Soil structure assessment under Subsurface Drip Irrigation; and comparison of wet sieving method and dynamic laser diffraction method of aggregate stability

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Declaration

I declare that this thesis does not contain material which has been accepted for a degree or diploma by the University or any other institution. To the best of my knowledge, the materials in this thesis were not previously published or written by another person except where due references are made in the text of -the thesis, nor does the materials in the thesis infringe copyright.

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Summary

Differences in soil properties were investigated under long-term Subsurface Drip Irrigation (SDI) system used for tomato production in northern Victoria. The samples were collected from same depths of 0-10, 10-20 and 20-30 cm but different locations along the dripper tape and from emitter. These locations were under emitter, 22.5 cm and 45 cm towards bed edge from emitter, between emitters (25 cm from emitters) and diagonally extreme point which is the farthest point from emitter.

Water Stable Aggregate (WSA), Electrical Conductivity (EC), Soil pH, Exchangeable Cations (Ca, Mg, K and Na), ESP, Ca:Mg ratio and CEC were measured. The results of most of the tested properties showed positive (increasing) relationship with distance away from emitter, excluding no trend for potassium result and negative trend for Ca:Mg ratio. Among all locations effects were only statistically significant to EC ($P<0.01$), soil pH ($P<0.01$), exchangeable sodium ($P<0.01$) and ESP ($P<0.05$). These results indicated that cations were possibly washed away from emitter during 4 years of SDI practise.

The decreasing ratio of exchangeable calcium to exchangeable magnesium (Ca:Mg) with increasing distance away the emitter may relate to calcium supplied via the fertigation system. The exchangeable sodium increased to more than double that of under emitter toward the extreme point suggests either leaching of sodium or general loss of CEC due to clay migration. The extent of the increment was higher in the direction towards bed edge than along the row.

Particle Size Distribution (PSD) of samples from under emitter and between emitters were also compared. The PSD result indicated that samples from between emitters were higher in proportion of ‘fines’, i.e. particles <100 µm, but lower in coarser fraction, especially sizes ranging between 650 – 1500 µm (coarse sands). This suggests that very fine sand, silt and clay might be migrating away from under the emitters with irrigation water to more distal areas of the bed.

These findings indicate real chemical and physical changes can occur to soils under longer term subsurface drippers and suggest general leaching and migration of finer particles in the irrigation waters.

In comparison to the aggregate stability results from the wet sieving method, Laser Diffraction (LD) can be used for aggregate stability test. According to the change of the disintegration rate of aggregate over time, the aggregate dynamic disintegration curve was separated into two part called Phase-1 and Phase-2. Comparison using the complete disintegration curve was not appropriate as aggregate size was closed to the particle size after entering Phase-2 of disintegration. However, direct comparison of complete disintegration curve of samples with different SOC from the same site showed good SOC to stability relationship. This indicated that LD is sensitive to soil property variations of samples from the same soil type as they are similar in particle size distribution (PSD).

In Phase-1, aggregate stability units (MVD and Median) and aggregate disintegration rate decreased rapidly. The result of wet sieving was positively correlated to the average disintegration rate of first four repeated measurements (parameter- m) and the modelled aggregate stability index (parameter- c). Parameter- c is a better stability index as it is the y-intercept point related to average disintegration rate and aggregate sizes of first four repeated measurement. Both disintegration rate and stability units ‘stabilized’ after 400 seconds, which was the Phase-2 of the disintegration curve. Therefore it is recommended that for Laser Diffraction applications to aggregation studies a limit of 400 seconds be applied.

Table of Contents

List of Figures

List of Tables

1 Introduction

This literature review and thesis aims to;

1) Evaluate the reasons for purported declining soil health and the potential use of a locally marketed liquid gypsum product (Gyp-Flo) for use as a soil amendment to ameliorate soil degradation relating to agricultural activities (**Chapter 3**). This was undertaken by examining the soil properties at different locations relative to the emitters and different GYP-FLO treatment rates were compared. The soil properties assessed were Mean Weight Diameter, particle size distribution, exchangeable cations (Ca, Mg, K and Na), soil pH and EC (salinity), ESP (exchangeable sodium percentage) and Ca: Mg ratios;

2) Evaluate the use and application of Laser Diffraction (LD) for aggregate stability assessment (**Chapter 4**) using samples with range (0.9 – 3.0%) of soil organic carbon contents. The LD method was compared with the tradition aggregate wet sieving method. The study thus aimed to determine the relationship between LD result and wet sieving result.

Application of Subsurface Drip Irrigation

Australian processing tomato production is mainly cultivated in NSW and Victoria, along the Murray-Darling basin. Depletion of water resources is one of the major threats to agricultural industry in semiarid area. In order to achieve higher water use efficiency, subsurface drip irrigation (SDI) system is widely used in processing tomato cropping.

Camp (1998) concluded that crop yields under subsurface system were equal to or better than other surface based irrigation systems; however, water use efficiency is much higher under SDI rather than other irrigation system. A 15 year SDI study conducted by water management research laboratory came out with same conclusion (Ayars *et al.*, 1999). The reason behind that can be explained by high irrigation frequency which resulted in better soil water and wetting patterns and thus improved yield and water use efficiency for SDI compare to other systems (Wang *et al.*, 2006). Furthermore, it reduced water percolation from root zone and increased water used from groundwater when the crops are nurtured on high water table land (Ayars *et al.*, 1999). Benefits of high frequency irrigation were also explained by other studies (Camp, 1998, Freeman *et al.*, 1976, Segal *et al.*, 2000, Wang *et al.*, 2006). Meanwhile, lower canopy humidity resulted in prevention against pathogen development, leaf burn and weed establishment (Lamm, 2003, Yarwood, 1978).

Subsurface drip irrigation has been widely installed for processing tomato plantation in Victoria over the last 14 years. The cultivation area with SDI increased from 53% in 2000 to 95 % in 2013 (Mann, 2014). The irrigation system commonly last for 10 years after installation unless severely damaged by tillage or harvesting operations. However, reduction in tomato yield is reported with the increasing number of years of the subsurface drip system in place. It has been suggested that the reduction is a result of soil physical and chemical property deterioration following a few years of subsurface drip irrigation and cultivation pressure.

Laser Diffraction

Aggregate stability has being studied since this idea was purposed by Yoder (1938). Various methods, such as wet sieving (Amezketa *et al.*, 1996, Yoder, 1936) raindrop impact (Loch, 1994a), ultrasonic dispersion (Fristensky and Grismer, 2008), immersion (Emerson, 1967), and dry sieving (Kemper and Chepil, 1965), have been successively developed for aggregate stability study. This work indicates that this research in soil structural quantification (aggregation) is continuing and under development with no single method proving fully satisfactorily. Le Bissonnais Le Bissonnais (1996) suggested a combination of different existing methods into three treatments to have comprehensive measurement of aggregate stability. Primarily, this is because of soil aggregate stability is the function of various soil physical and chemical properties. Different methods are sensitive differently to various properties based on the type (dry or wet) and strength (ultrasonic or immersion) of the energy used by the method.

Since 1970’s laser diffraction (LD) technique has been extensively used for Particle Size Distribution analysis (Ma *et al.*, 2000, Kelly *et al.*, 2006, Ryżak and Bieganowski, 2011, Zobeck, 2004). These studies gave sound support to LD as the most effective method to measure PSD. In comparison to traditional particle size distribution techniques, LD has two major advantages, which are, able to complete a measurement in an hour rather than days; PSD curve can be obtained by using LD machine, instead of points result from traditional methods.

In recent years, researchers proposed the potential of using LD to measure aggregate stability. The studies included direct application and methodological research of aggregate stability measurement by using LD method (Bieganowski *et al.*, 2010, Ma *et al.*, 2000, Mason *et al.*, 2011, Westerhof *et al.*, 1999, Rawlins *et al.*, 2013). LD can deliver consistent amount of disruptive forces throughout the measurement by pre-programmed stirring speed, pumping speed and ultrasonic level during the measurement. This is the fundamental requirement for aggregate stability study (Kemper and Rosenau, 1986). Most importantly, LD is able to monitor aggregate disintegration dynamic and aggregate size distribution, which are the most interested features and so far can't be achieved by any method.

In present, there are limited methodological studies of LD method for aggregate stability test around the world. There are barely any standard methodology is concluded from these studies. The relationships between aggregate stability result from LD method, other soil stability measurement methods and soil properties are still unknown. In this study, soil organic carbon and wet sieving method were chosen for comparison since they were the most common soil property and method used for soil structural indication.

2 Literature Review- Liquid Gypsum as soil amendment to ameliorate irrigation-induced soil degradation via subsurface drip irrigation systems

2.1 Abstract

Subsurface drip irrigation system is extensively used for processing tomato growing in Victoria, in order to achieve better water use efficiency (WUE) while maintaining the crop yield. Despite that, reduction in tomato yields is reported with the increasing number of years of the subsurface drip system in place.

Gypsum can efficiently supply plant available sulphur and calcium due to its moderate solubility. Gypsum is also commonly applied as soil amendment to ameliorate soil properties by means of the electrolyte and cation exchange effect. It increases the electrolyte concentration of soil solution and thus prevents soil dispersion. Furthermore, more stable particle bonding can be achieved by replacing exchangeable Na with Ca. However, the effect of gypsum is very soil type-dependent such properties as soil texture, organic matter and the amount of soluble electrolyte in the soil.

This review discusses the impacts of water chemistry on the soil properties and the relation between soil degradation and both salinity and sodicity levels of the soil. The negative impacts of sodicity commonly reduced the clay flocculation, hydraulic conductivity and indirectly can cause soil degradation such as tunnel erosion, surface sealing, reduced porosity and so increased surface runoff. It will also examine the potential soil degradation due to application of SDI and its potential mitigation by using gypsum to improve soil properties.

Laser diffraction (LD) is a well-known industrial testing method, developed in the 1980s for rapid and precise particle size distribution (PSD) for products like chocolate, milk and has now been extended to soils and sediments. LD can also deliver a consistent amount of disruptive energy whilst measuring PSA which is the fundamental requirement for aggregate stability testing. Thus the application of the LD method to aggregate stability has increased in recent years. Introduction and limitations of this method will be discussed as the last objective of this study.

2.2 Content

2.2.1 Soil structure and plant growth

Marshall (1962) defined soil structure as “the arrangement of the soil particles and of the pores in between them” or more simply “the arrangement and size of pores”. Besides the size and number of soil pores, continuity of these pores also plays many important roles in soil. Marshall *et al.* (1988) stated that soils with good structure are soft and friable, but also more resistant against degradation by water and traffic, and are thus more productive and amenable to management.

Soil porosity is composed of textural porosity and structural porosity (Murray and Grant, 2007). Textural porosity is formed between simple arrangement of particles of various shapes and sizes – in essence a packing arrangement. The rearrangement of these particles is pushed by physical, chemical and biological processes such as shrinking and swelling, water movement, root growth and soil organisms. They are bounded together by organic matter, cementing materials, microorganism, roots, clay and silt. The structural porosity is referring macroscopic soil structure in which aggregated particles form into larger ‘aggregates’ or ‘peds’ which are separated by cracks and flaws. It is also a basic characteristic of each particular soil. Naturally, development of soil structure is very time consuming, however, extrinsic factors can either positively or adversely alter soil properties in short period such as soil amendment, irrigation method, water quality, wetting rate and antecedent water content (Currie *et al.*, 2006).

Table 2.1 Scale in pore size (Marshall *et al.*, 1988)

Pore size	**Description**	**function**
0.1-10 mm	Macro-pores- formed by organism activities (root growth, soil fauna), soil tillage, and soil shrinkage	Fast draining, most of the time filled with air
0.01 mm	Meso-pores- mainly within aggregates	Coarsest water storage pores
1 um (0.001	Too small for bacteria to access	Water storage pores

mm)		
0.1 um	Micro-pores- finest water storage pores	However, water is unavailable for plants growth
<0.01um	Pores within clay and fine clay matrix	Sites for cations exchange, water-filled all of the time

Arvidsson and Håkansson (1991) and Stirzaker *et al.* (1996) indicated that plants grow best on soil with intermediate bulk density. Hard soils restrict water and nutrient uptake by plants by resisting root penetration in soil. In soft soil, root growth will not be affected, however, it is believed that the contact area between root and soil is too low for efficient nutrients uptake in soft or overly loose soils (Kooistra *et al.*, 1992). The presence of continuous macropores would promoted the incidence of clumped roots in occasional macropores which severely constricted supplement of nutrients and water (Passioura, 1991). Smith *et al.* (1983) noted that reduction in infiltration rate over time by infiltrating water into flood-irrigated, fine-textured soils lead to prolonged wetness periods and anoxia conditions. This caused retardation of crop growth and implied the importance of permeability, soil pores and soil strength to plant growth. These soil physical properties are the indices of good soil structure (Murray and Grant, 2007). Permeability reflects the total porosity and orientation of these channels in soil. It has significant impact upon infiltration, drainage, runoff, salt accumulation, nutrient availability, biological activities and heat transfer. Soil water content is largely affected by presence of water storage pores ranged between 0.2 and 3 µm. Soil texture plays important role in determining abundance of these pores. Soil strength has significant impact on seed germination and root growth, and indicating level of compaction in the field.

Agricultural practices and stresses from natural events puts pressure upon the soil structural aggregates. The ability of soil to retain its arrangement of solids and voids under stresses is called structural stability (Kay and Lal, 1998). Structural stability is supported by soil properties such as organic matter, cementing agents, cation concentrations, mineralogy and clay content. Kay (1990) defined structural resilience as “the ability of soil to recover its structure by natural processes when stresses are reduced”, such as shrinkage- swelling features of reactive clay. It is an important characteristic of soil that indicates the extent of, the possibility of and rate of soil structural recovery in place. Kay *et al.* (1994) combined the concept of structural resilience and structural stability in the term 'structural vulnerability' as a measurement of overall inability of soil structure to deal with common stresses.

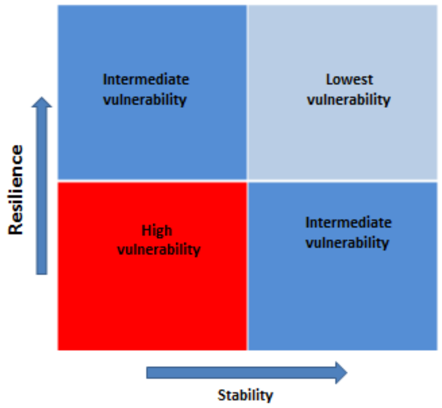


Figure 2.1 Variation of Vulnerability with stability and resilience (Kay and Lal, 1998)

Potential reasons causing decline in soil structure and soil properties under long-term SDI

Excluding the influence of irrigation water's quality (chemistry), water applied via SDI can induce negative changes in soil. The soil around the drippers experiences higher water volume flow and higher flow rate generated by the SDI dripper spacing leading to differences in soil water potential along the drip tape. Furthermore, wetting fronts are concentrated around the sphere of emitter. It would cause uneven distribution of water content across the landscape. The soil that is close to the emitter could suffer oversaturation if irrigation strategies are impropriate, such as high dripping rate in short time vs low dripping rate in long time (better). The SDI system can maintain soil water content within desirable ranges for plant growth with high WUE when monitored and administered appropriately.

However the continuous application conditions can favour the occurrence of slaking and dispersion which are strongly related to soil degradation during wetting. Soil strength is also inversely correlated with soil water content; therefore, soil with high water content is generally more vulnerable to structural degradation resulting from applied forces (Murray and Grant, 2007). Marshall *et al.* (1996) stated that wet soil is more vulnerable to damage due to the weakening of cohesion between particles and softening of cementing agents. Weaker soil consistency will result from high water content condition and soil could completely lose their structure leading to high strength on drying (Murray and Grant, 2007). However, the changes of soil consistency over ranges of water content vary between soil textures. Soil under SDI system experience prolonged wetness period and both high irrigation frequencies and emission rates.

Slaking is the process of disintegration of macro-aggregates into micro-aggregates through the pressures generated from wetting (Murray and Grant, 2007). Loch (1994b) noted that wetting rate has more significant effect than energy generated from rain and irrigation drop on aggregate stability. Lyles *et al.* (1974) indicated that dry soil will detach (slaking) twice as much as wet soil when they were applied with same amount of raindrop impact. Soil is unable to bear high entry rate of water into aggregates drawn by capillary forces at this leads to entrapment and compression of air and uneven swelling. Kemper *et al.* (1975) demonstrated adverse relationship between wetting rate, crust strength and the persistence of large soil pores. The magnitude of pressures produced by increased wetting rate and consequences to the extent of slaking were pointed out in other studies (Kay, 1990, Quirk and Panbokke, 1962). Currie *et al.* (2006) indicated that wetting rates generated by drip irrigation are generally higher than those produced by natural rainfall events due to the concentrated emission of water from one point, and thus soil is more likely to slake under drip irrigation. A 4 litre/hour emission rate with a wetting zone sphere diameter of 30cm in a sandy loam soil is equivalent to a 60 mm/hr of tropical downpour. Inappropriate first irrigation with high rate on dry soil after long fallow is believed to cause structural damage to soil and has the potential to generate tunnel erosion following dispersion in sodic soils.

Coalescence is the process of aggregate cementing and results in hardening and strengthening of soil expressed as hardsetting. Coalescence has been reported as an important issue that causes restricted root growth, poor productivity, and low hydraulic conductivity within the soil matrix on zero and minimum tilled soil (Cockroft and Olsson, 2000). However, coalescence does not appear to significantly change in soil bulk density; meanwhile, development of coalescence is relatively slow, rather than abruptly (Murray and

Grant, 2007). Porosity of the soil is not reported in any of the studies (Cockroft and Olsson, 2000, Grant *et al.*, 2001). Increased cycle of wetting and drying have significant impact on synthesis of coalescence but mechanisms which caused this distinct form of soil hardening is not well known. Cockroft and Olsson (2000) indicated that water-stable aggregates, untrafficked soils, good soil drainage, and fertile soil are not immune to the formation of coalescence after 3 months of cultivation. However, several studies have found that organic matter content and composition have adverse effects on the susceptibility of soil to the coalescence (Cockroft and Olsson, 2000, Grant *et al.*, 2001). Theoretically, soil under SDI is more prone to development of coalescence due to more wetting-drying cycle and the fact that deep soil tillage is not practicable.

The potential soil degradation issues discussed above result from effects of simply water alone on soil. Degraded soil is susceptible to oncoming pressures from mechanical stresses or disturbances which lead to more severe structural degradation. Research on soil structural deterioration with SDI system has rarely been compared to other irrigation systems. Furthermore, the extent of degradation is highly soil type-dependent, examples will be discussed later, such that, better understanding of structural degradation induced by SDI is essential for further evaluation of gypsum response in this study.

2.2.2 Case study

Barber *et al.* (2001) studied the effects of long-term subsurface irrigation on soil chemical and physical properties. The SDI has been applied with good quality irrigation water on the self-mulching clay for six years and decreased in lateral water movement has been observed. The study could not find relationships between bulk density and the degradation. The results showed significant migration of clay content and minerals from emitters, increases in porosity around the emitters, alternation of soil colour and increasing slaking though the bed but no significant dispersion of soil. Accumulation of migrated clay and minerals is believed to be the reason that causing decreased lateral water movement and low hydraulic conductivity at the emitter.

