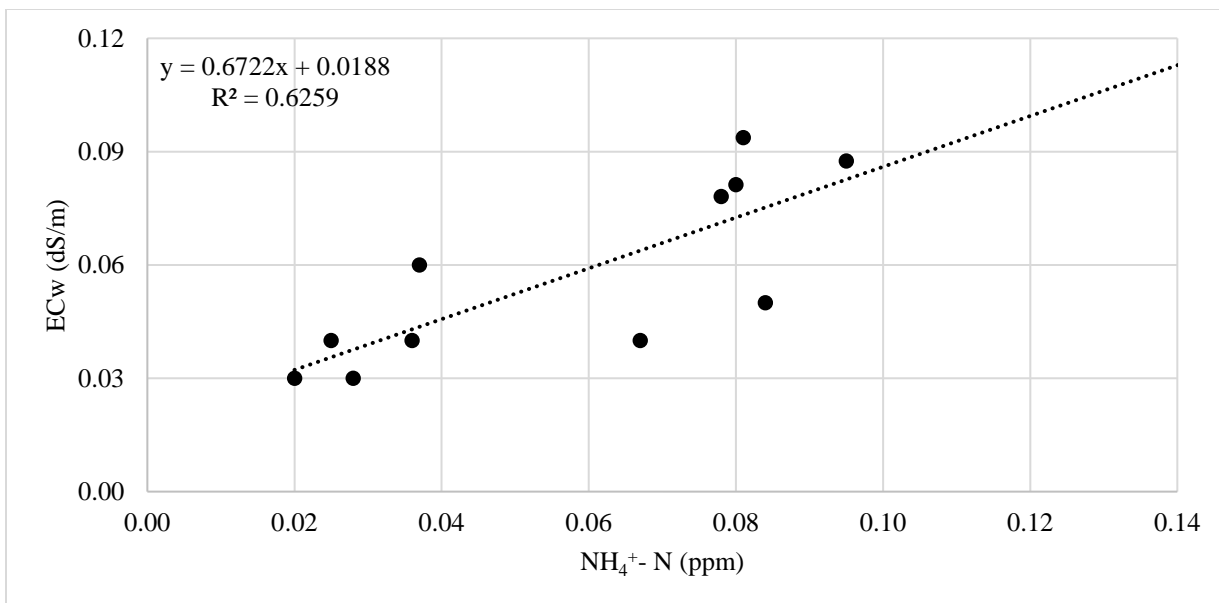


Figure 7 ECw vs NH₄⁺-N at 10-20 cm Depth for T1 (p=0.004).

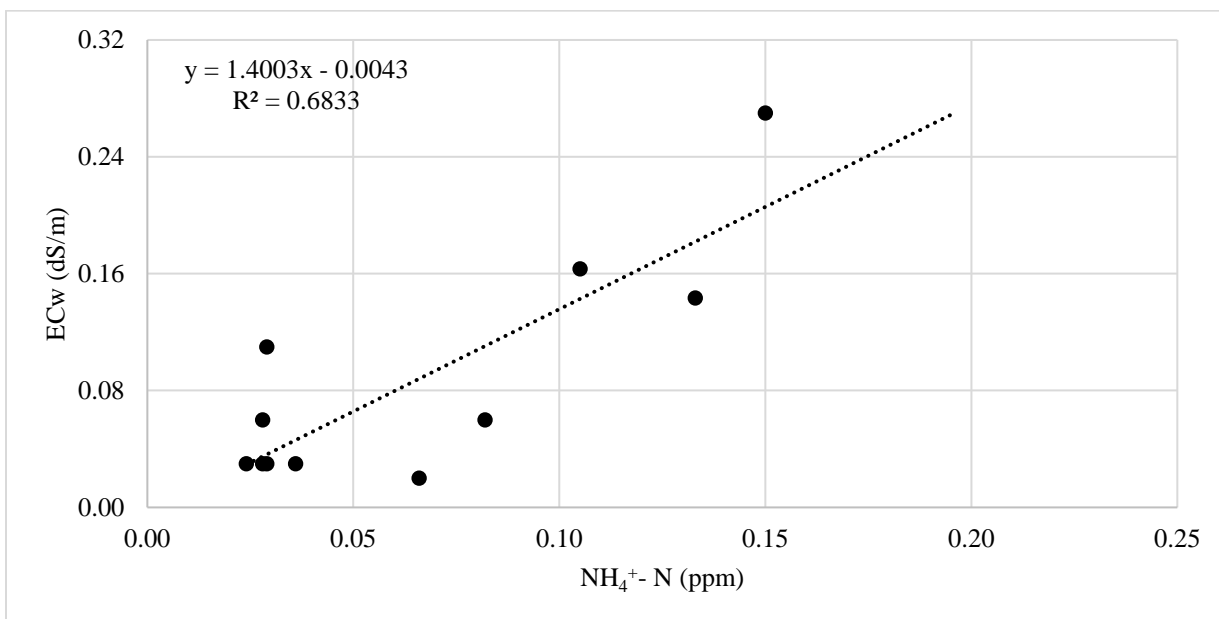


Figure 8 ECw vs NH₄⁺-N at 0-10 cm Depth for T2 (p=0.002).

