IMPACT OF LEONARDITE AMENDMENTS ON SOIL PHYSICAL

PROPERTIES OF TWO SOILS RELATED TO GRAIN PRODUCTION

BY

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A Thesis
Submitted to the Faculty of Graduate Studies
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THE UNIVERSITY OF MANITOBA

FACULTY OF GRADUATE STUDIES
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ABSTRACT

One of the major constraints to crop productivity is soil structure. A soil with good structure will have an optimum balance of pores conducive to infiltration. aeration and water retention. Soil structure can be changed with amendments and field management. The objectives of this thesis were: (1) to evaluate the effectiveness of leonardite amendment application on improving soil structure: (2) to determine whether leonardite application has the ability to improve crop productivity.

The evidence obtained suggests that the given forms of leonardite (an oxidized lignite) were ineffective in improving soil structure and crop productivity. Upon initial field application. the hydrophobic nature of the material was realized. This water repellency was carried over to the second field season. Leonardite was ineffective in altering soil porosity. soil strength. and water stable aggregation following one field season. Residual treatment effects evaluated following the next growth season showed there were no significant differences. Crop yield was also not affected by field applied leonardite. Due to the hydrophobic nature of the leonardite. it was suspected that there were little to no active functional groups on the surface of the leonardite. This was suspected to be the reason that leonardite was ineffective in altering soil structure.

An incubation study using a soluble powder form of leonardite resulted in no treatment effect on aggregate stability or crop productivity. The incubation study

revealed an increase in the organic carbon content of the soil with increasing leonardite

application. The chemical alteration of leonardite to a soluble form appeared to be

ineffective in increasing the quantity of surface active functional groups involved on soil

aggregation.

ACKNOWLEDGMENTS

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1. INTRODUCTION

1.1. Prologue

Soil structure is a physical property that describes how sand, silt and clay particles are arranged into stable structural units. These units are called aggregates or peds. Aggregate stability is dependent on cementing agents including organic matter (OM), secondary carbonates and in some cases, water films that bind together aggregates.

Soil structure has a great influence on soil erosion, crop productivity and yield. In general, a soil with favorable structure will exhibit a balance between macro- and micro-pores providing for a good combination of water infiltration, aeration and water retention (Brady 1990). High levels of water infiltration and low soil dispersion in structurally stable soils translate to low levels of water erosion. When aggregate stability is high, soil dispersion due to rapid excess wetting and consequent soil crusting is minimal (Hillel 1982). Well-structured soils tend to have a relatively low bulk density and strength, hence resistance to root penetration is also low (Thompson *et al.* 1987). Therefore, a well aggregated soil will exhibit favorable conditions for seed germination, emergence and plant growth.

Organic amendments have been applied to "less than ideal soils" in an attempt to improve structure and increase crop yield. Organic matter content in soils has been decreasing in the past few decades as a result of intensive cultivation and erosion. This organic matter must be replaced in order to maintain productive fields and decrease soil erosion. One possible means of replacement is through the use of organic amendments.

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A variety of organic treatments, such as sewage sludge, animal manures, and plant residues have been shown to improve soil structure, but these effects have not persisted. These materials have low microbial stability and are usually mineralized within one year, leaving only a small quantity of residue in the form of stable humic substances.

It has been demonstrated that soil humates can play an important role in soil aggregation. Humates similar to some of the extracts of soil organic matter may occur in weathered coal deposits (Stevenson and Shacklett 1988). This material should have a slower decomposition rate than less oxidized materials, and thereby offer long term protection from microbial breakdown. Therefore, by means of actions comparable to those of natural organic residues in the soil, mined humates may help in formation of stable soil aggregates.

Leonardite is an oxidized lignite (a low rank) coal. It has no commercial value as an energy source. Further, it is commonly found overlying coal seams, so it must be extracted to gain access to high energy yielding coal. Approximately 41 million tonnes of coal are produced annually by Luscar Ltd., Canada's largest coal company. Currently, leonardite is reincorporated with subsoil during mine reclamation.

1.2. Objectives

This project was started in an attempt to see if leonardite could have some commercial agronomic value. The leonardite used in all studies was extracted near coal seams in Northern Alberta. As leonardite is high in organic carbon it was hypothesized

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that it may improve crop productivity in two Manitoba soils as a result of its influence on soil structure. It was altered both physically and chemically to assess whether the raw leonardite material itself could influence aggregate stability or if chemical modification was necessary.

Leonardite may be a non-toxic highly organic material. It may have the potential to provide benefits to soil and primary production. The main objective of this study was to measure any improvement on soil structure and wheat productivity within two growing seasons related to leonardite amendment application.

1.3. Methodology

The study consisted of two main experiments: first. a field plot experiment which included several forms of a raw leonardite material: and second. an incubation study in which the amendment consisted of a soluble leonardite powder.

Soils that were representative of land commonly used for grain production in Southern Manitoba were chosen for the study. In Western Manitoba, areas of forest soils have been deforested and used for agriculture. The majority of these soils are Gray Luvisols. Heavy Clay Black Chernozemic soils are commonly found near and south of Winnipeg. The aggregates found in Luvisolic soils are highly susceptible to breakdown under rapid wetting and heavy clay soils are not well structured.

Two soils were chosen for the field study: a Waitville Clay Loam Dark Gray Luvisol and a Osborne Clay Humic Rego Gleysol. The Waitville clay loam (CL) was a

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degraded forest soil. Luvisol soils can severely limit crop production if environmental conditions conducive to surface crusting if environmental conditions conducive to surface crusting are evident. In 1998. the conditions were not conducive to crusting but in 1999. severe soil crusting had occurred. Surface crusting was caused by a packing rain followed by hot. dry weather. The emergence of canola was significantly affected by surface crusting as shown by the reduced emergence (Appendix A).

Waitville CL was characterized as a Luvisol by presence of an argillic Bt horizon (Ehrlich *et al.* 1956). This horizon indicated that clay from the A horizon directly above had been translocated and illuviated into the Bt horizon (Agriculture Canada Expert Committee on Soil Survey 1987). The Ae horizon lying above the Bt horizon was low in clay and had a platy structure. By adding organic carbon in the form of leonardite to the soil. it was postulated that granulation and structural stability would be promoted. as other studies have found positive relationships between the organic carbon content on the degree of roundness of aggregates (Dexter 1985) and structural stability (Kay 1998).

Previous studies have shown that the proportion of clay and organic matter originally in the soil affect the magnitude of change in soil structure related to addition of soil organic amendments (Kay 1997 and references cited therein). Higher amounts of each have been shown to reduce the overall amendment effect. Therefore. soil containing a high initial clay and organic carbon content. namely Osborne clay (C). was selected for comparative purposes against the Luvisol. As well. the Osborne C was prone to compaction as a result of its high clay content. and tests were conducted to determine if leonardite had the ability to reduce its susceptibility to compaction.

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The description and results of the field plot experiment are provided in Chapters 3 to 5. Chapters 3 and 4 examine the soil's physical properties. These chapters are divided based on the inter-relationships between the physical properties tested in the experiments within each chapter. In Chapter 3 an evaluation of the effect of the amendment on soil strength and density is included. Chapter 4 contains the results of leonardite on pore size distribution, aggregate stability and total organic carbon. Chapter 5 examines the influence of leonardite on crop productivity. The chapter contained the effects that leonardite had on wheat yield.

Description of the incubation experiment and the results are given in Chapters 6 and 7. In Chapter 6, the description and results of an experiment in which wheat was grown in Waitville CL are reported. Results of leonardite on physical properties relating to aggregate stability, chemical properties including pH, electrical conductivity and total organic carbon, in addition biological properties relating to crop biomass and yield were included. Chapter 7, contains results of a study in which wheat was grown in an Almasippi sandy loam soil. Root and shoot biomass as well as total wheat yield were examined.

Discussion of why the leonardite did not improve agronomic or structural conditions of the soil are given in Chapter 8. The main reason was thought to be that there were not enough active functional groups on the leonardite surface.

2. LITERATURE REVIEW

2.1. Introduction

Crop productivity and environmental quality are highly influenced by soil structure. Soil provides a mechanism of exchange between plants and the environment. Its structure is a dynamic property that plays a vital role in transport of water. gas and nutrients to plant roots. Soil erosion is lessened in soils with stable structure. Preventing erosion by improving structure will help to maintain productive fields and minimize eutrophication in nearby watersheds (Schindler 1974).

The physical structure of soil is dependent on texture and mineralogy as well as agronomic practice. biological and climatic factors (Hillel 1982). While the first two cannot be altered. the later three could change in a short time period.

By changing the agronomic practice through the application of organic amendments in an area where all other factors are fixed. it is possible to evaluate the amendment's effects on soil structure. This review deals with the formation of soil structure and is focused on how organic materials may influence it

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2.2. Soil Structure Formation

Soil structure results when external forces push individual particles together. The forces may include freezing-thawing, wetting and drying, root growth and tillage activities (Hassett and Banwart 1992). Individual soil particles are often arranged into larger secondary units called aggregates.

It has been suggested that soil aggregation takes place in a hierarchal order (Hadas 1987). The smaller units in the hierarchy, as well as the cementing agents binding them may significantly contribute to the development of the larger units. The internal strength within the units diminishes with an increase in size, so there is a general decrease in stability with an increase in aggregate diameter.

Four stages of aggregation have been proposed (Tisdall and Oades 1982). Individual clay platelets combine to form domains, quasi-crystals and floccules. These units are then arranged into clusters via coagulation and flocculation. Two or more clusters are bound together with polysaccharides, persistent humic substances and oxides of Al and Fe to form microaggregates. Through the action of plant roots and fungal hyphae, microaggregates (<250 µm in diameter) are combined to form macroaggregates (>250 µm in diameter). A larger unit called a clod is created when macroaggregates unite with no visible lines of weakness between the peds.

In soil aggregates, the principal cementing agents consist of soil organic matter (OM), secondary carbonates and water films (Hassett and Banwart 1992).

Soil OM includes fresh and incompletely decomposed plant and animal residues.

Kononova (1966) has discussed the terminology scheme for soil organic matter. Humus consists of humic substances and organic residues and products re-synthesized by microbes. Humic substances constitute a major fraction of the humus (up to 70%). They include humins and humic and fulvic acids, which are defined based on their solubility in acid and alkali solutions. Humins are insoluble at all pH's, humic acids are insoluble at pH $\leq$ 2 and fulvic acids are soluble in both acid and alkali (Jones and Bryan 1998).

According to Stevenson (1982), clay minerals adsorb organic complexes in several ways. The first was thought to involve Van der Waals forces or physical adsorption, i.e. a weak force involving dipole-dipole interaction. These forces are important for non-polar organic compounds. While the individual forces are low, they are additive. Hence, the concentration of the polymer would greatly affect the energy of interaction. The second interaction was proposed to involve chemical adsorption or electrostatic attraction of the cation exchange on clay particles. Here, positively charged organic compounds substitute for inorganic compounds attracted to negatively charged clays. Properties such as chain length and the type of cation on the colloidal exchange complex were thought to influence the degree of replacement. Thirdly, a pair of electronegative atoms could be linked through bonding with a single H atom through hydrogen bonding. This would occur on protonated edges of clay particles when the pH was low. Lastly, Stevenson (1982) postulated that a metal ion, e.g. Ca^{2+}, could bridge the organic molecule and the soil constituent in coordination complexes. This would be important for polyanion adsorption.

It is generally believed that cation bridging is the principal manner in which

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humic and fulvic acids as well as acidic polysaccharides are retained in the soil

(Stevenson 1982). This would occur where a salt bridge is formed between the COOH

group and the soil (clay - M - OOCR). Depending on the mechanism involved, the

association between humus and clay may vary for different soil types and even for

different clay fractions within soils (Turchenek and Oades 1979).

It has been shown that humic substances may be involved in clay flocculation and

coagulation. Theng (1979) suggested that an addition of an organic polymer in amounts

too small to be sufficient to cause aggregation of a dispersed charge stabilized system

may assist in coagulation of the soil. He argued that the particles may become sensitized

such that their susceptibility to coagulation by electrolytes was increased. In

investigations of colloidal suspensions, Kretzschmar *et al.* (1993) measured the critical

coagulation concentration (CCC) of kaolinitic soils in suspension. The CCC was defined

as the minimum concentration of an electrolyte needed to begin rapid flocculation of a

colloidal suspension. NaOCl treatment removed humic substances in soils which resulted

in decreased CCC values of a variety of kaolinitic soils at various pH levels. In another

study involving Na-montmorillonite, flocculation increased with increasing

concentrations of humic substances at all pH values (Tarchitzky *et al.* 1993). In these

studies, the relationship between concentration and coagulation, though positive, were not

linear.

2.2.1. Influence of Chemical Properties on Organo-Mineral Complexation

Humic acids unite with clay particles (< 2 μm) to form organo-mineral complexes and composites (Theng 1979). In addition to the nature of the humic substances, the degree of complexation is dependent on pH, concentration and type of background electrolytes, as well as mineral surface properties (Nayak *et al.* 1990).

2.2.1.1. pH.

Depending on the pH of the soil solution, humic acids (HA) have been shown to conform to different shapes. In solution, humic acids appeared as fibers or bundles at low pH, as a finely woven network at intermediate pH, and as plastic type sheets and fine grains at high pH (Senesi *et al.* 1977). According to Stevenson (1982) this might be caused by protonation and dissociation of the functional groups of HA, namely COOH and phenolic OH groups.

It has been suggested that HAs would undergo greater adsorption to soil when the pH is near the pK_a (or negative base 10 logarithm of the dissociation constant of an acid) of HA (Greenland 1971). Tarchitzky *et al.* (1993) observed that flocculation of Na-montmorillonite by humic substances was greater at low pH (4, 6 and 8) than higher pH levels (10). Another study showed that composites of montmorillonite and humic acids are formed in much greater amounts in acidic than basic solution (Ohashi and Nakazawa 1996). This was attributed to the dissociated nature of HA under alkaline conditions, which caused coulomb repulsion between molecules verses their protonated state in acidic environments.

An interaction effect on aggregation between the soil pH and salinity has been demonstrated. The was because the activity is influenced by the salt concentration. The

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pH at which dispersion of aggregated particles occurred was lowered as the concentration

of the background electrolytes increased (Spark *et al.* 1997). In the presence of HA the

critical pH for flocculation decreased with increasing humic concentration (Jones and

Bryan 1998). This suggested that humic acid was involved in stabilizing clay particles.

The apparent aggregation of HA particles at low pH has been attributed to Van der Waals

type intermolecular interactions, interactions between π-electron systems of adjacent

molecules, powerful H-bonding and /or homolytic reactions between free radicals (Senesi

et al. 1977). A pi bond is a "bond in which electrons are concentrated in orbitals which

are located off the internuclear axis: one bond in a double bond is a pi bond, and there are

two pi bonds in a triple bond" (Masterton *et al.* 1981). It was postulated that when the pH

rises, the binding forces diminish, and due to increasing ionization of acidic functional

groups, the particles separate. They begin to repel each other electrostatically, so that

molecular arrangements become smaller, thereby causing dispersion (Senesi *et al.* 1977).

2.2.1.2. Electrolyte. The concentration and nature of the background electrolyte have

been shown to strongly influence organic matter behavior on the soil. In an uncharged

solution, the humic polymer had the configuration of a random coil (Theng 1979). The

addition of NaOH increased dissociation of the carboxyl groups, which lead to uncoiling.

When the ionic strength of the solution increased further, there was recoiling due to

charge screening.

Studies involving clay - humic interaction conducted by Theng and Scharpensell

(1975) were summarized in Theng (1979). In general, the adsorption of humic acid

increased with valency of the saturating cation on montmorillonite. Furthermore, a good

linear relationship between humic acid adsorption affinity and the ionic potential of the saturating cation was found (Figure 1.1). The adsorption isotherm slope for monovalent cations was negative while that of di- and tri- valent cations was positive. This, it was argued, was due to the type of bonding involved: the humic acid was attached to monovalent ions through direct coordination of the functional groups while polyvalent ions were bonded via a water bridge.

Theng (1979) interpreted the relationship between the ionic potential of polyvalent cations and the degree of sorption of humic acids. Soil adsorbed cations are often surrounded by a hydration sphere, and initial adsorption of the humic acids is therefore, accompanied by a desorption of the water. There is an increase in the free energy of adsorption due to the increase in entropy associated with the displacement of the water molecules. Therefore, the free energy of adsorption would have been less negative with an increase in the polarizing power of the cation.

Bartoli et al. (1992) found that addition of poorly ordered Fe hydrous oxides had a positive influence on structure and stability of a friable temperate silt soil in the absence as well as the presence of humic macromolecules. In contrast, only a small aggregating effect was evident following addition of humic macromolecules alone. This demonstrated the importance of the polyvalent cation. Following organic matter addition, more preferential aggregation was detected in soils containing expanding clays, while soils containing Fe and Al oxides dispersed with organic matter addition.

As well, the presence of Fe or Al was shown to increase the amount of HA removed from solution by kaolinite and quartz and thereby favor aggregation (Arias et

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al. 1996). On the other hand, Ca adsorbs to clays much less strongly than either Al or Fe (Stevenson 1982). Arias *et al.* (1996) noted that the mineralogy had some influence on the amount removed. The percentage of HA the kaolinite samples retained ranged from 50.8-98.5% while the quartz samples retained 23.4-34.8%. Another study found that in a Ca-saturated montmorillonite suspension, sedimentation was facilitated by a dexitron polymer while flocculation was nearly non-existent when Mg $^{2-}$ was the exchangable cation present (Fuller *et al.* 1995).
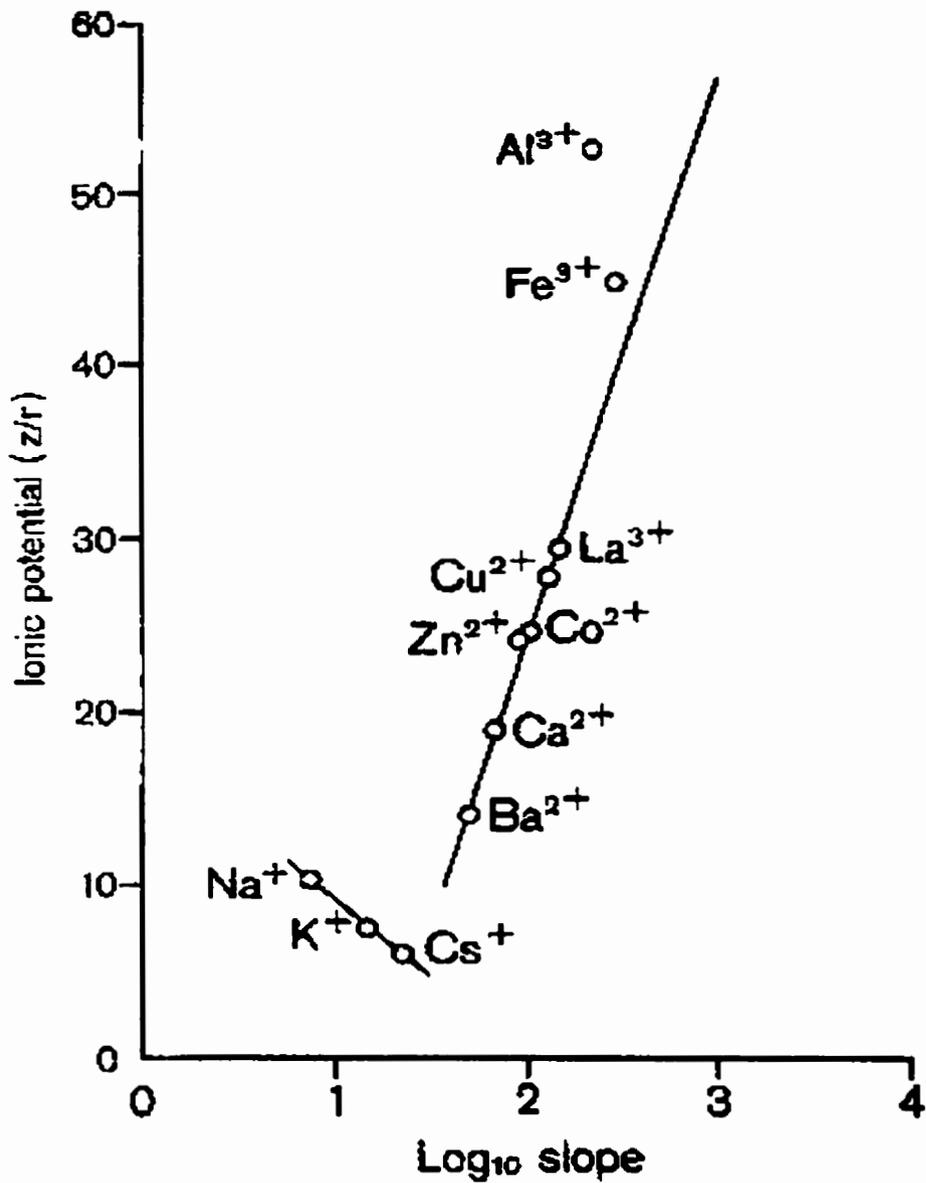
Figure 2.1. Relationship between the logarithm of the isotherm slope and the ionic potential of the saturating cation (Theng 1979). The log of the C type isotherm slope is a measure of solute surface affinity. The greater the value of log slope, the greater the affinity for adsorption.

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2.2.1.3. Mineralogy. Humic acid sorption has been shown to be related to the charge on the mineral surface. Surfaces with positive charges have shown higher sorbing capabilities than surfaces with negative charges (Spark *et al.* 1997). Adsorption of HA to Al and Si oxides on minerals including mordenite, kaolinite, and montmorillonite was described as pH dependent (Schulthess and Huang 1991). Furthermore, multivalent cations formed organo-metallic complexes that significantly increased adsorption, principally on Si sites. These observations lead to the conclusion that, in nature, the adsorption of aqueous compounds was highly dependent on the type of surface at the outermost layer where the solid phase was in contact with the liquid phase.

Optical and scanning electron microscopy further demonstrated the importance of the mineral surface. Caillier and Visser (1988) observed structural changes of various clay/silica associations following a 10-month contact with HA-enriched water from a peat source. They found that HA-treated minerals exhibited more developed structure than controls. Size and shape of aggregates varied for kaolinite, vermiculite and montmorillonite following treatment. Kaolinite aggregate diameters were 200 to 250 μm ovoid-shaped aggregates. Vermiculite particles became aligned with the major particle axis. Montmorillonite particles were positioned around and formed bridges with silica particles.

The role that organic matter plays in soil structure development has been well documented. However, a number of external factors may influence the aggregation effect. It is therefore necessary to consider not only the consistency of the organic matter, but also the composition of the soil and environmental conditions involved.

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2.3. Organic Matter Responsible for Soil Aggregation

The mechanism by which organic matter (OM) affects soil physical properties has been a topic of great discussion. In an effort to determine the mechanisms responsible for improvement of soil aggregation by organic components, Gu and Doner (1993) studied the effect of organic polyanions (including a soil humic acid, a soil polysaccharide, and a commercial anionic polysaccharide) and hydroxy-Al polycations (Al-p) on soil clay dispersion, aggregation and hydraulic conductivity. They found that dispersion increased and aggregate stability of a sandy loam soil decreased with the addition of organic polyanions.

Addition of HA caused the greatest dispersion, followed by soil polysaccharide, and anionic polysaccharide. In addition, destruction of OM with H_2O_2 resulted in lowered dispersion. This would indicate that the organic matter was dispersing clay. However, with the addition of Al-p to all treatments, dispersion was substantially suppressed. Mean weight diameter (MWD), as observed with wet sieving, showed treatment with HA was similar to the control, and soils treated with Al-p and Al-p plus HA resulted in only slightly increased MWD.

Hydraulic conductivities were lowest in soils treated with HA. Furthermore, soils that did not receive Al-p treatment had lower hydraulic conductivities. They were two orders of magnitude lower than those treated with Al-p or its combinations with either anionic polysaccharide or HA. It was postulated that while HA (and other humic

substances) may not contribute to stable soil aggregation as such, the combination of both polyvalent cations and polyanions may be additive in minimizing soil clay dispersion. This confirmed the results of an earlier study which showed that organic matter bonded to the clay particles through association with Al or Fe was highly important for aggregate stability (Hamblin and Greenland 1977).

In another study, clays treated with extracted humic substances were more resistant to weathering (measured by release of Mg and K in dilute sulfuric acid) than those treated with H_2O_2 (Novak and Smeck 1989).

Humic acids have been shown to be very resistant to microbial degradation, so their influence on aggregate stability should have little variation with time. Surface-adsorbed HAs and glucose addition to soils both caused increased aggregate stability (Chaney and Swift 1986a). In the first treatment, aggregate stability decreased only slightly with time. By contrast, addition of glucose and soil incubation resulted in an initial increase in aggregate stability, which began to decline after 12 weeks and reached zero after 21 weeks. It was suspected that this was due to production and decomposition of microbial extracellular polysaccharides, including gums and mucilages, with time.

In summary, humic substances appear to be important for soil aggregation and structural stability. Therefore, application of an organic amendment is likely to have positive effects on the physical nature of the soil. However, there has not been enough conclusive evidence to suggest that the mere addition of humic substances to the soil will increase aggregation. Furthermore, the effects of humic acids on soil physical properties have not been well documented. There is a need to study effect that the individual

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components of organic matter have on the soil in order to determine the main mechanisms involved.

Soil organic matter consists of a variety of components at various stages of decomposition, including HAs. These acids are less susceptible to microbial decomposition than many other organic materials. Therefore, any impact that they may on the soil have is likely to have persistent effects.

2.4. Effects of Anthropogenically Added Humates on Soil Structure

There has been a decline in soil organic matter with time due to cultivation (Poyser 1950). Application of organic amendments not only replace some of the carbon that will inevitably be lost, but additionally, many researchers have claimed that humic substances play an important role in the maintenance of soil structure.

Effects of humic acids on soil physical properties are poorly documented and contradictory. Some researchers have found HA addition to soil improves its structure (Chaney and Swift 1989 a & b; Swift 1990; Piccolo and Mbagwu 1989), while others have shown it to degrade structure and disperse particles (Painuli and Pagliai 1990; Painuli et al. 1990; Visser and Caillier 1988).