Bulk density, soil strength and porosity of a Red Brown Earth were reported as being adversely affected by drip irrigation (Clark, 2004). Drip irrigation with borewater (EC = 2-3 dS/m) would result in increased salinity and sodicity, especially at the wetting front of the sphere. Salinity and sodicity would favour colloid dispersion and structural decline to occur when the salt is leached below a certain threshold. Currie *et al.* (2006) found the adverse impact of drip irrigation on soil structure of Red Brown Earth. Soil at the emitter has higher soil strength and lower permeability. The infiltration rate and field-saturated capacity are positively related to the distance from dripper, except field-saturated hydraulic conductivity of B1 horizon over 15 years of drip irrigation. There is no evidence to support that SDI alter the bulk density in the subsoil. Currie indicated that soil drying treatment could be a potential method to improve saturated hydraulic conductivity of heavily degraded soil by creating pore space within soil aggregates during soil shrinkage on drying.

The key concluding results of these studies is that long-term drip irrigation probably results in clay and mineral migration away from the emitters. Water quality and soil type do not seem to affect the tendency of migration but they have significant impact on consequence of migration. For example, Barber *et al.* (2001) irrigated the non-dispersive self-mulching soil with high quality water and this resulted in accumulation of CEC, slightly dispersive at the edge of the sphere, increased porosity around emitter but no evidence of bulk density change; however, Clark (2004) reported that irrigated texture contrast soil with high ESP water and resulted in change in soil classification from Chromosol to Sodosol which implied large alteration of properties, extensive dispersion and leading to increase in bulk density, clod density and reduce in porosity. Clay migration may associate with coalescence and hardsetting formation and result in higher soil strength, no change in bulk density but increased in porosity around emitter (Barber *et al.*, 2001, Currie *et al.*, 2006). Permeability testing is needed to confirm the continuity and structure of void space.

2.2.3 Gypsum

Gypsum, comprised of calcium sulphate (79% CaSO4) and two H_2O (Chen and Dick, 2011). It has been used in agricultural production and soil conditioner since late 18th century (Crocker, 1922). It is a great source of available calcium and sulphur for plant nutrition. Gypsum and lime often confuse laymen due to their similarity in agricultural benefits. However gypsum (2.5g/L) is 200 times more soluble than lime (CaCO3), and thus, gypsum is more readily available and able to reach and potential ‘reclaim’ the deeper soil horizons (Chen and Dick, 2011). Lime will also increase the pH of the treated soil but gypsum does not, however, lime is only effective in shallow soil profile and soils with pH value below 6

(Haynes and Naidu, 1998). Sumner (1990) indicated that yield of alfafa, peach, cotton, corn and soybean increased significantly with use of combination of gypsum and lime rather than use of gypsum alone. Gypsum is most used to reclaim sodic soils by releasing calcium to the soil and, in turn, increase electrolyte concentration in soil solution to maintain adequate water infiltration (Oster, 1982). According to the desired change in exchangeable sodium fraction and cation exchangeable capacity (CEC) of soil, different application rates are recommended (Richards, 1954).

2.2.4 Gyp-Flo

Gyp-Flo is a fine particle size product compared to traditional gypsum. Traditional gypsum has particle size of approximately 1000 micron with solubility of 2.5 g/L in water. According to the product brochure, the "liquid" gypsum has particle size of only 5-micron. It is said to have 300 g of active gypsum in 1L of diluted irrigation water. Watts and Dick (2014) stated that flue gas desulfurization (FGD) gypsum may have greater soil improvement benefits than mined gypsum due to its smaller and more uniform particle size distribution. Shainberg *et al.* (1989) concluded solubility of gypsum over different particle sizes of products. Thus, liquid gypsum is believed to be more superior in soil amelioration, however, durability of each input is unclear.

Table 2.2 Active components of liquid gypsum (GYP-FLO). Data is calculated from product brochure of Gyp-Flo

Gyp-Flo	w/v (%)	g/L
active Ca (MW=40g)	35	350
active S (MW= 32.05g)	25	250
active gypsum (MW=172.17g)	~150	~1500
1:5 dilution	30	300

2.2.5 As nutrient supplement

Sulphur deficiency is predicted to be widespread due to the substitution of higher S-containing fertilizer (superphosphate) and pesticides with high analysis products (triple super and DAP and MAP) and less pesticide use or different formulations. In addition, increasing production pressure also contribute to greater amount of S removal from soil (Chen *et al.*, 2008). Gypsum can provide very quick response to sulphur requirement as it contains S as sulphate form which is readily available for uptake of plant root (Pivot, 2010).

Gypsum also contains calcium and movement of calcium within plants is very slow requiring good and regular supply. There is only a small amount of calcium that can reach the end line of the transportation, the fruit, therefore, available Ca must be always presented in root zone for plant uptake. Root tips are also highly sensitive to calcium deficits (Fisher, 2011). Calcium cannot be transported from other plant tissues to root tissue, therefore, gypsum is a better option rather than lime as gypsum is moderately soluble and it can move through the root zone easily. Recent studies have indicated that addition gypsum as soil amendment would improve the quality of horticultural crops. Incidence of blossom-end rot of tomato and watermelons under saline conditions decreased with gypsum treatment (Franco *et al.*, 1994). Tomato yields increased with gypsum input under both saline and non-saline conditions (Saeed and Ahmad, 2009), root rot of avocado caused by pathogen and bitter pit in apples are reduced by adding gypsum as regulator (Scott *et al.*, 1993). However, there are examples of yield penalty after gypsum treatment in citrus (Anderson, 1968, Jones *et al.*, 1963) probably relating to their sensitivity to salinity as gypsum can increase the EC of the soil.

2.2.6 Clay swelling

Clay is the most important component which determines soil hydraulic properties due to its large surface area. Swelling of soil is caused by active absorption of water by clay particles to lower the ion concentration near charged surfaces (Oster, 1982). The higher ion concentration upon charged surface than bulk solution is result from attachment of exchangeable cations to the charged surface. In consequence, swelling occurred to prevent exchangeable ions diffuse from the surface into the bulk solution. Soils with high exchangeable sodium percentage (ESP) are under large swelling pressures develop between clay particles due to monovalent sodium dominated charged site. Clay with calcium ion has fewer tendencies to swell due to stronger divalent bonding between clay surfaces. The calcium clays tend to aggregate into tactoid (clay platelets stacked parallel to each other); its stability is independent of the electrolyte concentration.

2.2.7 Dispersion

Dispersive soils are susceptible to aggregate break down due to their tendency to swell and then disperse forming clay suspensions. Dispersion is caused when a more diffuse cation distribution occurs between negatively charged between particles and resulted in mutual repulsion between particles. Consequently, it leads to structural degradation such as surface crusting, reduced water infiltration and hydraulic conductivity (HC), runoff, erosion and ultimately limited plant establishment and growth. High pH, extensive amount of hydrated monovalent sodium, and low electrolyte concentration in bulk solution all significantly enhance the extent of dispersion.

Flocculation is the opposite process of dispersion. It allows coagulation of the clay particles into microaggregates by compressing the electric double layer. This process is necessary for the formation and stabilization of soil structure. Gypsum also causes flocculation indirectly which is an adverse process that compressed the electric double layer and allow coagulation of the clay particles into microaggregates (Chen and Dick, 2011). This process is necessary for the formation and stabilization of soil structure, thus increase water infiltration and percolation. Furthermore, formation of surface crusts and the strength of surface crusts on drying can be altered by the use of gypsum amendment, thus improved crop and pasture yields.

2.2.8 Clay mineral and aggregate stability

In semiarid regions, the clay minerals are dominated by smectites, illite and vermiculite. These minerals are mainly permanent charged due to isomorphous substitution uniformly distributed over the planar surface. Anderson (1968), Oster (1982) and Shainberg *et al.* (1989)have reviewed the relationship between these clay minerals, sodium absorption ratio (Cassel Sharmasarkar *et al.*), flocculation value and electrolyte concentration from various studies. Vermiculite and llite have higher selectivity of absorbed sodium than montmorillonite (smectites), therefore, flocculation values required for each clay minerals vary significantly under same sodium adsorption ratio (Shainberg *et al.*, 1980, Rhoades, 1967). For soils containing a mixture of clay minerals that include montmorillonite dispersion increased with increasing increments of exchangeable sodium absorbed on external surfaces. The swelling between external surfaces is enhanced and weakens interparticle bonds and, in turn, enhancing the freedom of adjacent soil particles to move, in other words disperse.

2.2.9 Effect of Electrolyte concentration on dispersion

Although sodicity is often a consequence of a period of prior saline soil conditions, it is harder to eliminate from soil than salinity (Oster, 1994). Hydraulic conductivity, K, of several soils with different soil mineralogy with small swelling tendency were reduced after irrigated with distilled water (Frenkel *et al.*, 1978, Pupisky and Shainberg, 1979). Consequently, pore blockage and clay migration are promoted after reduction of K. Electrolyte concentration (EC) has positive impact in preventing soil dispersion with certain SAR. The concept of threshold concentration is introduced (Quirk and Schofield, 1955). It is the concentration required to maintain less than 10 to 15 % decrease in soil permeability at a given ESP value. Beside the EC value of the soil, the susceptibility of soil to soil degradation at a given EC and ESP value, increased with an increase in swelling clay, soil bulk density, and clay content (Frenkel *et al.*, 1978, McNeal and Coleman, 1966, McNeal *et al.*, 1968). Soils with high sesquioxides content are more resistant to sodic condition (McNeal *et al.*, 1968); it expressed the importance of clay mineralogy in threshold concentration at a given SAR.

Felhendler et al. (1974) studied the hydraulic conductivity (HC) of a sandy loam and silt loam soil as a function of the SAR and EC of the bulk solution. They found that HC is only slightly affected in both soils by the SAR of the bulk solution up to 20 when the EC value exceeded 10 mmol l^{-1}. However, HC decreased dramatically after they reduced the EC value of salt solution to distilled water level (1 mmol l^{-1}). While, calcareous silt loam has higher reduced HC ranged from 42 to 18 % of the initial HC than non-calcareous sandy loam soil with lower than 5% of initial HC value. Consequently, increased in mobility of clay was observed in the sandy loam soil but not in the silt loam soil. HC of a low ESP soil was appreciably reduced by leaching with distilled water to remove the salts and reduce EC (Shainberg *et al.*, 1981a). The adverse impact of ESP of up to 10 on HC on this soil was prevented by the percolating solution having and EC of 2 mmol l^{-1}. Percolation solution with EC 3 mmol l^{-1} prevented the adverse effect of 15 % Na on the exchange complex. These examples show how dispersion and associated permeability of soils are affected not just by the sodicity levels (ESP/SAR) but also the electrolyte concentration of the soil water (EC).

Shainberg *et al.* (1981b) reported that the EC of solution composition of a calcareous soil at a given ESP is ranged between 2 and 3 mmol l^{-1}, when placed in contact with distilled water. They hypothesized that sensitivity of sodic soils to solution with low EC is dependent on the rate of salt released from soil by mineral dissolution, which causing different responses of various sodic soils to the low electrolyte leaching water. Distilled water will not easily cause dispersion of moderate sodic soils if they containing minerals that readily release soluble electrolytes because high EC is maintained in the soil solution. Conversely, sodic soils with limited amount of readily release soluble electrolytes are more susceptible to low EC water and thus dispersion more readily occurs.

2.2.10 Gypsum responsive soil

Gypsum does not benefit all soil types. Soils suffer from sodicity, acidic subsoils, poor drainage- clayey soils, Ca and S deficiencies that are most likely benefit from gypsum amendment. Furthermore, benefits from gypsum are mostly indirect to soils, except adjustment of nutrient deficiency, thus significant improvement may only occur after a few years of continuous application (Watts and Dick, 2014).

As discussed above, some soils are resistant to soil dispersion due to presence of readily release soluble electrolytes in soil. Conversely, Shainberg *et al.* (1982) reported that these soils with extensive soluble electrolytes have very low response to the gypsum amelioration. The result indicated that non-calcareous Golan soil, a highly weathered Rhodoxeralf, occur under 1000 mm of annual water, was highly sensitive to the type of Ca amendments added (CaCl2 and CaSO4). The HC is maintained at high level with application of gypsum. Another studied soil is calcareous Naha-Oz soil. Ca amendments do not have significant effect on the HC of this soil. Shainberg *et al.* (1982) indicated the electrolyte effect brought by gypsum is soil type dependent. The two main ameliorate effects of gypsum – cation exchange and electrolyte effect are working on the clay component of the soil, thus, the clayey texture soils should have better response to gypsum treatment.

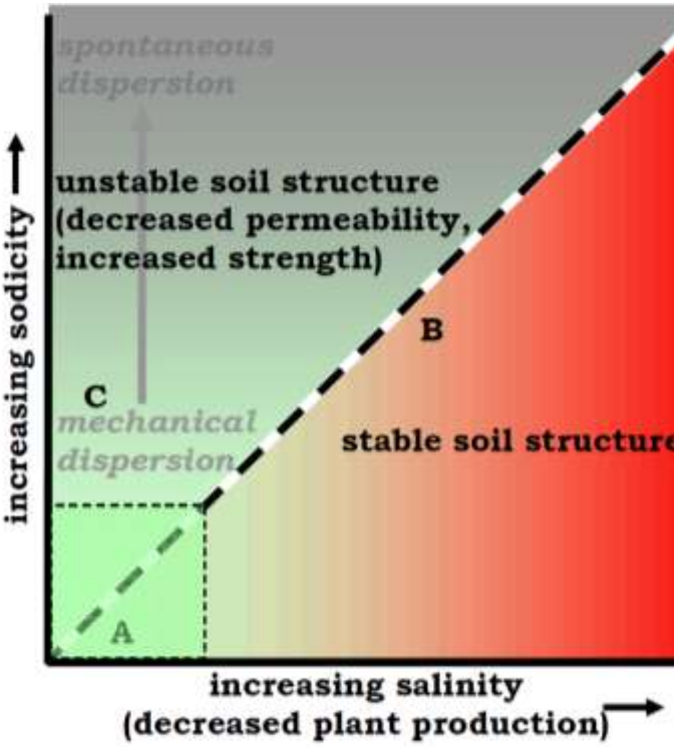


Figure 2.2 Impact of interaction of sodicity and salinity to soil structure (Murray and Grant, 2007).

2.2.11 Soil amelioration by using gypsum

Both EC and cation exchange effects are the two main means to prevent potential risk of soil degradation of sodic soil by using gypsum as amendment (Loveday, 1976). If the cation exchange effect can prevent clay dispersion and swelling of soil, and flocculation of soil, the amount of gypsum required depends on the amount of exchangeable Na in a selected depth of soil. Conversely, if the electrolyte effect is more important than the cation exchange, the amount of gypsum required to ameliorate the soil depends on the quantity and quality of irrigation water and the dissolution rate of gypsum.

Since gypsum is applied through SDI system in this study, thus, effect of gypsum on infiltration rate, soil crusting, surface soil sealing and surface runoff are not discussed here. However, by improving soil condition of the subsoil should also indirectly relief the pressure of these soil degradation issues to some extent. Reduction in swelling and dispersion are directly promoted by gypsum, in turn, improvement of structural stability, HC, soil tilth, drainage, permeability, porosity and soil strength indirectly achieved.

Gypsum improves or maintains the permeability of soil to allow water movement in the soil profile by increasing the electrolyte concentration in the soil solution, meanwhile substitutes the exchangeable Na with Ca. By comparing result of two studies of actual EC of soils with gypsum treatment and without gypsum treatment, EC of gypsum treated soil is at least 3-fold to 16-fold higher than EC of soils without gypsum treatment (Oster, 1982, Quirk and Schofield, 1955).

McIntyre *et al.* (1982) reported that HC in the upper profile of soil was increased after gypsum treatment. Leaching of soluble salts is promoted by gypsum treatment due to increase in extra profile drainage. Their results indicated that 10 tonnes of gypsum per hectare increased the infiltration rate 3 fold for ponded water experiments to 2 m depth. They also showed chloride salts were leached to 1 and 3 m respectively in the untreated and gypsum treated soils

2.2.12 Gypsum amendment and acidic soil

Soil acidification and leaching of nitrate are observed in orchard under SDI system (Stork *et al.*, 2003, Haynes, 1985, Vázquez *et al.*, 2006). Devasirvatham (2009) suggesting that it is the result of over-irrigation, greater drainage and fertigation. The level of acidity tend to increase with soil depth.

Lime is well known in ameliorate acidic soil by increasing soil pH, meanwhile, its ability to reduce the extensive level of exchangeable Al by converting it into less active Al precipitates as hydroxyl-Al polymers. However, effectiveness of liming is often limited to topsoil due to the low solubility of lime. Gypsum is 200 times more soluble than lime, which make it more available in subsoil. Shainberg *et al.* (1989) and Sumner (1990) have reviewed the amelioration effect of gypsum on the acidic soil. Potential mechanisms that promote soil amelioration are studied (Sumner, 1990).

The role of calcium plays in root elongation is studied in Hanson (1984). Sufficient calcium available at the zone of elongation is essential for healthy root growth. Hanson (1984) pointed out the inverse relationship between exchangeable Ca and Al. However, whether Ca deficiency is the main reason restricting root growth in acidic subsoil is unclear. Ion pair formation is said to be the primary effect of gypsum which reducing Al toxicity (Pavan *et al.*, 1982). The sulphate in the gypsum has greater ability to compete with the hydroxyl anion and forms less toxic form of $AlSO_4^+$. $AlSO_4^+$ is not soluble and is readily leached from the root zone by deep percolation. Soil degradation in acidic soil are observed after short period of liming and gypsum application (Haynes and Naidu, 1998, Roth *et al.*, 1988). Acidic soil normally is greatly flocculated due to high available Al activity in soil solution promoted compression of the double layer and flocculation of clays. Activity of available Al is decline with gypsum as it is converted into Al precipitates as aluminium sulphate minerals. However, long-term gypsum treatment promoted higher aggregate stability and infiltration rate (Haynes and Naidu, 1998, Roth *et al.*, 1988). The biological activity was improved, resulting from improved Ca and P nutrition, the compensation effect is more than enough to cover negative effect due to the stabilization of aggregates by organic matter.

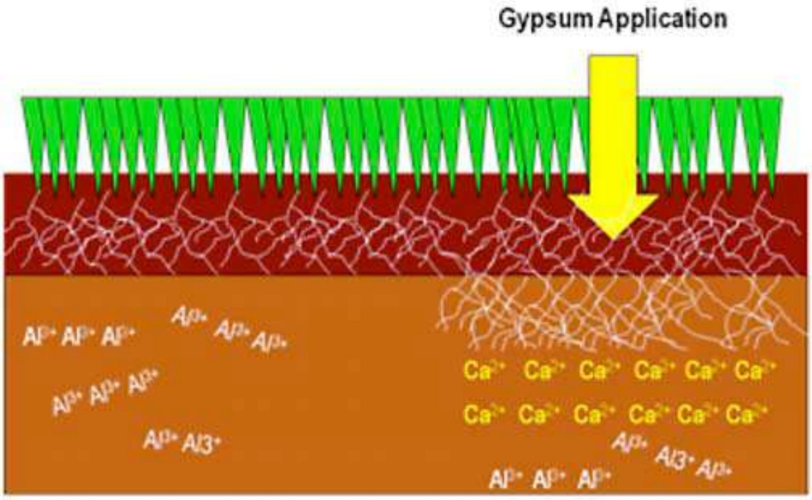


Figure 2.3 Root growth is restricted by Al toxicity in acidic soil (left); root elongated to deeper soil as exchangeable Al leached to deeper profile due to amelioration effect gypsum treatment (right). (Chen and Dick, 2011)

2.2.13 Laser diffraction

LD is one of the recently developed methods (electro-resistance particle counting, time of transition, image analysis) for grain-size analysis (Goossens, 2008). These methods are advanced in generating a wide range of grain sizes but low time-consuming, and less quantity of samples is needed in comparison to traditional PSD measurement (sedimentation methods).