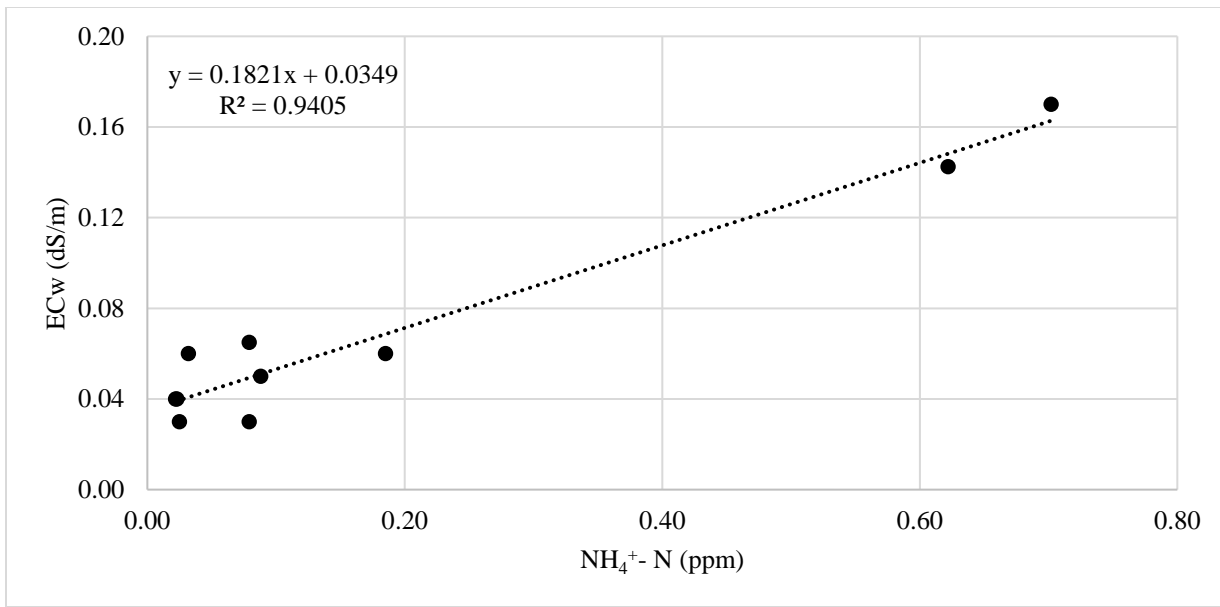


Figure 9 ECw vs NH₄⁺-N at 0-10 cm Depth for T3 (p=0.000).

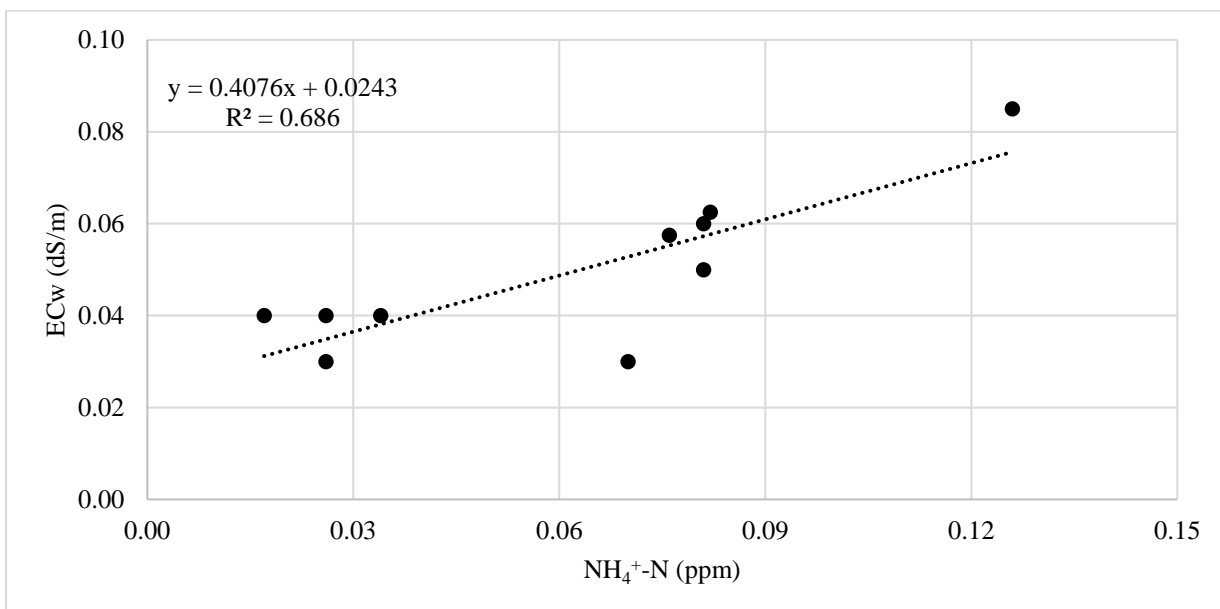


Figure 10 ECw vs NH₄⁺-N at 10-20 cm Depth for T3 (p=0.003).