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2.4.1. Positive Effect of Humic Acids on Soil Aggregation

A number of studies have shown that HAs have positive effects on soil structure. Soils amended at rates of 2 to 4 Mg/ha with nitration-transformed-HA extracted from peat showed increased soil aggregate stability as assessed by the dry sieving method (Almendros 1994). Gati (1982) summarized findings of Tomko (1973), who also found that HA (extracted from brown coal) increased the number of water stable aggregates. Piccolo and Mbagwu (1989) investigated differing rates of leonardite-extracted HAs on aggregate stability of SL and C soils. The positive effect was most pronounced in the sandy loam soil.

Brandsma *et al.* (1999) measured effects of a HA based conditioner "Humus" (Humus of America Inc., Texas) on physical properties of a Bridgnorth Series loamy sand in England. The rate of application was 2.4 L/ha. Results suggested that the material had no effect on crust strength (measured with a penetrometer) or aggregate stability (fraction remaining on a 1 mm sieve following rainfall simulation). However, the same material resulted in a decreased bulk density (measured with cores), increased total porosity (calculated using bulk and particle density values), and increased retention of soil moisture by top soil.

2.4.2. Dispersion of Soil Aggregates by Humic Acid

Dispersion of soil via HA addition may occur in a manner similar to that of calgon (Greenland 1971). If the concentration of the polymer is very high, the polymers saturate colloidal particles and increase osmotic pressure between the particles. In effect, the electrostatic double layer is extended. Oades (1984) thought that this was due to formation of complexes between organic anions and metal ions or by the abnormally high concentration of acidic functional groups in HA. It was believed that these groups, which complex Fe and Al cations, caused instability by increasing the negative charge. He proposed that the negative charge extends the diffuse layer and thereby favors dispersion. As well, cations such as Ca^{2+} and H^+ could have caused precipitation of HAs so that they were unable to interact with clays (Painuli and Pagliai 1990). However, organic matter repels water so it should countercheck the forces and decrease the dispersion to some extent. On the other hand, at lower concentrations, the polymer may extend beyond the electrostatic barrier (which is dependent on the thickness of the diffuse double layer) and flocculation may occur.

2.4.3. Modification of Humic Acid and Soil Surface Properties

It is believed that the chemically altered HAs may result in more pronounced effects on soil physical properties than those that are unaltered. Almendros (1994) modified the reactivity and/or structure of HAs extracted from peat, which changed molecular weight, functional groups, aromatic to aliphatic ratio and total amount of N. This caused increased water solubility in a number of samples. In most cases, the effect

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of the transformed HA on soil structure was more pronounced than that of the original HA.

Ammonia fixation (AMO) was one of the HA transformations conducted in the previous study. The HA was combined with aqueous ammonia mixed with water and oven dried at 95°C for 5 hours. The transformation resulted in increased water solubility and N enrichment. Compared to an untreated soil and a soil treated with the original HA, application of AMO resulted in increased mean weight diameter (using the dry sieving method) and aggregate stability to water following benzene pretreatment. Application rates were 2 and 4 Mg/ha.

Another study used ammonia to improve effects of HA on soil stability. HAs extracted from coal react with ammonia to produce ammonium humate salts (Whiteley 1993). A dispersion of lignite made using 0.1 M aqueous ammonia was added to air dry soil. The slurry, which was added to a subsoil low in organic matter at mixing ratios of 5 to 10 % (w/w), resulted in significant increases in aggregate stability measured by wet sieving.

The soil/HA mixture, itself, has also been modified in past studies. In a study conducted by Chaney and Swift (1986b), a NaOH/HA slurry was added to mono-ionic soils. Soils were leached with distilled water followed by $CaCl_2$ to convert the soil-humic complex to the calcium form. While physical addition of soil derived HAs to soil showed no significant improvement in structure, addition of surface-adsorbed HAs showed increased aggregate stability of Sterling soils. It should be noted, however, that this method would not be possible to carry out in large scale agricultural practices.

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2.4.4. Effect of Origin of Humic Acid on Soil Structure

The effects that humic acids have on soil structure were found to be a function of

the parent material from which they were derived (Malcolm and MacCarthy 1986;

Canarutto et al. 1996) and the rate of application (Visser and Caillier 1988; Piccolo and

Mbagwu 1989). It is generally believed that humates from varied parent materials have

different elemental and structural compositions with different functional groups.

Terrestrial humic matter has been shown to contain mainly lignoprotein complexes with

humic and fulvic acids being the major components (Lobartini et al. 1992; Tan 1993).

However. geologic humic matter (found in geologic deposits such as lignite and

leonardite) contains mainly HAs. as most of the fulvic acids have been polymerized. As

well. the HAs obtained from less evolved materials (including sludge and compost) have

a higher nitrogen compound content. lower oxidation. more heterogeneous composition

and a higher aliphatic nature than those derived from the more evolved materials (peat

and leonardite) (Ayuso et al.. 1997).

Malcolm and MacCarthy (1986) studied seven commerical "humic acids"

obtained from five different suppliers. as well as HAs isolated from streams. soils. peat.

leonardite and doplerite. They concluded that commercial HAs were all quite similar

irrespective of the supplier. but were markedly different than humic substances obtained

from soil and water. Additionally. commercial HAs are similar to leonardite humic acid

and to doplerite.

The molecular weight of the organic compound would have some bearing on the

amount adsorbed to the soil (Theng 1979). In general, on non-porous adsorbants, an increase in the molecular weight of the polymer would lead to an increase in the surface segment bonds. This is due to the increased probability of the neighboring segments (and/or functional groups) being attached following the attachment of one segment. However, soils contain pores and there may be an inverse relationship between adsorption and molecular weight of the polymer. The entry of the polymer into the interlayer spaces of the clay system and/or inter-domain spaces of a soil aggregate (in effect, the pore spaces) may be limited by the polymer size.

The origin and characteristics of the humus fractions have some bearing on their influence on soil structure. Humates from green waste compost within the range of 1000 to 8000 mg/kg improved structure of a silty clay soil (Canarutto *et al.* 1996). On the same soil, rates of 4000 to 8000 mg/kg of a leonardite HA extraction negatively affected soil shrinkage by increasing the area of cracks produced following drying from a slurry. These rates also caused disaggregation of water-stable particles (measured with the laser scattering technique used by Pini and Guidi (1989)). On the other hand, HAs of brown coal copolymerized with vinyl monomers increased the proportion of water-stable aggregates from approximately 1-2% for the controls to 33-99% for treated samples (Dzhanpeisov *et al.* 1984). The proportion of water-stable aggregates was influenced by the amount of amendment applied, their storage time, the way in which they were synthesized, and the nature of the soil and of the carbohydrate monomer grafted to HAs. Positive results were observed when concentration of the monomer was low and the rates were in the order of 2-3% by weight. The conditioning effect declined and aggregate

stability was similar to the controls after 3 months. Effect on aggregate stability of grafted monomers ranged from high to low in the following order: acrylamide. methylmetacrylate. vinyl butyl ester. monovinyl ester of glycol. and acrylidic acid. However. the addition of physical conditioners from sources not found in nature is a declining proposition from the point of view of soil environmental health.

Comparison of effects of humic and fulvic extractions from a manure (FHM) and a peat (FHP) on sandy loam and clay soil aggregation further demonstrated the importance of the source (Fortun *et al.* 1990). Both humic extractions had molecular weights greater than 200.000 and both treatments caused the appearance of large irregular pores and narrowed planes in the soils indicating structural improvement. However. the effect on soil structure varied: the FHM converted small aggregates to large aggregates. while the FHP increased the numerical density of smaller aggregates. It was postulated that because the FHM was more aliphatic. there were a higher number of linkages. This could have caused binding between large particles and planes to fill in. The FHP had a greater proportion of polymerized molecules with less free carboxyl groups. Due to this. formation of organo-mineral complexes was slower. As well. there was only an increase in binding between clay particles. and not by binding between large particles as was apparent for the FHM. This explained the high occurrence of small aggregates.

2.4.5. Results of Different Rates of Amendment

The amount of HA added may affect the degree of soil aggregation or dispersion. Dispersion of clay in CL soil occurred when concentrations of soil-extracted HAs were in the range of 1-400 mg/l with an optimum of 40 mg/l (Visser and Caillier 1988). Dispersion was most efficient for concentrations in the range of 25 to 100 mg/l; higher concentrations resulted in flocculation, which was increased with the quantity of HA. Similarly, clay particle dispersion had been correlated with the amount of organic matter adsorption. High organic matter (3.4-4.4% C) corresponded to a high degree of aggregation, while clays containing a small amount of organic matter (0.5-2.5% C) were highly dispersed (Dong et al. 1983). Kuznetsova (1998) suggested that the proportion of water-stable aggregates in Chernozems was optimized when organic carbon was greater than 3% and the humic acid to fuvic acid ratio was greater than 1.5.

Piccolo and Mbagwu (1989) investigated differing rates of leonardite-extracted HAs on aggregate stability of sandy loam and clay soils. HA increased the amount of water-stable aggregates with increasing rates (0, 10, 50, 100, 1000, and 10 000 mg/kg). The effect was most pronounced in the sandy loam soil.

2.4.6. Surfactants on Effectiveness of Humic Acid

Surfactants and HA may have interactive effects on soil structure (Piccolo and Mbagwu 1989). A surfactant is a substance that lowers the surface tension of a liquid. It has a hydrophilic and hydrophobic end. Nonionic surfactants have a neutral charge on the hydrophilic end and have a small positive to no effect on aggregate stability while

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anionic surfactants have a negative charge on the hydrophilic end and reduce aggregate stability.

Nonionic surfactants (NS) and leonardite-extracted HA had a positively synergistic effect, increasing micro-and macro-aggregate stability in temperate soils (Piccolo and Mbagwu 1989). The effect was slightly higher in clay than the sandy loam soil. However, in the same study, the interaction between anionic surfactants (AS) and HA lowered the stability of SL, yet improved that of the clay soil at both macro- and micro-levels of aggregation. Differing results may be due to the surfactants' hydrophilicity. The NSs may have left a hydrophobic coating on particle surfaces, while ASs caused increased water infiltration. The researchers suggested that in clays, HA may have the ability to suppress the tendency of AS additions to decrease soil aggregate stability.

In a more recent study, similar results were found for tropical soils (Piccolo and Mbagwu 1994). HA alone increased macro-aggregate stability in both a weakly aggregated Entisol and a strongly aggregated Ultisol. AS caused clay dispersion, but HA limited this. NS caused increased aggregation, and the effect was enhanced when applied in combination with HA. However, HA caused increased dispersion in the Ultisol. As well, all combinations reduced microaggregate stabilty of the Ultisol compared to the controls. The researchers suggested that these findings may be useful to consider in zero tillage systems, where surfactants are commonly found in herbicide formulations.

In summary, organic amendments have been used to improve soil structure in an endeavor to increase productivity of the land. The effects of amendment of humates

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have been variable. Therefore. it is necessary to examine a number of application rates

and forms of the amendment in order to find the most optimal usage.

2.5. Effect of Lignite Coal on Soil Structure

Lignite from different sources and/or at different levels of oxidation will exhibit

different physio-chemical properties (Sharma *et al.* 1993). A degraded lignite. such as

leonardite. has had high oxidation of lignin. It also has a high occurrence of waxy

compounds. similar in behavior and appearance to those found in Mor (soils under forest

vegetation) litter layers which also have a large amount of highly water repellent surfaces

(Richardson and Wollenhaupt 1983). The water repellency in the Mor layers. however. is

mainly due to the appearance of fungal mycelia (Richardson and Hole 1978). The

hydrophobic properties of oxidized lignite may influence water and nutrient uptake as

well as water movement due to its high water repellency. Commercial lignite has higher

water repellency than degraded lignite due to undecomposed lignin-type organic

polymers on its surface (Sharma *et al.* 1993).

Lignite addition has been shown to have a favorable effect on soil physical

properties. Rates of lignite addition of 3.57. 5.95 and 8.33 ton/ha on sandy soil caused an

increase in initial surface infiltration rates of 1.3 to 3.4 times those of control plots (El-

Abedine and Hosny 1982). As well. field capacity was increased. While increase in crop

yield was not statistically correlated to field capacity or infiltration rate, it was correlated

to the surface infiltration rates at the end of the season.

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Some studies have shown that lignite should be altered prior to soil application.

A study was carried out in which physical addition of lignite in powder form had no

effect on soil aggregate stability (Whiteley 1993). In the same study, surface adsorbed

lignite, based on methods of Chaney and Swift (1986b), promoted aggregation in soil

containing a large clay fraction, however no significant treatment effect was evident for

coarser soils, the latter probably due to reduced surface adsorption of lignite.

Additionally, Gati (1982) cited findings of Barna (1973 and 1976) that lignite which had

been decomposed into a colloidic state, increased mechanical stability of the soil surface.

It formed a thin, "film-like layer" on the soil which decreased evaporation losses from the

soil while not affecting infiltration.

2.5.1. Interaction of Polyacrylamide and Lignite on Yield

Organic amendments can be costly to purchase and apply. It may be

economically advantageous to consider combinations of amendments to reduce the total

amount of amendment needed. Both soil organic matter (OM) and polyacrylamide

(PAM) have been shown to bind clay particles causing aggregation (Oades 1984), but

PAM can accomplish the same aggregation in smaller amounts than soil OM.

Interactions between PAM and lignite gave additive and synergistic effects on plant

growth, supposedly due to improvement in soil physical properties of Yolo Loam and

Xerothents subsoil (Wallace and Wallace 1986). The greatest interaction effect was

observed when tomatoes were grown in the Xerothents subsoil. In the absence of lignite,

112 kg/ha PAM had only a minimal effect on yield compared to controls. However,

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when PAM was used in combination with 2.2 Mg/ha lignite the yield almost tripled. This suggested that addition of lignite had a sparing effect on the amount of PAM required for soil conditioning. Differences between crop yield were attributed to the fact that different plants require different degrees of aeration.

It has been shown that lignite amendment could influence soil aggregation. Therefore, it is hypothesized that a degraded lignite may have similar effects. In summary, synergistic effects between lignite and other soil conditioners have demonstrated. As well, it appears that soils low in organic matter may show more response to lignite than those which had contained significant amounts of it prior to amendment addition.

2.6. Influence of Humic Acid on Crop Productivity

The main benefits of soil organic matter on plant growth result indirectly through improvement of soil structure, however, plant growth could be affected in other ways. Organic compounds have been shown to decrease metal toxicity, and stimulate root growth and germination (Tan 1993).

2.6.1. Influence of Humic Acid on Germination

The addition of humic substances has shown varied response on germination for different species. The most advantageous concentration is largely dependent on the seed under consideration and the composition of the original material (Ayuso et al. 1996).

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For the more evolved humic substances (including leonardite, peat and commercial

"humic acid") stimulatory effects were found at high rates (500 and 100 mg C/liter) for

watercress and tobacco and lower rates (200 and 500 mg C/litre) in the case of barley.

For the less evolved humic substances (such as sewage sludge and compost), stimulation

occurred for germination of watercress and barley, but not tobacco. Optimum amounts

differed substantially for different seed types. Humic substances obtained from more

evolved materials gave more consistent results than those from less humified organic

materials. Further, the less humified materials had a more negative effect on germination.

probably because of the appearance of phytotoxic compounds in the younger materials.

Another observation in the study was that humic substances had a more stimulatory effect

than humic acid extractions of identical materials. This suggests that some active.

alleropathic material may be lost in extraction.

2.6.2. Influence of Humic Acids on Root Growth

Many studies have shown that HA can influence root growth. The effect appears to

be dependent on the origin of the HA and the plant species. Vaughan (1974) postulated

that humic substances might form strong Fe complexes with HAs and thereby reduce

wall-bound hydroxyproline, so that cell membrane permeability is increased. As well.

humates found in leonardite may contain small amounts of polyphenolic compounds.

possibly providing auxin activity, thereby increasing root growth and abundance

(O'Donnel 1973).

At low concentrations. HAs may have a stimulatory effect, while at very high

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concentrations they may inhibit root growth . Tobacco root number and length was increased as the concentration of HA increased from 0 to 100 ppm and then declined as concentration increased further to 1000 ppm (Mylonas and Mccants 1980).

Malik and Azam (1984) observed a five fold increase in wheat root length when 54 mg/l of soil-extracted HA was added to water as compared to wheat grown in water alone. Further, there was increased plant growth and biomass. Shoots were elongated due to increased moisture uptake as influenced by enlarged root surface area. However, humates had very little effect on container-grown Turkish hazelnut (Kelting et al. 1997). Only one of four treatments (granular humate) fertilized with 2.5g N/container showed increased root length.

2.6.3. Influence of Root Growth on Soil Structure

Soil macroaggregation may be increased indirectly with addition of HAs. Studies have indicated that HAs stimulate root growth (Ayuso et al. 1996; Mylonas and Mccants 1980). This stimulation may reflect on soil macroaggregation, which is mainly influenced by temporary binding agents including root exudates and fungal hyphae (Tisdall and Oades 1982). Aggregate stability has been shown to be greatly influenced by the growth and activities of living roots (Reid and Goss 1981).

Tisdall and Oades (1982) have documented the mechanisms by which root growth influences soil structure. Roots enmesh soil particles to form stable macroaggregates. As well, root residues add carbon to the soil system. In this manner, food is provided for soil organisms including earthworms. Earthworms contribute to soil structure through mixing

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soil with organic materials in the gut. As roots absorb water, localized drying around roots causes soil shrinkage, bringing soil particles together and thus increasing the amount of water stable aggregates (Harris *et al.* 1965). If root growth can influence formation of stable aggregates, then a substance that can stimulate root growth could indirectly have some bearing on soil structure.

Past studies have indicated that addition of amendments to soil can influence soil structure and crop growth. In summary, humic acids may effect crop production either by having a direct influence on the crop itself, or an indirect influence due to a change in the soil structure.

3. EFFECT OF LEONARDITE FIELD APPLICATIONS ON SOIL STRENGTH AND DENSITY

Abstract

Measurements were taken to determine if leonardite could improve root growth by decreasing soil strength. Effects of a single, spring application of leonardite were measured two weeks prior to fall harvest using a handheld Bush penetrometer. During this time. gravimetric moisture was evaluated. Following fall harvest. soil samples were extracted for bulk density and modulus of rupture measurements.

No significant treatment effect was evident from penetrometer readings or gravimetric moisture content in 1998. In addition. leonardite treatment had no significant effect on bulk density or modulus of rupture.

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3.1. Introduction

Crop productivity has been shown to be related to soil physical properties. Soil strength as measured by penetrometer resistance is an index of a soil's compaction, moisture content, texture and the type of clay in the soil (Baver *et al.* 1972). Penetrometer resistance (PR) and bulk density (BD) are good predictors of root system proliferation (Thompson *et al.* 1987), as they are an indication of soil strength. Bulk density has an indirect effect on emergence in that it has been shown to affect oxygen diffusion rate, and the corresponding air pore space. Air pore space was shown to be a limiting factor for plant growth (Hanks and Thorp 1956).

Overall soil strength is a critical impedance factor controlling root penetration (Taylor and Gardner 1963). Studies have found that proportion of wheat roots penetrating the soil was inversely related to PR (Martino and Shaykewich 1994). Strength is affected by both soil moisture and bulk density (Taylor and Gardner 1963).

Previous studies have shown that HA based conditioner was able to increase moisture retention by top soil (Brandsma *et al.* 1999). Further, increasing rates of manure increased plant available water (PAW) in sandy and clay soils (Hafez 1974) while PAW was decreased in a dark brown Chernozem (Sommerfeldt and Chang 1986). An increase in PAW would have increased the water available for plant roots. This positive effect was expected in the current experiment since soil organic matter has been shown to increase the water holding capacity of the soil (Hassett and Banwart 1992).

Bulk density (BD) is a measure of the density of an undisturbed soils. Therefore an

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alteration in the total porosity of the soil would be observed through measurement of BD. Tillage and reduction in organic matter often to lead to soil compaction. In these cases, it is desirable to increase the total pore space in soils. An increase in porosity translates to a reduction in BD. Often, BD can be reduced with organic amendments (Tester 1990; Mbagwu and Piccolo 1990), while some studies (Black Chernozem) have shown it to be unaffected (Campbell *et al.* 1986).

The modulus of rupture measurement has been used as an indicator of soil crust strength. It is determined through measuring the force required to break a briquette formed from wet soil which is dried in an low temperature oven. It is based on two assumptions: (1) the force required to break the briquette is in essence the force that emerging seedlings must expend to break soil crusts; (2) the soil briquette's physical properties simulate those of naturally formed crusts (Baver *et al.* 1972). Modulus of rupture has been altered with organic amendments (Hafez 1974).

The study had two objectives: first, to examine the impact that leonardite amendment had on soil strength; and second, to determine the effect that leonardite had on soil density. It was hypothesized that leonardite would lower soil strength and bulk density.

3.2. Materials And Methods

Several experiments were conducted on Manitoba field plots to evaluate the effect of field applied leonardite amendment on soil physical properties. Two of these experiments (named Penetration Resistance Determinations and Modulus of Rupture

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Measurements) were selected for the present study. The experiments were conducted on soils from the same field plots. Selected soil properties for these sites are given in Appendix A.

The field plots were established on two soil types: Osborne Clay Humic Rego Gleysol (Figure A.3.) and Waitville Clay Loam Dark Gray Luvisol (Figure A.4.). (legal locations were NW 6-5-2W and SW 2-17-17W. respectively). Soils were chosen with the view of improving their poor physical properties following the application of leonardite. The soil high in clay was prone to compaction while the Luvisol was highly susceptible to surface crusting.

3.2.1. Field Plot Design

The field plots had dimensions of 25.5 m by 46 m. They were arranged in a randomized complete block design with split plots which were fertilized (+F) and not fertilized (-F). There were 4 replicates. each containing three treatments and one control (C) (Appendix A). Each treatment subplot was 3.2 m by 5 m.

On May 5. and May 22. 1998 leonardite amendments were added to Waitville clay loam (CL) and Osborne clay (C) soils. respectively. immediately prior to seeding wheat (*Triticum aestivum* L.). Leonardite was added to the soil surface and rototilled to approximately 7 cm. Treatments included an insoluble pulverized leonardite powder (P), a ground leonardite material <2 mm diameter (M). and a liquid leonardite suspension that was thought to be contain high amounts of humic acid[1] (H). The P amendment was

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[1] The majority of the extraction was humic acids, but fulvic acids were also found to be present.

mixed with soil a small quantity of soil from the subplots prior addition as during application it was highly susceptible to wind erosion due to its fine particle size. The M was added directly to the soil surface and the H suspension was hand mixed immediately prior to its application on the soil surface. Selected properties of the leonardite amendments are contained in Appendix B.

Application rates of P. M and H were 150 kg/ha, 1000 kg/ha and 100 L/ha respectively. The quantities of P and M amendments applied were based on previous studies conducted in Lethbridge. In these studies a number of application rates (including 0, 100, 500 and 2000 kg/ha) of P and M were evaluated. According to the findings, rates of 150 kg/ha P. and 1000 kg/ha M were predicted to be the most beneficial to crop productivity[2]. The H amendment was a new product that had not previously been studied. The application rate of 100 L/ha used in the study was based on the organic carbon content of the H amendment.

3.2.2. Field Operations

In year one. AC Barrie wheat was seeded May 5. and May 22, 1998 and harvested August 25 and August 27, 1998 in the Waitville clay loam (CL) and Osborne clay (C) soils, respectively. In year two, wheat was seeded June 2 and harvested October 6, 1999 in the Waitville CL soil. Flooding prevented seeding on Osborne C. and second year analysis of the soil was not acceptable.

Spring fertilizer application rates were as follows. In 1998, the Osborne C soil

[2]Research conducted in Lethbridge by L. Cramer, W. Akinremi and H. Janzen, Agriculture Canada.

received 20 kg/ha nitrogen (85 kg/ha had been applied in the previous fall) and 20 kg/ha

phosphorous. The Waitville CL soil received 125 kg/ha nitrogen. 20 kg/ha phosphorous.

and 24 kg/ha sulphur. In 1999. 150 kg/ha nitrogen. 40 kg/ha phosphorous and 24 kg of

sulfur was applied to Waitville CL. Nitrogen was broadcast in the form of urea.

phosphorous was applied with the seed in the form of monoammonium phosphate. and

sulphur was applied as ammonium sulphate.

3.2.3. Penetration Resistance Determinations

Penetrometer resistance readings (PR) were only conducted in 1998. Readings were

taken approximately two weeks prior to harvest (August 13 and 10 for Osborne C and

Waitville CL respectively) when average gravimetric water content was 25 % and 23% in

the Osborne and Waitville soils respectively. A Bush penetrometer (Findlay. Irvine Ltd..

Penicuik. Scotland) measured soil resistance to penetration of a 12.9 mm diameter cone

with a 15° semiangle. Resistance of 10 depths at intervals of 3.5 cm was measured. Four

determinations for each treatment within the same replicate were taken. To evaluate

gravimetric soil moisture. two soil samples per treatment within each replicate were

extracted using a backsaver probe for depths of 0-5 cm. 5-10 cm and 10-20 cm. Soil was

weighed. oven dried at 110°C for 48 hours and re-weighed.

3.2.3.1. Soil Moisture and Density. Following fall harvest. soil samples were extracted

at depths of 0-5 and 5-10 cm using copper cores 1.8 cm in height and 1.95 cm radius.

Soil was transferred into pre-weighed plastic bottles and dried at 110°C. Bulk density

(BD) is the ratio of the mass of an undisturbed oven dry soil sample to the volume of space it occupied. Bulk density was calculated according to Eq [1]:

$$D = m/V \qquad [1]$$

where: D = Density
m = Mass
V = Volume

Particle density (PD), defined as the mass of solids per unit volume, was assessed using pycnometers (Blake 1986). Pycnometers and their stoppers were filled with de-aired distilled water at room temperature and mass was measured. Half of the water was removed, pycnometers without stoppers were weighed and approximately 5 grams of soil was added. Total mass was measured and soil mass was calculated according to Eq. [2]. To remove dissolved air, the soil water mixture was boiled for 10 minutes and cooled to room temperature. Pycnometers were filled with water and capped with the stopper. Mass was recorded and mass of water displaced from soil was calculated. Based on Eq. [1], and using the density of water for the given temperature, the volume of water displaced (in effect the volume of soil added) was calculated. Applying values for mass and volume of soil, particle density was calculated according to Eq. [3].

$$(m_{PSWI.2} - m_{PWI.2})/(1 + w) = m_S \qquad [2]$$

where: $m_{PSWI.2}$ = mass of pycnometer half full of water plus soil
$m_{PWI.2}$ = mass pycnometer half full of water
w = fraction air dry water content of soil
m_S = mass oven dry soil

$$m_{WD} = m_{PW} - (m_{PSEST} - m_S) \qquad\qquad [3]$$

where: m_{WD} = mass water displaced
        m_{PW} = mass pycnometer and water full
        m_{PSEST} = mass pycnometer. soil. water and stopper
        m_S = mass oven dry soil

3.2.4. Modulus of Rupture Measurements

Samples of Osborne C and Waitville CL were collected July 9 and 10. 1998.

respectively for baseline analysis. The outcome of the following analysis indicated

whether soil crusting was a limiting factor for plant germination in either of the soils.