When a light strikes a particle, the angle of light scattered by particle is inversely proportional to particle size. The intensity of the scattered light at any angle represents the number of particles with a specific cross-sectional area in the optical path. LD utilises light intensity received by detectors located at different position around the reaction room to calculate particle size distribution. As the size of particles can vary widely in samples, LD commonly uses two types of monochromatic light - red and blue, which are different in wavelength for PSD.

Fraunhofer theory and the Mie theory are commonly used for calculation of particle sizes from light intensity sensed by detectors (Gee and Or, 2002). However, the Mie theory is preferred as Fraunhofer theory was found often overestimated clay content and becomes inapplicable when the particle's diameter is close to the wavelength of light. In order to use Mie theory, knowledge of refractive index (RI) is required. However, Soil is comprised of various types of materials. Each of them could have different RI; furthermore, different soils are different in composition and proportion of components.

Some assumptions were made for LD method (Konert and Vandenberghe, 1997):

1. The light source comes only from one direction during measurement, therefore, transformation of scattered light to grain size is based on the light scattered from a cross-sectional area of particles;
2. The particles were assumed to be spherical;
3. The particle orientation is assumed to be randomly distributed, though particles may be oriented according to its shape in the continuous suspension.

The use of LD method for aggregate stability test raised the question of accuracy of aggregate distribution measured by LD machine. Buurman *et al.* (1997) pointed out that LD tend to underestimate the clay content compare to sedimentation methods and the error increased with increasing clay content. However, Campbell (2003) found opposite result when studying the accuracy of measurement with mixture of different portion of fine and coarse particles. Therefore, the variation is rather dependent on the properties of clay content than quantity of clay. These studies suggested that irregular shape of particles is the major factor causing underestimation of smaller particles. The particles are assigned to larger size fractions of PSD when the light is scattered by larger cross-sectional of particles.

However, sedimentation method will also affect by the sphericity of particles and causing overestimation of clay content (Di Stefano *et al.*, 2010). Konert and Vandenberghe (1997) concluded that results of both methods were comparable for blocky quartz but vary significantly for platy clay particles. Therefore, the difference between both methods' PSD is not necessarily from underestimation of LD method.

During aggregate disintegration in LD, the shape of aggregate will probably change randomly and never has perfect spheres, and changes between soil types and within same subsamples. Furthermore, clay/silt size aggregates will increase with length of the test. Therefore, underestimation or overestimation is likely to increase with duration of testing. The dynamics of soil aggregation is the most powerful feature that LD could achieve that other traditional methods lack. Meanwhile, it raises the difficulty in comparing the different between samples.

There are only a few numbers of publication of aggregate stability test by using LD method but they reveal the limitations that are awaiting to be resolve. Mason *et al.* (2011) studied dynamic of seven soil samples with 2 pre-treatments in detail (14 samples in total). The study is more observational rather than statistical as there were lot of data from 1 samples. Conversely, wet sieving will only have 1 data from 1 soil.

Bortoluzzi *et al.* (2010) and Bieganowski *et al.* (2010) compared aggregate stability result from LD and traditional wet sieving. Bortoluzzi *et al.* (2010) studied effect of lime on medium textured Acrisol and found improvement in large size aggregate of lime treated soil in LD result (observational) and no significant different in wet sieving method. Bieganowski *et al.* (2010) presented soil disintegration dynamic of the median of aggregate size distribution (median decreased with treatment time- expotential curve). Although the result is still observational, it raised the potential to compare the rate of disintegration from the median. The wet sieving result was not positively related to the organic carbon content of three studied soils, possibly due to similar organic carbon content in three soils (2.7-3.1 %). Both studies could not find any relation between soil stability and organic matter content which is the major factor determining aggregate stability in wet sieving. Nevertheless, aggregate stability studies by using LD do show different in aggregate size distribution, though the soil properties that determine the stability were still remained unknown.

Furthermore, the LD method is advantaged in highly repeatable and reproducible, and less time consuming than traditional methods.

2.3 Conclusion

Cropping with the SDI system can result in benefits from high WUE, crop yield and less management requirements than other irrigation methods. However, soil degradation is likely to occur with application of SDI systems.

Water content of soil under SDI system commonly ranges between field capacity and refill point (normally 100kpa) in cropping season. Soil has less dry periods which is important for natural soil amelioration by creating cracks and voids after shrinkage. Water always enters the subsoil through the emitters with an initial high rate. Prolonged wetness and water injection are likely to cause mechanical soil degradation, such as slaking, swelling and dispersion, and thus, result in reduced hydraulic conductivity and permeability, increased soil strength and compaction, migration of clay minerals and soil pore blockage.

Soil dispersion firstly depends on the clay mineralogy of soil; in turn, EC and SAR of soil. In America, soil is classified as sodic with ESP value of 15, however, Australian soil can suffer from severe physical degradation with ESP as low as 6 if the EC is low in soil. It is found that the EC value of the water source vary significantly. The role of EC in soil dispersion is thus critical. However, soil with high EC values have adverse impacts on crop yields. Parida and Das (2005) reviewed the responses of plants to salinity stress. Soil management should Soil's sodic level and natural rainfall in consideration. From **Figure 2.2**, migration of clay of naturally non-sodic soil in Barber *et al.* (2001) can be explained by the mechanical dispersion posted by the stress from low EC water wetting events. Gypsum can be a potential solution by increasing EC, and thus, minimize soil degradation by mechanical dispersion.

Gypsum is a great nutrient source of calcium and sulphur. They are presented as plant available form. Gypsum treatment increases the electrolyte concentration and available Ca in the soil solution. Gypsum can directly prevent swelling and dispersion and indirectly, in turn, improve structural stability, HC, soil tilth, drainage, permeability, porosity and soil strength. Supplement of available calcium is not only a good source of nutrient, it also acts as a barrier to push available sodium and aluminium into deeper soils by leaching and thus reduces the level of sodium and aluminium toxicity. However, the effectiveness of gypsum treatment is highly dependent on clay mineralogy and content, soil conditions such as present of soluble electrolyte, sodium and aluminium level and water quality.

3 Soil structure assessment under Subsurface Drip Irrigation (SDI)

3.1 Introduction

The previous review indicated that there was limited or none published study of liquid gypsum. There were extensive study about surface drip irrigation on soil structural degradation but limited study about SDI system. Most of the SDI study discussed the benefits brought by SDI system. This study was conducted to investigate whether the particular liquid gypsum product (GYP-FLO) is an economically worthy product for soil amelioration and fertilization; and whether soil degradation was the consequence of continuing negative impact of long term SDI practise.

3.2 Methods and Materials

3.2.1 Site selection and trial preparation

The field experiment was conducted on Carinya Farm, which is located between Echuca and Rochester in Victoria (36°15'30" S 144°42'13" E). The total area of the trial site is 9.34 hectare and has four years of SDI practise history.

The trial was consisted of two liquid gypsum (Gyp-Flo) treatments, control and high (150 L/ha), with the completely randomized block design. The tomato transplant was sown on one meter width bed on 16th October. Each treatment was comprised by three rows. The edge rows, the two outer rows, of each treatment were the buffer between the two adjacent treatments. The liquid gypsum (Gyp-Flo) was diluted one part to a minimum of five parts of irrigation water during injection by pressure sprayer. Injection of high rate treatment was separated into two equal parts and applied on two separate dates. The first 75 L of high rate treatment was injected on 6th November, another 75 L of high rate treatment was injected on 11th December. Irrigation schedule also depends on the seasonal weather variations, plant growth stages and evapo-transpiration. The distance between adjacent emitters is 0.5 m with discharge rate of 1.65 L/h.

Figure 3.1 Displays the view of field site of gypsum trial

Block 1		Block 2		Block 3		Block 4	
Control	150L/ha GYP-FLO	150L/ha GYP-FLO	Control	Control	150L/ha GYP-FLO	150L/ha GYP-FLO	Control

Figure 3.2 Field design of the liquid gypsum trial

3.2.2 Soil Collection

The soil was sampled after six weeks of liquid gypsum treatment. Unfortunately the site had irrigation just before sampling due to a miscommunication with farmer. The sampling points were randomly selected on the middle row of each treatment. The drip-lines were typically buried at depth of 25 cm. The samples were collected at 5 cm below the drip-lines. Five samples were collected from each treatment and the selection is based on the distance from the emitter. The selected sampling locations were directly under an emitter, at right-angles out from that emitter; at both 22.5 cm and 45 cm and between the emitters (at 25 cm) and then at a diagonally extreme point, being furthest from adjacent emitters, where the likely SDI impact would minimal (see figure below).

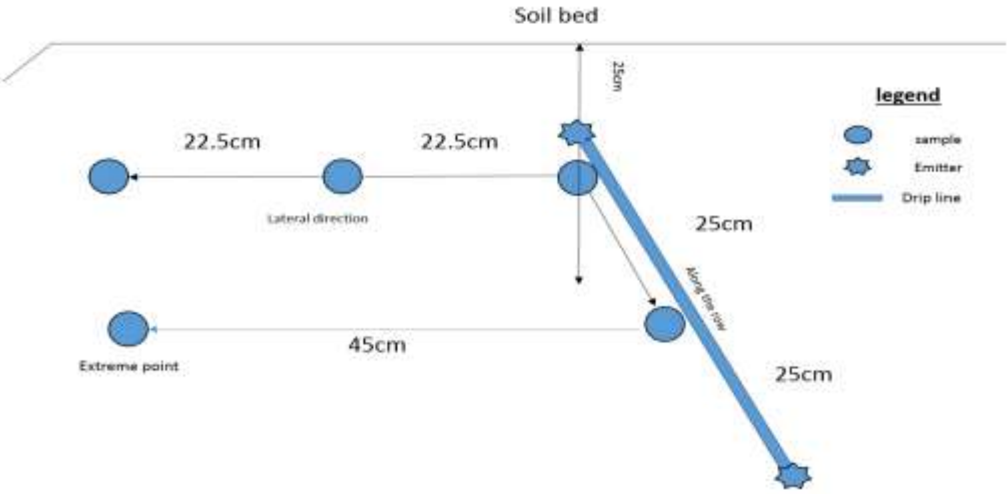


Figure 3.3 Design of sampling for study of impact of SDI to soil away from emitter. The drip-line is located at depth of 25 cm from surface. The samples from five locations were collected from same depth.

3.2.3 Soil preparation before analysis

The bulk soil samples were air-dried in oven at 40 degrees. The air dried samples were ground by using Van Gelder Crushers. The opening of the crusher was approximately between 1 and 10 mm. The breakdown aggregates were separated into two portions, which one was sieved for chemical properties analysis and another was sieved for physical properties analysis.

The chemical portion was hand sieved by using a 2 mm sieve. Representative samples were obtained by breaking down large aggregates (>2 mm) with hand-crusher (metal pestle) on the sample before being sieved at 2 mm.

For the physical portion, samples were dry-sieved on the EFL2 Mk3 shaker. The aggregate classes obtained from dry-sieving were >4 mm, 2 – 4 mm, 1 – 2 mm and <1 mm.

Figure 3.4 EEL2 mk3 Shaker.

Figure 3.5 The opening of the crusher hopper showing the smallest gap (left) and largest gap-opening (right).

Figure 3.6 The sequence of crushering of the soil samples. 1) Bulk samples air-dried (left), 2) Crushing hopper and 3) Aggregate samples after crushing (right)

Figure 3.7 Ground aggregates were further broken-down by hand-crushing for chemical properties analysis with a metal pestle

Figure 3.8 Hand sieving for aggregate samples size below 2mm (chemical portion)

3.2.4 Emerson Dispersion and Remoulded Test (Emerson, 1967)

According to McKenzie *et al.* (2002), large size air-dried aggregates (5 – 7 mm) were dropped into distilled water. There were three replication from one subsample. Degree of dispersion and slaking were recorded according to the Emerson dispersion classification after 2 hours and 20 hours.

Large size air-dried aggregates were remoulded into approximately 5 mm cube with spatula. Three replication were made from one subsample. More than half of samples (40 subsamples) were remould in first day and remains were mould in second day. All remoulded samples were rewetted by drops of distilled water (~field capacity) before put into bottles of distilled water. Degree of dispersion were recorded according to Emerson dispersion classification after 20 hours.

3.2.5 Wet Sieving

The method was modified from Amezketa *et al.* (1996) and Yoder (1936). 4 g of 1 – 2 mm air-dried aggregates were wet-sieved upon three sizes of sieves – 1 mm, 0.5 mm and 0.25 mm. The wet-sieving machine has 10 sets of sieving units and so can conduct 10 tests at once. Three replication were made from each subsample. The samples were pre-wetted by immersing the samples in distilled water for five minutes. After that, samples were wet-sieved in water with 1.5 cm of vertical oscillation for 10 minutes. The speed was set as 36 oscillation cycles per minutes. The aggregates retained on each sieve were oven-dried at 105^{o}C for a day.

Mean weight diameter was calculated from the proportion of aggregates retained on each sieves and passed through the sieves (0 – 0.25 mm, 0.25 – 0.5 mm, 0.5 – 1 mm and 1 – 2 mm).

3.2.6 Particle Size Analysis (PSA)

Particle size distribution of samples at two locations were completed; 1) under the emitter and 2) between two adjacent emitters. Four pairs of samples were selected from the four field blocks. The particle size distribution was measured using the 'Mastersizer 2000' attached with the 800 ml 'Hydro 2000G'. The refractive index and particle absorption index used were 1.52 and 0.01 respectively. Pump speed, stirrer speed and ultrasonic level of 2000 rpm, 800 rpm and 100% were inputted during measurement.

4 g of 1 – 2 mm aggregates were mixed with 40 ml of distilled water and 8 ml of sodium hexa-meta-phosphate. The suspensions were treated with 30 kHz ultrasonic bath for 30 minutes and lastly were shake more than 24 hours in end-over-end shaker.

The samples were diluted and riffled into 16 equal parts using 800 ml ‘Hydro 2000G’. In each measurement, two parts are required to maintain optimum obscuration range (10-20%). Measurement were made over four replications. Each replication has eight repeated measurement in one run. The results of eight repeated measurements were averaged to form result of one replication. Field results were made by averaging four replications and location results were made by averaging four field replication.

3.2.7 Chemical properties

1 – 2 mm soil aggregates were sent to DEPI Chemical lab for chemical properties analysis. EC and soil pH were tested in 1:5 soil: water suspension; CEC (Ca, Mg, Na, and K) were tested by either Tucker method or Ammonium acetate method. Methods selected for cations analysis were dependent on the soil pH and. ESP and Ca: Mg ratio were calculated from cation exchange capacity (CEC).

3.2.8 GYP-FLO analysis

GYP-FLO samples were extracted from five batch of products. Two of them were sent to SWEP Analytical laboratories (Victoria) and three of them were sent to Mark Wainwright Analytical Centre (New South Wales). Total Sulphur and Calcium were tested. Different methods were used by two lab.

Table 3.1 The methods that were used for GYP-FLO analysis by SWEP lab and Mark Wainwright.

	SWEP LAB TEST 1	SWEP LAB TEST 2	MARK WAINWRIGHT
Sulphur	HCl evaporation, ICPAES	Perchloric/ Nitric acid digestion (PN)	Leco CNS analyser
Calcium	HCl evaporation, ICPAES	Perchloric/ Nitric acid digestion	XRF

3.2.9 Statistical analysis

The comparison between GYP-FLO treatments and between locations treatments were analysed by ANOVA analysis function of statistical program- SPSS 22.

Analyze> general Linear Model> Univariate

3.2.10 Chemical properties

The result of chemical properties indicated that the soil type from Field Block 3 is quite different from the soil type from the other blocks. Soil pH (water), electrical conductivity (EC) and exchangeable calcium content vary significantly between soil from Field Block 3 and the soil in the other blocks. Therefore, Field Block 3 was excluded from all soil analysis as it is obviously not comparable with soil from other blocks.

Table 3.2 EC, exchangeable calcium content and soil pH (water) range of soil from Field Block 3 and other Field Blocks (1, 2 and 4)

Chemical properties	Block 3	Other blocks
pH (water)	7.6 – 8.7	6.0 – 7.0
EC (ds/m)	0.21-0.3	0.11-0.20
Calcium content (meq/100g)	17 – 27	7 – 9.6

Chemical properties of each block were tested without making any replication due to budgetary limitations. The statistical result is not reliable without sample replication. The block effect of each tested property was tested. Result indicated that block effect has no significant influence ($p>0.1$) on the most of the properties except the sodium content. Therefore, field replications were considered as the sample replications due to similarity from all block. The statistical result of sodium content was tested based on block design.

Overall, most of the chemical properties from the control treatment were higher than the 150 L/ha GYP-FLO treatment for soil pH, EC (salinity), exchangeable cations (Ca, Mg, Na, K), CEC and ESP (Exchangeable Sodium Percentage), while the Ca:Mg ratios were lower. GLY-FLO has significant impact on Mg content, CEC and Ca/Mg ratio. Soil pH and EC of the soil are within the tolerant ranges of 6 – 7.5 and <0.9 dS/m. The control soil is high in all exchangeable cations. However, the high sodium value resulted in sodicity and there is also an imbalance in calcium to magnesium ratio. Soil after the GYP-FLO treatment, the soils showed an improvement indicated by reducing ESP values ($p>0.05$) and an increase in the ratio between calcium and magnesium ($p<0.05$).

Results from different locations indicated that four years of SDI practise on the site there is an increase in cations away from emitter, excluding potassium. Among all tested properties, changes in the EC, pH and exchangeable sodium were significant. Although the difference between other properties were not statistically significant, they also showed the same cation leaching patterns as soil chemical properties were significantly different between locations. The chemical properties of samples were always higher at 22.5 cm toward bed edge from emitter than the samples at between two emitters although both were about equal distance away from emitter. The result showed that the extreme point had the highest values as it was the farthest place away from probable leaching effects of the emitters.

Table 3.3 Comparison between GYP-FLO treatments and between soil locations. The control treatment tend to have higher value in every tested properties than 150L/ha GYP-FLO treatments. The chemical properties and MWD tend to increase with distance away from emitter except the potassium content. ($p>0.05$= NS; $p<0.05$= *; $p<0.01$= **; Duncan test subset= abc).