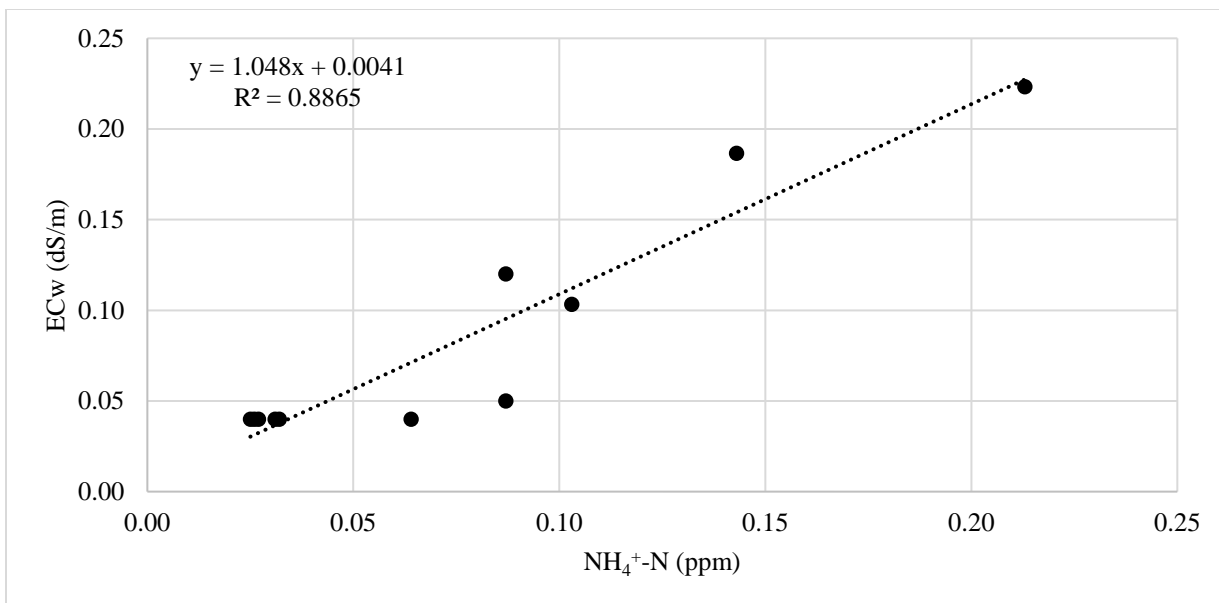


Figure 11 ECw vs NH₄⁺-N at 0-10 cm Depth for T4 (p=0.001).