Four baseline samples were collected adjacent to the experimental plots. where no

fertilizer had been applied. In addition. samples were collected immediately following

fall harvest.

Using the method of Reeve (1965). modulus of rupture was determined. The soil

obtained from bulk sampling was ground and passed through a 1 mm sieve in preparation

for modulus of rupture (B') determination. Soil was poured into lubricated briquette

molds underlined with filter paper on a screen lined tray. Molds had dimensions for

length. width and height of approximately 7. 3.5 and 1 cm. Briquettes were wetted at

their base. soaked for one hour and oven dried for 24 hours at 40°C. Force required to

break the molds was determined using an Ottawa Texture Measuring System for 1998

samples. This measuring system resulted in large error and as a result. 1998 values are

not included in this chapter. In 1999. force was measured using a Hoskin Scientific

Limited Modulus of Rupture Apparatus. Measurements of the width and thickness of the

briquette as well as the distance between the two lower supports (Figure 3.1.) were taken

to one tenth of a millimeter using calipers. Modulus of Rupture was calculated using Eq. [4].

$$B' = 3FL/(2bd^2) \qquad\qquad [4]$$

where:

    B' = the modulus of rupture (dynes/cm^2)
    F = the breaking force (dynes)
    L = the distance between the two lower supports (cm)
    b = the width of the briquet (cm)
    d = the thickness of the briquet (cm)
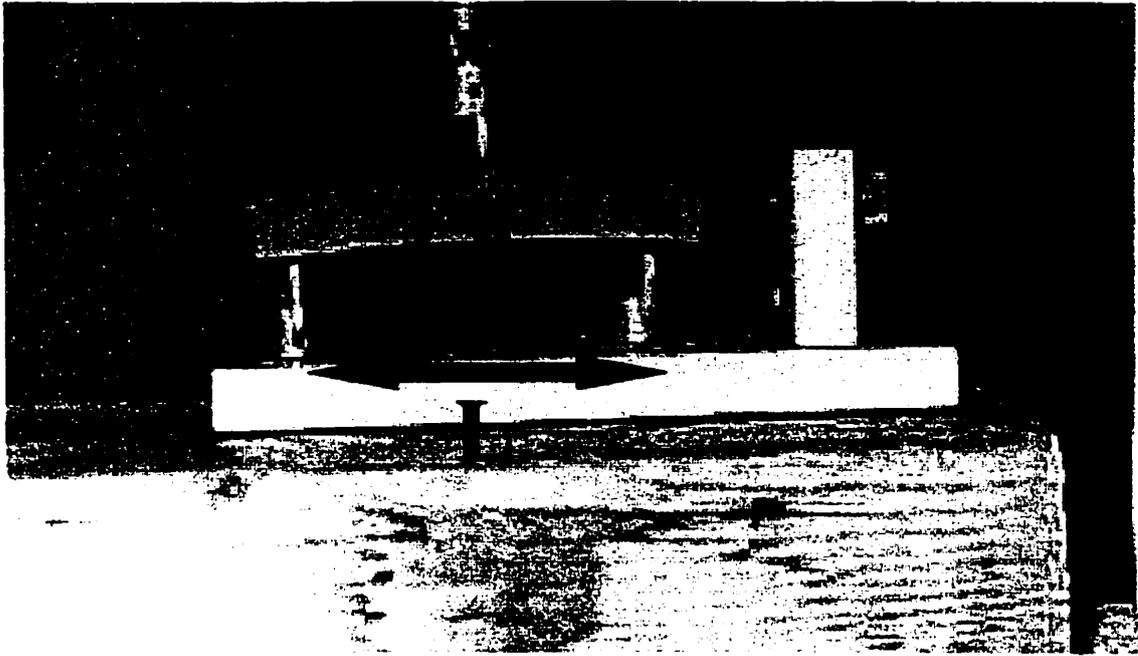
Figure 3.1. Modulus of rupture assembly for quantifying crust strength according to Eq. [4]. The rupture force is applied to a soil briquet of width (b) and thickness (d) at the midpoint of the length between the two lower supports (L).

3.2.5. Statistical Analyses

Analyses of variance (ANOVA) tests were performed using JMP IN software (SAS Institute 1997). Fertilized and unfertilized plots were examined separately. For each soil depth, penetration resistance, soil moisture, bulk density and modulus of rupture data were analyzed individually. The LSD test was used to compare treatments found to be significantly different in ANOVA at the 95% level of significance.

3.3. Results and Discussion

3.3.1. Effect of Leonardite on Penetration Resistance in the Soils

Organic material has been shown to increase soil aggregation and contribute to structural stability. Increased aggregation should result in greater macro-porosity (porosity between individual aggregates). As such, it was believed that the soil should be loosened with organic amendment addition. Soil strength is dependent on macro-porosity and it was hypothesized that resistance to penetration would be lowered with organic amendment addition. If that had been the case, resistance for treated soils would be less than the control soils for equal penetration depths.

It has been suggested that the effect of organic carbon on soil strength is more dramatic in fine than in coarse textured soils (Kay 1997). In soils high in swelling clays including montmorillonite, such as the Osborne C, the strength of bonds between aggregate particles was dominated by clay which acts as a cementing agent when dry. Organic carbon was thought to reduce the dispersibility of the clay. Therefore, it was hypothesized that the Osborne C soil would show most response to the leonardite treatment.

Figures 3.2.-3.3. show the results of penetration resistance (PR) obtained for the Osborne C leonardite treated soils. It was hypothesized that the amendment would not flow freely in the soil, and as variability in penetration resistance appears to increase with depth, data below a 14 cm depth were thought to be unreliable. All measurements between 0 cm and the above depth (both fertilized and unfertilized) showed there to be no

differences between treatments or the treatments verses the controls.

Figures 3.4.-3.5. show the results of penetration resistance (PR) obtained for the Waitville CL leonardite treated soils. All measurements between 0 cm and the 14 cm (both fertilized and unfertilized) showed there to no treatment effect in either the fertilized or unfertilized soils.

The Mean Square Error (MSE) was extremely high and may have masked any small treatment effect. Possible causes of variability include problems inherent in the instrument design (Lowery 1986). To obtain accurate readings. the cone penetrometer should be inserted into the soil at a constant force. This is extremely difficult to do manually. Further error could be caused by failure to hold the mechanism vertical during insertion. Penetrometer resistance had been shown to vary markedly with time between readings as a direct result of changes in moisture content (Martino and Shaykewich 1994). As the water content was measured separate from the penetrometer. error due to spatial variation is likely (Young et al., 2000). A decrease in matric potential results in an increase in cone resistance. This change should be approximately linear in soil where matric potential is greater than -15 kPa; beyond this, high variability should be expected (Steinhardt 1974).

Table 3.1. shows that the measured value was well below the -15 kPa gravimetric water content of the Osborne C. This low water content may have contributed to the high resistance. Consequently. high penetration variation may have resulted due to change in penetration speed. Penetrometers are a fairly crude method of analysis and it is possible that subtle conditioning changes in aggregation may have been missed using this type of

instrument (Brandsma *et al.* 1999). Therefore, a study was conducted to observe the

effect of leonardite on aggregate size distribution and stablility of the two soils as

assessed with dry and wet sieving, respectively (Chapter 4).

Based on the above, the PR experiment was excluded from the 1999 investigations.

Further, the results of the PR experiment in the 1998 season were inconclusive due to the

high degree of error. Error may have been minimized with the use of a mechanical

(rather than manual) insertion probe.
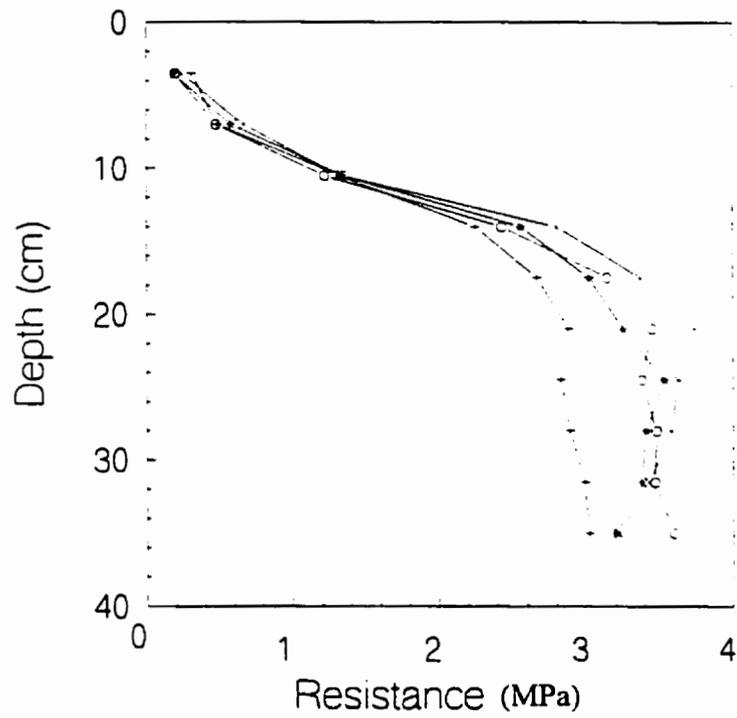
Figure 3.2. Mean soil penetration resistance (PR) profiles for each leonardite treatment applied at the fertilized Osborne clay study site 1998.
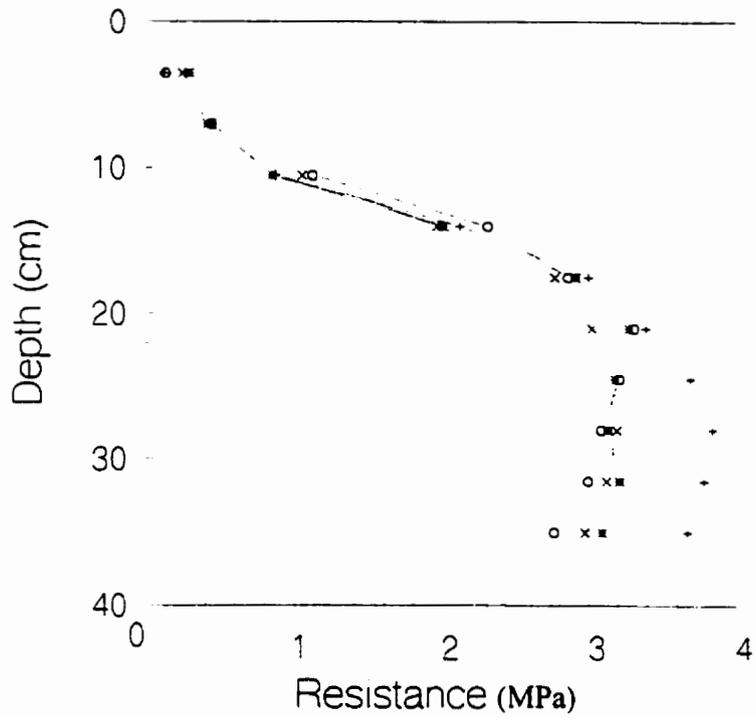
Figure 3.3. Mean soil penetration resistance (PR) profiles for each leonardite treatment applied at the unfertilized Osborne clay study site 1998.

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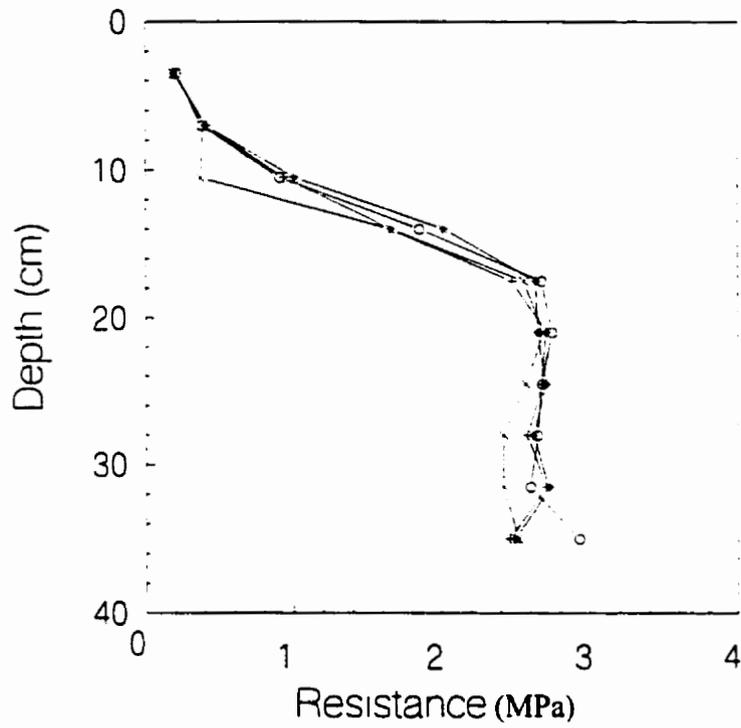
Figure 3.4. Mean soil penetration resistance (PR) profiles for each leonardite treatment applied at the fertilized Waitville clay loam study site 1998.
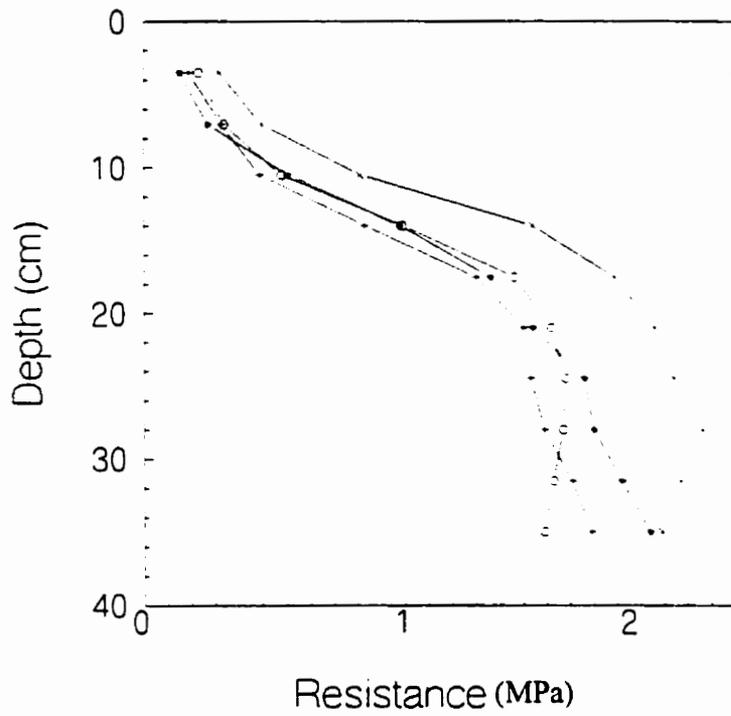
Figure 3.5. Mean soil penetration resistance (PR) profiles for each leonardite treatment applied at the unfertilized Waitville clay loam study site, 1998.

3.3.2. Leonardite Impact on Moisture and Density

Tables 3.1. and 3.2. show gravimetric moisture, BD and PD values for Osborne C and Waitville CL soils, respectively. There were no amendment effects on moisture, BD or PD.

The gravimetric moisture content of both soils was not significantly changed with amendment addition. However, the results herein only indicate the water content of the soil at a single point in time. In addition, the water content at the time of sampling was quite low. Studies have suggested that organic amendments have more influence on water retention when the water content is near the upper limit of plant available water than when it is below the lower limit of plant available water. It was hypothesized that although there was no treatment effect at this moisture level, the amendment may affect water holding capacity at another moisture level. Based on this hypothesis, further study of soil water holding capacity was conducted using ceramic tension plates (included in 4.3.1).

An improvement in soil structure would increase soil porosity and thereby reduce its BD. The apparent low values for soil BD may be due to a number of factors. The cores were extracted only to a maximum depth of 10 cm. Therefore, the soil was less compact than soil which would have been taken at a considerable depth. Another factor was that the soil contained montmorillonite, a swelling clay. The soil volume collected in the field that filled the cores was much higher than the volume of soil following drying. As the bulk density was calculated based on the volume of the core rather then the volume of the soil following drying, it is not surprising that such small bulk density volumes were

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calculated. Therefore the BD was highly dependent on the moisture condition at the time

of sampling. However, all treatments appeared to have similar values to the controls.

This shows that the bulk density was not changed with the amendment addition.

Although OM has a lower density than mineral material, the rates of addition of HA

were not high enough to substantially change the overall PD of the soil. The porosity of

the soil does not influence PD. Therefore, the addition of organic matter to the soil at the

given rates of application should not affect PD. No amendment effects on PD were

expected in the current experiment. Further, it was not expected that the PD would have

any variation throughout the top 10 cm of the soil. Therefore, it was considered sufficient

to test only the top 5 cm of the soil and the 5-10 cm depth was excluded from the

analysis.

Table 3.1. Gravimetric moisture (w),[*] bulk density (BD)[¥] and particle density (PD)[¥] taken for each treatment at different depths in the Osborne clay study site 1998.

	w(%)			BD (g/cm^3)	PD (g/cm^3)
		Depth (mm)			
Treatment	0-5 cm	5-10 cm	10-20 cm	0-5 cm	0-5 cm
C – F	25.8 ± 9.9	25.0 ± 2.6	24.5 ± 9.4	0.73 ± 0.01	2.61 ± 0.05
P – F	26.1 ± 4.5	26.2 ± 2.3	24.6 ± 2.3	0.75 ± 0.07	2.60 ± 0.01
M – F	25.7 ± 2.8	25.4 ± 1.8	25.1 ± 2.0	0.76 ± 0.08	2.59 ± 0.04
H – F	25.6 ± 4.9	26.3 ± 3.3	25.7 ± 3.0	0.71 ± 0.08	2.62 ± 0.04
C - F	24.3 ± 2.6	26.6 ± 3.3	26.8 ± 3.1	0.79 ± 0.10	2.63 ± 0.05
P - F	24.7 ± 3.5	25.8 ± 3.3	26.7 ± 4.7	0.77 ± 0.08	2.67 ± 0
M - F	23.0 ± 2.3	25.7 ± 1.6	25.3 ± 2.1	0.81 ± 0.07	2.63 ± 0.02
H - F	22.8 ± 3.3	25.6 ± 1.9	25.1 ± 2.6	0.74 ± 0.07	2.62 ± 0.01

[*]Average = standard deviation of 4 samples per treatment within each replicate.
[¥]Average = standard deviation of 2 samples per treatment.

Table 3.2. Gravimetric moisture (w)*, bulk density (BD)^y and particle density (PD)^y taken for each treatment at different depths in the Waitville clay loam study site 1998.

	w (%)			BD (g/cm^3)	PD (g/cm^3)
	Depth (mm)				
Treatment	0.5 cm	5-10 cm	10-20 cm	0-5 cm	0-5 cm
C + F	22.8 ± 3.5	19.0 ± 2.6	20.4 ± 4.0	0.91 ± 0.11	2.51 ± 0.02
P - F	23.1 ± 1.7	17.8 ± 1.5	19.4 ± 2.9	0.92 ± 0.17	2.49 ± 0.01
M + F	23.4 ± 2.6	18.9 ± 2.6	20.0 ± 4.3	0.91 ± 0.10	2.53 ± 0.04
H + F	23.7 ± 2.3	19.3 ± 2.0	20.5 ± 4.8	0.91 ± 0.14	2.50 ± 0.02
C - F	24.8 ± 1.1	22.2 ± 1.4	21.4 ± 2.7	0.91 ± 0.11	2.48 ± 0.03
P - F	23.9 ± 2.4	21.7 ± 2.1	20.8 ± 2.4	0.83 ± 0.15	2.48 ± 0.04
M - F	23.4 ± 1.5	21.3 ± 2.3	20.7 ± 3.0	0.89 ± 0.15	2.55 ± 0.02
H - F	24.0 ± 2.8	21.8 ± 2.2	21.6 ± 4.2	0.52 ± 0.15	2.53 ± 0.04

*Average ± standard deviation of 4 samples per treatment within each replicate.
^{y}Average ± standard deviation of 4 samples per treatment within each replicate.

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3.3.3. Leonardite Effects on Modulus of Rupture

Crop productivity is negatively influenced by soil crust strength which can be measured with the modulus of rupture method. This relationship depends on the thickness of the crust, soil moisture, crop species and depth of seed placement (Hillel1982).

Crusting occurs more commonly in soils with poor aggregate stability to water. Under certain conditions, such as heavy precipitation events, surface crusts could develop. As a result of water pooling on the soil surface, individual soil particles within the aggregate become dispersed. The dispersed layer clogs macropores at the surface of the soil and thereby retards gas exchange and water infiltration. Upon drying a crust of structureless soil accrues on its surface. This crust may impede emerging seedlings and tears roots as it cracks.

Materials which increase aggregate stability to water should act to lower soil dispersion and thereby lower surface crusting. This achievement has been demonstrated for a number of organic amendments (Pagliai and Antisari 1993; Hafez 1974).

Results from baseline analysis (Table 3.3) reveal that both soils have relatively high B' values, which could, under certain conditions inhibit plant growth. Therefore any modification that would lower crust strength would be beneficial. However, due to the high standard deviation, any small amendment effects may have been masked by variability between samples. Higher variability in Osborne C samples was due to shrink/swell properties which caused cracking.

Due to mechanical error, 1998 measurements resulted in high variability. The values,

therefore, are not included below. Residual treatment effects were measured using a

different device in 1999 on Waitville clay loam (Table 3.4.). The values show

that at both depths no leonardite treatment effects were found. Some of the aggregates

were destroyed in the process of creating the soil briquettes. Therefore, it was desirable

to study soil in its aggregated state. This was one reason that experiments in Chapter 4

were conducted.

Table 3.3. Baseline Modulus of Rupture[*] (millibars) values for two study sites taken at two depths in 1998.

Soil Type	0-5 cm	5-10 cm
Osborne C	605 ± 347	712 ± 388
Waitville CL	458 ± 241	409 ± 174

[*]Average ± standard deviation of 5 samples per depth within each of 4 replicates per site

Table 3.4. Modulus of Rupture[‡] (millibars) values for the Waitville clay loam study site, 1999.

Treatment	Soil Depth	
	0-5 cm	5-10 cm
C+F	332 ± 116	438 ± 84
P+F	245 ± 69	252 ± 107
H+F	371 ± 99	554 ± 186
M+F	387 ± 238	300 ± 250
C-F	463 ± 184	484 ± 226
P-F	352 ± 174	540 ± 292
H-F	337 ± 105	352 ± 78
M-F	293 ± 141	518 ± 262

[‡]Mean ± standard deviation of 8 samples per treatment within each replicate

3.4 Conclusions

The objectives of the above experiment were to determine if leonardite could reduce soil strength and density. All forms of leonardite appeared to be ineffective in reducing soil strength.

PR and soil moisture measurements exhibited no significant treatment effect. As a result of the high variability in the above measurements, the PR study was not continued in 1999. The results of the PR experiment were inconclusive due to the experimental error involved. However, other methods of analysis conducted herein, provide evidence to conclude that there was no treatment effect on soil strength.

Bulk and particle densities (also only measured in 1998) were not altered by leonardite amendment. As bulk density has a great effect on soil strength the outcome of

this experiment leads us to conclude that there was no significant effect of leonardite on soil strength.

Surface crusting, as tested with the modulus of rupture is another measure of soil strength. The results of which were not changed with amendment application.

Further study was initiated to assess the effect of leonardite on soil aggregation, pore size distribution and total organic carbon (Chapter 4).

4. INFLUENCE OF FIELD APPLIED LEONARDITE ON PORE SIZE DISTRIBUTION, AGGREGATE STABILITY AND TOTAL ORGANIC CARBON

Abstract

A field experiment was conducted to evaluate the effect of leonardite on pore size distribution. aggregate stability and total organic carbon. Tension plates (for pore size distribution) as well as wet and dry sieving (to evaluate aggregate stability) were used to assess effects of a single, springtime application of leonardite.

No significant treatment effect for pore size distribution in 1998 or residual treatment effect in 1999 was evident. Wet and dry sieving tests also showed that the leonardite did not alter aggregate stability. There was no treatment effect on total organic carbon.

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4.1. Introduction

Physical processes including water and air movement in the soil and important for the resistance of the soil to erosion by wind and water are highly influenced by soil structure. In addition, researchers have shown that increased mean diameter of aggregates has a positive relationship (r = +0.52) with crop (tomato) yield (Doyle and Hamlyn 1960). Therefore, by some means, a change in soil physical properties could result in a change in crop productivity.

In order to understand the formation and stabilization of soil structure, it is important to distinguish between the mechanisms involved (either individually or in concert) in the initial stages of aggregation (Fuller 1995). Both of these mechanisms involving sedimentation of clays have been described by Theng (1979).

In the process of coagulation, electrolyte addition induces particle aggregation through reducing the thickness of the diffuse double layer.

In flocculation, a polymer chain links and bridges several particles. In this mechanism, the chain spans the inter-particle distance. Therefore, the longer the polymer, the greater the probability that flocculation would occur. Flocculation has been shown to be facilitated by the presence of polymeric bio-molecules (Tisdale and Oades 1982).

The extent of flocculation was thought to depend on the dominant cations on the exchange (Theng 1979; Fuller 1995). It was suggested that adsorption of HAs was due to displacement of water around a cation (Hayes and Himes 1986). Water desorption lead to

increased entropy (ΔS), which translated to an increased adsorption energy (-ΔG).

Therefore, the ionization potential (charge:size ratio) of the cation affected the polymer

adsorption (Theng 1979).

It was hypothesized that, based on the dominant cations (Appendix A), the addition of

organic matter would facilitate in flocculation of the clay particles via cation bridging, as

had been previously demonstrated (Kretzschmer *et al.* 1993; Tarchitzky *et al.* 1993). The

humic acids found in leonardite were mature, of high molecular weight, and therefore

composed of large chains which would have been expected to span the interparticle

distance and cause flocculation.

In general, an increase in the molecular weight of the polymer would lead to an

increase in the surface segment bonds on non-porous adsorbants (Theng 1979).