Treatment	**MWD ^{NS} (Lamm)**	**EC ^{NS} (dS/m)**	**pH ^{NS} (water)**	**Ca ^{NS} (cmol(+)/kg)**	**Mg ** (cmol(+)/kg)**	**K ^{NS} (cmol(+)/kg)**	**Na ^{NS} (cmol(+)/kg)**	**CEC * (cmol(+)/kg)**	**Preferred <6 ESP ^{NS}**	**Preferred >2 Ca/ Mg ratio ***
150L/ha	0.406	0.147	6.95	8.14	8.18	0.59	1.17	18.07	6.13	1.05
control	0.445	0.161	7.18	8.47	10.28	0.60	1.45	20.60	6.83	0.833
locations	**MWD ^{NS} (Lamm)**	**EC ** (dS/m)-**	**pH ** (water)**	**Ca ^{NS} (cmol(+)/kg)**	**Mg ^{NS} (cmol(+)/kg)**	**K ^{NS} (cmol(+)/kg)**	**Na ** (cmol(+)/kg)**	**CEC ^{NS} (cmol(+)/kg)**	**Preferred <6 ESP ***	**Preferred >2 Ca/Mg ratio ^{NS}**
under emitter	0.412	0.135^{a}	6.40^{a}	8.15	8.50	0.64	0.73	18.00	3.97^{a}	0.96
22.5cm away from emitter	0.427	0.142^{a}	6.98^{ab}	8.33	9.52	0.56	1.18	19.50	5.85^{a}	0.88
between adjacent emitters (25cm)	0.437	0.148^{a}	6.70^{a}	8.20	9.17	0.60	0.97	18.67	5.00^{a}	0.89
45cm away from emitter	0.451	0.170^{b}	7.53^{bc}	8.37	9.28	0.61	1.80	20.00	8.79^{b}	0.90
extreme	0.401	0.177^{b}	7.70^{c}	8.47	9.68	0.56	1.86	20.50	8.79^{b}	0.88

3.3 Results

3.3.1 Wet sieving results

The location effects and Liquid GLY-FLO application effects were not significant (**Table 3.3**).

A practical issue was found during wet sieving test. When the sieve columns were placed into the water, there was a possibility entrapment of an air bubble under the mesh. The air bubble was likely to stay at the same place as long as the sieves were oscillated vertically. This air entrapment created a barrier between soil above the bubble and upward water when the sieve was moving downward. Thus, the soil aggregates above the air bubble would not receive much disruption energy when the sieves were moving downward. **Figure 3.9** shows the consequence of the air bubble effect after the sieving. The aggregates that were above an air trap were not properly being sieved and remained on the sieve. The size of aggregates on the top were visually smaller than the sieve size which indicated that the aggregates did receive forces and breakdown but was not able to pass through the sieve due to the blockage. The reason, bubble's size and chance of trapping a bubble were still unknown. However, the sieve was more likely to trap an air bubble when it was still wet. Those results with aggregates assembled together (**Figure 3.9**) were discarded and retested.

Figure 3.9 The aggregates that were above a trapped air bubble, could not pass through the sieve due to blockage of pathways. The aggregates assembled did breakdown by applied energy and the sizes were visually smaller than the sieve size (<1mm).

3.3.2 Particle size distribution

The PSD results indicated that the samples sampled between emitters were higher in portion of smaller sized particles than samples sampled under emitter. In **Figure 3.11,** the PSD shifted from right- the average result (n=3) of samples from under emitter to left- the average result (n=3) of samples from between emitters. **Figure 3.10** shows the detailed differences in PSD between two locations by separating particle size into groups. Samples from between emitters were always more commonly in particle size groups that were less than 45 µm, between 90 – 110 µm and 650 – 1500 µm. Samples from under the emitters were extremely high in particles size groups between 200 – 650 µm

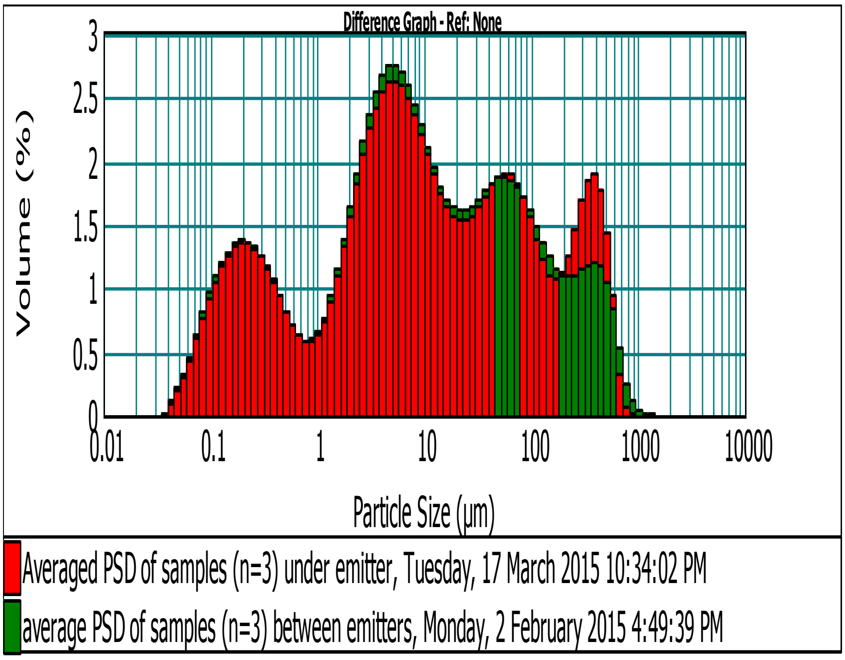


Figure 3.10 Comparison of average particle size distribution (PSD) of samples from two locations in histogram form (n=3, replication=4). Samples sampled between emitters have higher portion of small size particles than samples sampled under emitter.

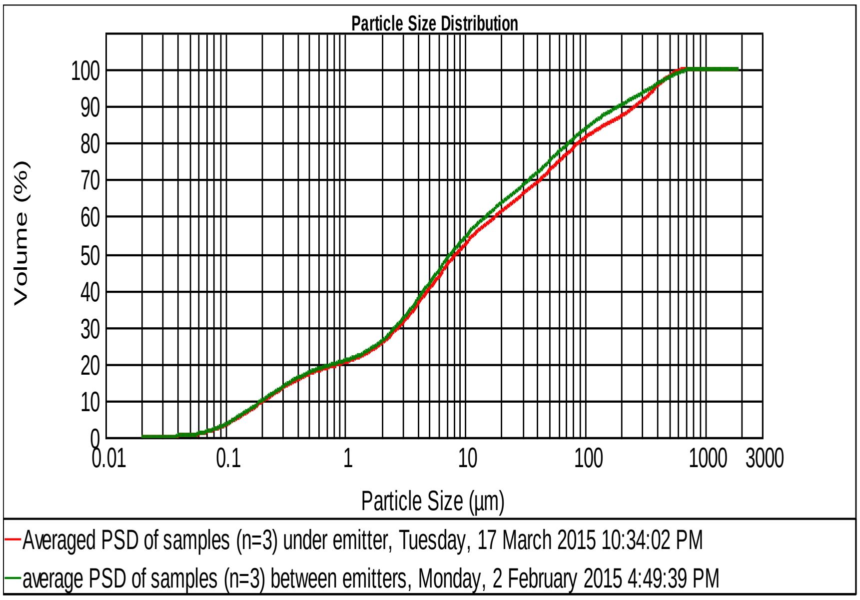


Figure 3.11 Average accumulative particle size distribution (n=3, replication= 4) of samples from two locations.

3.3.3 Dispersion test

For the 5 mm aggregates dispersion test, all tested samples were severely slaked. The aggregates collapsed into either angular form or smaller rounded form which are the class 2 and class 3 slaking respectively (reference).

Samples from the extreme point and 45cm toward bed edge were suffered from severe dispersion. The dispersion classes ranged between nil and dispersion of more than 50% of aggregate. This was followed by samples from 22.5 cm towards bed edge, which ranged between slightly dispersed and less than 50% dispersed. Samples sampled under emitter and between emitters were the most stable aggregates in the test. The dispersion class ranged between nil and slightly dispersed. The remoulding test has similar result trend as Emerson test, though samples from between emitters were more susceptible to dispersion and has same dispersion ranges as samples from 22.5 cm toward bed edge.

The samples from GYP-FLO treated soil were more stable than control samples. The differences were obvious by comparing samples from same location (**Table 3.4**).

Table 3.4 Location effects and Liquid GYP-FLO application effects on Emerson Dispersion Tests (n=3; 4 replications). Nil= slaking, not dispersed; 1= slightly dispersed; 2= obvious dispersion but less than 50% is affected; 3= obvious dispersion and more than 50% is affected.

Emerson test- 24 hours									
Under emitters		22.5 cm toward bed edge		45 cm toward bed edge		Between emitters		Extreme point	
Control	150 L/ha	Control	150 L/ha	Control	150 L/ha	Control	150 L/ha	Control	150 L/ha
Nil	Nil	Nil-2	Nil-1	1-3	Nil-3	Nil-1	Nil-1	1-3	Nil-3

Remoulding test- 24 hours									
Under emitters		22.5 cm toward bed edge		45 cm toward bed edge		Between emitters		Extreme point	
Control	150 L/ha	Control	150 L/ha	Control	150 L/ha	Control	150 L/ha	Control	150 L/ha
2-3	Nil	2-3	1-3	3	3	2-3	1-3	3	3

3.3.4 GYP-FLO

The total sulphur and calcium content measured by three methods were overall lower than the content stated on product flyer. Thus, the lower contents were not caused by underestimation of using inappropriate methods. The calcium content measured by using perchloric/Nitric acid digestion and XRF analysis were similar to the recommended content. It was suggesting that the tested products did contain moderate quantities of calcium but were very low in sulphur content being only a fifth of that specified in my samples, at 5.7%, and only in one of the product distributor's samples which showed 19.7% S, still 5% below the level stated on the label. This highest test samples was determined on an older product by using LECO CNS analyser, while the data range from 13.9 to 19.7%.

The moisture content of the product were about 60% w/v and the density of the product was 1.6 kg/L which is equivalent to 160% w/v. The sum of the moisture content. Sulphur and Calcium carbonate of the five samples were approximately 150% w/v for batch a, b, c, and d; and 160% w/v for batch e.

Table 3.5 The results of GYP-FLO test by using different methods and the standard content stated on product flyer. (Number of sample batch= $_{abcde}$). The density- 1.6kg/L; moisture content- 38~39%, which is equivalent to 60 (w/v).

Component	**Product flyer**	**Perchloric/ Nitric acid digestion (W/V)**	**Leco CNS analyser (W/V)%**	**XRF analysis (W/V)%**
Sulphur	25%	4.98_a, 5.71_b	19.68_c, 18.72_d, 13.86_e	-
Calcium	35%	33.92_a, 33.12_b	-	28.64_c, 34.72_d, 30.72_e

3.4 Discussion

3.4.1 Product description

According to the written product statements made by the manufacturer it is implied GYP-FLO is a form of liquid gypsum. However, gypsum is not the primary ingredient of the product. In fact calcium carbonate and elemental sulphur are the two main component of GYP-FLO. The label states the product has equivalent of approximately 35% w/v of calcium and 25% w/v sulphur respectively. Apparently the mechanism for deriving the 'liquid gypsum' is that elemental sulphur will be oxidized to sulphate in soil and followed by substitution reaction with calcium carbonate and thus converted into gypsum. However, the analytical result indicated that both calcium and sulphur content in the five tested samples were variable and significantly lower than the content told by product description.

The sum of the major components of the GYP-FLO samples were approximately 150% w/v out of the 160%w/v in total, i.e. the liquid S.G. 1.6. There were something unknown, which occupied 10% w/v of the sample. This product is unlikely to contain the stated 25% and 35% w/v of sulphur and calcium (from calcium carbonate) respectively if the density (SG) and moisture content is 1.6 kg/L and 38% w/w respectively, as the total of these major component will exceed 160% w/v.

3.4.2 GYP-FLO as alternative gypsum fertilizer

Assuming that the product does contain 25% w/v of elemental sulphur, the effectiveness of the GYP-FLO is conditional as it is indirectly provides gypsum based on the oxidation of elemental sulphur to plant available form sulphate. Freney (1967) reviewed the conditions favoured oxidation of elemental sulphur in soil. Among the conditions, the concentration and type of the microorganism existed in the soil is the fundamental factor that pushing sulphur oxidation. If the sites have limited population of autotrophs- *Thiobacillus* group or recently fumigated are not likely to response to the GYP-FLO application. Furthermore, the oxidation rate of the sulphur was suggested to be moderate (10-70%) in different soils after 2 months of incubation experiments (Nor and Tabatabai, 1977, Vitolins and Swaby, 1969).

Therefore the “gypsum effect” and sulphur supplement brought by the product was not likely to occur immediately after application.

3.4.3 GYP-FLO effect

Based on the reasoning discussed above, calcium carbonate in the GYP-FLO is the only component that is likely to cause difference between the GYP-FLO treatments in the trial. Although wet sieving result did not show significant difference between GYP-FLO treatments, the Emerson test indicated that the samples after GYP-FLO treatment were more stable than the samples from control treatment. This can be explained by the substitution and leaching of the exchangeable magnesium and sodium by calcium from the lime supplied in the GYP-FLO, this is known to occur in sites rich in exchangeable magnesium (Bohn *et al.*, 2002). Therefore, ESP was lower in samples from GYP-FLO treated soil and also higher in Ca:Mg Ratio. Application of some lime products can increase both exchangeable calcium and magnesium in soil as dolomitic lime products also contain certain amounts of magnesium (Fageria *et al.*, 2014, Chatzistathis *et al.*, 2015, Meiwes *et al.*, 2002, Mosquera-Losada *et al.*, 2012). However, there was only trace amount of magnesium in GYP-FLO. Injection of GYP-FLO enriched the total calcium concentration in the soil and continuously pushing the substitution of exchangeable magnesium with calcium from the charge site. This process can be slow as calcium carbonate is not soluble in high soil pH, however, Chatzistathis *et al.* (2015) reported that exchangeable calcium and Ca:Mg ratio increased dramatically in neutral soils after lime application.

3.4.4 Impact of SDI on soil properties

According to the soil physical and chemical properties, the results of the different location treatments, were indicated that the clay and exchangeable cations (Ca, Mg, and Na) might translocate away from emitter with the irrigation water. Furthermore, the soil was initially low in Electrical Conductivity. The literature has discussed the positive the impact of EC on reducing soil dispersion. Therefore, the low EC of the study soils and medium to low ESP values (4-8%) might make them susceptible to dispersion. The Emerson result confirmed this. As a consequence it seems some dispersion of clays and very fine particles (silts) could migrate and block the conductive pores in the soil, particular away from the emitters. (Helalia *et al.*, 1988, Pupisky and Shainberg, 1979, Bodman and Harradine, 1939).

Meanwhile, increases in the percentage of finer particles at great distances from emitter was correlated with incremental increases in exchangeable calcium, sodium and magnesium. However these limited and preliminary findings were not statistically significant, there trend is not unexpected. They indicate that Ca, Mg, and Na might be leached away from emitters with irrigation water, along with fine particles (clays and silt).

Increases in exchangeable sodium content away from the emitters was the most pronounced of the three cations, indeed they doubled at the farthest location. Also the Ca:Mg ratio decreased with the distance away from emitter showing that the increases in exchangeable magnesium were greater than that of calcium. These phenomena suggest that incremental changes in the exchangeable cation contents away from emitters, in all treatments, were primarily positively related to number of valent of the cation and secondary negatively related to the area of charge on outer hydrated surface of the cations (hydrated radii are Na^+ = 0.36, Mg^{2+} = 0.43 and Ca^{2+} 0.41 nM)

In both the GYP-FLO treatments and the location treatments there were no significant differences in wet sieving results. However, the Emerson tests did show that dispersion level of aggregates and remoulded cubes were positively related to the distance away from emitter. This suggested that the minor cation and ESP differences in the samples were not large enough to cause differences in the wet sieving results at the different locations.

These are interesting somewhat contradictory findings and may suggest different mechanisms driving each test. The 24 hours Emerson test may better allow the expression of the changes in the ESP and Ca:Mg ratios. While the shorter (10 min) wet sieving might only be reflecting the coarser particle-SOM bonds and water entry dynamics and hence not allowing expression of the key difference in the soils, i.e. the cation levels and ratios.

3.5 Conclusion

Sulphur and calcium content of the gypsum samples did not meet the product description provided by the manufacturer. It is unlikely to have 25% and 35% of sulphur and calcium respectively, based on the result of the moisture content and product density.

The availability of GYP-FLO as sulphur or gypsum amendment could not be confirmed as the sulphur ingredient is not plant uptake available form. The efficiency of GYP-FLO is conditional. It is dependent on the characteristic of microbial population, soil temperature and pH, and particle size distribution. The conversion of elemental sulphur to plant available form takes months. Therefore use of GYP-FLO ought to be made in the knowledge it is calcium carbonate and sulphur, not gypsum, and so ought to be applied during pre-season for tomatoes, so that soil has sufficient time to convert the elemental sulphur to plant available form sulphate.

Subsurface Drip Irrigation (SDI) might potentially causing clay and exchangeable cations to translocation away from the the emitters. Extent of cation translocation is species dependent. It was found to be primarily positively related to number of valent of the cation and secondary negatively related to the area of charge on outer hydrated surface of the cations. As a consequence the decreased proportion of exchangeable calcium, and accumulation of dispersed clay and exchangeable sodium resulted in increment of dispersion class of aggregates in Emerson test. Clay migration may lead to reductions in hydraulic conductivity (not measured) as they would block the conductive pores of soil.

Finally it is suggested aggregate testing always include the longer Emerson test if cation ratios are relevant to structural stability of the soils under consideration.

4 Comparison of aggregate stability test of wet sieving and Laser Diffraction technique

4.1 Introduction

Aggregate stability of twelve samples were measured by using traditional wet sieving and a Laser Diffraction (LD) based particle sizing machine. The twelve samples covered a wide range of SOC content (0.9 – 3.0%). Results of LD measurement were compared to the SOC of the samples and results of traditional wet sieving of aggregates. This study was aimed to evaluate the correlation between the aggregate stability measurements using LD method and wet sieving methods. It also allowed examination of the impact of SOC on aggregate stability results from both methods and SOC content.

4.2 Methods and Materials

4.2.1 Soil samples

Six paired soil samples collected from different locations across Victoria were selected for this study. The six pairs were previously studied to investigate the impacts of soil organic matter on traditional water stable aggregation. Each pair from the six soil types were sampled from different soil organic matter treatments. One sample from each of the pairs had a higher organic matter input compared to the other sample of the pair. At each site, soil samples were taken from 0 – 10, 10 – 20, and 20 – 30 cm depths. Soil organic carbon contents were provided from the previous study by Vic-DEPI. This prior work was undertaken using 4 g of standard size aggregates (2 – 4 mm) for wet-sieving test. The current study used 1 – 2 mm aggregates for both wet sieving and laser diffraction tests.

Table 4.1 Comparison of average soil carbon content and average water stable aggregates (n=3) in previous study.