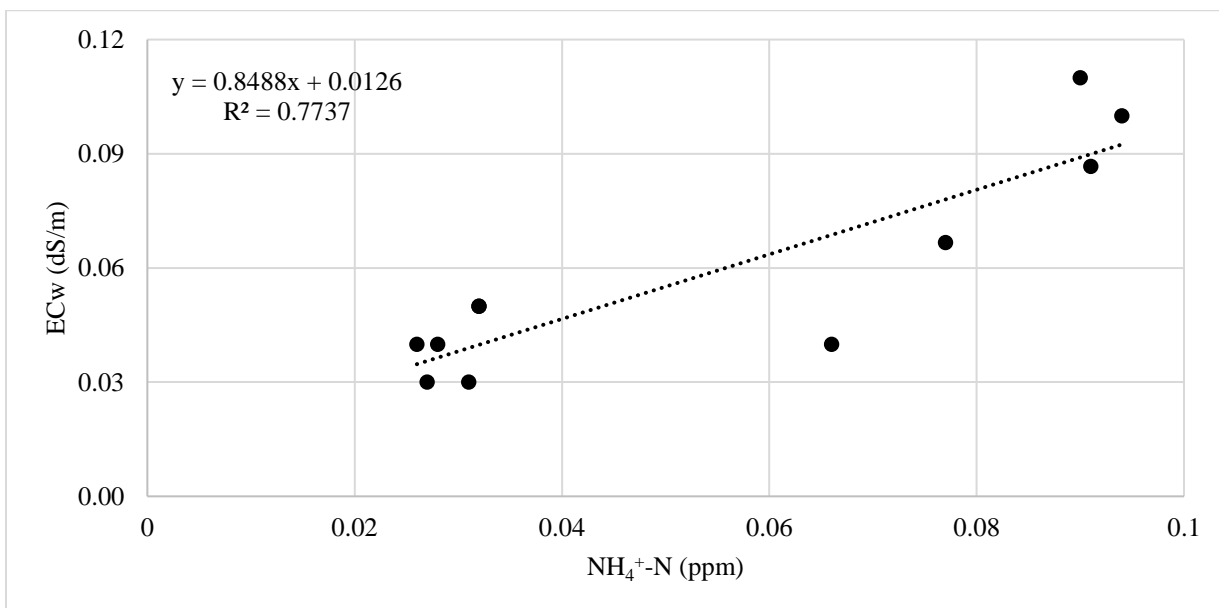


Figure 12 ECw vs NH₄⁺-N at 10-20 cm Depth for T4 (p=0.008).

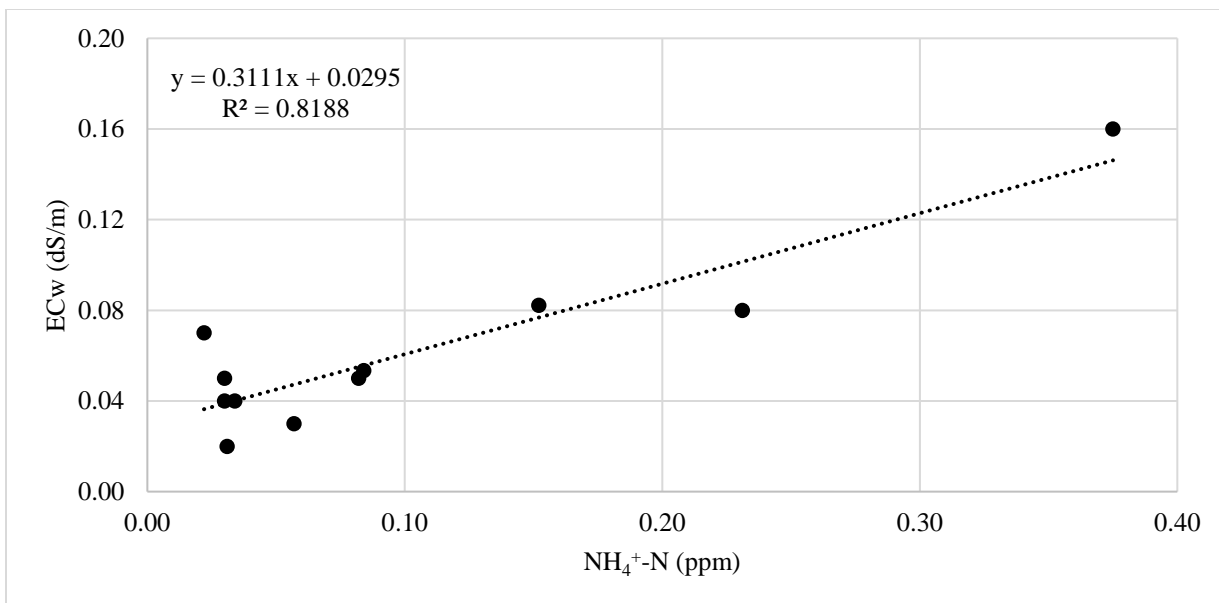


Figure 13 ECw vs NH₄⁺-N at 0-10 cm Depth for T5 (p=0.02).