However, Theng (1979) also noted that soils contain pores, and entry of the polymer into

the pore spaces may be limited by polymer size. This would therefore limit sorption.

The flexibility of the polymer chain was also thought to be a contributory factor affected

sorption of organic polymers.

A number of studies have shown organic amendments to influence aggregate stability

(Pagliai and Antisari 1993; Hafez 1974). Soil aggregation refers to the aggregation of the

primary particles of soil into larger structural units (Hasset and Banwart 1992). The action

of wetting can cause aggregates to slake. This may happen when there is unequal strain

due to swelling and/or when external forces such as the pressure of entrapped air in pores

exceed the force of cohesion between soil particles.

Soil OM influences soil aggregation at two levels: the macro-scale and micro-scale

(Tisdall and Oades 1982). At the micro-scale, simple and complex sugars and reactive humic substances act as bridges between clay particles in the soil, to create fine micro-aggregates. At the macro-scale, fungal mycelia and roots bind small aggregates to form larger aggregates.

Aggregate stability appears to be dependent on the amendment material and soil type. Organic waste application has been shown to improve aggregate stability in sandy loam and sandy clay Italian soils, while decreasing stability in heavy clay soils (Mbagwu *et al.* 1991).

Distribution of aggregate size is closely related to that of the pore sizes within the aggregates (Oades 1984). A well aggregated soil has a different pore size distribution than a compact soil (Figure 4.1.)(Hillel 1982). Pore radii > 50 μm are associated with aggregates with radii > 500 μm. These pores are responsible for soil aeration, rapid water movement and amplified root growth. Pore radii of 12.5 to 50 μm are associated with aggregates with radii between 125 and 500 μm. These are the pores involved in conduction of capillary water and gas exchange. Pores below 12.5 μm are important for water retention. A pore radius ≤0.1 μm is associated with water held below the lower limit of available water.

Soil pore size distribution is an indicator of the pores that correspond to drainage, aeration and water holding capacity (Kay 1998). Total porosity required for growth depends on the crop and soil type. This is important as air pore space has been demonstrated to be a limiting factor for plant growth (Hanks and Thorp 1956). An ideal soil for growth would contain equal parts pore space and solid space. The pore space

would be 50 % macropores (containing the soil atmosphere and responsible for drainage) and 50 % micropores (responsible for water retention)(Hasset and Banwart 1992). At optimum moisture, a well granulated medium textured soil with 50% of its volume consisting of pore space shared equally by air and water would have a pore size distribution similar to that shown in Figure 4.2. (Brady 1990).

The addition of organic matter (OM) amendment has been shown to influence distribution of pores. Amendments in the form of sewage sludges and livestock effluents caused increased micro- and macro-porosity of silty clay and sandy loam soils (Pagliai and Antisari 1993). These increases were attributed to increases in micro- and macro-aggregation and were accompanied by decreased surface crusting.

Pore size distribution has an influence on plant available water (AW). Plant AW could be altered with addition of OM. It decreased with increasing rates of manure in a Dark Brown Chernozem (Sommerfeldt and Chang 1986). and increased for both sandy and clay soils (Hafez 1974). Mean volumetric soil water content increased with increased organic matter on Hanslope series clay loam in Cambridge, UK at 25 and 50 cm tensions (Davies 1985).

The lower limit of soil water available for plant growth. termed permanent wilting percentage. is the volumetric water content at which plants cannot extract water well enough to maintain turgor. Most researchers agree that it can be evaluated by measuring the water retained at a water potential of -1.5 Mpa (Klute 1996). This represents the soil pores ≤ 0.1 μm. While organic matter seems to have a fairly high correlation with the upper limit of available water (termed field capacity), the appearance of organic matter

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does not have a lot of bearing on permanent wilting point in soils found in Manitoba

(Shaykewich and Zwarich 1968). This has been demonstrated in Italian soils as well: pig

and cattle slurry sludge application resulted in slight increases in water retention at

pressures of 0.03 MPa, but no effect was apparent at pressures of 1.5 MPa (Mbagwu and

Piccolo 1990). Elsewhere, however, the 1.5 MPa moisture has been altered through the

use of some soil conditioners (Doyle and Hamlyn 1960). Others have shown that

microporosity is highly influenced by the organic carbon content of the soil (Kay 1997).

Soil aggregation, and therefore, soil porosity is highly influenced by the amount of

organic carbon in the soil (Kay 1998 and references cited therein). In general, an increase

in organic carbon leads to an increase in the degree of aggregation and aggregate stability.

This also may lead to a change in the pore size distribution of the soil.

It was hypothesized that with the addition of leonardite, there would be an increase in

the total organic carbon content of the soil. This possible increase in organic carbon was

hypothesized to contribute to the soil aggregate stability and pore size distribution. It was

hypothesized that aggregate stability and total soil porosity would increase as a result of

leonardite amendment application.

The objectives of the following experiment were to evaluate if leonardite amendments

could: (1) increase the porosity and influence pore size distribution of the soil: (2)

increase aggregate stability to water: and (3) increase the total organic carbon content of
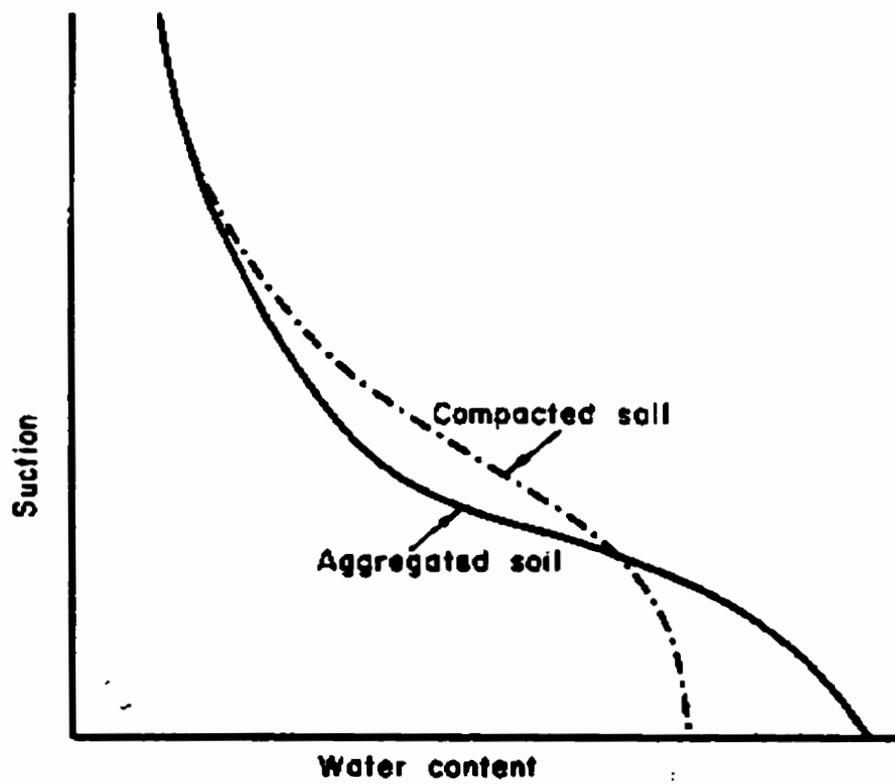
the soil.

Figure 4.1. The effect of soil structure on soil-water retention (Hillel 1982).
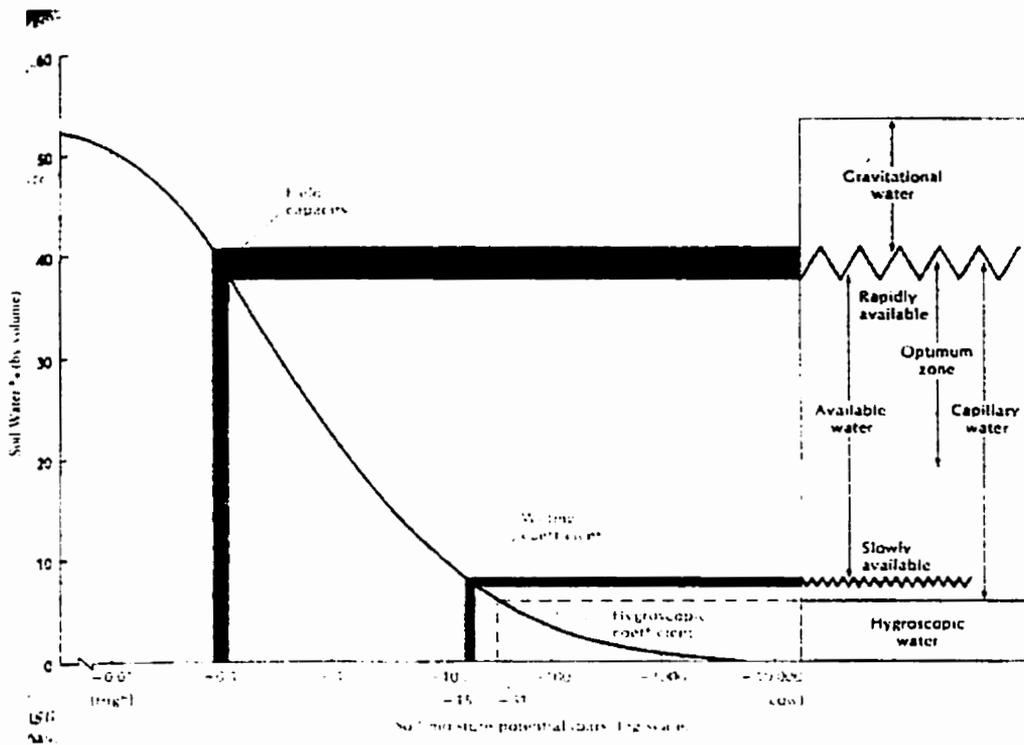
Figure 4.2. Potential moisture curve of a loam soil with an ideal pore size distribution for plant growth. Different terms to describe water in the soil are shown on the right hand side of the diagram. The wavy lines suggest that the measurements are no completely quantitative, but assist in the qualitative description of the moisture in soils (Brady 1990).

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4.2. Materials And Method

Three experiments evaluating the effect of field applied leonardite on soil structure were selected for this study: tension plate experiment, 15 atmosphere test and aggregate stability. Selected soil properties for these sites are shown in Table A.1.

4.2.1. Field Plot Design

The field plots had dimensions of 25.5 m by 46 m. They were arranged in a randomized complete block design with split plots which were fertilized (+F) and not fertilized (-F). There were 4 replicates, each containing three treatments and one control (C) (Appendix A). Each subplot was 3.2 m by 5 m.

On May 5, and May 22, 1998 leonardite amendments were added to Waitville clay loam (CL) and Osborne clay (C) soils, respectively, immediately prior to seeding wheat (*Triticum aestivum* L.). Leonardite was added to the soil surface and rototilled to approximately 7 cm. Treatments included an insoluble pulverized leonardite powder (P), a ground leonardite material <2 mm diameter (M), and a liquid leonardite suspension that was thought to be contain high amounts of humic acid[3] (H). The P amendment was mixed with soil a small quantity of soil from the subplots prior addition as it during application it was highly susceptible to wind erosion due to its fine particle size. The M was added directly to the soil surface and the H suspension was hand mixed immediately prior to its application. Selected properties of the leonardite amendments are contained in

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[3] The majority of the extraction was humic acids, but fulvic acids were also found to be present.

Appendix B.

Application rates of P, M and H were 150 kg/ha, 1000 kg/ha and 100 L/ha

respectively. The quantities of P and M amendments applied were based on previous

studies conducted in Lethbridge. In these studies a number of application rates (including

0, 100, 500 and 2000 kg/ha) of P and M were evaluated. According to the findings, rates

of 150 kg/ha P, and 1000 kg/ha M were predicted to be the most beneficial to crop

productivity[4]. The H amendment was a new product that had not previously been

studied. The application rate of 100 L/ha used in the study was based on the organic

carbon content of the H amendment.

4.2.2. Field Operations

Chapter 3 contains a detailed description of the field operations. In year one, AC

Barrie wheat was seeded May 5, and May 22, 1998 and harvested August 25 and August

27, 1998 in the Waitville CL and Osborne C soils, respectively. In year two, wheat was

seeded June 2 and harvested October 6, 1999 in the Waitville CL soil. Flooding

prevented seeding on Osborne C, and second year analysis of the soil was not acceptable.

Spring fertilizer application rates were as follows. In 1998, the Osborne C soil

received 20 kg/ha nitrogen (85 kg/ha had been applied in the previous fall) and 20 kg/ha

phosphorous. The Waitville CL soil received 125 kg/ha nitrogen, 20 kg/ha phosphorous,

and 24 kg/ha sulphur. In 1999, 150 kg/ha nitrogen, 40 kg/ha phosphorous and 24 kg of

sulfur was applied to Waitville CL. Nitrogen was broadcast in the form of urea,

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[4]Research conducted in Lethbridge by L. Cramer, W. Akinremi and H. Janzen, Agriculture Canada.

phosphorous was applied with the seed in the form of monoammonium phosphate. and

sulphur was applied as ammonium sulphate.

4.2.3. Pore Size Distribution

Two different experiments were involved in evaluating the pore size distribution of

the two soils. The tension plate experiment was used to determine pore sizes above the

lower limit of plant available water. Due to the porosity of the ceramic tension plates. the

smaller pore sizes had to be evaluated with a pressure membrane apparatus.

The capillary model is generally accepted for representation of soil pore space (Brady

1990) . In the following method of measuring pore size distribution. it is assumed that

pores in the soil are similar to a number of capillary tubes each containing both an open

and closed end. Following saturation. the force of tension holds water within the pores

until it is exceeded by an opposing force (in this case suction).

Liquid will rise in a capillary tube when it is wetted (Figure 4.3). Forces of adhesion

will cause the liquid to be attracted to the side of the capillary tube. The height (h) of the

rise is directly proportional to the surface tension. which depends on the cohesion

between the liquid molecules (Brady 1990). The height will be inversely proportional to

the radius (r) of the tube. The water will stop rising when the upward force due to surface

tension is equal to the downward force due to gravity. When the forces are equal (Klute

1986):

$$2\pi rT\cos\alpha = \pi r^2 hdg \qquad\qquad [1]$$

where: r = radius of the tube
   α = contact angle between the tube and the liquid
   h = height of the rise
   d = density of the liquid
   g = acceleration due to gravity (980 dynes/cm^2)
   T = surface tension of the liquid (72.5 dynes/cm for water)

The left hand side of Eq. [1] describes the surface tension acting around the circumference. That is, the length over which surface tension is acting ($2\pi r$) multiplied by the vertical component of that force ($T\cos.\alpha$). The right hand side of the equation describes the force of gravity. That is the acceleration due to gravity multiplied by the mass of the liquid ($d\pi r^2 h$).

Rearranging Eq. [1] and assuming that the contact angle is zero, it is found that:

$$h\ (cm) = 2T/rdg \qquad\qquad [2]$$

Through the use of the Eq. [2], it is possible to determine the suction, h, required to drain pores having an effective radius greater than the corresponding r value. In a saturated soil sample, the volume of pores is equal to the volume of water extracted.
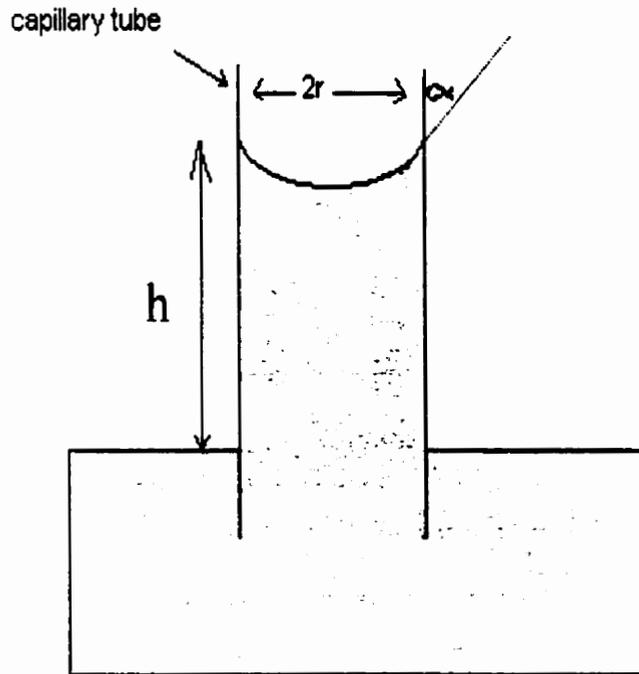
Figure 4.3. Illustration of the capillary tube concept. Liquid will stop rising in a tube when the force of gravity is equal to the surface tension acting around the circumference. This is dependent on the radius of the tube (r), the contact angle (α), as well as the surface tension (T) and the density of the liquid.(d).

After equilibrium was reached for a given suction, mass was measured. Following gravimetric determination for all suctions, samples were placed in pre-weighed beakers in a 110°C oven for 48 hours. Beakers were removed, weighed, and gravimetric soil moisture at the given suctions were calculated (Eq. [3]).

$$w = m_w/m_s \qquad\qquad [3]$$

where:
  m_w = mass of moist soil
  m_s = mass of oven dry soil
  w = gravimetric water content

Using bulk density, volumetric soil moisture was calculated (Eq. [4]).

$$\theta = (w)(BD) \qquad\qquad [4]$$

where:

  θ = volumetric soil moisture content
  w = gravimetric soil moisture content
  BD = soil bulk density

4.2.3.1. Tension Plate Experiment. Following fall harvest. soil samples were extracted

using copper cylinders (22 cm^3 volume. height 1.8 cm. diameter 3.9 cm) at soil depths of

0-5 cm and 5-10 cm. to be used for pore size distribution determination according to

methods of Klute (1986). Cores were hermetically sealed at 4°C prior to use. Water

retention was determined at tensions of 0, 10. 25. 50 and 100 cm (or pressures of

approximately 0. 1.0, 2.5, 5.0 and 10.0 kpa) using tension plates. When the mass was at

equilibrium. the pores corresponding to a given suction had drained. Pore radius was

calculated using the capillary rise equation, and pore size distribution was determined.

The 0 cm tension revealed the total porosity of the soil and the 10 cm tension

represented the diameter of wheat roots (radius of 148 μm). Tensions of 25 (radius of 59

μm) and 50 cm (radius of 29.6 μm) were chosen to represent poorly drained. and mole

drained grassland water tables. respectively (Davies 1985). A tension of 100 cm

corresponds to a radius of 14.8 μm.

Oversize pore volume (OPV) fraction was plotted against log pore radius to obtain the pore size distribution of the soils. The OPV represented the fraction of the total pore volume that was greater than the particular radius it was plotted against.

4.2.3.2. 15 Atmosphere Test. Lower limit of soil plant available water was estimated using the

15 atmosphere test with a pressure membrane apparatus (100 Bar Pressure Membrane Extractor

Cat # 1700, Soil Moisture Equipment Co., Santa Barbara, California) (Lehane and Staple 1960).

Soil from each treatment was collected between 0-10 cm depth and air dried for 1 week. It was

ground to pass through a 2 mm sieve. Soil was then spooned into cloth lined plastic cores

(height 0.8 cm, diameter 4.5 cm), soaked in water for 24 hours, and then placed in a cellulose

membrane lined apparatus (Figure 4.5.). Using N_2 gas, 15 atmospheres (1.5 Mpa) of air pressure

were applied to soil in order to extract water from the soil. After equilibrium was reached, when

the number measured on the capillary tube scale remained constant, cores were removed and

weighed, oven-dried for 48 hours at 110°C. Gravimetric water content, volumetric water and

pore radii were calculated.
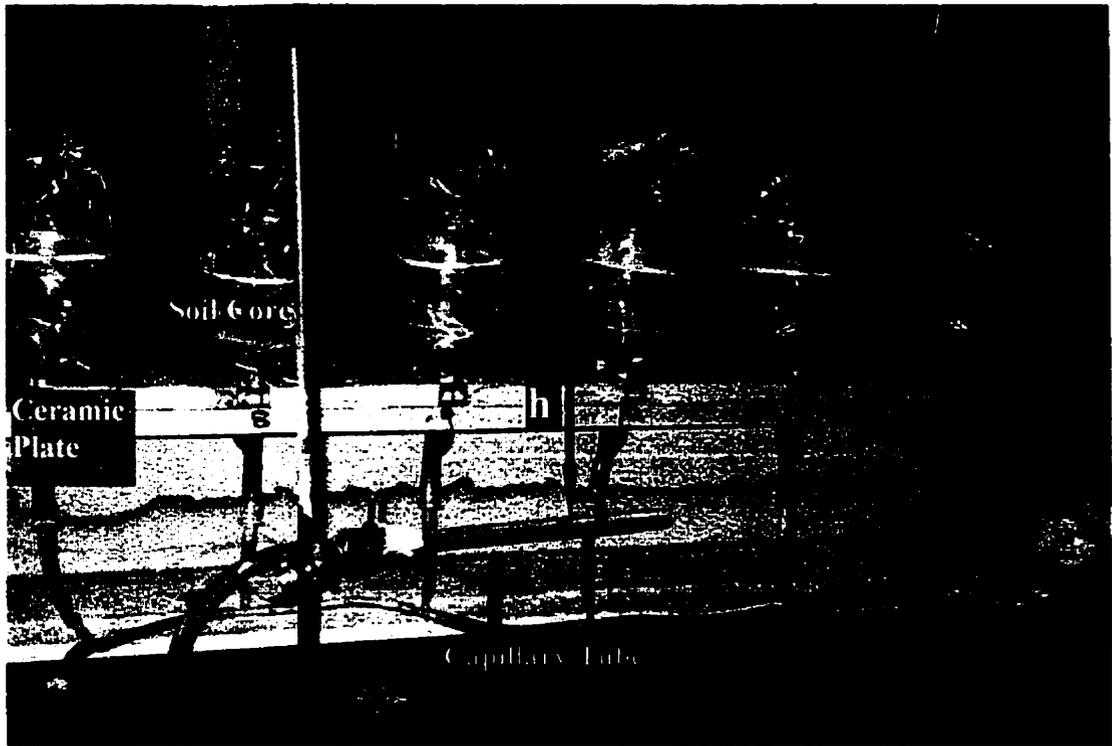
Figure 4.4. Illustration of the tension plate apparatus. The capillary tube was lowered to a level (h) where the acceleration due to gravity (g) drained water with surface tension (T) and density (d) from pores of radii (r) according to Equation [2].
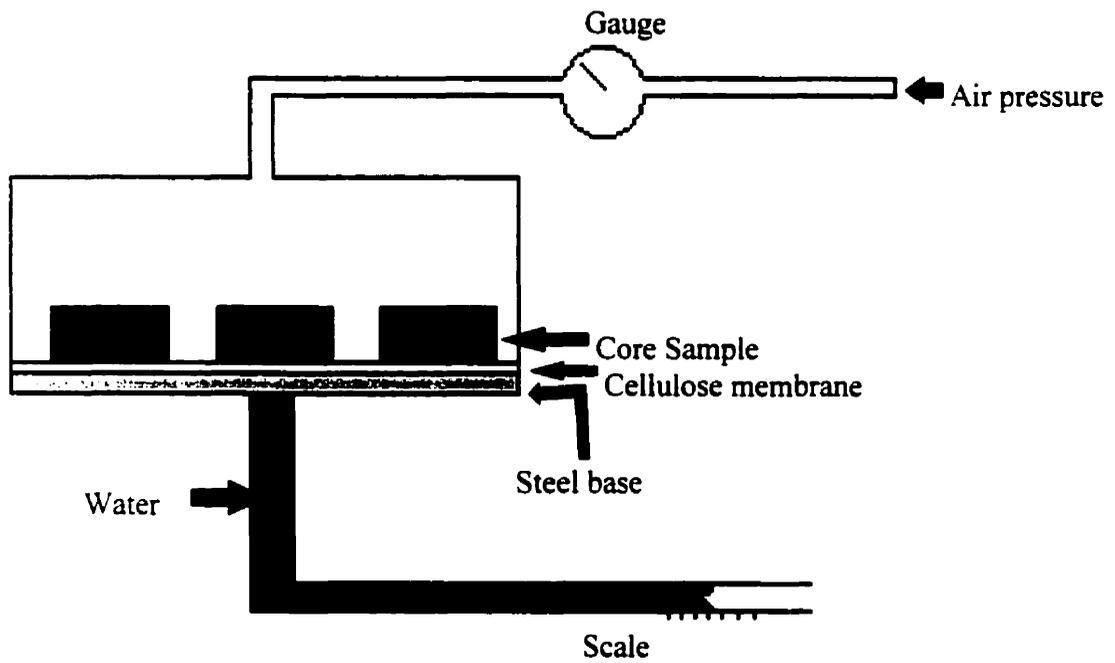
Figure 4.5. Apparatus for measuring soil water content retained when soil was subjected to 15 atmospheres of pressure.

4.2.4. Aggregate Stability Analysis

Baseline samples of Osborne C and Waitville CL were collected July 9 and 10, 1998, respectively. Four samples were collected adjacent to the experimental plots, where fertilizer had not been applied.

Following fall harvest, bulk soil samples were taken using a shovel and trowel. Samples were separated by depth (0-5 and 5-10 cm), air dried, broken into aggregates (<9.5 mm in diameter) in preparation for dry sieving (White 1993) and wet sieving (Kemper and Rosenau 1986). Two samples from each treatment were analyzed. Soil samples weighing approximately 50 g were agitated on a nest of sieves with mesh of size 4, 2, 1, 0.5 and 0.25 cm. Following dry sieving, mass of soil was measured to determine proportion of the total soil larger than the sieve size. Soil <0.25 mm was collected below the nest. After wet sieving, soil collected over each sieve was washed into previously weighed beakers and oven dried for 48 hours at 110°C. Using the initial sample weight, and moisture content of air dry soil, the mass of oven dry soil <0.25 mm was calculated (Eq. [3]).

For both wet and dry sieving, the characteristic mean diameter (CMD) of the aggregates was calculated. For most soils, the frequency distribution of the logarithm of aggregate size follows a normal distribution. Thus, a plot of percent oversize on a probability scale against aggregate size should produce a straight line (Gardner 1956).

Particles in the soil which remain aggregated had greater cohesive forces than the disruptive forces acting on them. Dry sieving was performed only for a short period of time (15 seconds). This was to obtain the overall size distribution of dry aggregates

without breaking them. The wet sieving simulated slaking, the breakdown of aggregates under rapid wetting in the field. The difference between the CMD obtained from dry and wet sieving is expressed as the change in characteristic mean diameter (CH) which is a measure of aggregate stability to water. Thus, the smaller the CH, the greater the aggregate stability towards slaking.