SiteID	OM_ID	Water stable aggregates (%)	Soil Carbon %
2	H	85.4	2.39
2	L	67.7	1.90
5	H	73.5	2.21
5	L	51.3	1.63
6	H	84.3	3.03
6	L	89.8	2.33
7	H	49.6	0.90
7	L	64.6	0.91

12	H	84.1	1.44
12	L	83.9	1.36
14	H	55.7	1.20
14	L	72.9	1.14

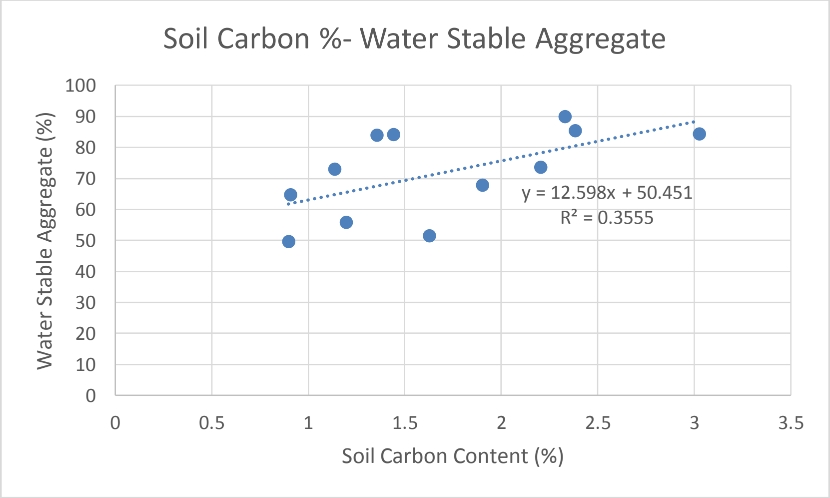


Figure 4.1 Correlation between average soil carbon content and average water stable aggregates. However, the regression analysis of raw samples was not significant (n=36, p>0.05).

4.2.2 Wet sieving

The method was modified from Amezketa *et al.* (1996) and Yoder (1936) with 4 g of 1 – 2 mm air-dried aggregates being wet-sieved through sieves of 1 mm, 0.5 mm and 0.25 mm mesh diameter. The wet-sieving machine has 10 holders so 10 tests can be conducted simultaneously (**Figure 4.2**). Three replications were made from each sample. These subsamples were pre-wetted by immersing in reverse osmosis (RO) water for five minutes. After that, samples were wet-sieved in RO water with 1.5 cm of vertical oscillation for 10 minutes. The speed was set as 36 oscillation cycles per minutes. The aggregates retained on each sieve were oven-dried at 105^{o}C for 24 hours.

Water stable aggregate (WSA) was calculated as the portion of aggregates above 0.25mm after 10 minutes of sieving. Mean weight diameter (MWD) was calculated from the proportion of aggregates retained on each sieves (w_i) and the mean diameter of each aggregate size group (Wang *et al.*) (0 – 0.25 mm, 0.25 – 0.5 mm, 0.5 – 1 mm and 1 – 2 mm).

$$MWD = \sum_{i-1}^{n} x_i \, w_i$$

Figure 4.2 Tradition aggregate wet sieving machine

4.2.3 Laser diffraction (LD)

The dynamic aggregate disintegration was measured using the 'Mastersizer 2000' attached with the 800 ml 'Hydro 2000G' dispersion unit (**Figure 4.3**). The refractive index and particle absorption index used were 1.52 and 0.01 respectively. Pump speed and stirrer speed of 2000 rpm and 800 rpm were applied during measurement. The background signalling of the dispersant, distilled water was tested for 30 seconds. Each repeated measurement was 13 – 14 seconds, which consisted of four seconds of red light measurement followed by four seconds of blue light measurement and few seconds of alignment and delay.

The 1 – 2 mm air-dried aggregates were immediately dropped into the 'Hydro 2000G' after measurement was started. The amount of aggregates used for each run ranged between 0.5 and 1 g depending on the recommended instrument parameters, namely 'obscuration' (discussed below). Each run consisted of 90 consecutive measurements. Changes of aggregates size distribution over time were recorded to compare the dynamic disintegration between different soil types.

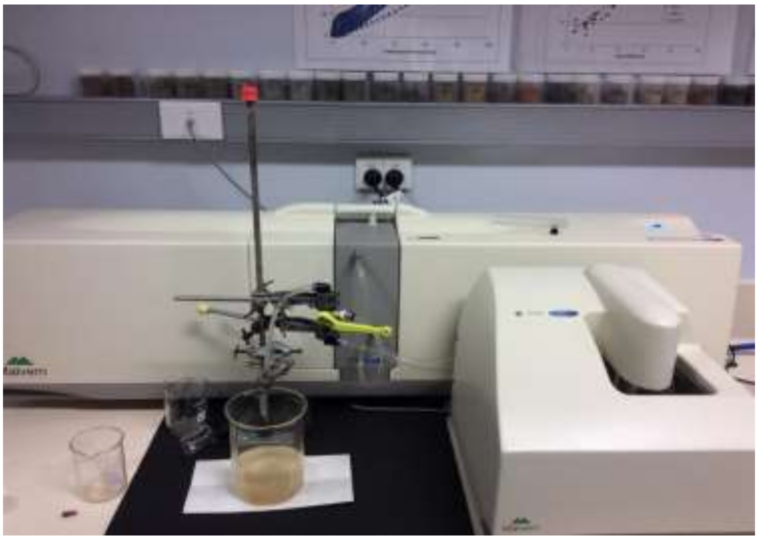


Figure 4.3 Laser diffraction particle sizing machine

4.2.4 Analysis of Laser diffraction result

The aggregate stability was measured by comparing changes of median and changes of Mean Volume Diameter (MVD) over consecutive measurements.

The median represents the size at which 50% of the volume population is below this size and 50% of the volume population is above this size.

Mean Volume Diameter (MVD) is modified from the concept of Mean Weight Diameter. MVD is calculated by dividing the particle size distribution into a number of size categories, then multiplying the middle size value of category (Wang *et al.*) by the proportion of volume in that particular size group (v_i). The value from all size groups are then summed up to get MVD. The advantage of using MVD is all aggregates from different aggregate size classes are included. The average size of aggregates from one defined group was assumed to be the middle of that size group. The aggregate size groups used were 0 – 25, 25 – 250 (micro- aggregate), 250 – 500, 500 – 1000 and 1000 – 2000 µm.

$$MVD = \sum_{i-1}^{n} x_i \, v_i$$

Obscuration is a measure of the proportion on light in the main beam that does not reaches the end detector due to scattering or adsorption. It is a measure of the soil concentration in the optical window, and needs to be maintained within limits if data quality is not to be reduced. Some of the samples have obscuration values lower than 10% in the first repeated measurement which is outside the manufactures recommended range. However, our results with obscuration value lower than 10%

still had good light scattering signal compared to background signal, which indicated that the results were reliable and were not interrupt by weak scattering of light due to low numbers of aggregates. The disintegration of 1 – 2 mm aggregates over time formed a nonlinear curve. However a linear equation was fitted for the first four consecutive measurements over time.

$y = ax^b$------- Nonlinear curve equation

$y = mx + c$ ------ Linear equation

The parameters from the curve and linear equations were compared to the soil carbon content and the results from wet sieving.

4.2.5 Statistically analysis of regression

The significant level of regressions between any two factors were analysed by using

Statistical program- SPSS 22.

Process linear regression- Analyze> Regression> Linear

Non-linear regression- Analyze> Regression> curve estimation

4.3 Results

4.3.1 Wet sieving

The result of wet sieving from the prior DEPI study were presented as WSA while the current study was using MWD. Therefore, MWD and WSA, of the present study, has to be correlated to prove that both results are comparable. **Figure 4.4** confirms that MWD and WSA are strongly correlated (R square= 0.91).

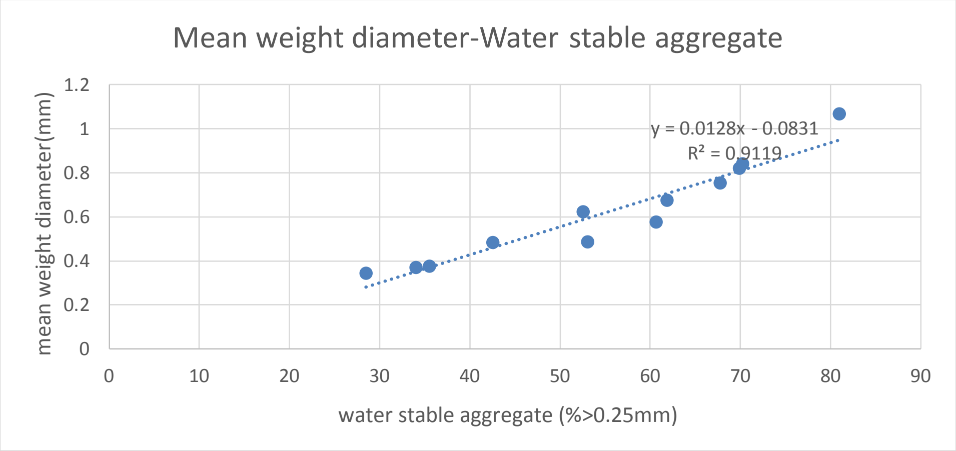


Figure 4.4 MWD and WSA are strongly correlated (n=36, p<0.01).

The aggregate stability (MWD & WSA) and soil carbon content were moderately correlated (R square= 0.45-MWD & 0.46- WSA). However, aggregate stability was more carbon dependent in this study than the results from previous study (R square= 0.33).

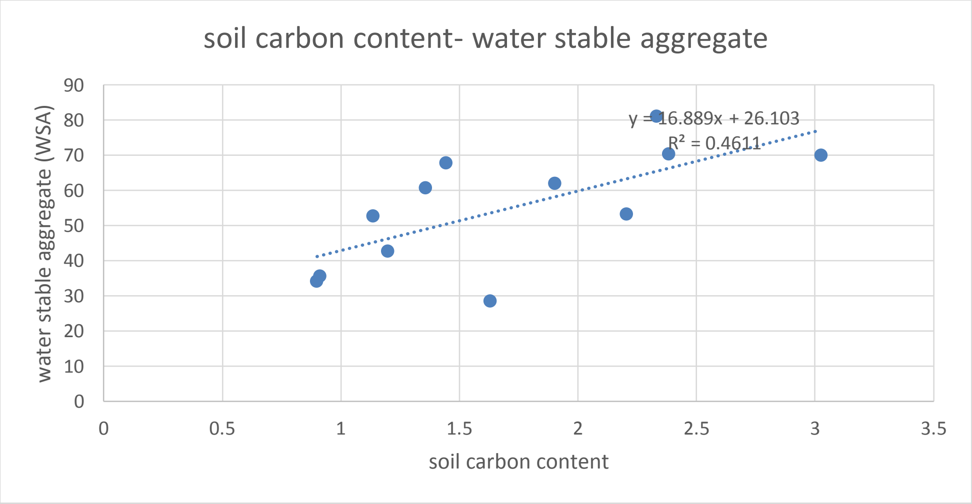


Figure 4.5 Average Soil Carbon Content and average Water Stable Aggregate were positively related (n=36, $p<0.01$).

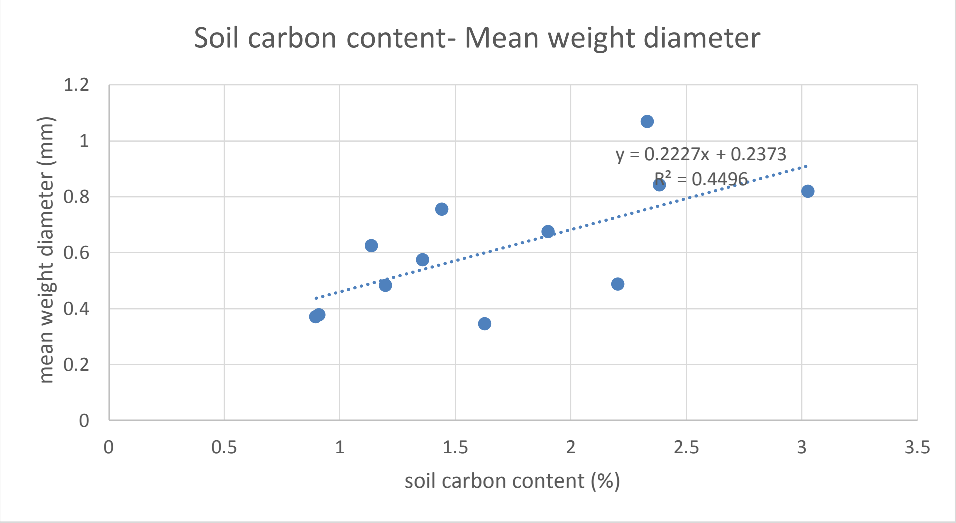


Figure 4.6 Average Soil Carbon Content and average Mean Weight Diameter are positively related (n=36, $p<0.01$).

4.3.2 LD – Obscuration calibration

In aggregate stability testing with Laser Diffraction the length of the test is one of the most important factors that needs to be standardised. Low obscuration was hard to avoid in the initial measurements as the sample contained larger aggregate sizes and was thus a lower number of aggregates causing low obscuration values. However,

Figure 4.7 indicates that the samples with low obscuration have good light scattering signals compared to background signals. It is thus a reliable measure of aggregate sizing.

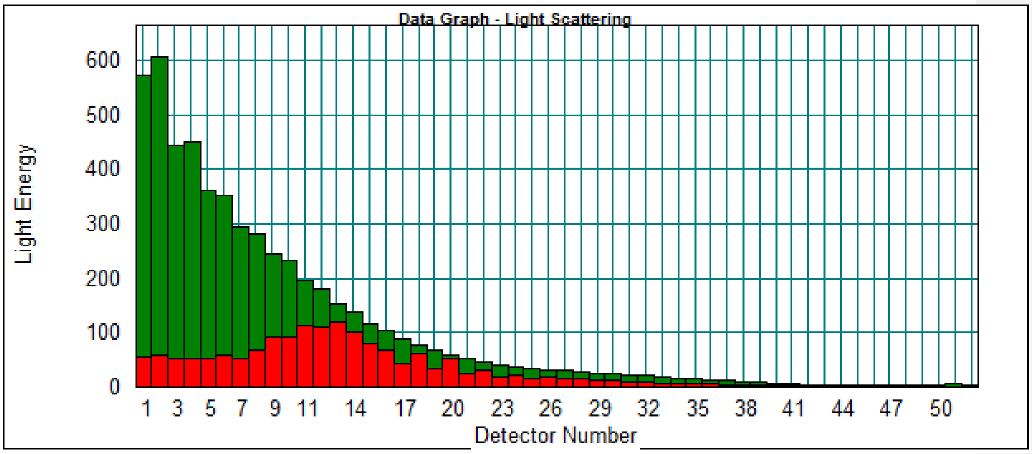


Figure 4.7 One of the result of comparison of the light scattering pattern between sample's signal (Mason *et al.*) and background signal (Lamm) in the initial measurements (obscuration= 5.68%).

Inversely, disintegration of aggregates causes the obscuration value to increase. The test was suspended and the suspension was diluted until the obscuration value approached 20%. Bieganowski *et al.* (2010) showed that dilution of the suspension during measurements did not affect the dynamic of disintegration. (Example: See **Figure 4.8** & **4.9**).

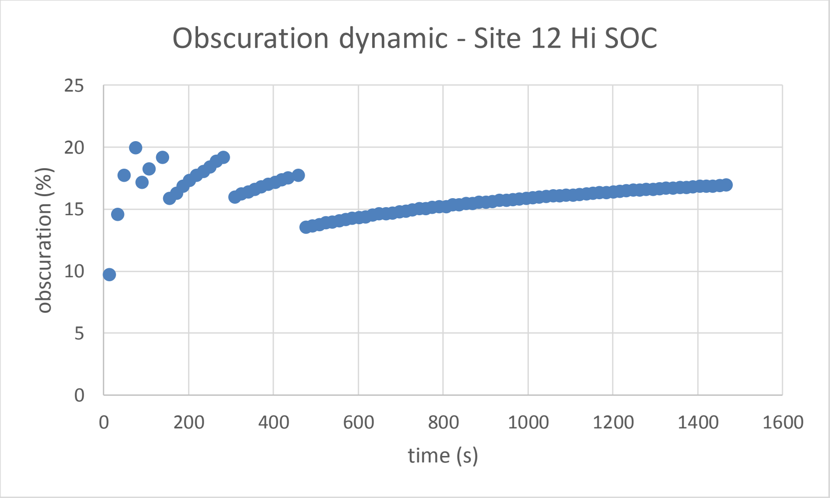


Figure 4.8 The suspension was diluted when the obscuration was approaching 20% and thus the obscuration was reduced. There was 4 times dilution shown in the figure. **Figure 4.8** & **4.9** were plotted from same source.

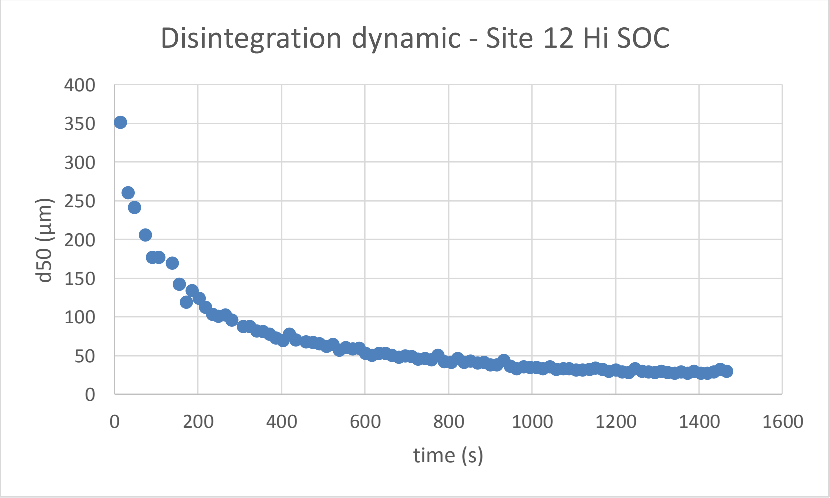


Figure 4.9 Change of median was not affected by dilution by referring Figure 5. **Figure 4.8** & **4.9** were plotted from same source.

4.3.3 Dynamic disintegration

LD results from Site 14 were excluded from this study as they showed randomly fluctuating d(90) over time (**Figure 4.10**). The d(90) results of other samples were similar to the trend of the median particle size. This may indicate that the samples from Site 14 contain large aggregates that were not disintegrable by LD measurement, such as gravel, but this has not been confirmed.

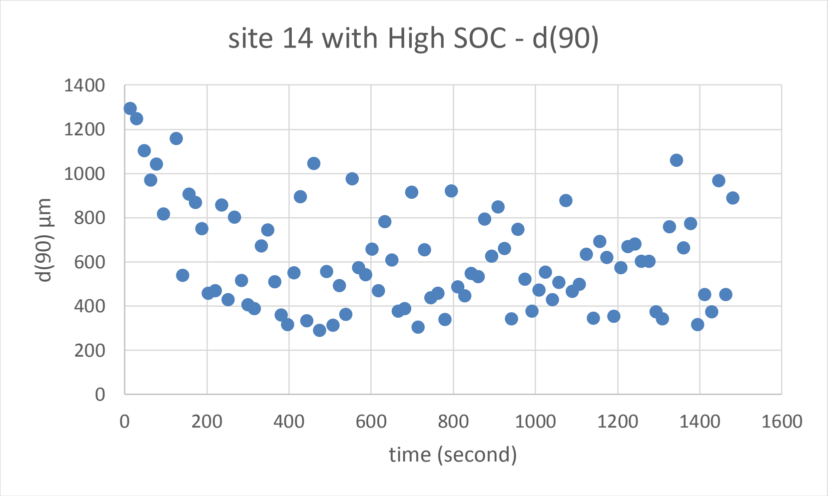


Figure 4.10 Abnormal d(90) trend of site 14 with high SOC.