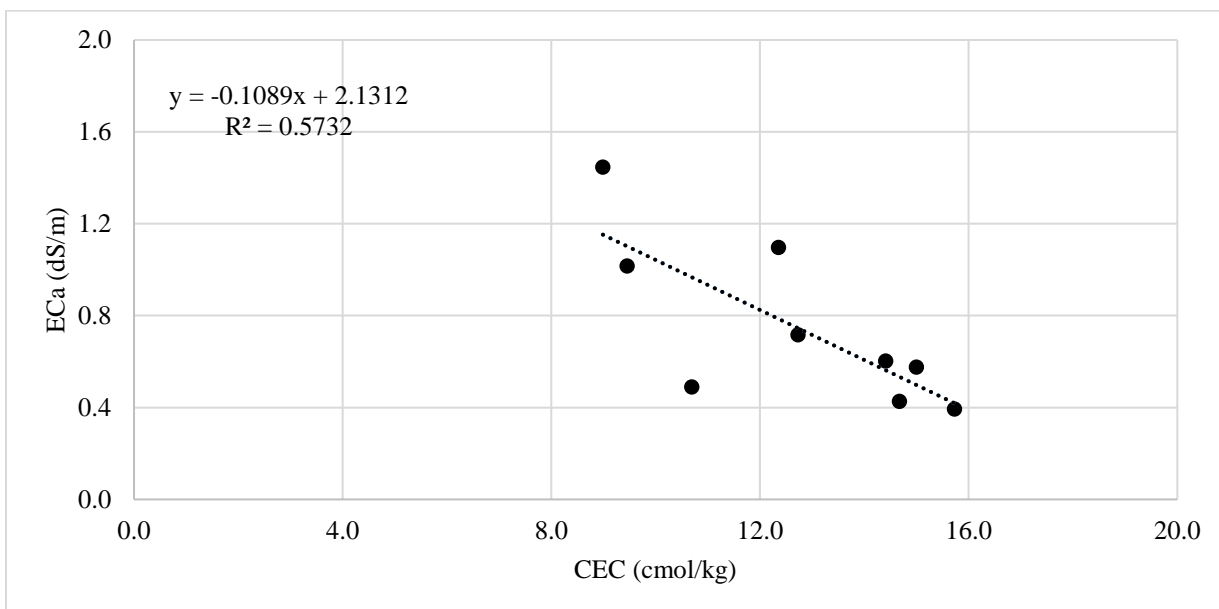


Figure 14 ECa vs CEC for T1 (p=0.02).

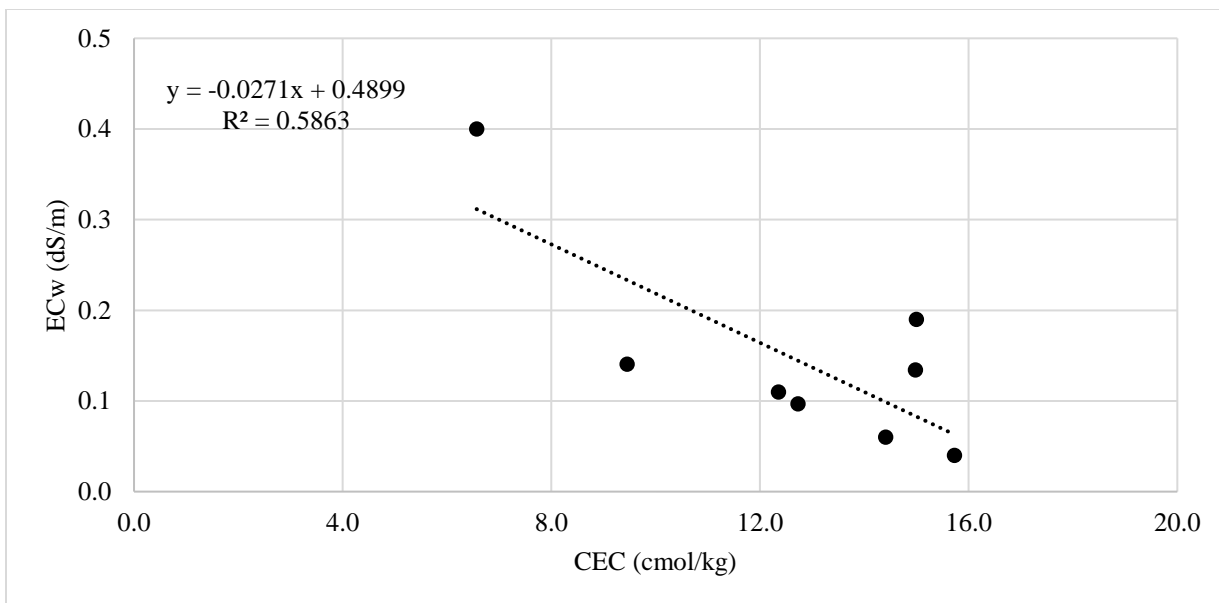


Figure 15 EC_w vs CEC at 0-10 cm Depth for T1 (p=0.03).