4.2.5. Total Organic Carbon

Soil from bulk samples described in 4.2.5. was ground to <250 μm. Using the Walkley-Black method (Allison 1983) total organic carbon (OC) was determined. Each soil sample (0.5 grams) was added to a 500 mL flask along with one blank. Following this, 10 mL of 1.0 N $K_2Cr_2O_7$ and 20 mL of concentrated H_2SO_4 were added. The OC was oxidized by chromic acid. After 30 minutes, 10 mL of H_3PO_4 and 200 mL of distilled H_2O were added. An indicator (0-phenanthroline-ferrons complex) was added and the mixture was titrated with 0.5 N $FeSO_4$, a reducing agent, until the mixture changed color. This back titration was used to determine the amount of $K_2Cr_2O_7$ used. The chromic acid used in excess to that of the blank was an indication of the proportion involved in the oxidation of OC.

4.2.6. Statistical Analyses

Analyses of variance (ANOVA) tests were performed using JMP IN software (SAS Institute 1997). Fertilized and unfertilized plots were examined separately. For each separate soil depth, in effect the 0-5 and 5-10 cm depth, the data was analyzed

individually - excluding the 15 atmosphere test in which only one depth (0-10 cm) was used. The LSD test was used to compare treatments found to be significantly different in ANOVA at the 95% level of significance.

4.3. Results And Discussion

4.3.1. Pore Size Distribution

4.3.1.1. Tension Plate Experiment. Analysis of the porosity of the two soils sampled in 1998 revealed that, as expected, the total porosity of the fine textured Osborne C soil was higher than that of the Waitville CL soil. There was no treatment effect on total porosity for both soils at all depths (Table 4.1.).

Figures 4.6. through 4.17. were based on the proportion of pores of the total soil volume greater than or equal to a given radii. This provided an indication of the distribution of oversize pore volume (OPV) relation to pore size.

The Osborne C pore sizes are shown in Figures 4.6. to 4.9. Roughly 20 - 25 % of the Osborne C soil volume was associated with pores >59 μm. These pores were responsible for soil aeration, rapid water flow and enhanced root growth. Approximately 5 - 7 % of the soil volume was associated with pores in the 14.8 - 59 μm range which allowed for gas exchange and conduction of water. Results in Table 4.3. show that a great deal of the pores held water below the lower limit of available water (approximately 24% of the total soil volume), while about 25 % of the total soil volume consisted of pores responsible for water retention available to roots.

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The Osborne C had the appearance of having a fairly high proportion of macropores. This was due to shrink/swell properties of the dominant clay. montmorillonite, which caused cracking in the sample and between the soil and the walls of the core in dry conditions and swelling beyond the volume of the sampling cores at low suctions. As a result. the total porosity and porosity at low suctions was very high. The pore size distribution was not optimal for plant growth. A high proportion of water held in the soil was below the lower limit of plant available water.

The graphs in Figures 4.6. through 4.9. suggested that the pore size distribution of the Osborne C was not changed with leonardite application. Even so. in the fertilized 5-10 cm depth soil there is a statistically significant treatment effect at the 10 cm tension (ie. 148 μm pore radius). It appears that the control (C) has a lower proportion of pores of this size. It was hypothesized that this is due to experimental error. This could be due to cracks in the sample or included crop residue which would have appeared to increase the macroporosity of the samples. This conclusion was drawn based on the high F ratio determined for replicated effects (Appendix C). The F ratio was 7.61 and for 3.78 for replicates and treatments. respectively. It would have been desirable to test the Osborne C again in 1998 and 1999 to substantiate this claim. Unfortunately. as the soil cores are destroyed in the experiment. that was not possible.

The magnitude of porosity increase as a result of increased levels of organic carbon has been shown to be greater in soil with a coarse texture compared to finer textured soils (Kay 1997). Therefore. it was hypothesized that the leonardite amendment may have impacted the pore size distribution of the coarser textured Waitville CL.

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Distributions of the 1998 Waitville CL soil pore sizes are shown in Figures 4.10. through 4.13. Proportions were 15 - 20 % and 10 %, respectively for pores > 59 μm and between 14.8 - 59 μm. About 20% of the total soil volume contained pores responsible for water retention and 11 % were below the lower limit of plant available water. Based on the above findings, the Waitville CL soil exhibited a good pore size distribution for plant growth.

The figures showed that, in general, there was no significant treatment effect on the Waitville CL. In the unfertilized 5-10 cm depth soil (Figure 4.12.) at the 25 cm tension (ie. 59 μm pore radius) ANOVA does suggest a treatment effect. The liquid (H) treatment appears to have lowered porosity. However, as the effect is not evident for the entire pore size distribution, or from the other graphs of Waitville CL soil pore profiles, it is speculated that there was no treatment effect, and rather the apparent effect is due to experimental error.

Table 4.1. Effect of treatment on total volumetric porosity percent[+] of soils collected from 2 study sites at 2 depths 1998.

Treatment	Waitville CL		Osborne C	
	Depth (cm)			
	0-5	5-10	0-5	5-10
C+F	63 ± 12	64 ± 15	72 ± 12	71 ± 21
P+F	63 ± 7	70 ± 4	83 ± 13	89 ± 10
M+F	64 ± 10	69 ± 14	68 ± 5	81 ± 11
H+F	59 ± 9	63 ± 12	73 ± 9	79 ± 21
C-F	66 ± 6	68 ± 10	78 ± 19	91 ± 19
P-F	63 ± 13	59 ± 10	73 ± 8	64 ± 16
M-F	67 ± 11	62 ± 4	70 ± 5	75 ± 6
H-F	65 ± 5	60 ± 6	78 ± 10	77 ± 10

[+]Mean of 1 sample per treatment within each replicate.

Figures 4.14 through 4.17. show no residual treatment effects on Waitville CL pore size distribution in 1999. Springtime precipitation conditions in Manitoba were well above average. and it was only possible to seed and sample Waitville CL soil as Osborne C was flooded. These conditions promoted soil dispersion and surface crusting as demonstrated by the decrease in pores responsible for aeration (> 59 μm) and conduction (14.8 - 59 μm) compared to samples collected in the previous year.

Roughly 12.5 - 17.5 % of the Waitville CL soil volume was associated with pores >59 μm. Approximately 7.5 % of the OPV was associated with pores in the 14.8 - 59 μm range. Based on Table 4.3. where approximately 11 % of the total soil volume was associated with pores below the lower limit of plant available water. it was calculated that 30 % of the total soil volume contained pores responsible for water retention.

Although Figure 4.14. appears to show that the P treatment had some effect on OPV. ANOVA showed there to be no significant effect. All other graphs had no treatment effect as well.

Replicate effects were high for both soils. This may have been due to variation within the field or experimental error. A number of tension plates were used in the study. Therefore. it was extremely difficult to lower tensions to precisely the same level. As well. only approximately 50 samples could be analyzed at one time. and the experiment was carried out during all seasons. Although the laboratory was climate controlled. there may have been variations in temperature. As temperature affects viscosity of water. drainage of the pores may have varied throughout the two years of study. and consequently. pore size distribution may have appeared to change.

Table 4.2. Effect of treatment on total volumetric porosity percent[a] of Waitville CL collected at 2 depths 1999.

Treatment	0-5 cm	5-10 cm
C+F	64 ± 11	64 ± 11
P+F	78 ± 12	66 ± 9
M+F	65 ± 1	64 ± 6
H+F	64 ± 11	66 ± 13
C-F	68 ± 8	68 ± 8
P-F	64 ± 9	64 ± 9
M-F	64 ± 4	64 ± 4
H-F	70 ± 8	70 ± 8
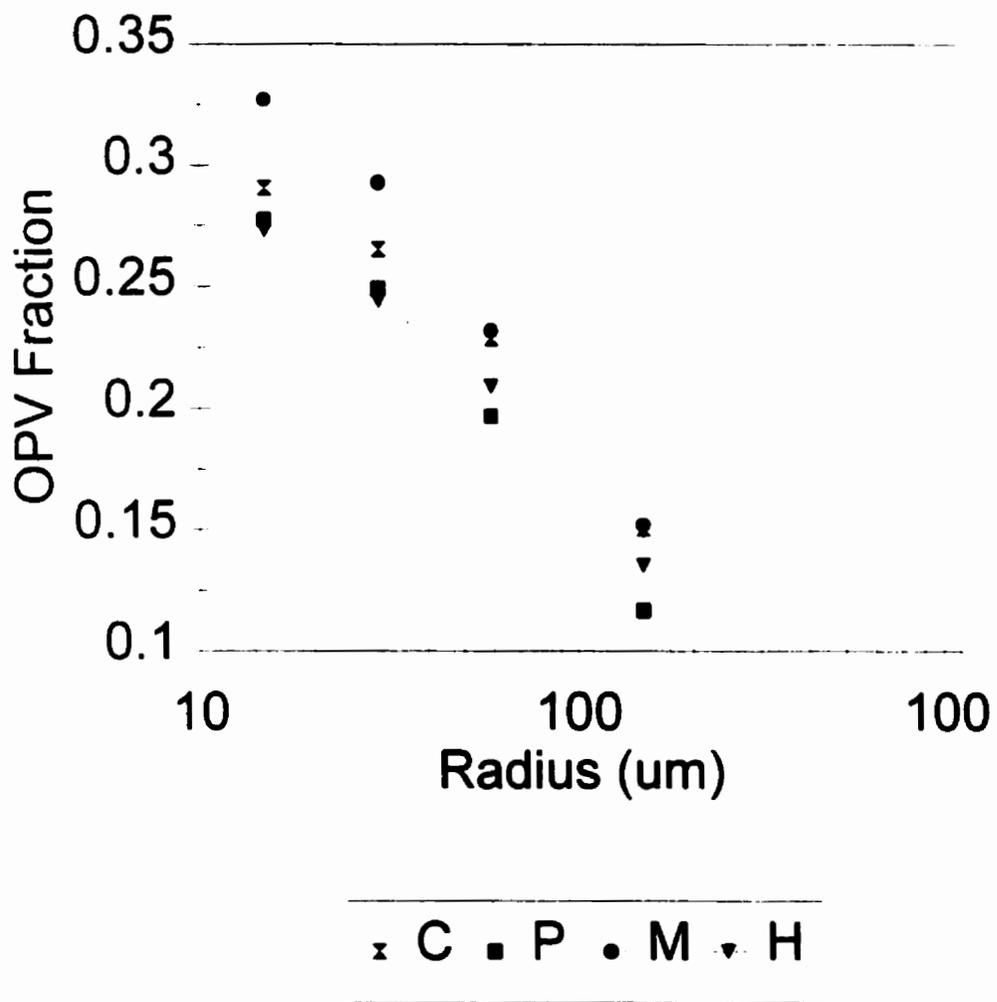
Figure 4.6. Soil pore size distribution of the 0-5 cm depth of fertilized Osborne clay soil treated with leonardite, 1998.
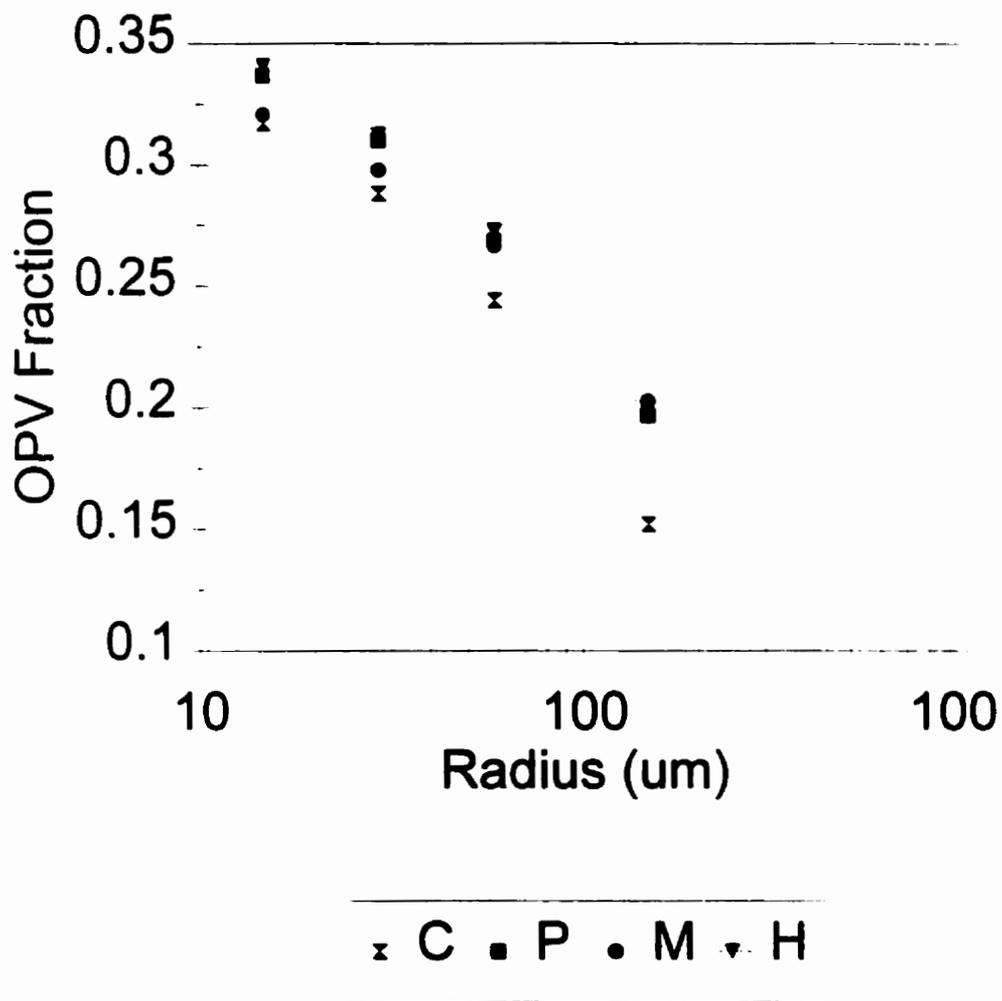
Figure 4.7. Soil pore size distribution of the 5-10 cm depth of fertilized Osborne clay soil treated with leonardite, 1998.
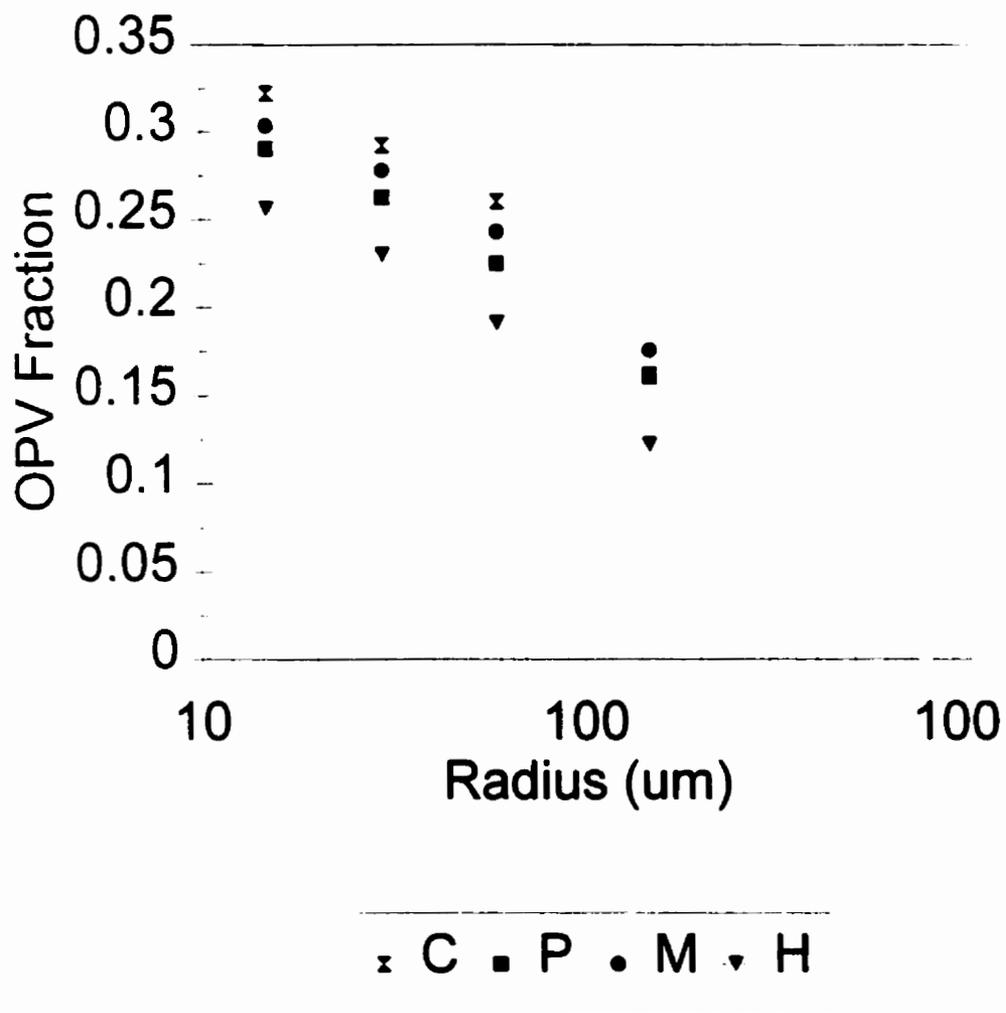
Figure 4.8. Soil pore size distribution of the 0-5 cm depth of unfertilized Osborne clay soil treated with leonardite, 1998.
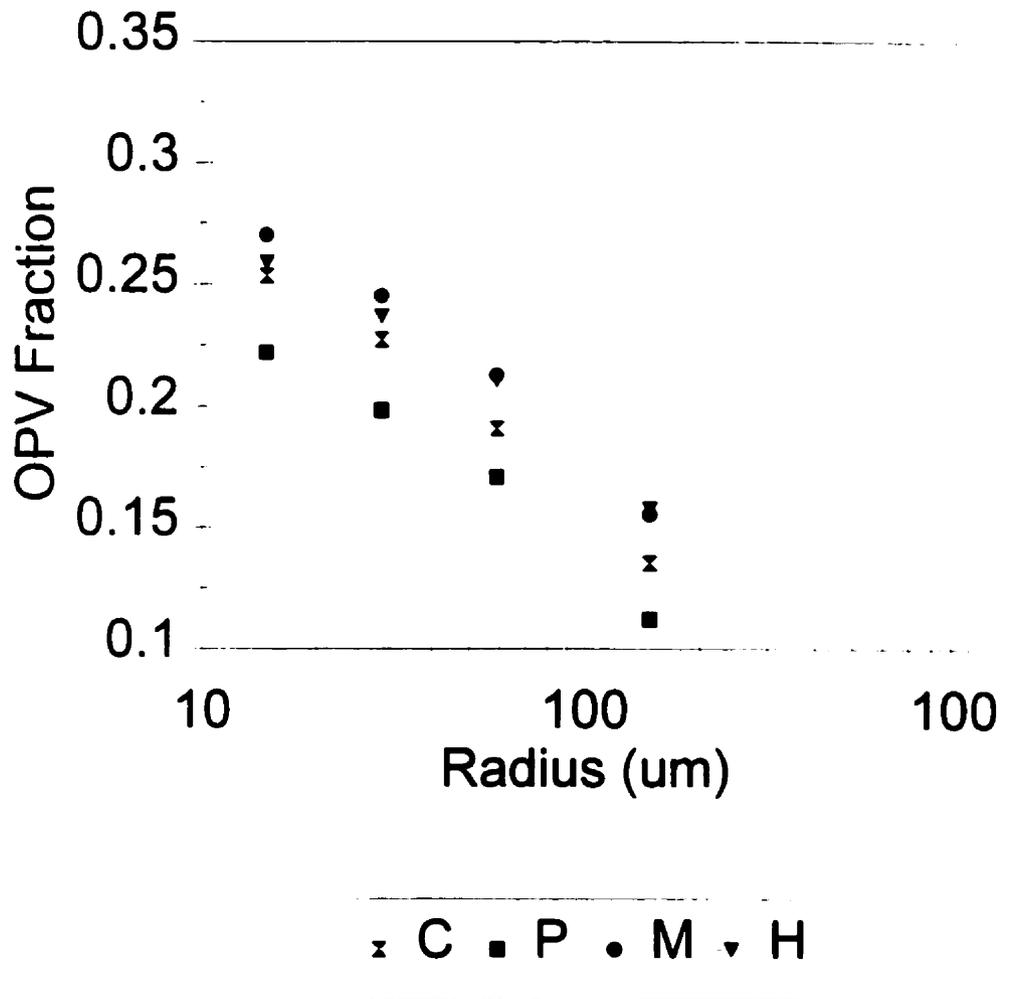
Figure 4.9. Soil pore size distribution of the 5-10 cm depth of unfertilized Osborne clay soil treated with leonardite, 1998.
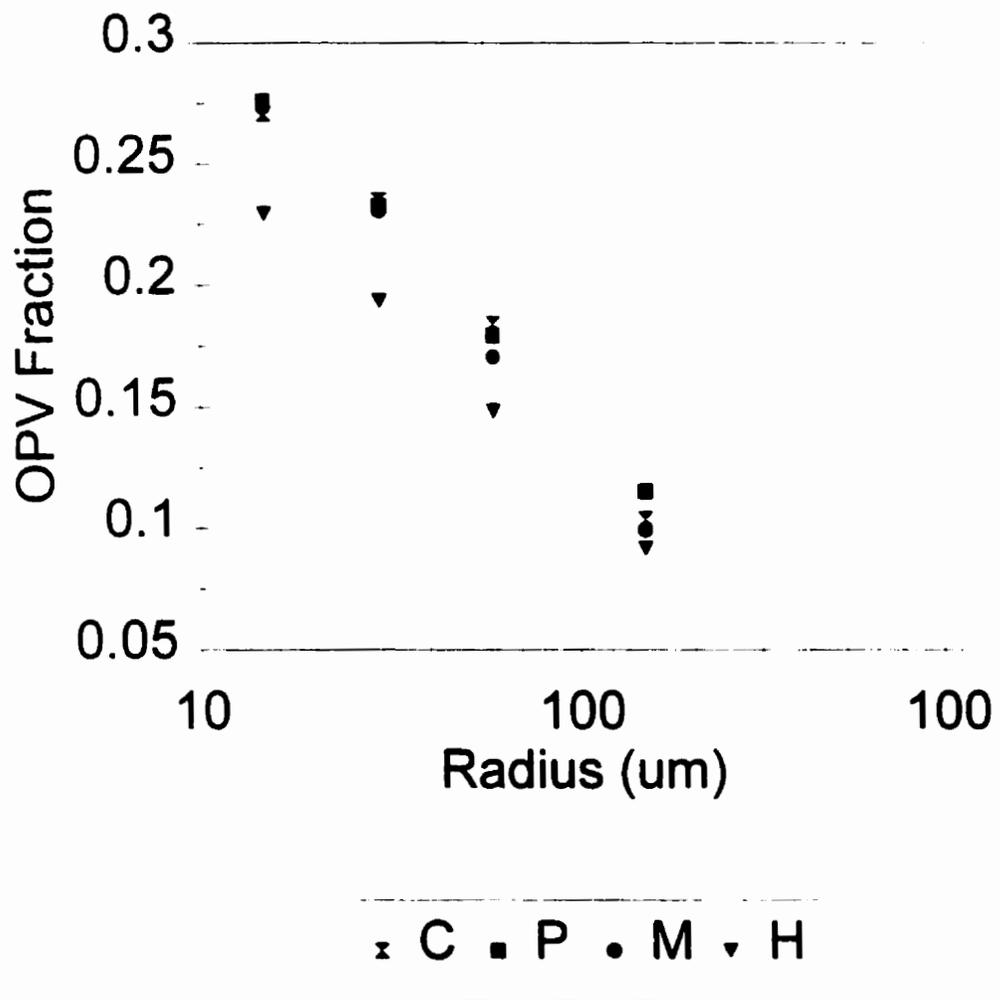
Figure 4.10. Soil pore size distribution of the 0-5 cm depth of fertilized Waitville clay loam soil treated with leonardite, 1998.
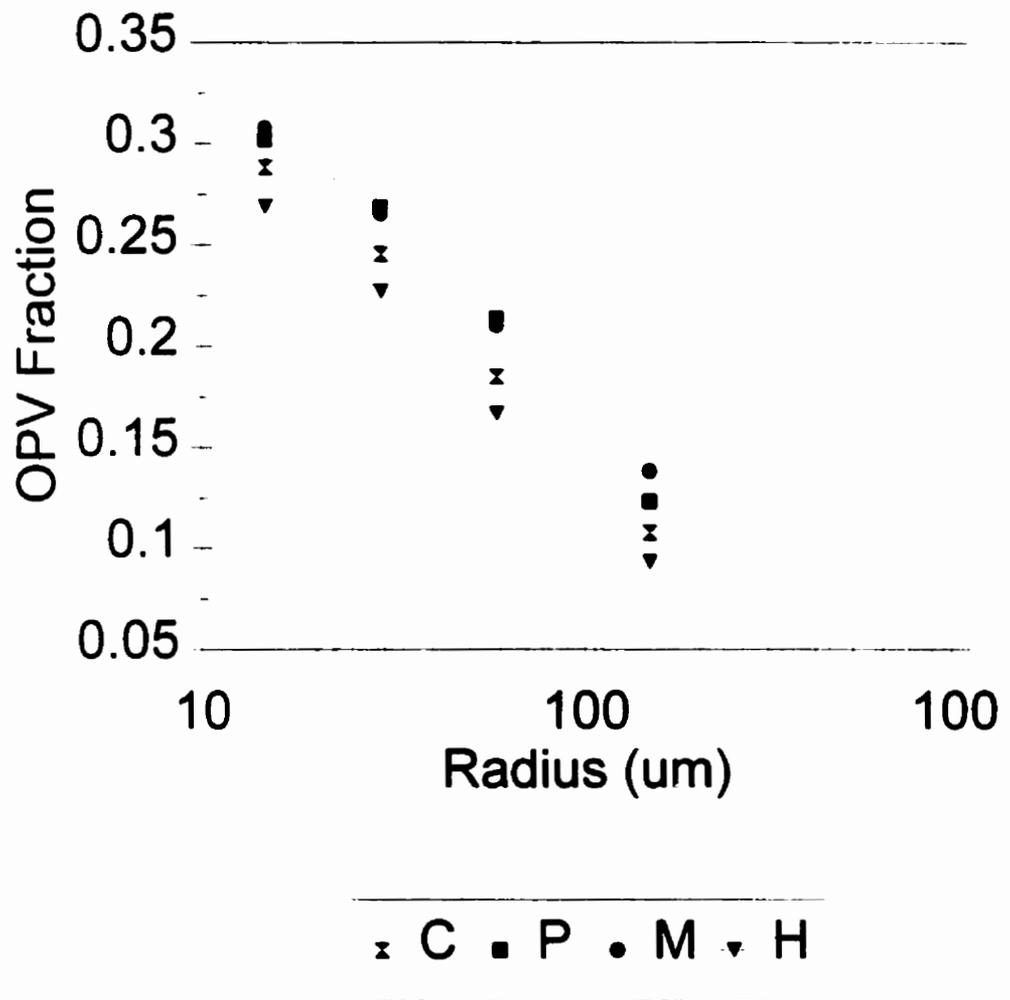
Figure 4.11. Soil pore size distribution of the 0-5 cm depth of unfertilized Waitville clay loam soil treated with leonardite, 1998.
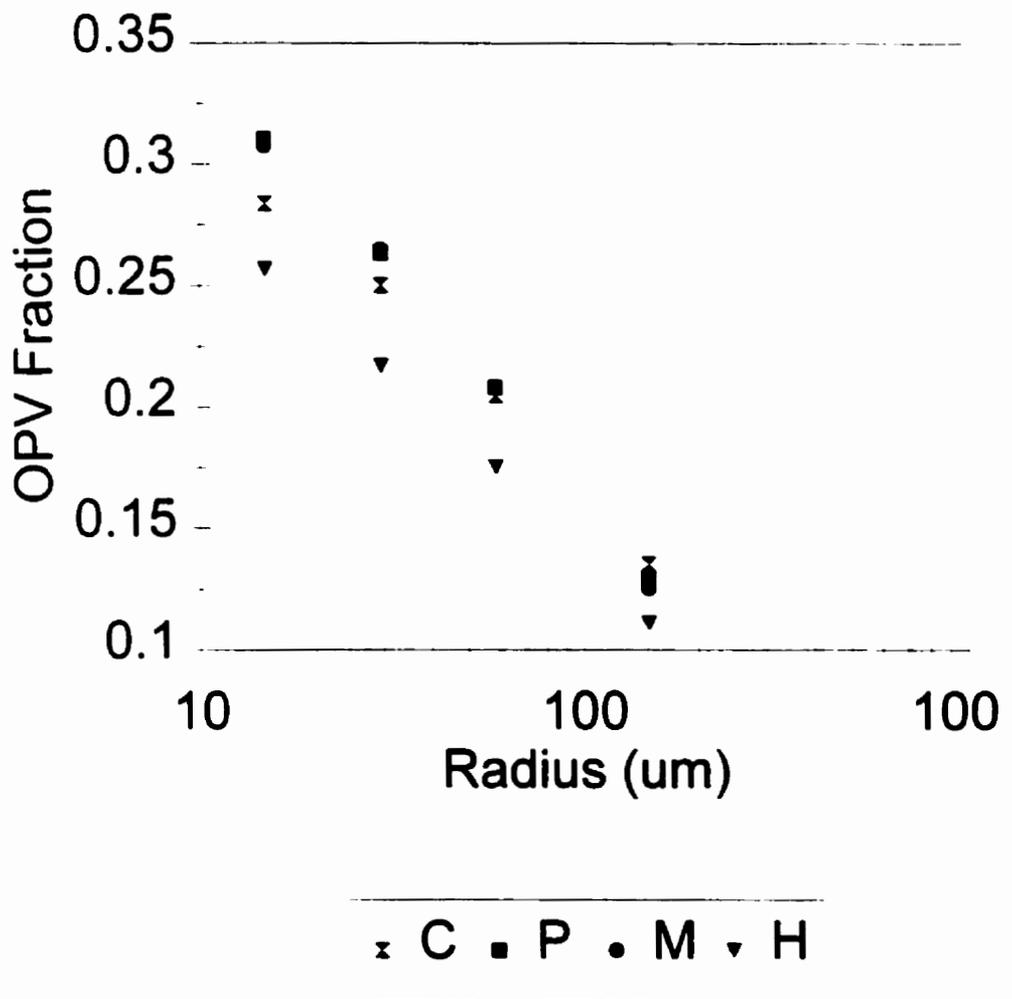
Figure 4.12. Soil pore size distribution of the 5-10 cm depth of fertilized Waitville clay loam soil treated with leonardite, 1998.
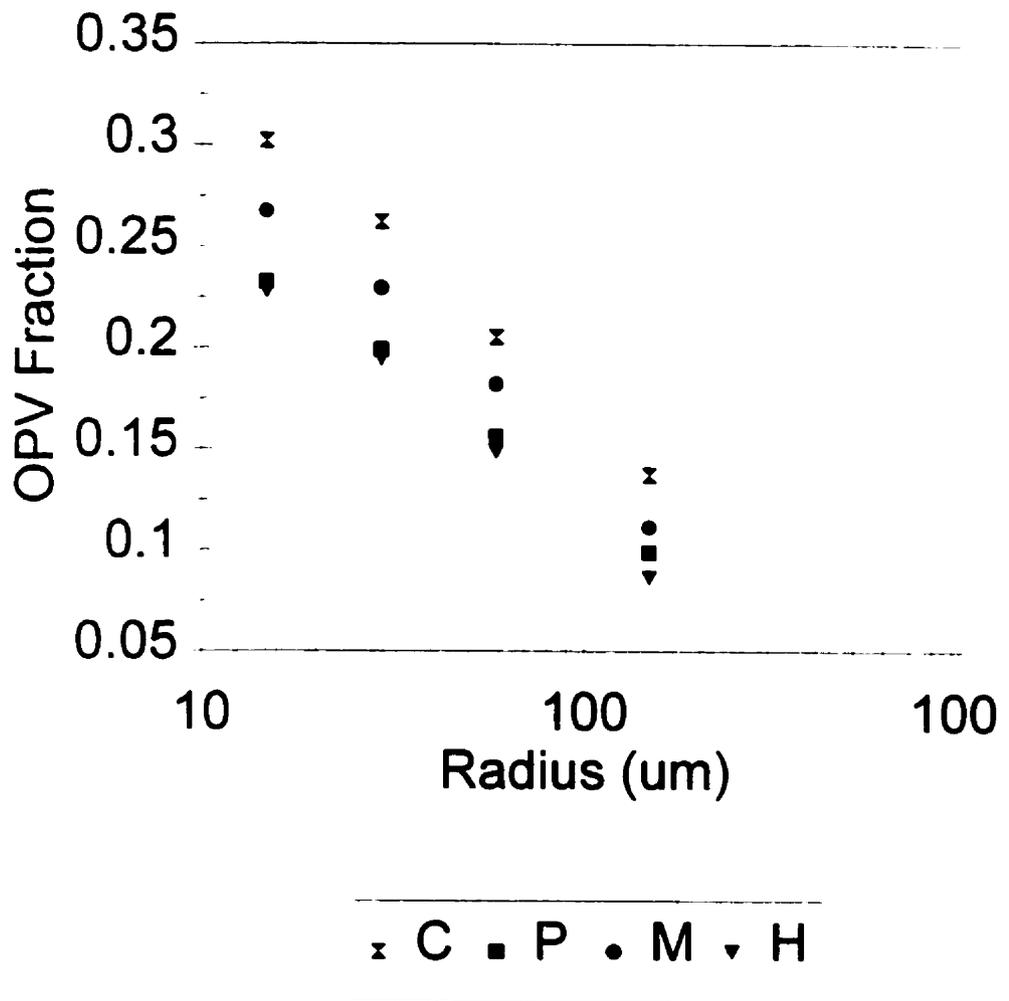
Figure 4.13. Soil pore size distribution of the 5-10 cm depth of unfertilized Waitville clay loam soil treated with leonardite, 1998.
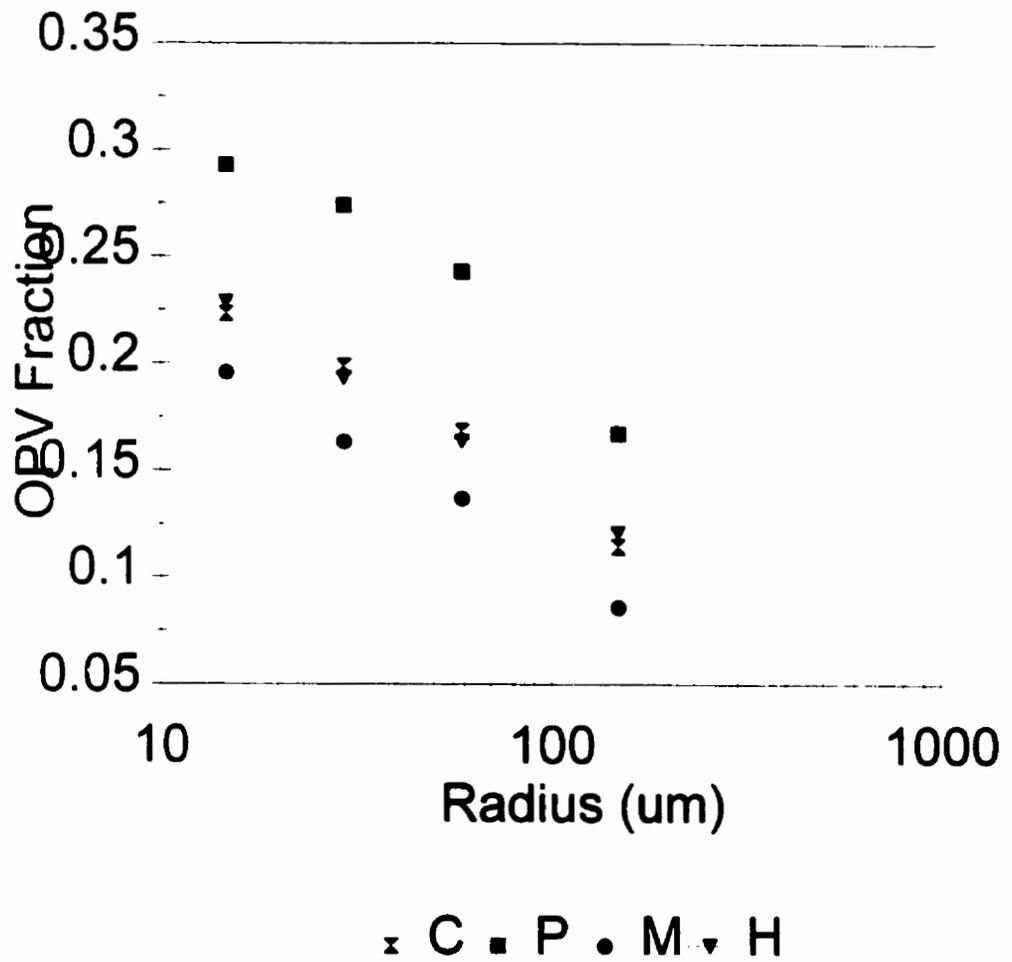
Figure 4.14. Soil pore size distribution of the 0-5 cm depth of fertilized Waitville clay loam soil treated with leonardite, 1999.
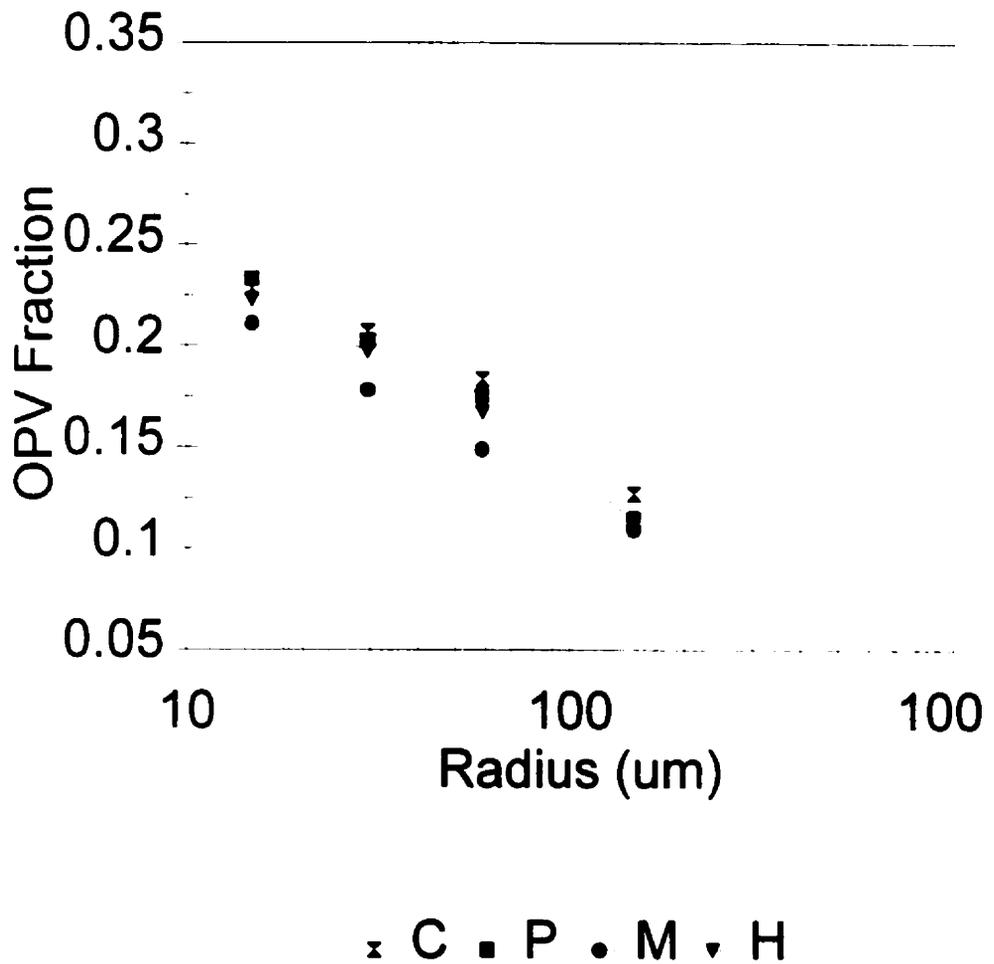
Figure 4.15. Soil pore size distribution of the 5-10 cm depth of fertilized Waitville clay loam soil treated with leonardite 1999.
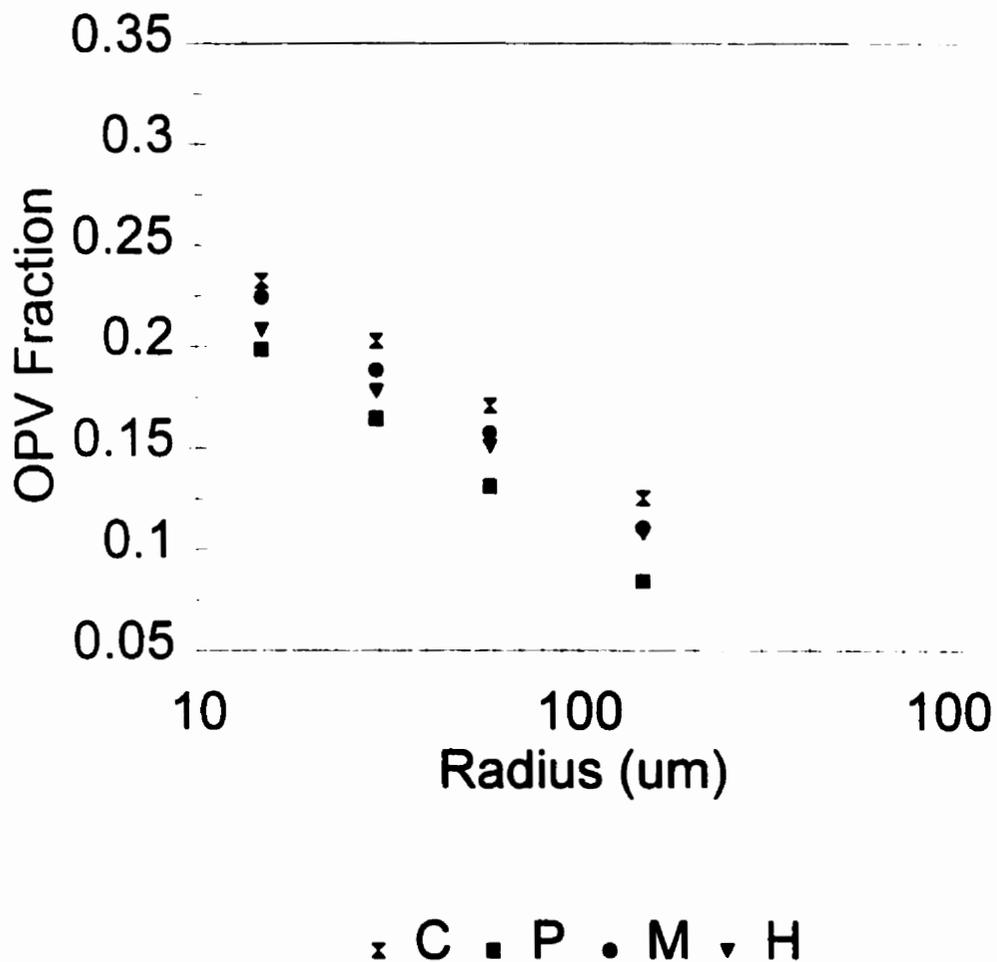
Figure 4.16. Soil pore size distribution of the 0-5 cm depth of unfertilized Waitville clay loam soil treated with leonardite 1999.