According to **Figure 4.11**, curve of dynamic disintegration can be separated into two phases. In Phase-1, disintegration of aggregates was rapid and the curve declined exponentially. With time the aggregate disintegration approached a quazi-stable phase following the initial rapid period of aggregate disintergration (in both Median and MVD). We call this the beginning of Phase-2. Disintegration rate of Phase-2 was not equal or close to zero, but the acceleration of the disintegration rate was slowed down. The MVD, Median and Soil Organic Carbon were arranged in order in **Table 4.2**. In comparison the differences between orders, results of samples from Site 7 were highly unrelated to soil organic carbon (<1%). In wet sieving result, both samples from Site 7 have the lowest MWD. Therefore, MVD and Median of Phase-2 were unlikely to show good relation with SOC.

Comment [PF1]: Don't think this is the right word, may be "a linear phase"

In Phase-1, rate of disintegration could barely be distinguished by plotting disintegration curve of 10 samples in one graph (**Figure 4.11**). The linear equation was plotted from first four repeated measurement of complete measurement over time. The parameters of the linear equation were compared to the SOC and Aggregate Stability result of Wet Sieving (discussed in next section).

Table 4.2 The aggregate stability units- median and MVD of samples in phase two and Soil Organic Content (SOC) of samples were arranged in order. (H = high SOC; L = low SOC)

	Decreasing >>>>>>									
SOC	6 H	2 H	6 L	5 H	2 L	5 L	12 H	12 L	7 L	7 H
MVD	6 H	7 H	6 L	2 H	2 L	7 L	5 H	5 L	12 H	12 L
median	6 H	7 H	6 L	7 L	5 H	2 H	12 H	2 L	5 L	12 L

In comparison Median and MVD of samples from same site, the curves of MVD and Median of high SOC samples were always above the curves of low SOC samples, excluding the comparison between samples from Site 6. The difference between curves were little for most of the site but some were obvious (**Figure 4.12**). The difference of dynamic Median between high and low SOC soils from Site 7 were the largest and the difference of dynamic MVD between high and low SOC soils from Site 2 were the largest. The Median and MVD of high SOC sample from Site 6 were lower than Median and MVD of low SOC sample during Phase one. However, high SOC sample started to lead the dominant after entering Phase-2 as the disintegration rate of low SOC sample was relatively higher in Phase-2.

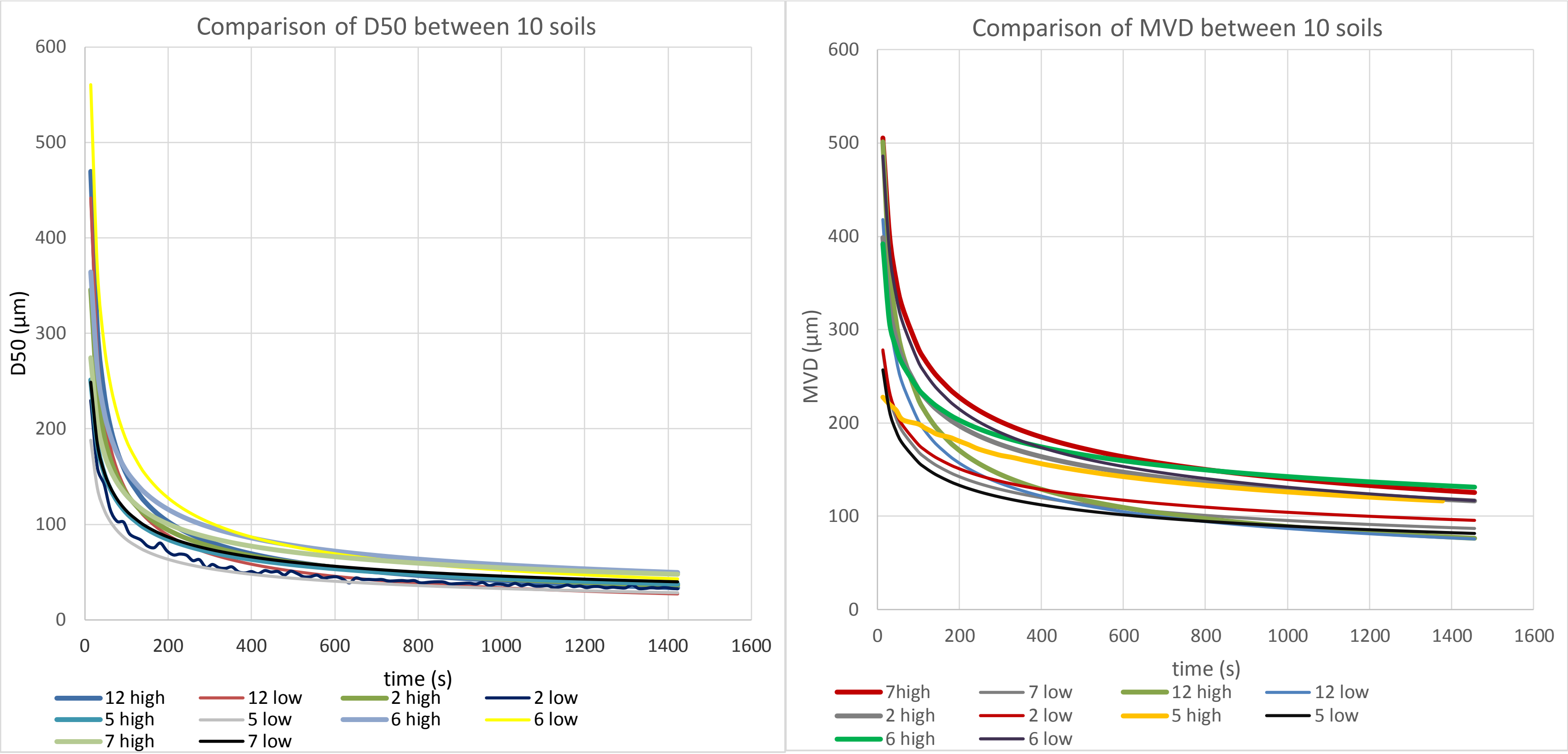
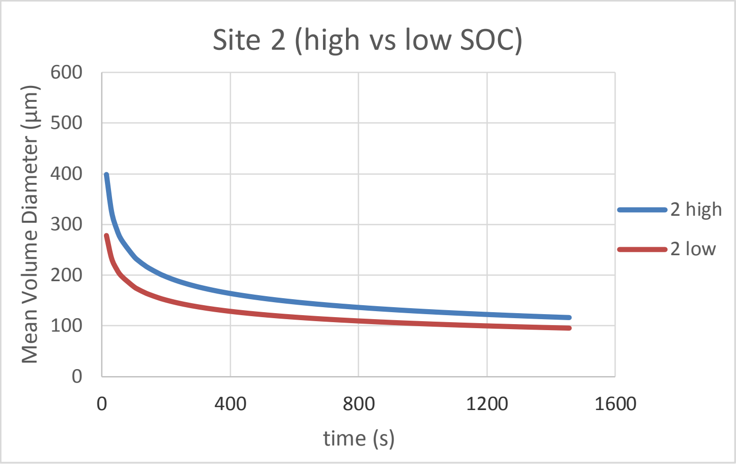
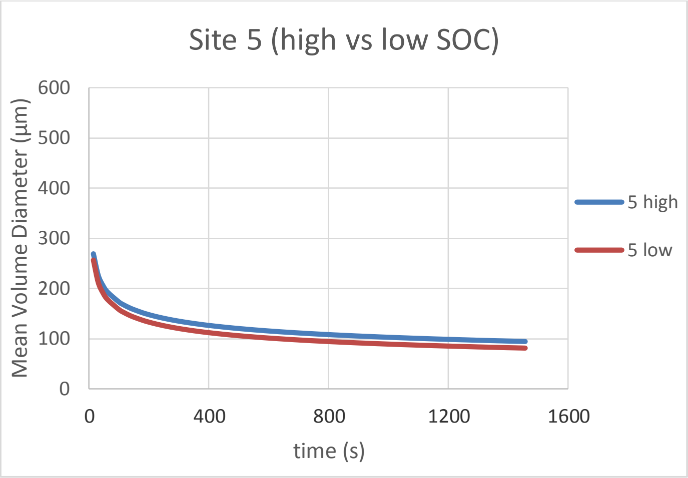


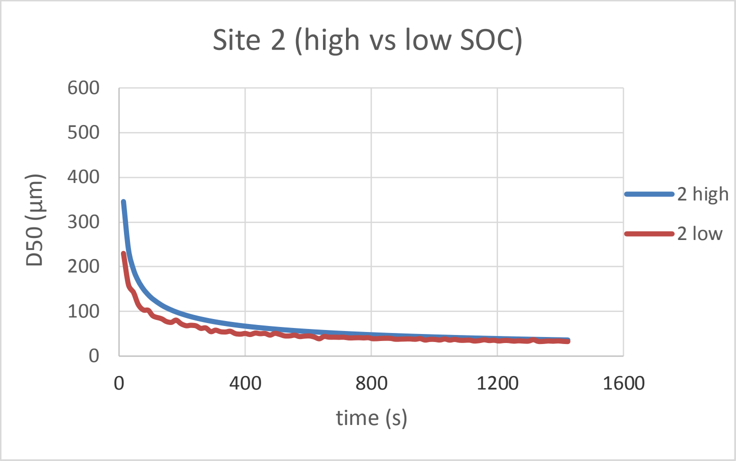
Figure 4.11 Comparison between both graphs indicated that MVD were always higher than median when the aggregate disintegration was approaching stabilization as MVD takes multiple groups of aggregate into account rather than 50th percent of population. (Bieganowski *et al.*, 2010)

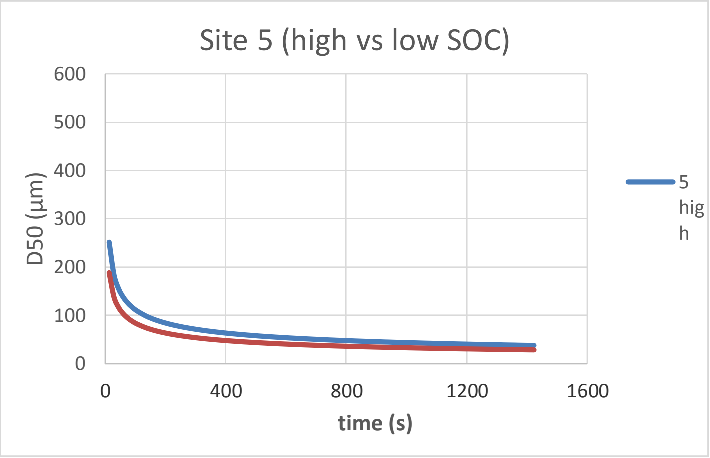
MVD


Site 2 (high vs low SOC)
Mean Volume Diameter (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
2 high
2 low

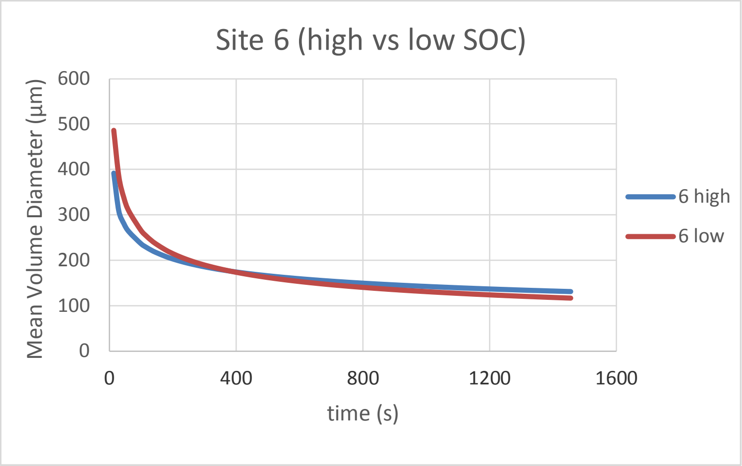

Site 5 (high vs low SOC)
Mean Volume Diameter (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
5 high
5 low

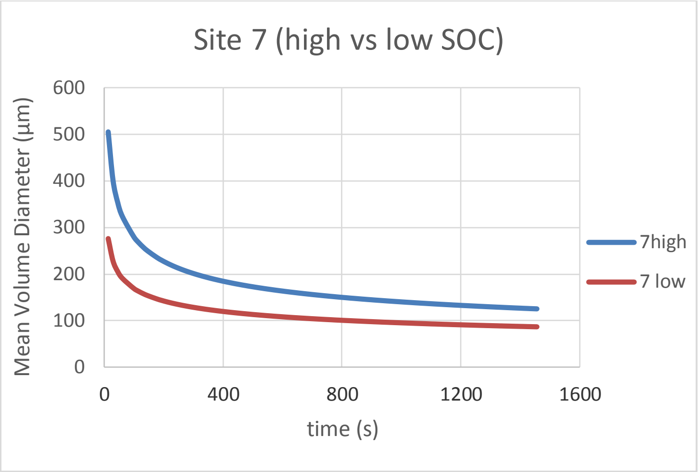
Median


Site 2 (high vs low SOC)
D50 (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
2 high
2 low

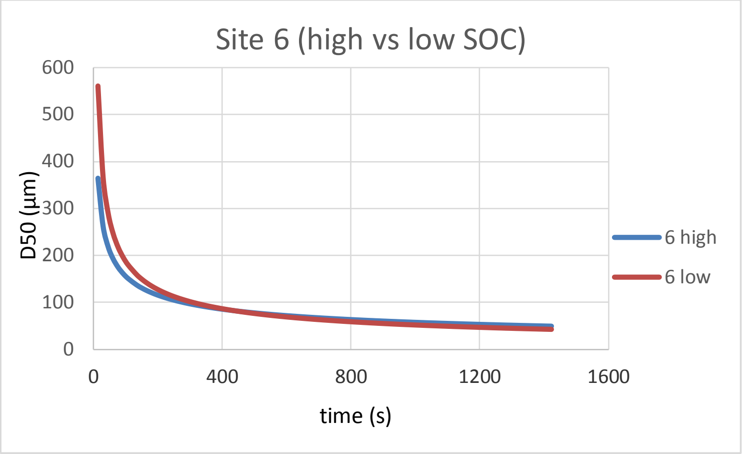

Site 5 (high vs low SOC)
D50 (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
5 high

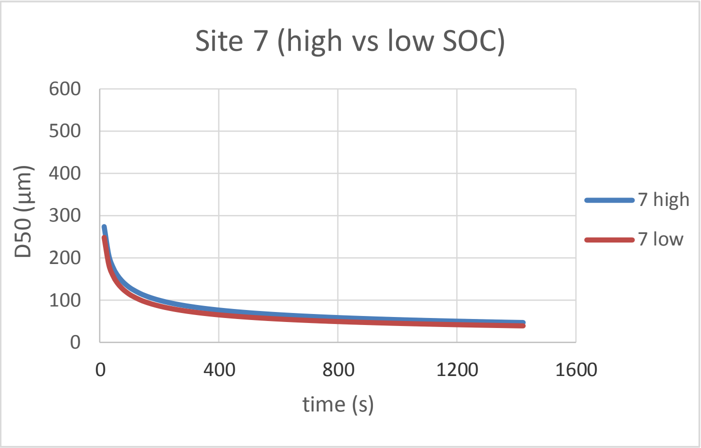
MVD

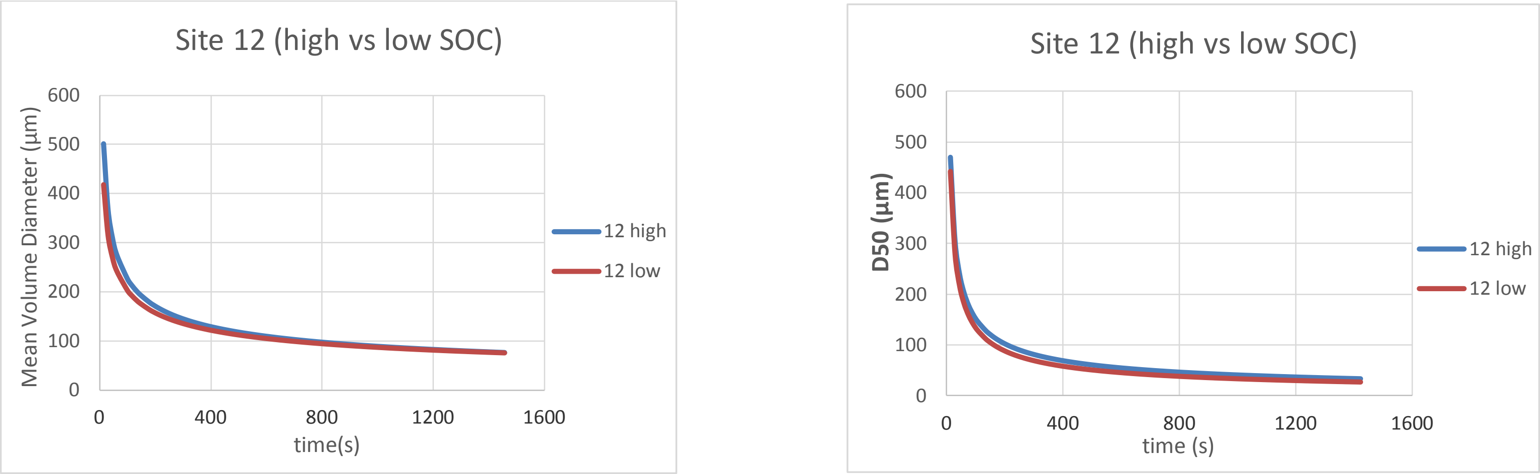

Site 6 (high vs low SOC)
Mean Volume Diameter (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
6 high
6 low


Site 7 (high vs low SOC)
Mean Volume Diameter (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
7high
7 low

Median


Site 6 (high vs low SOC)
D50 (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
6 high
6 low


Site 7 (high vs low SOC)
D50 (µm)
600
500
400
300
200
100
0
0
400
800
1200
1600
time (s)
7 high
7 low



MVD

Median

Figure 4.12 Comparison of median and MVD between soils from same site but different in Soil organic carbon (10 graphs). (H = high SOC; L = low SOC)

4.3.4 Correlation between Aggregate Stability, SOC and Parameters of disintegration equation of Phase 1 + Phase

The parameters of the entire power curve equation (Phases 1 + 2) and the subset linear equation (Phase-1, or first four points) were compared to the SOC and the MWD of Wet Sieving results (**Table 4.3 & Table 4.4**).

Bieganowski *et al.* (2010) discussed the limitation of using the power function because y approaches infinity when x approaches 0. Meaning no reliable value can be determined for the y intercept. While the power model was used to describe the aggregate breakdown this might not be appropriate or helpful in defining aggregate stability as;

1) The disintegration rate of the two phases vary significantly; and

2) The aggregate stability index ‘c’ as determined from the linear equation, was similar to the lower MWD values in the wet sieving data. This suggests the destructive energy of the LD machine is significantly greater than wet sieving as most breakdown is occurring within 60s.

Therefore, Phase-1 (~60s) is a suitable segment to model aggregate stability comparisons.

Table 4.3 Comparison between Soil Organic Carbon, MWD, and parameters of Linear and Curve equation of Median against time.