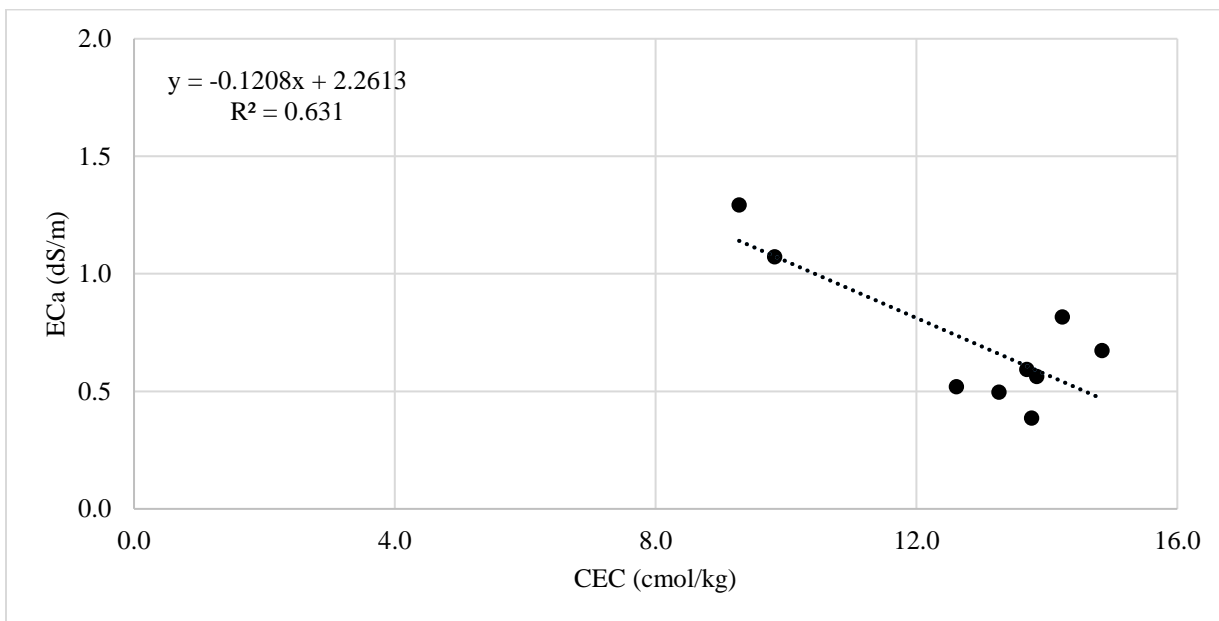


Figure 16 EC_a vs CEC for T3 (p=0.01).

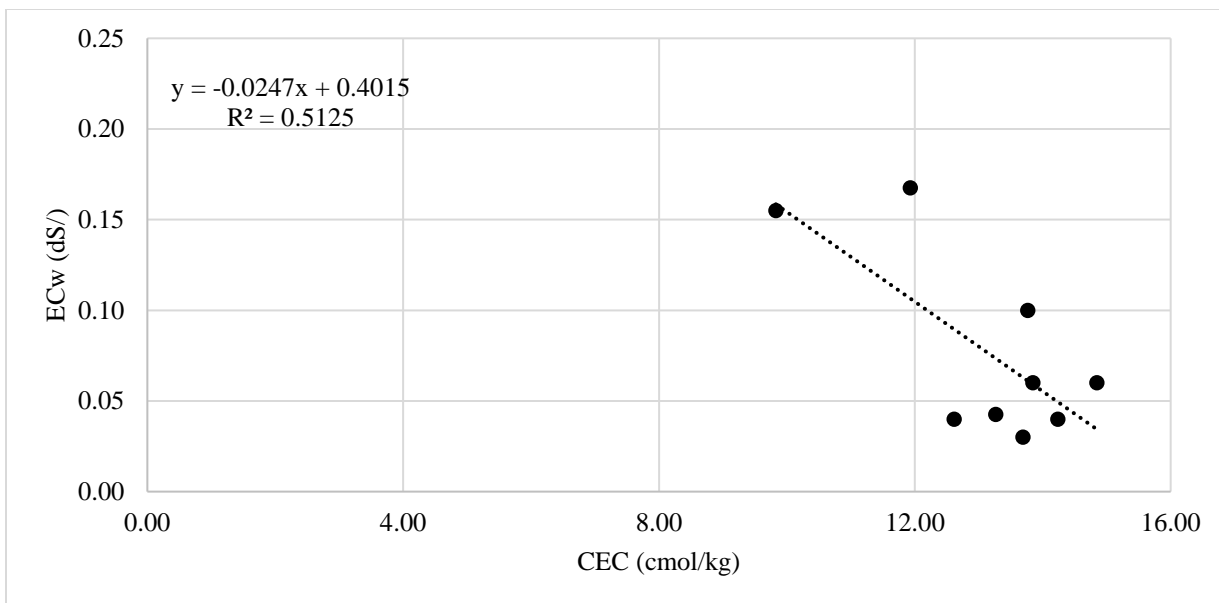


Figure 17 ECw vs CEC at 0-10 cm Depth for T3 (p=0.03).