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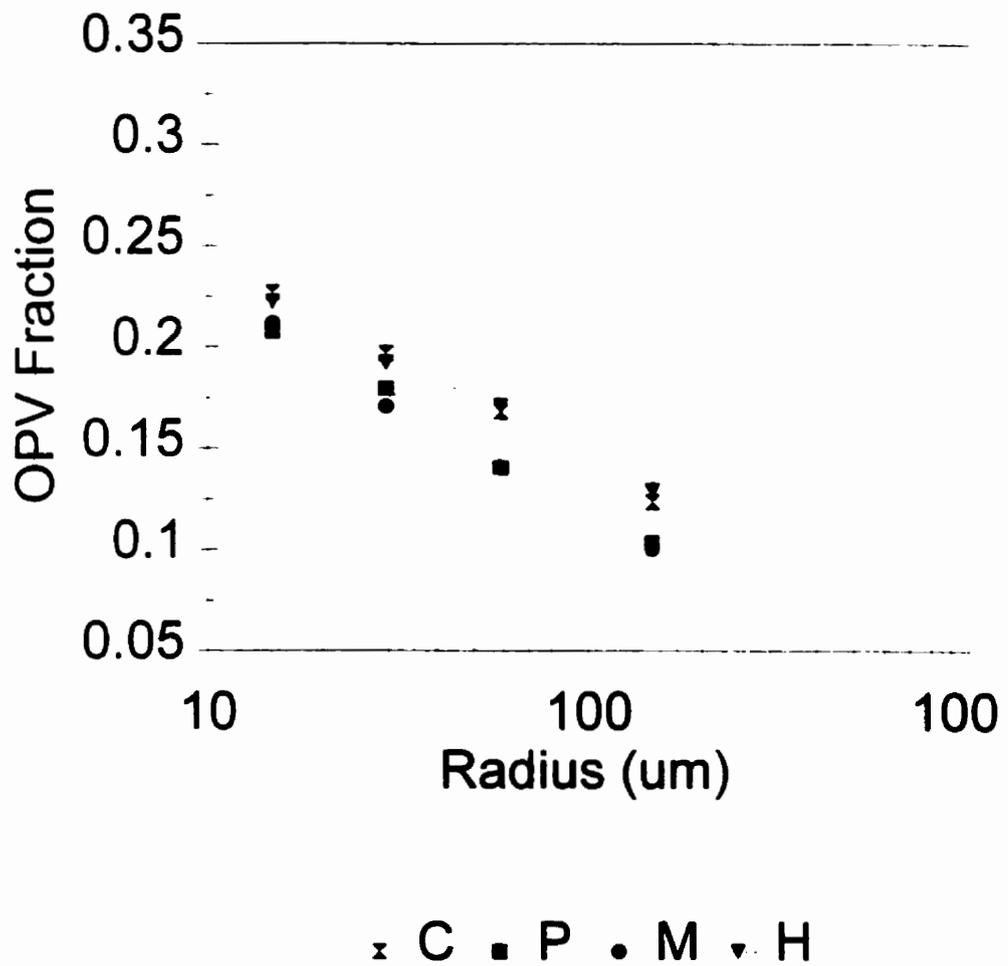
Figure 4.17. Soil pore size distribution of the 5-10 cm depth of unfertilized Waitville clay loam soil treated with leonardite 1999.

4.3.1.2. 15 Atmosphere Test. Results from 4.3.1.1. revealed porosity associated with low tensions (<1000 kPa), while results from this section (Table. 4.3.) demonstrated the porosity associated with high tensions. Between approximately 0-1000 kPa, suction is strongly affected by the soil structure (Hillel 1982). At these low matric suction values the capillary effect is primarily responsible for matric suction.

At high suction values, such as data from the 15 atmosphere data presented herein, the texture and specific surface area of the Manitoba soils are important for the soil moisture retained (Shaykewich and Zwarich 1968). Soil structure at high suction values is less consequential. Therefore, although others have found organic carbon to influence the proportion of micropores (Kay 1997), it was hypothesized that the organic amendment would have little to no effect on the permanent wilting coefficient. The results from the first year suggested that the hypothesis was correct and the experiment was not repeated in the next subsequent year.

When data were split into fertilized/unfertilized and different soil types, there was little variation between treatments and treatments versus control soils, showing that no significant treatment effect was found (Table 4.3.). As an indication of the proportion of the total pore space that was below the lower limit of plant available water, the total porosity of the soils (found from the 0 cm tension in the pore size distribution study) was also analyzed (Table 4.1.). The results herein were expected, as the permanent wilting point has been shown to be influenced by soil texture much more than by organic matter content (Shaykewich and Zwarich 1968).

Kay (1997 and references cited therein) discussed the influence of carbon on soil

macro- . meso- and micro-porosity. Mesoporosity was defined as the pores between the

upper and lower limit of plant available water. In general. an increase in organic carbon

leads to an increase in porosity at all levels. The magnitude of the increase was lower for

the macroporosity than micro- and meso-porosity. They also noted that in soils

containing swelling clays such as montmorillonite there was a negative relationship

between organic carbon and mesoporosity. This suggested that the mesoporosity was less

responsive to the organic carbon concentration than the swelling forces. Other findings

suggested that the influence of organic carbon was greatest in medium textured soils.

This was the reason for using two soils of different textural classifications.

Table 4.3. Effect of treatment on volumetric moisture percent[*] of 2 soils collected
between 0-10 cm depth subjected to 15 atmospheres of pressure.

Treatment	Waitville CL	Osborne C
C+F	11.69 ± 2.07	24.50 ± 4.19
P+F	11.99 ± 1.67	24.27 ± 3.59
M+F	10.72 ± 0.75	23.62 ± 3.14
H+F	11.19 ± 1.12	23.35 ± 2.89
C-F	11.24 ± 1.51	22.30 ± 2.98
P-F	12.75 ± 4.10	23.92 ± 2.05
M-F	12.28 ± 2.29	25.87 ± 4.06
H-F	12.60 ± 2.59	22.30 ± 2.98

[*]Mean of 2 samples per treatment within each replicate.

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4.3.2. Aggregate Stability

Baseline analysis on aggregate stability is given in Table 4.4. As shown there was

high spatial variance within the field. Aggregates are heterogenous, and therefore it was

expected that there may be some spacial variation.

Table 4.4. Change in characteristic mean diameter[*] (mm) of untreated soils taken from 2
depths within the 2 study sites 1998.

Soil	0-5 cm	5-10 cm
Osborne clay	2.75 ± 0.75	4.10 ± 1.40
Waitville clay loam	1.10 ± 0.73	0.44 ± 0.96

[*] Mean = standard deviation of 2 samples per depth within each of 4 replicates.

As expected, results from baseline analysis show that both soils are quite low in

aggregate stability. Deterioration of aggregates results when the disruptive forces acting

on the soil overpower the binding forces within the aggregate. There are highly

disruptive forces acting on the Osborne C soil. The soil contains a high fraction of

montmorillonite clay. Upon rapid wetting, water quickly enters voids in the soil. The

higher the content of clay, the higher the suction and possibility of air entrapment and

consequential pressure on the walls of the micropores. As well, differential swelling due

to the high content of montmorillonite may lead to disaggregation. The binding forces in

the Waitville CL are low. This soil was classified as a Luvisol based on the appearance

of the Bt horizon. This indicates that the main cementing agents, clay and organic matter,

have been leached away from the upper horizons which has lead to a low stability within

the plow layer.

In 1998, wheat was seeded immediately prior to amendment application. Upon addition, the amendments appeared to be highly hydrophobic. This was further demonstrated in the laboratory (Figure B.1.). In year two, wheat was seeded June 2, 1999 in Waitville CL and residual amendment effects were tested.

Figures 4.18 through 4.29 show CMD values from wet and dry sieving for the two sites and different years. The CH is a measure of aggregate stability to water. Mean and standard deviation for CH in CMD of are also shown in Tables 4.5 and 4.6.

If the hypothesis that application of leonardite would improve the soil structure had been true, this would have been seen as an increase in the number of, size of, and/or the stability of the macroaggregates. An increase in the aggregate stability and size would have resulted in higher CMD numbers for W and D, respectively. Improved stability would also have been seen as a reduction in the CH value relative to controls. A lower CH characteristic mean diameter number would have indicated that the aggregates that had undergone wet sieving had been more resistant to water erosion, and were closer in size to those that had not been subjected to the disruptive forces of water.

ANOVA was performed on CMD values for W, D, and CH. It was analyzed separately on groups of data based on fertilizer application, depth and sampling time.

In Osborne C soils sampled in 1998, results from W and D sieving are shown in Figures 4.18.- 4.21. Both wet and dry sieving results indicate no significant treatment effect for all depths, both fertilized and unfertilized (α=0.05). However, some block effects were evident. In the W sieving results, the fertilized soil had a replicate effect at the 0-5 cm depth. Both depths of unfertilized soil had block effect when D sieved.