Median			Linear equation- Median $y = mx + c$		Curve equation- Median $\mathrm{y} = \mathrm{a}x^{b} \equiv \ln(\mathrm{y}) = \ln(\mathrm{a}) + \mathrm{b}\ln(\mathrm{x})$		
Site	SOC (%)	MWD(Lamm)	m	c	ln(a)	a	b
Site 6 high	3.028	0.817	-3.224	387.27	7.04	1139.53	-0.432
Site 2 high	2.385	0.840	-3.6524	374.2	7.14	1260.33	-0.49
Site 6 low	2.333	1.066	-7.2076	636.73	7.57	1938.40	-0.518
Site 5 high	2.205	0.485	-2.7902	285.61	6.61	745.47	-0.412
Site 2 low	1.904	0.673	-1.9308	240.78	6.47	646.03	-0.414
Site 5 low	1.63	0.343	-1.3852	190.21	6.32	555.00	-0.41
Site 12 high	1.444	0.753	-2.2419	360.41	7.66	2125.70	-0.572
Site 12 low	1.359	0.573	-4.0924	451.5	7.69	2181.73	-0.605
Site 7 low	0.9116	0.375	-1.2338	207.35	6.56	709.59	-0.397
Site 7 high	0.8987	0.368	-1.7649	271.15	6.62	746.31	-0.379

Table 4.4 Comparison between Soil Organic Carbon, MWD, and parameters of Linear and Curve equation of Mean Volume Diameter against time.

MVD			Linear equation- MVD $y = mx + c$		Curve equation- MVD ($\mathrm{y} = \mathrm{a}x^{b}$) $\equiv$ [$\ln(\mathrm{y}) = \ln(\mathrm{a}) + \mathrm{b}\ln(\mathrm{x})$]		
site	SOC (%)	MWD(Lamm)	m	c	ln(a)	a	b
Site 6 high	3.03	0.82	-4.26	567.77	6.61	742.04	-0.24
Site 2 high	2.39	0.84	-2.82	515.09	7.35	1563.32	-0.37
Site 6 low	2.33	1.07	-7.67	773.46	7.17	1296.41	-0.33
Site 5 high	2.21	0.49	-5.34	489.75	6.88	968.67	-0.34
Site 2 low	1.90	0.67	-3.63	419.19	6.74	841.42	-0.31
Site 5 low	1.63	0.34	-3.65	389.84	6.33	559.30	-0.27
Site 12 high	1.44	0.75	-3.69	505.37	7.45	1713.17	-0.43
Site 12 low	1.36	0.57	-5.98	636.65	7.85	2577.35	-0.51
Site 7 low	0.91	0.38	-3.00	345.92	6.31	552.78	-0.26
Site 7 high	0.90	0.37	-3.44	473.49	7.02	1115.49	-0.30

Table 4.5 The meaning of parameters of linear and curve equation

Parameters	Name
m	Average disintegration rate of first four measurements (phase one)
c	Modelled Median/MVD – aggregate stability index from first 4 repeated measurement (phase one)
a	-
b	Disintergration rate (complete)

Table 4.6 summarises the level of significance of correlation of parameters of MVD and Median to the MWD and SOC. The correlations between SOC and parameter ln(a) and b, and between MWD and parameter ln(a) and b were statistically insignificant, excluding the correlation between ln(a) and MWD (R square= 0.45). According to the limitations of the power curve equation discussed above, the curve - $y = ax^b$ was not suitable to express aggregate stability from wet sieving.

There was no evidence to support correlation between SOC and curve parameters as there was no significant correlation between LD results and SOC.

Correlations between parameters of MVD's equations and MWD & SOC separately were mostly insignificant, excluding the correlation between aggregate stability index (c) and MWD (**Figure 4.14,** R square =0.56).

Table 4.6 indicates that majority of the parameters of Median were significantly moderately (0.41<R square< 0.67) correlated to MWD (**Figure 4.15**).

Table 4.6 Correlation between aggregate stability and parameters, and between Soil Carbon Content and parameters of linear and nonlinear regression. (**= p<0.01; *= p<0.05; ns= p>0.05)

regression	MVD		D50	
	SOC (%)	MWD	SOC	MWD
m	ns	ns	ns	**
c	ns	*	ns	**
ln(a)	ns	ns	ns	*
b	ns	ns	ns	ns

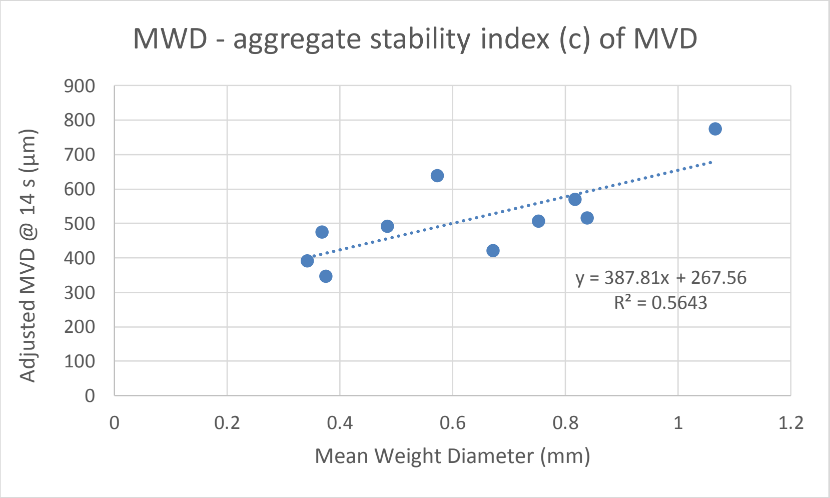
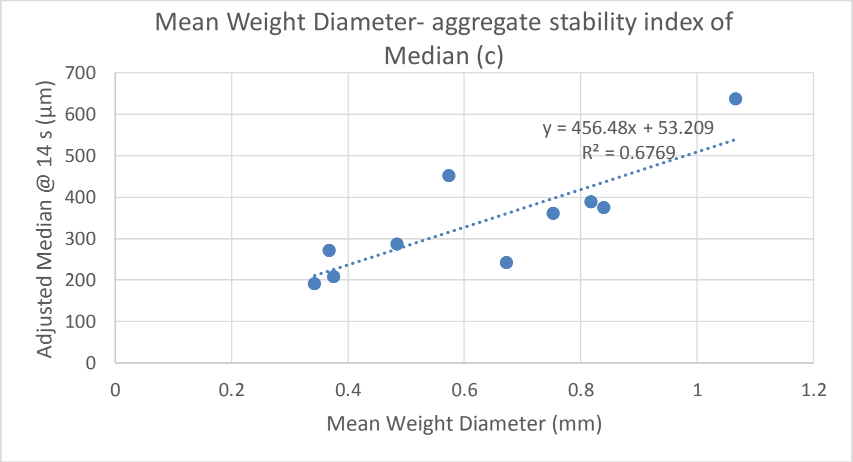
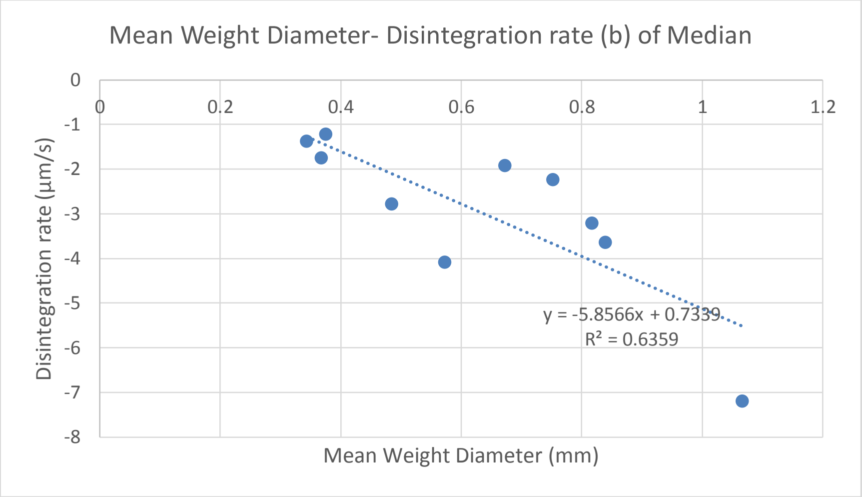


Figure 4.13~~14~~ c was the only parameter of equations of MVD that was significantly correlated to Wet Sieving result- Mean Weight Diameter (MWD).


Mean Weight Diameter- aggregate stability index of Median (c)
Adjusted Median @ 14 s (µm)
700
600
500
400
300
200
100
0
0
0.2
0.4
0.6
0.8
1
1.2
Mean Weight Diameter (mm)
y = 456.48x + 53.209
R² = 0.6769


Mean Weight Diameter- Disintegration rate (b) of Median
Disintegration rate (µm/s)
0
-1
-2
-3
-4
-5
-6
-7
-8
0
0.2
0.4
0.6
0.8
1
1.2
Mean Weight Diameter (mm)
y = -5.8566x + 0.7339
R² = 0.6359

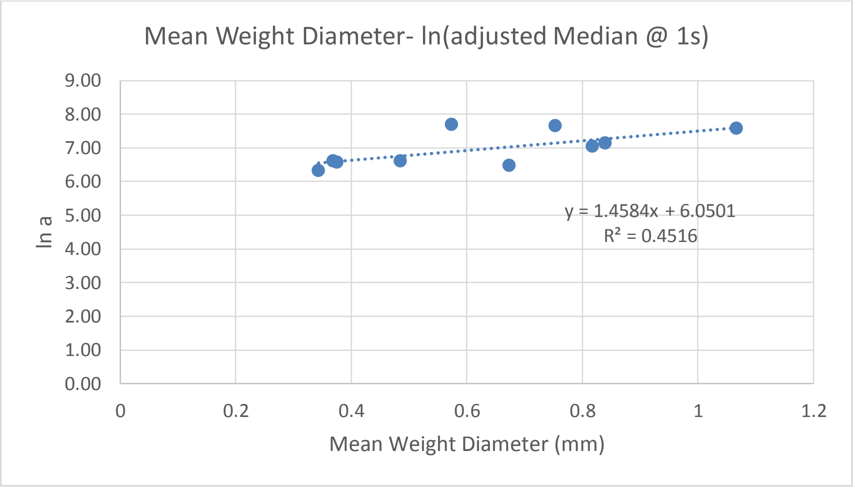


Figure 4.14~~15~~ Parameters of equations that were significantly correlated to Soil Carbon Content (SOC) and Wet Sieving result- Mean Weight Diameter (MWD)

4.4 Discussion

4.4.1 Wet sieving result

In comparison with a previous study by Peter Fisher *et al.* (2009) the present work has shown a strongly correlation between WSA and SOC levels in the samples re-tested. Aggregate stabilities of samples of Peter Fisher *et al.* (2009) were overall higher and less SOC-dependent than aggregate stabilities in present study. Furthermore, the correlation between SOC and aggregate stability was significant ($p<0.01$) in this study but was not significant in previous study ($p>0.05$). However the Peter Fisher *et al.* (2009) study used larger aggregate sizes (2 – 4 mm) than this current work (1 – 2 mm). Kemper and Rosenau (1986) reviewed the positive relation between aggregate size and aggregate stability. Therefore, it could be that the reason is that more energy is needed to breakdown bonding between micro-aggregates (<0.25mm) than that of the coarse aggregates (>0.25 mm).

Secondly, the samples were taken in 2006. Aumann *et al.* (2006) indicated that active and labile organic carbon has a half-life ranging from a few weeks to few years. Thus, although the samples had been air dried, some of this labile carbon might have degraded during the eight years of storage hence affecting the current analysis.

Thirdly, SOM is not the only factor that determined aggregate stability. Studies reported the influence of clay mineralogy, cation exchange capacity, exchangeable sodium percentage and electrical conductivity to the aggregate stability (Goldberg *et al.*, 1988, García-Orenes *et al.*, 2005, Kemper and Koch, 1966, Dimoyiannis *et al.*, 1998, Lehrsch *et al.*, 1991).
The result suggest that positive influences of other soil properties may possibly be less significant in the smaller aggregates used in our tests.

Soil with lower SOC suffered a greater reduction in the WSA than soil with high SOC. Therefore, this suggests that there were more than one factor causing a decline in aggregate stability, though the factors and extent that each contributed to aggregate stability were unknown.

4.4.2 Low obscuration (LD)

In Particle Size Analysis (PSA) by using LD, a minimum obscuration value of 10 is recommended by the manufacturer as a suitable value. A lower obscuration than this implies the numbers of particle in the suspension is insufficient to be representative. Thus the obscuration was always checked and set to vary between 10 and 20%. In aggregate stability testing the aggregates (1 – 2 mm) were tipped into the dispersion tank and immediately followed by the LD measurements. Any low obscuration values were probably as a result of low quantities of aggregates in the measurement cell, as initially the aggregates are large and few, thus, light scattering is low, but also with some light being absorbed by aggregates.

4.4.3 Dynamic disintegration- Phase-2

When the aggregate disintegration was approaching stabilization, the size of most of the aggregates were similar to the primary particle sizes. This explains why the Median and MVD of samples from Site 7 at the end of the Phase-2 were unrelated to SOC. Also different soil types all have different Particle Size Distributions (PSDs). Some soils were high in sand content and thus, the Median and MVD were higher for these soils with high coarse particle contents. Therefore, LD measurement for aggregate stability may take less time.

The Median index samples tended to enter Phase 2 after about 200 – 400 seconds of measurement, while MVD index tended to enter Phase 2 after 400 seconds of measurement (**Figure 4.8**). Considering that some aggregates might be very similar to the size of the coarser particles after 400 seconds of measurement, therefore, the length of aggregate stability test for dynamic study purposes is suggested not to exceed 400 seconds.

4.4.4 How is the parameters of Linear equation correlated to the Aggregate Stability?

From the results we knew that the parameters of the linear equation (first 4 repeated measurements) of the Median showed significant correlation with Mean Weight Diameter. However, comparison between soils from same site (**Figure 4.8**) indicated that both indexes-MVD and Median showed good relationship with SOC.

Dilution of the suspensions was always undertake after the first four readings in the continuous measurement run. Therefore, comparison of aggregate stability using the results of the first four readings was standard to all measurements. The parameter-‘c’ can be treated as the aggregate stability index as consequence of LD measurement in shorter timeframes (~60s). It is the y-intercept point that reflected aggregate stability based on the Median or MVD values of first four continuous measurements. The results of samples with poor aggregate stability indicated that aggregate stability index (c) of median were similar amount of aggregates below 0.25 mm with the result from wet sieving due to large disruptive energy input. There were five samples have Water Stable Aggregates (0.25 mm) less than 50% (**Figure 4.3**) and five samples have aggregate stability index (c) of Median around 0.25 mm. This indicates that measurement in shorter time frames seems a reasonable method to undertake.

Aggregates disintegrated exponentially over time. Therefore, the disintegration rate of aggregates was decreasing with the size of aggregate. This suggests that the dynamic of disintegration rate was primarily affected by the aggregate size of the population and secondarily affected by the stability of the aggregate due to large disruptive energy input.

4.5 Conclusion

The Laser Diffraction (LD) method can be used for aggregate stability measurement based on the significant correlation between mean weight diameter (MWD) using tradition wet sieving and the size and disintegration of aggregates in the first minute of LD run. Aggregate stability comparisons by using parameter- c (modelled aggregate stability index of first four repeated measurement was recommended as it is related to both average integration rate and aggregate sizes in first four repeated measurements. The SOC was correlated with most parameters of the both equations though were mostly insignificant. This can be explained by the multiple soil properties impacting on aggregate stability discussed in wet sieving section. LD measurement was limited to no longer than 400 seconds as the aggregates might reduce to very close to the size of the primary particles in Phase 2 of the disintegration process. The study methodology require only a few minutes to conduct a test which was very time effective compared to the methodology of wet sieving methods (requiring at least one day).

Direct comparison of complete disintegration curve (Phase-1+ Phase-2) of samples from different site were not recommended due to the Phase-2 effect. However, direct comparison of samples with different SOC from same site showed that samples with high SOC or MWD (site 6) always have better stability curve than samples with low SOC or MWD. This indicated that LD might be sensitive to the change in soil properties of sample from soil type.

Due to time limitations, I have not been able to separate the dynamic disintegration of aggregates seen in Phase 1 (1st 400 s) from those of the primary particles see in Phase 2. Undertaking this subtraction might improve the resolution of the findings. Also a further study of more soil types is recommended and might confirm the positive correlations found in this study. Furthermore, whether the method is universal for all soils could not be guarantee due to the results of Site 14 which indicated that soil characteristic would potentially affect aggregate stability measurement using LD method.

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Appendix

Chapter 3

Anova analysis of chemical properties- EC, ESP, soil pH, exchangeable cations (Ca, Na, Mg, K), Ca:Mg ratios between different location treatment and between GYP-FLO treatment (See **Table 3.2**)

Tests of Between-Subjects Effects

Dependent Variable: EC (ds/m)

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	.711	1	.711	551.535
	Error	.003	2	.001[a]	
treatment	Hypothesis	.001	1	.001	10.811
	Error	.000	2	.000[b]	
location	Hypothesis	.008	4	.002	8.354
	Error	.002	8	.000[c]	
block	Hypothesis	.003	2	.001	7.442
	Error	.000	1.555	.000[d]	
treatment * location	Hypothesis	.004	4	.001	5.658
	Error	.002	8	.000[e]	
treatment * block	Hypothesis	.000	2	.000	.649
	Error	.002	8	.000[e]	
location * block	Hypothesis	.002	8	.000	1.263
	Error	.002	8	.000[e]	
treatment * location * block	Hypothesis	.002	8	.000	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: EC (ds/m)

Source		Sig.
Intercept	Hypothesis	.002

	Error	
treatment	Hypothesis	.081
	Error	
location	Hypothesis	.006
	Error	
block	Hypothesis	.160
	Error	
treatment * location	Hypothesis	.018
	Error	
treatment * block	Hypothesis	.548
	Error	
location * block	Hypothesis	.375
	Error	
treatment * location * block	Hypothesis	.
	Error	

Estimates

Dependent Variable: EC (ds/m)

			95% Confidence Interval	
treatment	Mean	Std. Error	Lower Bound	Upper Bound
high	.147	.	.	.
low	.161	.	.	.

Tests of Between-Subjects Effects

Dependent Variable: EC (ds/m)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.014[a]	9	.002	4.842	.002
Intercept	.711	1	.711	2270.681	.000
treatment	.001	1	.001	4.255	.052
location	.008	4	.002	6.399	.002
treatment * location	.004	4	.001	3.431	.027
Error	.006	20	.000		
Total	.731	30			
Corrected Total	.020	29			

a. R Squared = .685 (Adjusted R Squared = .544)

Estimates

Dependent Variable: EC (ds/m)

treatment	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
high	.147	.005	.138	.157
low	.161	.005	.151	.170

EC (ds/m)

location	N	Subset	
		1	2

Duncan[a,b]	1.0	6	.1350	
	2.0	6	.1417	
	4.0	6	.1467	
	3.0	6		.1700
	5.0	6		.1767
	Sig.		.293	.522

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = .000.

a. Uses Harmonic Mean Sample Size = 6.000.

b. Alpha = .05.