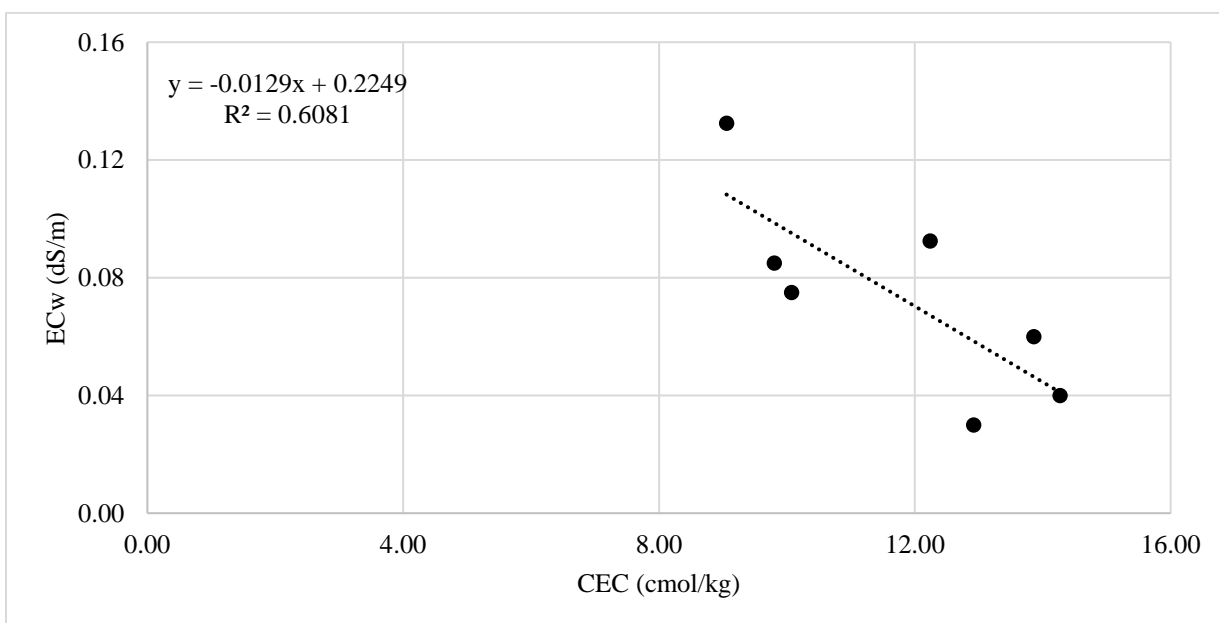


Figure 18 ECw vs CEC at 10-20 cm Depth for T3 (p=0.04).

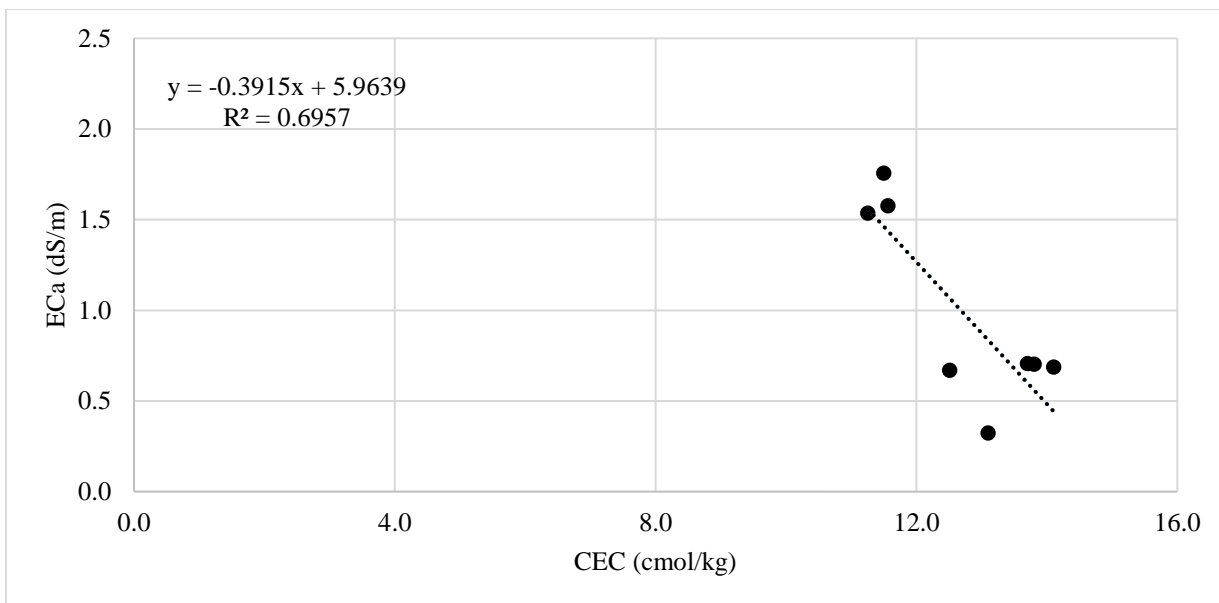


Figure 19 ECa vs CEC for T4 (p=0.01).