Results for CH in CMD are given in Table 4.5. The results indicate that there is no significant treatment effect on aggregate stability for fertilized soils at both depths. In unfertilized soil, there was no significant treatment effect at the 5-10 cm depth. The results for the 0-5 cm depth show that the M treatment reduced aggregate stability compared to all other treatments and controls. There were block effects in unfertilized soils at both depths.
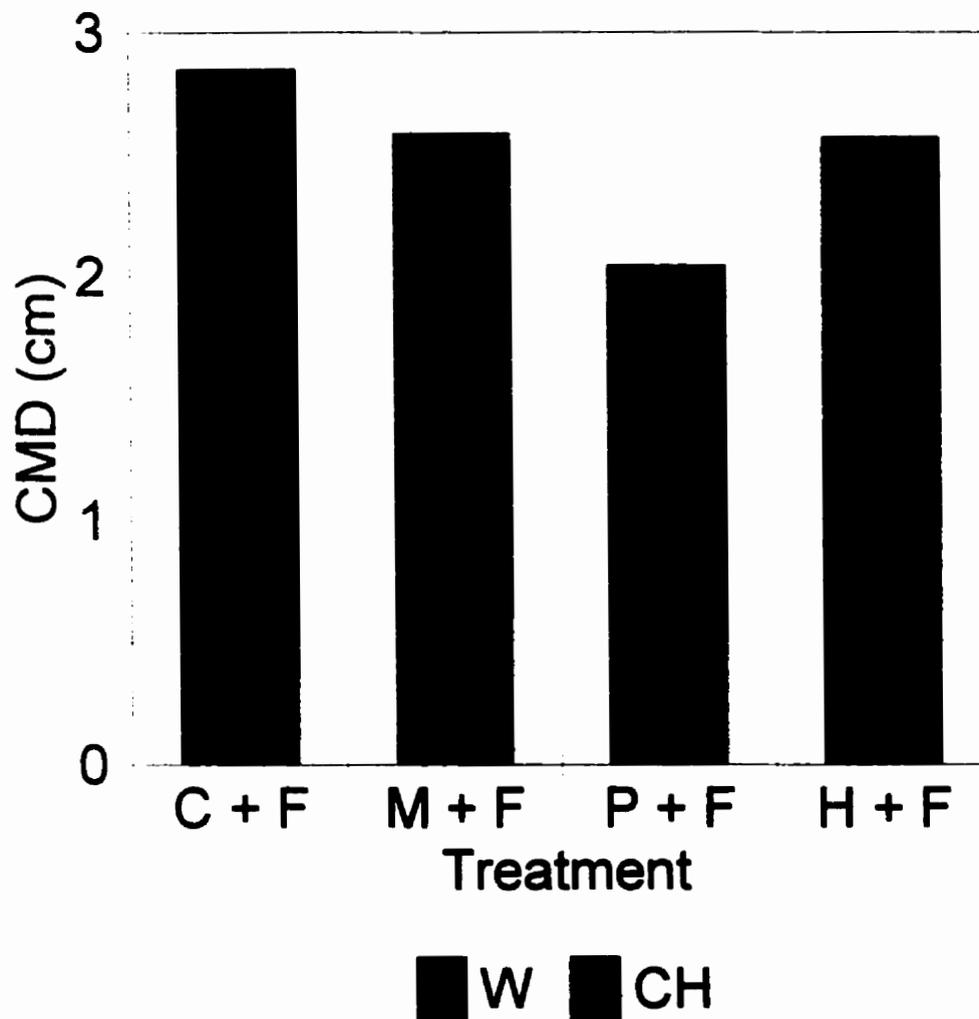
Figure 4.18. Characteristic Mean Diameters (CMD) of the 0-5 cm depth of leonardite treated soil obtained from the fertilized Osborne clay study site, 1998. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
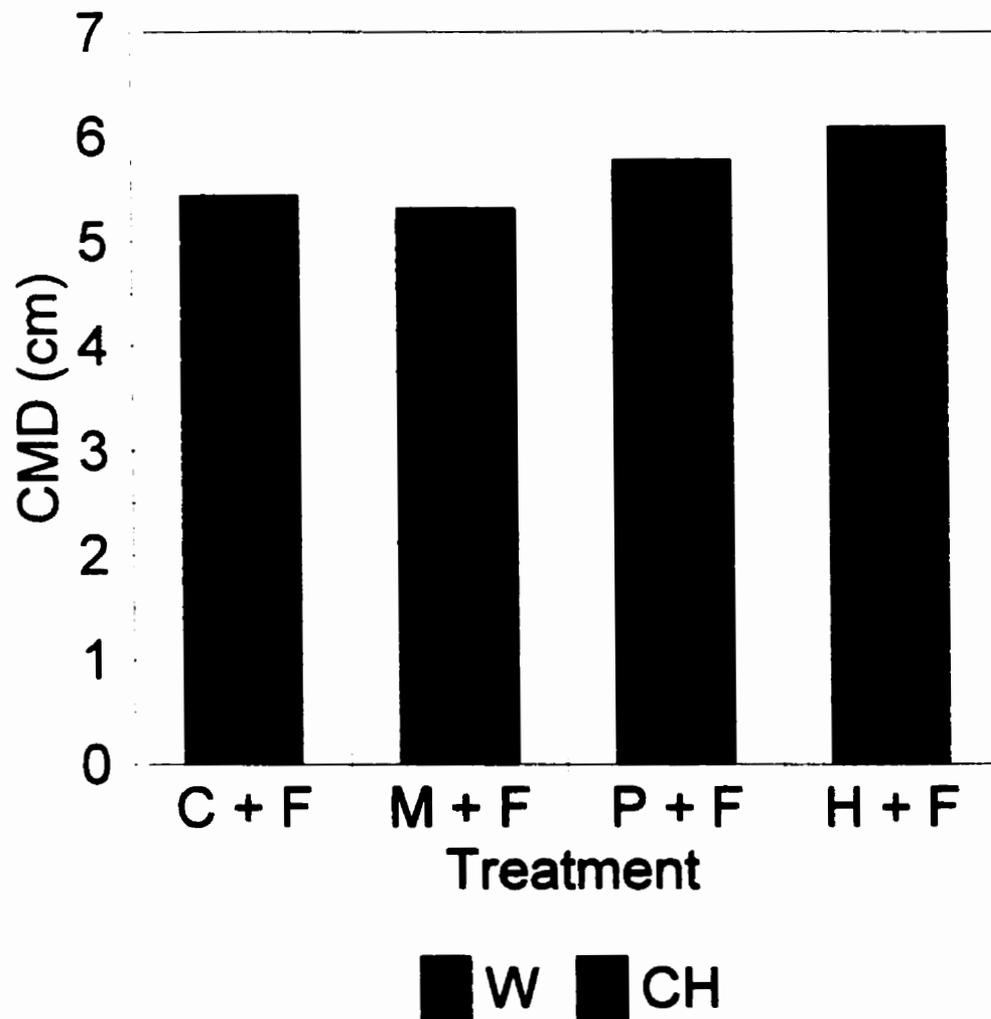
Figure 4.19. Characteristic Mean Diameters (CMD) of the 5-10 cm depth of leonardite treated soil obtained from the fertilized Osborne clay study site, 1998. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
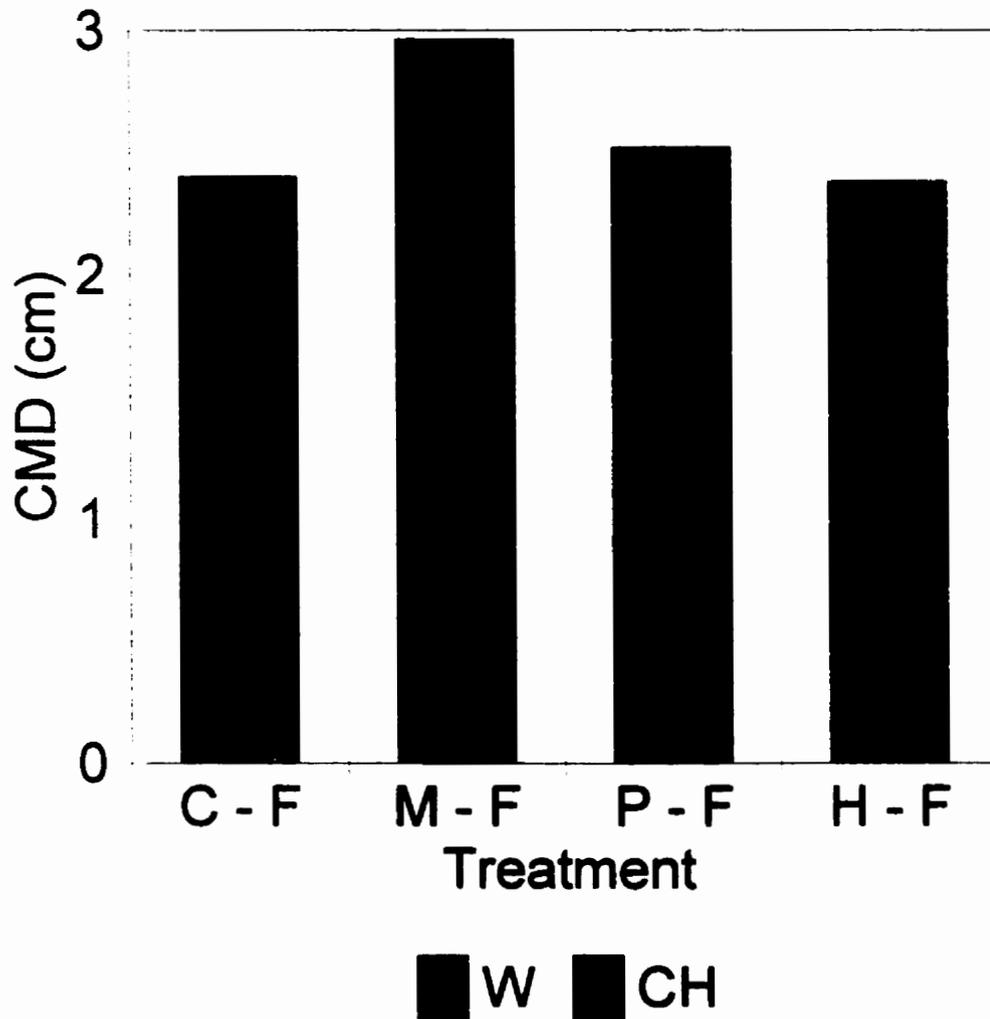
Figure 4.20. Characteristic Mean Diameters (CMD) for the 0-5 cm depth of leonardite treated soil obtained from the unfertilized Osborne clay study site, 1998. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
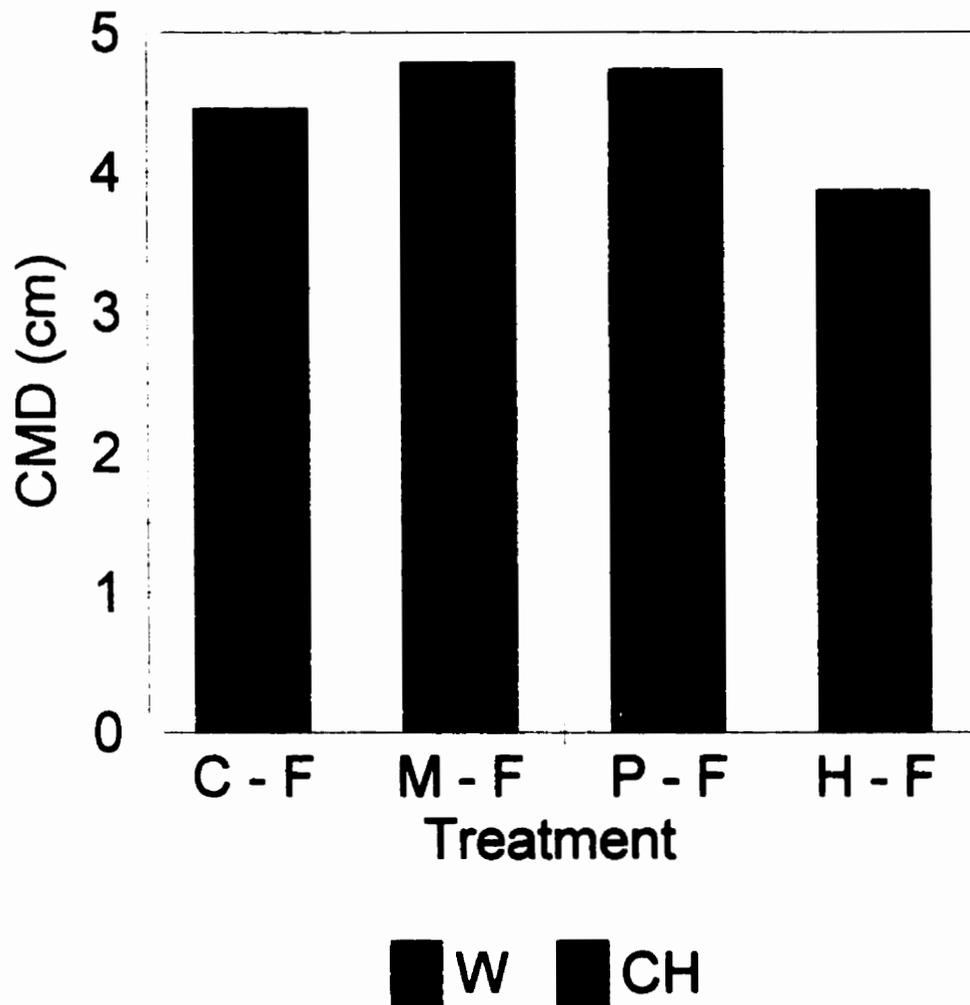
Figure 4.21. Characteristic Mean Diameters (CMD) for the 5-10 cm depth of leonardite treated soil obtained from the Osborne clay study site, 1998. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

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Table 4.5. Change in characteristic mean diameter[*] (mm) for Osborne clay soil at 2 depths. 1998.

Treatment	0-5 cm depth	5-10 cm depth
C + F	2.35 ± 1.09	5.17 ± 2.94
P + F	1.26 ± 0.32	5.40 ± 1.02
M + F	1.97 ± 0.80	5.00 ± 2.18
H + F	1.97 ± 0.55	5.54 ± 1.55
C - F	1.75 ± 0.44	4.01 ± 1.50
P - F	1.65 ± 0.71	4.36 ± 1.15
M - F	2.67 ± 0.84	4.38 ± 1.13
H - F	1.87 ± 0.53	3.42 ± 1.37

[*]Mean of 2 samples per test (wet and dry sieve), per treatment within each replicate.

In Waitville CL soils sampled in 1998, results from W and D sieving are shown in Figures 4.22-4.25. Both wet and dry sieving results indicate no significant treatment or block effect for all depths, both fertilized and unfertilized (α=0.05). Table 4.6. gives values for CH in CMD for Waitville CL for all treatments sampled in 1998. The data indicate that there was no significant treatment effect on aggregate stability for fertilized soils at both depths. In unfertilized soil, there was no significant treatment effect at either depth.
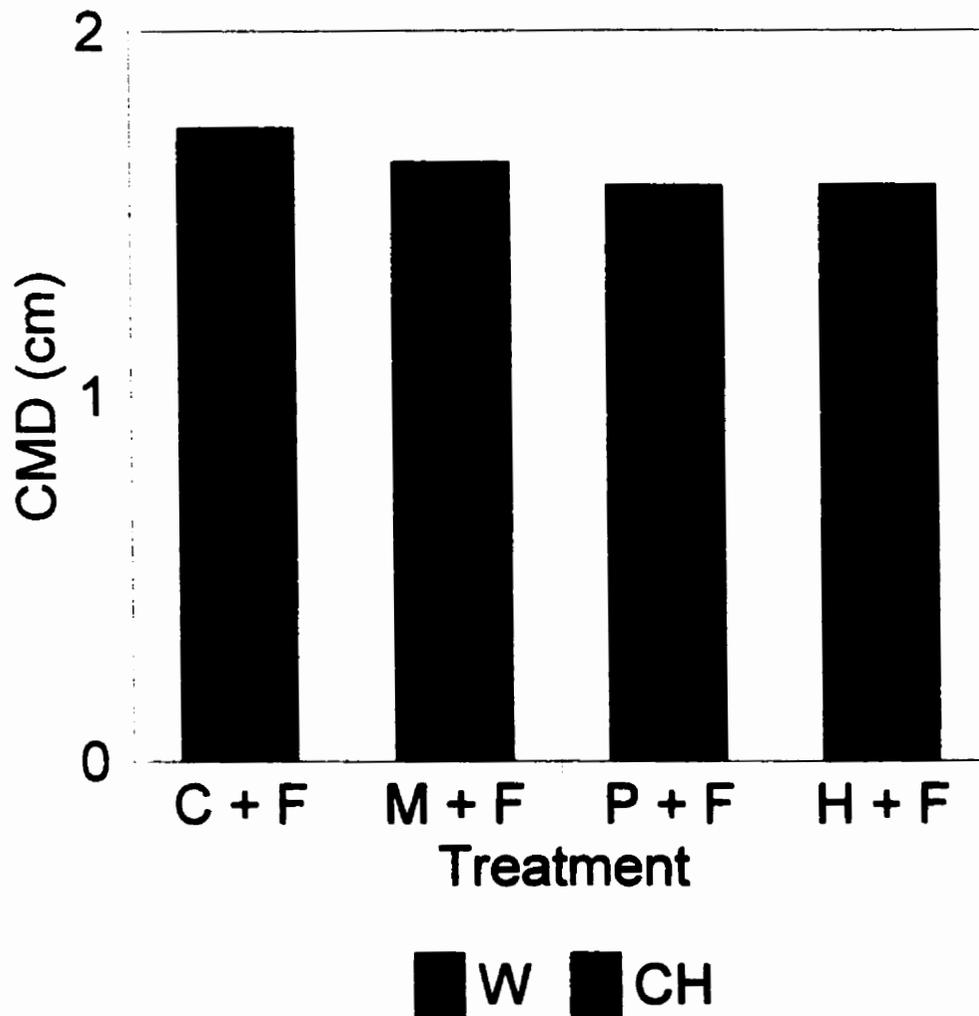
Figure 4.22. Characteristic Mean Diameters (CMD) for the 0-5 cm depth of leonardite treated soil obtained from the fertilized Waitville clay loam study site, 1998. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
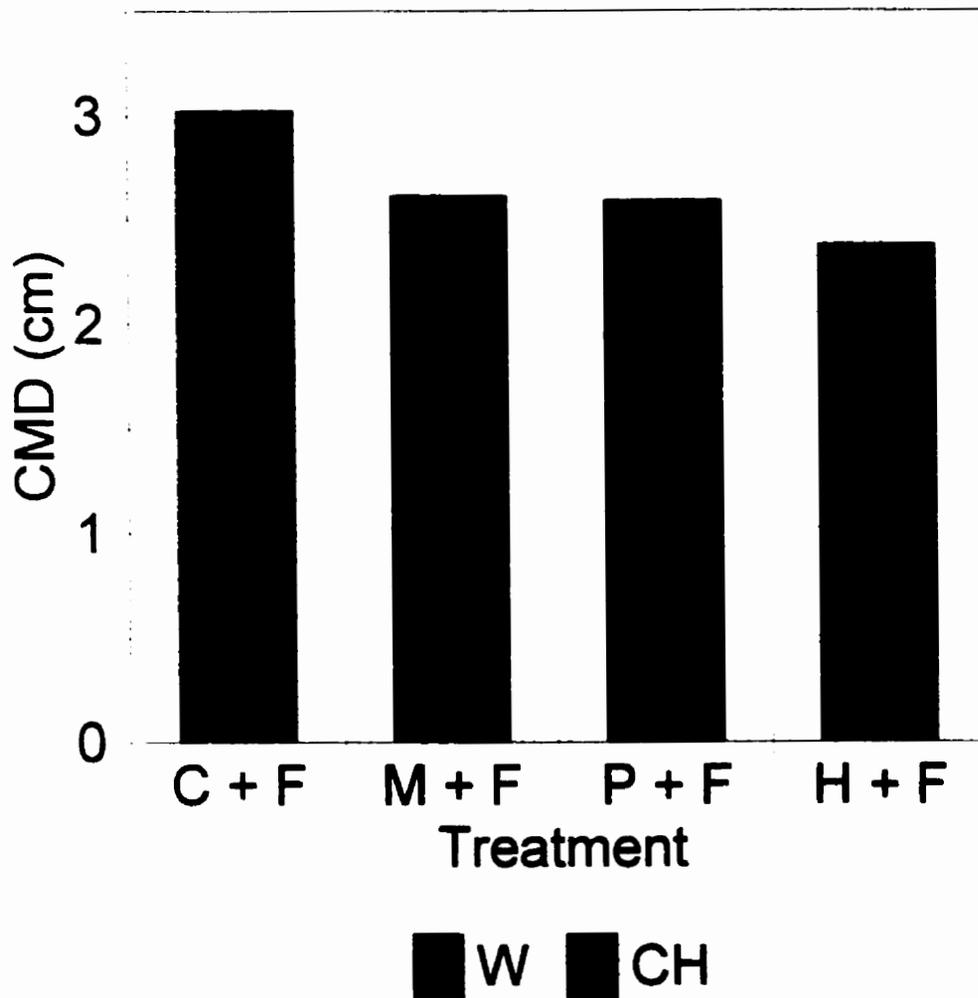
Figure 4.23. Characteristic Mean Diameters (CMD) for the 5-10 cm depth of leonardite treated soil obtained from the fertilized Waitville clay loam study site, 1998. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

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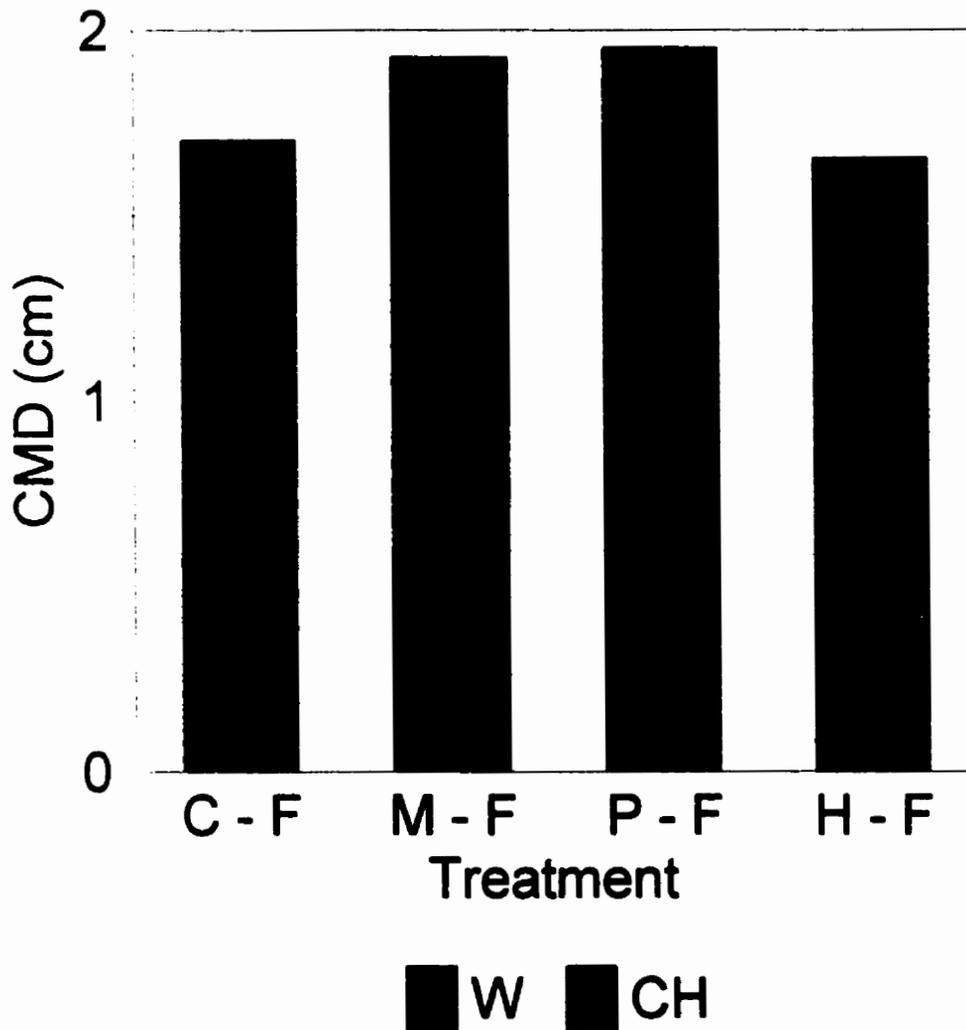
Figure 4.24. Characteristic Mean Diameters (CMD) for the 0-5 cm depth of leonardite treated soil obtained from the unfertilized Waitville clay loam study site, 1998. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
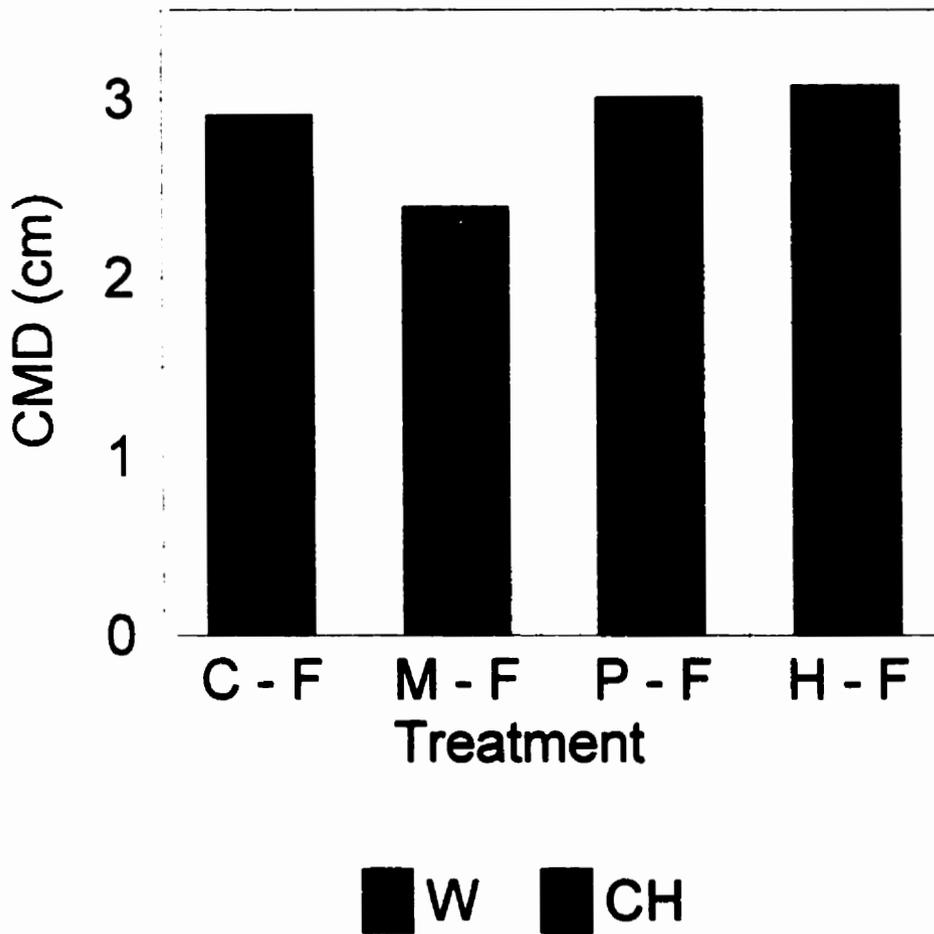
Figure 4.25. Characteristic Mean Diameters (CMD) for the 5-10 cm depth of leonardite treated soil obtained from the unfertilized Waitville clay loam study site, 1998.CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

Table 4.6. Change in characteristic mean diameter[*] (mm) for Waitville clay loam soil at 2 depths. 1998.