Tests of Between-Subjects Effects

Dependent Variable: pH(water)

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	1496.720	1	1496.720	1255.287
	Error	2.385	2	1.192[a]	
treatment	Hypothesis	.408	1	.408	1.267
	Error	.645	2	.322[b]	
location	Hypothesis	7.228	4	1.807	16.390
	Error	.882	8	.110[c]	
block	Hypothesis	2.385	2	1.192	4.903
	Error	.248	1.020	.243[d]	
treatment * location	Hypothesis	.227	4	.057	.299
	Error	1.515	8	.189[e]	
treatment * block	Hypothesis	.645	2	.322	1.702
	Error	1.515	8	.189[e]	
location * block	Hypothesis	.882	8	.110	.582

	Error	1.515	8	.189[e]	
treatment * location * block	Hypothesis	1.515	8	.189	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: pH(water)

Source		Sig.
Intercept	Hypothesis	.001
	Error	
Treatment	Hypothesis	.377
	Error	
Location	Hypothesis	.001
	Error	
Block	Hypothesis	.300
	Error	
treatment * location	Hypothesis	.871
	Error	
treatment * block	Hypothesis	.242
	Error	
location * block	Hypothesis	.770
	Error	
treatment * location * block	Hypothesis	.
	Error	

Tests of Between-Subjects Effects

Dependent Variable: pH(water)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	7.863[a]	9	.874	3.220	.014
Intercept	1496.720	1	1496.720	5516.168	.000

treatment	.408	1	.408	1.505	.234
location	7.228	4	1.807	6.660	.001
treatment * location	.227	4	.057	.209	.930
Error	5.427	20	.271		
Total	1510.010	30			
Corrected Total	13.290	29			

a. R Squared = .592 (Adjusted R Squared = .408)

Estimates

Dependent Variable: pH(water)

			95% Confidence Interval	
treatment	Mean	Std. Error	Lower Bound	Upper Bound
high	6.947	.134	6.666	7.227
low	7.180	.134	6.899	7.461

pH(water)

			Subset		
	location	N	1	2	3
Duncan[a,b]	1.0	6	6.400		
	4.0	6	6.700		
	2.0	6	6.983	6.983	
	3.0	6		7.533	7.533
	5.0	6			7.700

Sig.		.080	.082	.586

a. Uses Harmonic Mean Sample Size = 6.000.

b. Alpha = .05.

Tests of Between-Subjects Effects

Dependent Variable: Calcium (meq/100g)

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	2068.360	1	2068.360	1890.064
	Error	2.189	2	1.094[a]	
treatment	Hypothesis	.800	1	.800	.349
	Error	4.581	2	2.290[b]	
location	Hypothesis	.395	4	.099	.351
	Error	2.251	8	.281[c]	
block	Hypothesis	2.189	2	1.094	.470
	Error	4.782	2.054	2.328[d]	
treatment * location	Hypothesis	1.608	4	.402	1.653
	Error	1.946	8	.243[e]	
treatment * block	Hypothesis	4.581	2	2.290	9.416
	Error	1.946	8	.243[e]	
location * block	Hypothesis	2.251	8	.281	1.157
	Error	1.946	8	.243[e]	
treatment * location * block	Hypothesis	1.946	8	.243	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: Calcium (meq/100g)

Source		Sig.
Intercept	Hypothesis	.001
	Error	
treatment	Hypothesis	.614
	Error	
location	Hypothesis	.837
	Error	
block	Hypothesis	.679
	Error	
treatment * location	Hypothesis	.253
	Error	
treatment * block	Hypothesis	.008
	Error	
location * block	Hypothesis	.421
	Error	
treatment * location * block	Hypothesis	.
	Error	

Dependent Variable: Calcium (meq/100g)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2.803[a]	9	.311	.568	.807
Intercept	2068.360	1	2068.360	3772.086	.000
treatment	.800	1	.800	1.460	.241
location	.395	4	.099	.180	.946
treatment * location	1.608	4	.402	.733	.580
Error	10.967	20	.548		
Total	2082.130	30			
Corrected Total	13.770	29			

Estimates

Dependent Variable: Calcium (meq/100g)

treatment	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
high	8.140	.191	7.741	8.539
low	8.467	.191	8.068	8.865

Calcium (meq/100g)

	location	N	Subset
			1
Duncan[a,b]	1.0	6	8.150
	4.0	6	8.200
	2.0	6	8.333
	3.0	6	8.367
	5.0	6	8.467
	Sig.		.514

Tests of Between-Subjects Effects

Dependent Variable: Calcium Magnesium ratio

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	26.696	1	26.696	91.322
	Error	.585	2	.292[a]	
treatment	Hypothesis	.363	1	.363	2.074
	Error	.350	2	.175[b]	
location	Hypothesis	.035	4	.009	1.828
	Error	.039	8	.005[c]	

block	Hypothesis	.585	2	.292	1.688
	Error	.339	1.957	.173[d]	
treatment * location	Hypothesis	.009	4	.002	.325
	Error	.053	8	.007[e]	
treatment * block	Hypothesis	.350	2	.175	26.250
	Error	.053	8	.007[e]	
location * block	Hypothesis	.039	8	.005	.725
	Error	.053	8	.007[e]	
treatment * location * block	Hypothesis	.053	8	.007	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: Calcium Magnesium ratio

Source		Sig.
Intercept	Hypothesis	.011
	Error	
treatment	Hypothesis	.286
	Error	
location	Hypothesis	.217
	Error	
block	Hypothesis	.375
	Error	
treatment * location	Hypothesis	.854
	Error	
treatment * block	Hypothesis	.000
	Error	
location * block	Hypothesis	.670
	Error	
treatment * location * block	Hypothesis	.

Error	

Tests of Between-Subjects Effects

Dependent Variable: Calcium Magnesium ratio

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.407[a]	9	.045	.881	.558
Intercept	26.696	1	26.696	520.058	.000
treatment	.363	1	.363	7.071	.015
location	.035	4	.009	.172	.950
treatment * location	.009	4	.002	.042	.996
Error	1.027	20	.051		
Total	28.130	30			
Corrected Total	1.434	29			

Estimates

Dependent Variable: Calcium Magnesium ratio

treatment	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
high	1.053	.058	.931	1.175
low	.833	.058	.711	.955

Calcium Magnesium ratio

	location	N	Subset
			1
Duncan[a,b]	2.0	6	.883

	5.0	6	.933
	4.0	6	.950
	3.0	6	.967
	1.0	6	.983
	Sig.		.501

Tests of Between-Subjects Effects

Dependent Variable: Magnesium(meq/100g)

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	2555.787	1	2555.787	154.326
	Error	33.122	2	16.561[a]	
treatment	Hypothesis	33.075	1	33.075	3.256
	Error	20.318	2	10.159[b]	
location	Hypothesis	4.965	4	1.241	.910
	Error	10.911	8	1.364[c]	
block	Hypothesis	33.122	2	16.561	1.556
	Error	23.223	2.182	10.644[d]	
treatment * location	Hypothesis	3.003	4	.751	.855
	Error	7.029	8	.879[e]	
treatment * block	Hypothesis	20.318	2	10.159	11.563
	Error	7.029	8	.879[e]	
location * block	Hypothesis	10.911	8	1.364	1.552
	Error	7.029	8	.879[e]	
treatment * location * block	Hypothesis	7.029	8	.879	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: Magnesium(meq/100g)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	41.043[a]	9	4.560	1.278	.308
Intercept	2555.787	1	2555.787	716.107	.000
treatment	33.075	1	33.075	9.267	.006
location	4.965	4	1.241	.348	.842
treatment * location	3.003	4	.751	.210	.930
Error	71.380	20	3.569		
Total	2668.210	30			
Corrected Total	112.423	29			

a. R Squared = .365 (Adjusted R Squared = .079)

Estimates

Dependent Variable: Magnesium(meq/100g)

			95% Confidence Interval	
treatment	Mean	Std. Error	Lower Bound	Upper Bound
high	8.180	.488	7.163	9.197
low	10.280	.488	9.263	11.297

Magnesium(meq/100g)

			Subset
	location	N	1
Duncan[a,b]	1.0	6	8.500
	4.0	6	9.167

	3.0	6	9.283
	2.0	6	9.517
	5.0	6	9.683
	Sig.		.342

Dependent Variable: Potassium (meq/100g)

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	10.514	1	10.514	499.000
	Error	.042	2	.021[a]	
treatment	Hypothesis	.000	1	.000	.051
	Error	.008	2	.004[b]	
location	Hypothesis	.031	4	.008	1.810
	Error	.034	8	.004[c]	
block	Hypothesis	.042	2	.021	2.895
	Error	.034	4.678	.007[d]	
treatment * location	Hypothesis	.008	4	.002	1.673
	Error	.009	8	.001[e]	
treatment * block	Hypothesis	.008	2	.004	3.599
	Error	.009	8	.001[e]	
location * block	Hypothesis	.034	8	.004	3.604
	Error	.009	8	.001[e]	
treatment * location * block	Hypothesis	.009	8	.001	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: Potassium (meq/100g)

Source		Sig.
Intercept	Hypothesis	.002

	Error	
treatment	Hypothesis	.843
	Error	
location	Hypothesis	.220
	Error	
block	Hypothesis	.152
	Error	
treatment * location	Hypothesis	.248
	Error	
treatment * block	Hypothesis	.077
	Error	
location * block	Hypothesis	.044
	Error	
treatment * location * block	Hypothesis	.
	Error	

Tests of Between-Subjects Effects

Dependent Variable: Potassium (meq/100g)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	.039[a]	9	.004	.916	.531
Intercept	10.514	1	10.514	2241.774	.000
treatment	.000	1	.000	.045	.833
location	.031	4	.008	1.632	.205
treatment * location	.008	4	.002	.419	.793
Error	.094	20	.005		
Total	10.646	30			
Corrected Total	.132	29			

Dependent Variable: Potassium (meq/100g)

treatment	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
high	.589	.018	.552	.626
low	.595	.018	.558	.632

Potassium (meq/100g)

	location	N	Subset
			1
Duncan[a,b]	5.0	6	.5550
	2.0	6	.5617
	4.0	6	.5950
	3.0	6	.6050
	1.0	6	.6433
	Sig.		.057

Tests of Between-Subjects Effects

Dependent Variable: Sodium(meq/100g)

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	51.326	1	51.326	14.954
	Error	6.864	2	3.432[a]	
treatment	Hypothesis	.594	1	.594	9.936
	Error	.119	2	.060[b]	
location	Hypothesis	6.026	4	1.507	13.137
	Error	.917	8	.115[c]	
block	Hypothesis	6.864	2	3.432	33.882
	Error	.254	2.505	.101[d]	
treatment * location	Hypothesis	.391	4	.098	1.337

	Error	.585	8	.073[e]	
treatment * block	Hypothesis	.119	2	.060	.817
	Error	.585	8	.073[e]	
location * block	Hypothesis	.917	8	.115	1.568
	Error	.585	8	.073[e]	
treatment * location * block	Hypothesis	.585	8	.073	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: Sodium(meq/100g)

Source		Sig.
Intercept	Hypothesis	.061
	Error	
treatment	Hypothesis	.088
	Error	
location	Hypothesis	.001
	Error	
block	Hypothesis	.015
	Error	
treatment * location	Hypothesis	.336
	Error	
treatment * block	Hypothesis	.475
	Error	
location * block	Hypothesis	.269
	Error	
treatment * location * block	Hypothesis	.
	Error	

Tests of Between-Subjects Effects

Dependent Variable: CEC (meq/100g)

Source		Type III Sum of Squares	df	Mean Square	F
Intercept	Hypothesis	11213.333	1	11213.333	294.829
	Error	76.067	2	38.033[a]	
treatment	Hypothesis	48.133	1	48.133	2.529
	Error	38.067	2	19.033[b]	
location	Hypothesis	24.333	4	6.083	1.722
	Error	28.267	8	3.533[c]	
block	Hypothesis	76.067	2	38.033	1.867
	Error	46.089	2.263	20.367[d]	
treatment * location	Hypothesis	10.200	4	2.550	1.159
	Error	17.600	8	2.200[e]	
treatment * block	Hypothesis	38.067	2	19.033	8.652
	Error	17.600	8	2.200[e]	
location * block	Hypothesis	28.267	8	3.533	1.606
	Error	17.600	8	2.200[e]	
treatment * location * block	Hypothesis	17.600	8	2.200	.
	Error	.000	0	.[f]	

Tests of Between-Subjects Effects

Dependent Variable: CEC (meq/100g)

Source		Sig.
Intercept	Hypothesis	.003
	Error	
treatment	Hypothesis	.253
	Error	
location	Hypothesis	.238

	Error	
block	Hypothesis	.332
	Error	
treatment * location	Hypothesis	.396
	Error	
treatment * block	Hypothesis	.010
	Error	
location * block	Hypothesis	.259
	Error	
treatment * location * block	Hypothesis	.
	Error	

Tests of Between-Subjects Effects

Dependent Variable: CEC (meq/100g)

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	82.667[a]	9	9.185	1.148	.377
Intercept	11213.333	1	11213.333	1401.667	.000
treatment	48.133	1	48.133	6.017	.023
location	24.333	4	6.083	.760	.563
treatment * location	10.200	4	2.550	.319	.862
Error	160.000	20	8.000		
Total	11456.000	30			
Corrected Total	242.667	29			

Estimates

Dependent Variable: CEC (meq/100g)

treatment	Mean	Std. Error	95% Confidence Interval

			Lower Bound	Upper Bound
high	18.067	.730	16.543	19.590
low	20.600	.730	19.077	22.123

CEC (meq/100g)

	location	N	Subset
			1
Duncan[a,b]	1.0	6	18.000
	4.0	6	18.667
	2.0	6	19.500
	3.0	6	20.000
	5.0	6	20.500
	Sig.		.183

Tests of Between-Subjects Effects

Dependent Variable: ESP

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	125.443[a]	9	13.938	1.811	.129
Intercept	1259.291	1	1259.291	163.594	.000
treatment	3.583	1	3.583	.466	.503
location	117.438	4	29.360	3.814	.018
treatment * location	4.421	4	1.105	.144	.964
Error	153.953	20	7.698		
Total	1538.687	30			
Corrected Total	279.396	29			

Estimates

Dependent Variable: ESP

treatment	Mean	Std. Error	95% Confidence Interval	
			Lower Bound	Upper Bound
high	6.133	.716	4.639	7.628
low	6.825	.716	5.330	8.319

ESP

	location	N	Subset	
			1	2
Duncan[a,b]	1.0	6	3.9654	
	4.0	6	4.9990	
	2.0	6	5.8524	5.8524
	5.0	6		8.7862
	3.0	6		8.7917
	Sig.		.278	.097

Means for groups in homogeneous subsets are displayed.

Based on observed means.

The error term is Mean Square(Error) = 7.698.

a. Uses Harmonic Mean Sample Size = 6.000.

b. Alpha = .05.

Chapter 4

Significant level of regression between soil organic carbon and water stable aggregate of previous study- n=36. (See **Figure 4.1**)

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.009	1	.009	.305	.585[b]
	Residual	.996	34	.029		

	Total	1.005	35			

a. Dependent Variable: WSA

b. Predictors: (Constant), SOC

Significant level of regression between soil organic carbon and water stable aggregate of present study-N=36 . (See **Figure 4.4**)

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.736	1	.736	26.174	.000[b]
	Residual	.956	34	.028		
	Total	1.692	35			

a. Dependent Variable: MWD

b. Predictors: (Constant), SOC

Significant level of regression between Soil Organic Carbon and parameters of Median equations (See **Table 4.6**)

Parameter m SOC

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	6.980	1	6.980	2.611	.145[b]
	Residual	21.385	8	2.673		
	Total	28.365	9			

a. Dependent Variable: m

b. Predictors: (Constant), carbon

Parameter c- SOC

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	29208.848	1	29208.848	1.761	.221[b]

	Residual	132666.311	8	16583.289		
	Total	161875.159	9			

a. Dependent Variable: c

b. Predictors: (Constant), carbon

Parameter ln(a)- SOC

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.067	1	.067	.222	.650[b]
	Residual	2.414	8	.302		
	Total	2.481	9			

a. Dependent Variable: ln_a

b. Predictors: (Constant), carbon

Parameter b- SOC

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.000	1	.000	.018	.896[b]
	Residual	.056	8	.007		
	Total	.056	9			

a. Dependent Variable: B

b. Predictors: (Constant), carbon

Significant level of regression between Mean Weight Diameter and parameters of Median equations (See **Table 4.6**)

Parameter- m/ MWD

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	18.037	1	18.037	13.971	.006[b]

	Residual	10.328	8	1.291		
	Total	28.365	9			

a. Dependent Variable: m

b. Predictors: (Constant), MWD

Parameter c- MWD

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	109571.494	1	109571.494	16.759	.003[b]
	Residual	52303.666	8	6537.958		
	Total	161875.159	9			

a. Dependent Variable: c

b. Predictors: (Constant), MWD

Parameter ln(a)- MWD

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1.120	1	1.120	6.581	.033[b]
	Residual	1.361	8	.170		
	Total	2.481	9			

a. Dependent Variable: ln_a

b. Predictors: (Constant), MWD

Parameter b- MWD

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.015	1	.015	2.892	.127[b]
	Residual	.041	8	.005		

Model		Sum of Squares	df	Mean Square	F	Sig.
	Total	.056	9			

a. Dependent Variable: B

b. Predictors: (Constant), MWD

Significant level of regression between Soil organic carbon and parameters of MVD equations. (See **Table 4.6**)

Parameter m- SOC

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	1.766	1	1.766	.732	.417[b]
	Residual	19.309	8	2.414		
	Total	21.075	9			

a. Dependent Variable: m

b. Predictors: (Constant), carbon

Parameter c- SOC

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	27366.167	1	27366.167	1.941	.201[b]
	Residual	112792.792	8	14099.099		
	Total	140158.959	9			

a. Dependent Variable: c

b. Predictors: (Constant), carbon

Parameter ln(a)- SOC

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.002	1	.002	.006	.942[b]

	Residual	2.226	8	.278		
	Total	2.227	9			

a. Dependent Variable: ln_a

b. Predictors: (Constant), carbon

<u>Parameter b- SOC</u>

ANOVA^a

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.003	1	.003	.346	.573^b
	Residual	.062	8	.008		
	Total	.065	9			

a. Dependent Variable: B

b. Predictors: (Constant), carbon

Significant level of regression between Mean Weight Diamter and parameters of MVD equations. (See **Table 4.6**)

<u>Parameter m- MWD</u>

ANOVA^a

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	4.538	1	4.538	2.195	.177^b
	Residual	16.537	8	2.067		
	Total	21.075	9			

a. Dependent Variable: m

b. Predictors: (Constant), MWD

<u>Parameter c- MWD</u>

ANOVA^a

Model		Sum of Squares	df	Mean Square	F	Sig.

1	Regression	79085.409	1	79085.409	10.359	.012[b]
	Residual	61073.549	8	7634.194		
	Total	140158.959	9			

a. Dependent Variable: c

b. Predictors: (Constant), MWD

Parameter ln(a)- MWD

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.385	1	.385	1.672	.232[b]
	Residual	1.842	8	.230		
	Total	2.227	9			

a. Dependent Variable: ln_a

b. Predictors: (Constant), MWD

Parameter b- MWD

ANOVA[a]

Model		Sum of Squares	df	Mean Square	F	Sig.
1	Regression	.002	1	.002	.306	.595[b]
	Residual	.062	8	.008		
	Total	.065	9			

a. Dependent Variable: B

b. Predictors: (Constant), MWD