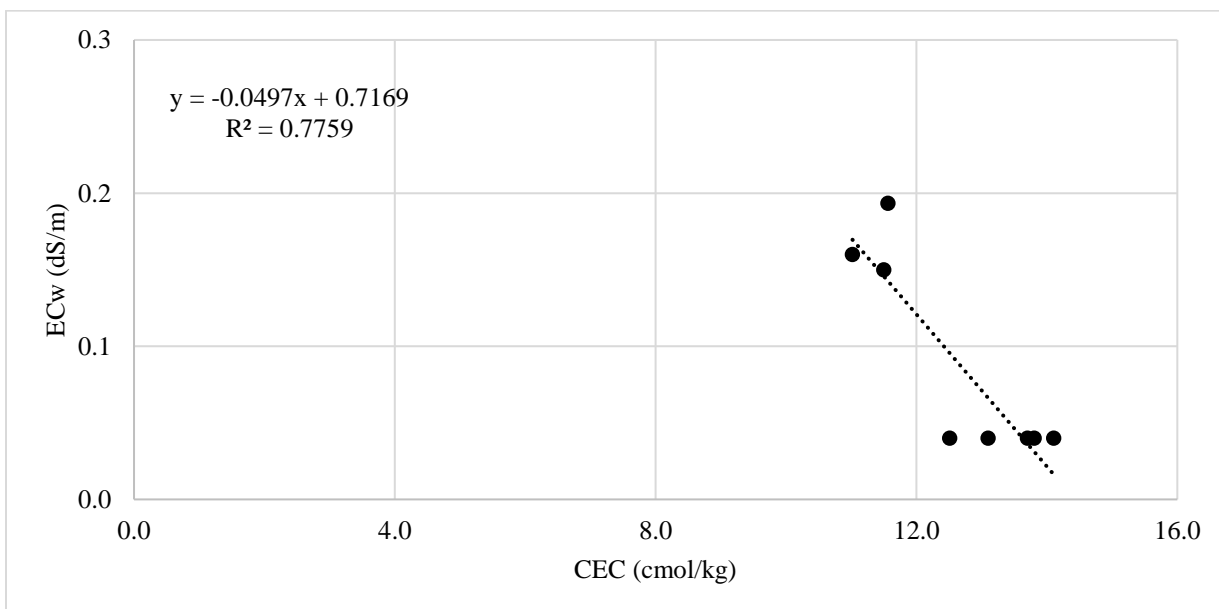


Figure 20 ECw vs CEC at 0-10 cm Depth for T4 (p=0.004).

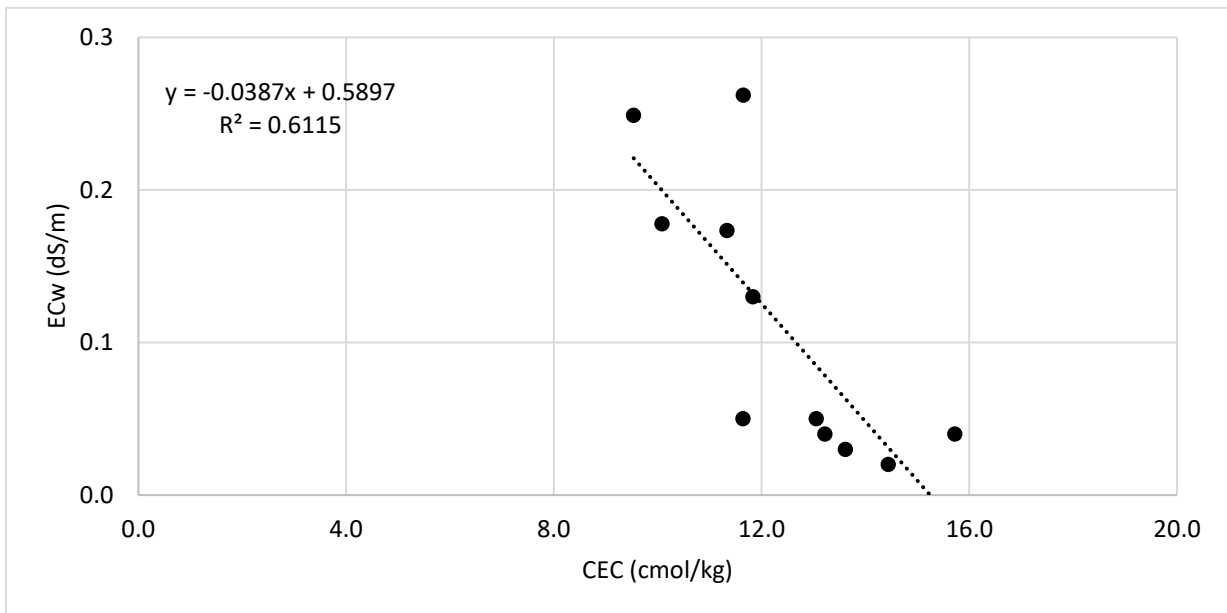


Figure 21 ECw vs CEC at 0-10 cm Depth for T5 (p=0.004).