Treatment	0-5 cm depth	5-10 cm depth
C + F	1.12 ± 0.40	1.32 ± 0.30
P + F	0.94 ± 0.59	0.81 ± 0.23
M + F	1.11 ± 0.27	1.05 ± 1.66
H + F	0.99 ± 0.20	1.09 ± 1.07
C - F	0.42 ± 0.83	0.93 ± 0.76
P - F	1.07 ± 0.45	1.11 ± 2.50
M - F	0.87 ± 0.38	0.74 ± 0.71
H - F	0.60 ± 0.14	1.48 ± 0.77

[*]Mean ± standard deviation of 2 samples per test (wet and dry sieve), per treatment within each replicate.

Figures 4.26-4.29. display results for W and D sieving of Waitville CL soils sampled in 1999. Both W and D sieving results indicate no significant treatment or block effect for all depths, both fertilized and unfertilized ($\alpha=0.05$). The change in CMD for Waitville CL for all treatments sampled in 1999 is given in Table 4.7. ANOVA indicates that there was no significant treatment effect on aggregate stability for fertilized soils at both depths. In unfertilized soil, there was no significant treatment effect at either depth.
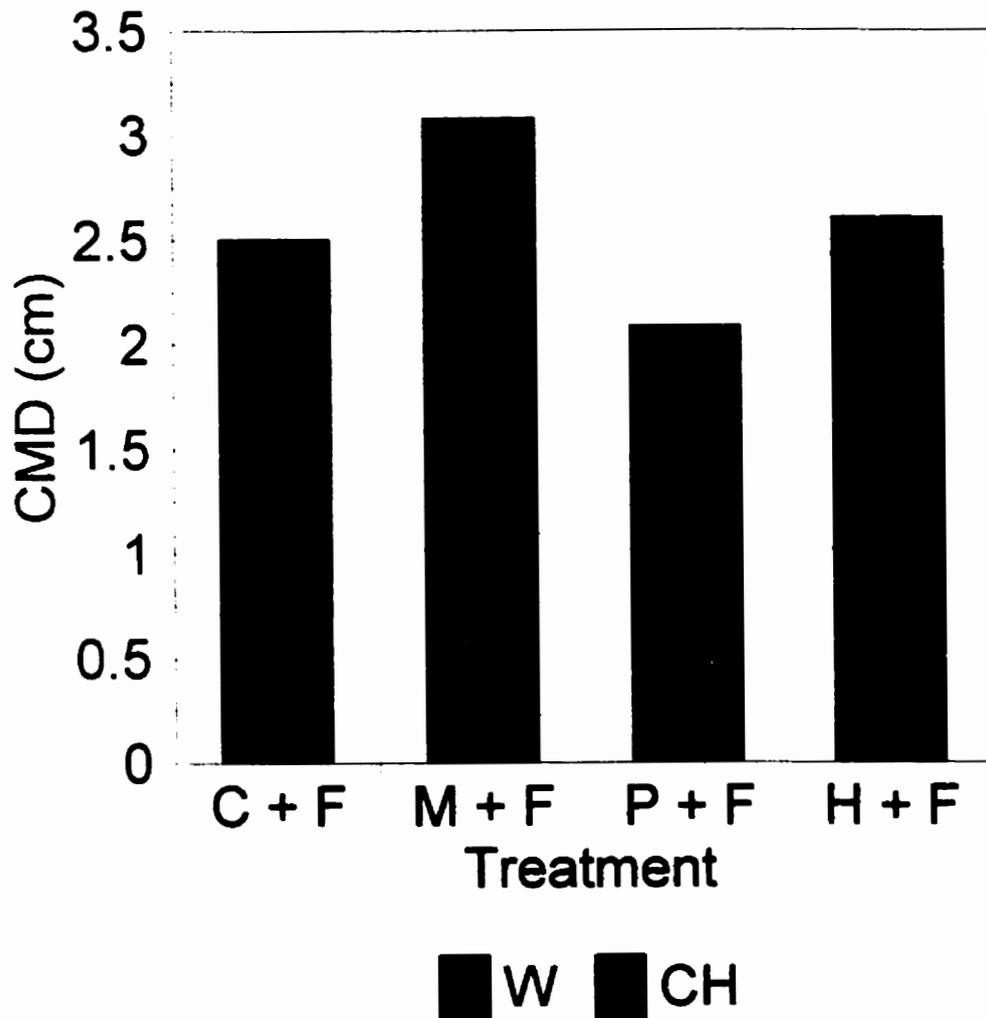
Figure 4.26. Characteristic Mean Diameters (CMD) for the 0-5 cm depth of leonardite treated soil obtained from the fertilized Waitville clay loam study site, 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
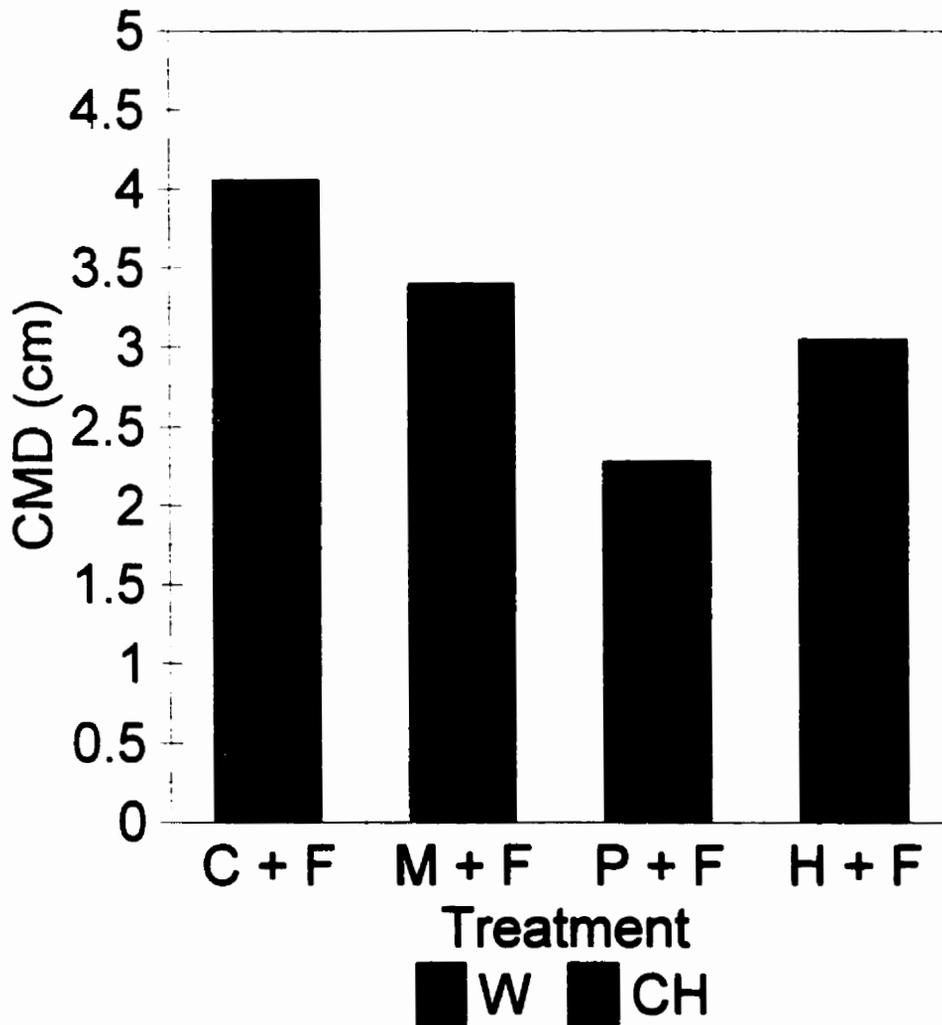
Figure 4.27. Characteristic Mean Diameters (CMD) for the 5-10 cm depth of leonardite treated soil obtained from the fertilized Waitville clay loam study site, 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
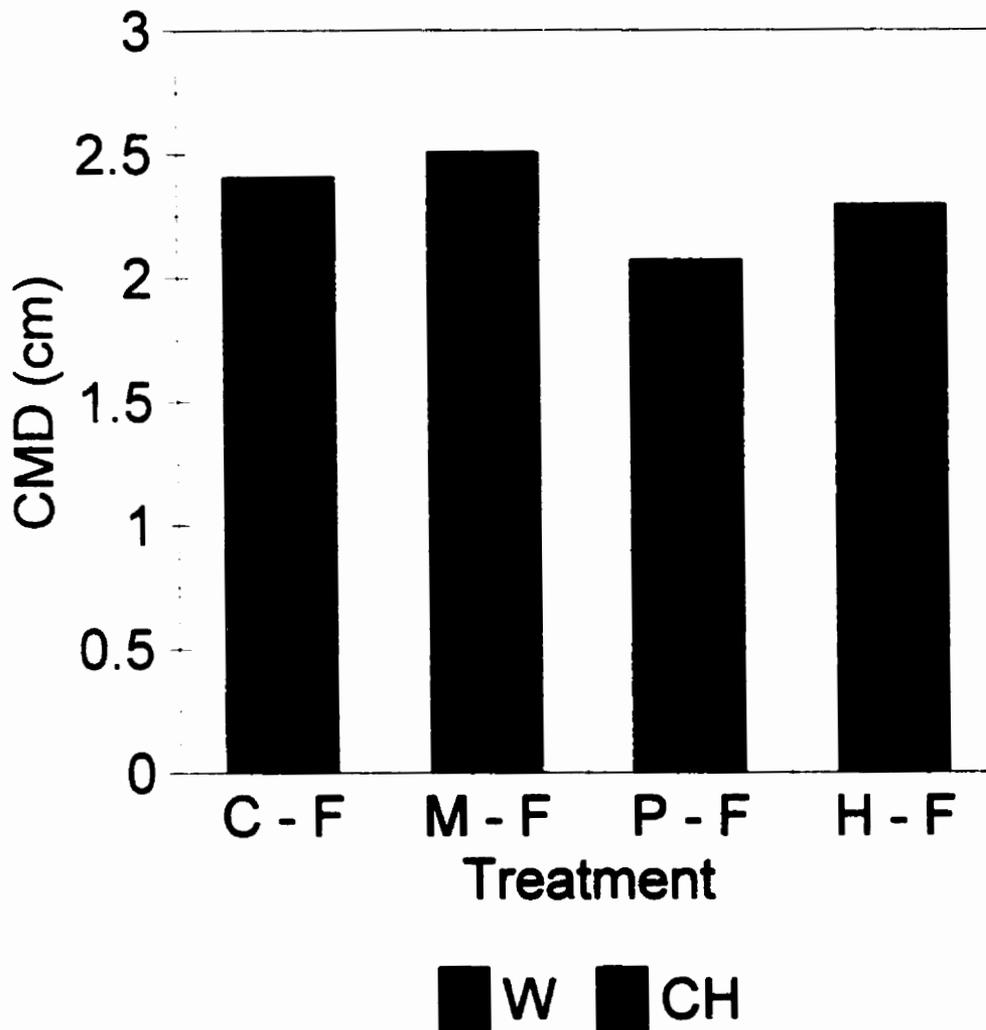
Figure 4.28. Characteristic Mean Diameters (CMD) of the 0-5 cm depth of leonardite treated soil obtained from the unfertilized Waitville clay loam study site, 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
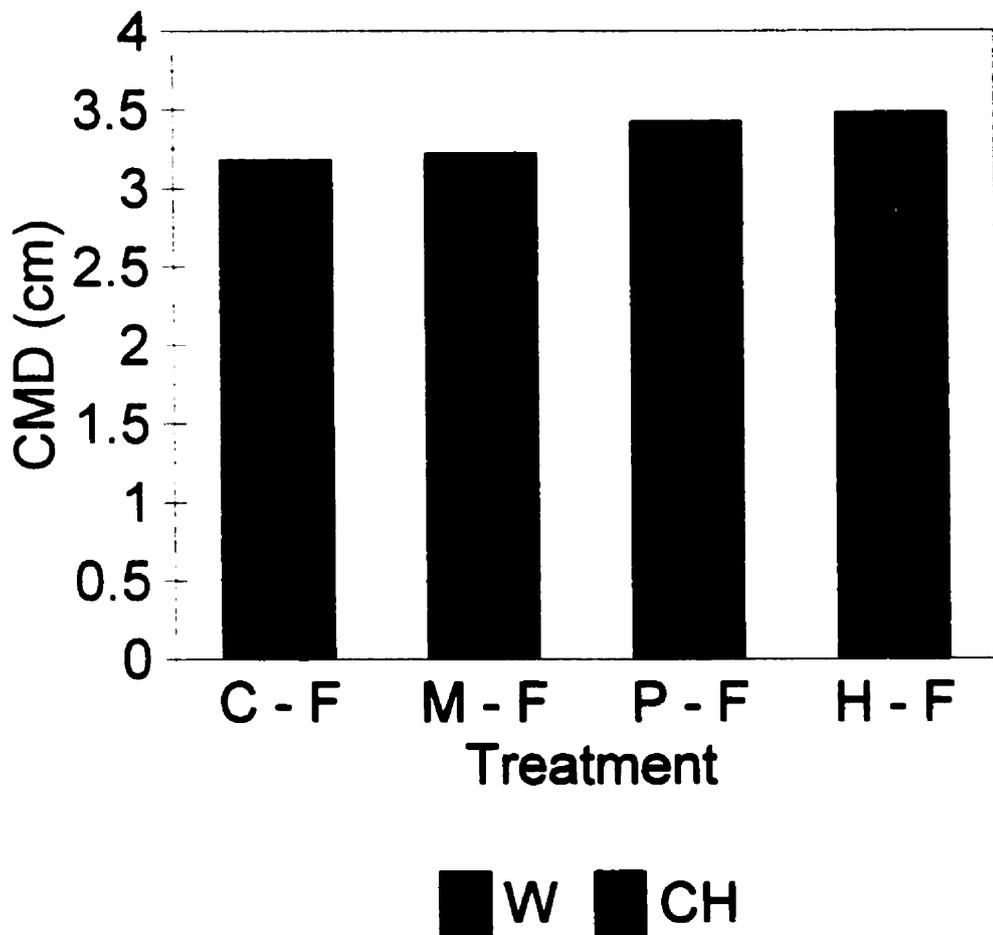
Figure 4.29. Characteristic Mean Diameters (CMD) of the 5-10 cm depth of leonardite treated soil obtained from the unfertilized Waitville clay loam study site, 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

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Table 4.7. Change in characteristic mean diameter[*] (mm) of leonardite amended
Waitville clay loam obtained from 2 sampling depths 1999.

Treatment	0-5 cm depth	5-10 cm depth
C + F	1.24 ± 0.76	2.38 ± 2.06
P + F	0.96 ± 0.23	0.67 ± 0.77
M + F	1.98 ± 0.79	1.58 ± 1.55
H + F	1.66 ± 0.71	1.39 ± 1.21
C - F	0.31 ± 0.17	0.71 ± 0.73
P - F	0.21 ± 0.51	0.86 ± 1.60
M - F	0.27 ± 0.91	0.93 ± 1.65
H - F	0.40 ± 0.38	0.70 ± 0.73

[*]Mean of 2 samples per test (wet and dry sieve), per treatment within each replicate.

Organic amendments interact with the soil (Brandsma *et al.* 1999). Unlike chemical

fertilizers which provide nutrients to crops regardless of the medium in which they are

grown. an amendment's performance is strongly dependent on soil type. There are a

multitude of reasons for the apparent inability of leonardite to influence soil structure in

this experiment.

The reason that the field applied raw leonardite amendment did not work was

unknown at this point. However. it was observed that the material was hydrophobic. It

was postulated that the inert nature of the leonardite amendment may have been attributed

to its hydrophobic nature. Therefore, it was felt that the material may behave differently

if it was altered to a more hydrophilic form. This was the rational which lead to the

development of the studies discussed in Chapters 6 and 7. In these studies the impact of a

impact of a modified soluble powder form of leonardite on soil physical properties and

crop productivity were examined.

4.3.3. Total Organic Carbon

The effects of selected leonardite amendments (H and P) on total organic carbon content of Waitville CL are shown in Table 4.8. In all cases, leonardite did not have any effect on organic carbon content.

In both the fertilized and unfertilized plots, there was a general decrease in OC with depth. This was expected as soil organic matter generally decreases with depth.

It appears that the addition of leonardite at the given application rates did not have the ability to alter the overall organic carbon content of the soils.

If there had been a great deal of variability in the total soil organic carbon in the field itself, the change in organic carbon due to addition of leonardite amendments may have been masked. This may have been the case as the coefficient of variation was quite high and as there was a difference in the depth of the Ap horizon within the replicates (Table A.6.). Experimental error could also have also contributed to the high variability. Only one sample from each treatment within each replicate was analyzed. Greater duplication of analysis could have possibly reduced the coefficient of variation if the variation was due to experimental error rather than variation within the field.

The effect of a soluble form of leonardite on total soil organic carbon was determined in an incubation study discussed in Chapter 6. To address the effect of field variability, soil used in the incubation study was taken from a small area that was thought to be relatively homogenous with respect to organic carbon content. As well a number of application rates on the soil and higher duplication of analysis was used, to determine if the total organic carbon content of the soil could be increased.

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Table 4.8. Effect of leonardite treatment on organic carbon[*] for 2 sampling depths of leonardite treated Waitville CL 1998.

| Treatment | Organic Carbon (%) Depth (cm) | |
	0-5	5-10
C+F	1.78 ± 0.57 a[‡]	1.78 ± 0.56 a
H+F	2.26 ± 0.61 a	1.67 ± 0.65 a
P+F	1.78 ± 0.57 a	1.14 ± 0.35 a
C-F	2.25 ± 0.20 a	1.36 ± 0.14 a
H-F	2.46 ± 0.73 a	1.38 ± 0.36 a
P-F	2.12 ± 0.75 a	1.04 ± 0.37 a

[*] Mean ± standard deviation of 1 sample per treatment within each replicate

[‡] Means followed by the same letter with similar fertilizer status within columns were not different (p>0.05)

4.4. Conclusion

Upon application the hydrophobic nature of the leonardite materials was apparent

(Appendix B). This water repellence was carried into the second field season.

Leonardite amendment did not significantly alter pore size distribution or aggregate

stability of the two soils analyzed. The tension plate experiment and D and W sieving

were used in 1998 to test treatment effects and 1999 to assess residual effects. In both

years, there was no significant treatment effect. The 15 Atmosphere test was only

conducted in 1998, as it was believed that the lower limit of available water would not be

altered with addition of organic matter. The organic carbon test (conduced on Waitville

CL 1998) samples showed that there was no leonardite treatment effect.

5. EFFECT OF FIELD APPLIED LEONARDITE ON WHEAT
(*Triticum aestivum* L.) YIELD

Abstract

The effect of several forms of leonardite on total wheat (*Triticum aestivum* L.) yield was determined.

Data from 1998 showed that there was no significant improvement in yield with any of the leonardite forms on either Osborne C or Waitville CL. Due to flooding, wheat could not be seeded on Osborne C in the second year. There were no significant residual treatment effects on the yield of wheat grown in Waitville CL in 1999.

5.1. Introduction

Studies have shown that humic acids (HAs) can have a positive effect on germination (Ayuso *et al.* 1996) as well as root density and length (Mylonas and Mccants 1980). It has been suggested that HAs influence plant root density due to there ability to increase availability of micronutrients (Clapp *et al.* 1998). In addition to indirect effects by improving soil structure, they have been shown to directly influence plant growth by their ability to limit soil aluminum toxicity and accelerate plant respiration by increasing cell permeability or by some hormonal response (O'Donnell 1973). However, some studies have shown HAs to have little effect on plant growth (Kelting *et al.* 1997). As well, previous researchers have indicated that a positive growth response to HA treatment could only be found when the a soil low in organic matter or nutrient solution (Clapp *et al.* 1998 and references cited therein). Further, a change in soil structure via amendment application may not alter crop yield (Almendros 1994).

A situation in which germination was improved would result in a greater number of seedlings per unit area. Increased plant root density and length would result in an enhanced ability to obtain water and nutrients. Increased respiration would result in greater production per plant. Under any of these situations, it would be expected that the overall crop yield would be increased.

Leonardite has a high content of HA. Therefore, although the leonardite amendment may not have affected soil stucture (Chapters 3 and 4) it was hypothesized that the soil amendment may still have been able to improve total yield as a result of some other crop

response.

Although previous researchers have suggested that HA treatment effects would not be seen on highly productive soils, the study was conducted in an attempt to evaluate if the addition of leonardite could improve primary productivity of soils which are presently used for agricultural purposes. The purpose of this experiment was to determine if leonardite treatment could result in an improvement in wheat (*Triticum aestivum* L.) yield grown in Obsorne C and Waitville CL soils, and if so, whether the improvement was due to improved soil physical properties as postulated by Wallace and Wallace (1990) or some other effect. The study was conducted simultaneously with those discussed in Chapters 3 and 4 in an effort to correlate any treatment effects on soil structure with those on crop yield.

5.2. Materials And Method

Several studies were conducted to evaluate the effect of leonardite amendment on soil physical properties (Chapters 3 and 4). The present study was conducted to observe if the treated soils showed any positive effects on crop productivity. Selected soil properties and climatic conditions for these sites are shown in Appendix A.

5.2.1. Field Plot Design

Wheat yield was determined for both Osborne clay (C) and Waitville clay loam (CL) soil types. AC Barrie wheat was seeded May 5 and May 22, 1998 and harvested August

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25 and August 27, 1998 in the Waitville CL and Osborne C soils, respectively in year

one. In year two, wheat was seeded on June 2, 1999 and harvested October 13, 1999 in

the Waitville CL soil. As a result of localized flooding, it could not be seeded in Osborne

C in the 1999 season.

The experiment had a randomized complete block design with split plots which were

fertilized (+F) and not fertilized (-F). There were 4 replicates, each containing three

treatments and one control (C). Treatments included a pulverized leonardite powder (P),

a ground leonardite material <2 mm diameter (M), and a liquid humic acid extraction

(H)[5]. Leonardite was added and rototilled to approximately 7 cm in the spring of 1998

immediately prior to seeding AC Barrie wheat (*Triticum aestivum* L.). Rates of P, M and

H were 150 kg/ha, 1000 kg/ha and 100 L/ha respectively. Residual effects of the

leonardite treatments on Waitville CL were assessed in 1999. Due to flooding, the

Osborne C soil was not analyzed.

5.2.2. Field Operations

Spring fertilizer application rates were as follows. In 1998, in the Osborne C soil 85

kg/ha nitrogen had been applied in the previous fall and 20 kg/ha nitrogen and 20 kg/ha

phosphorous fertilizers were applied in the spring. The Waitville CL soil received 125

kg/ha nitrogen, 20 kg/ha phosphorous, and 24 kg/ha sulphur. In 1999 the Osborne C

received 50 kg/ha of nitrogen and 40 kg/ha of phosphorous. The Waitville CL soil

received 150 kg/ha nitrogen, 40 kg/ha phosphorous and 24 kg of sulfur. Nitrogen was

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[5]The extraction contained mainly humic acids, however, fulvic acids were also present.

broadcast in the form of urea. Phosphorous was applied with the seed in the form of

MAT, and sulphur was applied as ammonium sulphate.

Harvest yield was estimated through sampling 2 rows three meters long for each

treatment. Following 48 hours of drying, total weight and grain weight measurements

were obtained.

5.2.3. Statistical Analyses

Analyses of variance (ANOVA) tests were performed using JMP IN software (SAS

Institute 1997). Fertilized and unfertilized plots were examined separately. The (LSD)

test was used to compare treatments found to be significantly different in ANOVA at the

95% level of significance.

5.3. Results And Discussion

The hydrophobic nature of the amendments was observed upon application. This was

further verified in the laboratory in a water solubility test (Appendix B).

Results for wheat yield in 1998 are shown in Table 5.1. The data indicated that there

was no significant treatment effect on crop yield at both sites in 1998.

The hydrophobic nature of the leonardite material was apparent upon field application

and in the laboratory (Appendix B). It is postulated that the material remained insoluble

throughout the entire growing season. This may have reduced the mobility of the

leonardite in the soil, and therefore reduced the contact of the material with the wheat,

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roots. Any effect of HA on the crop would be reduced as the distance between the root and the HA increased. Therefore, it was suspected that the hydrophobic nature of the material was the main reason that no effect was seen in the 1998 harvest yield. It was hypothesized that the solubility of the leonardite may increase with time due to microbial decomposition. The extent of and time required for the microbial breakdown of leonardite was unknown. Therefore, the residual effects of the leonardite on crop yield in 1999 were also assessed to determine if there was a treatment effect following two growing seasons.

Table 5.1. Effect of leonardite on wheat yield[+] (kg/ha) in 1998 grown in two leonardite amended study soils.

Treatment	Osborne clay	Waitville clay loam
C - F	2008 ± 394	3001 ± 244
P - F	1915 ± 476	3150 ± 258
M - F	1650 ± 464	3058 ± 868
H - F	1971 ± 300	3281 ± 216
C - F	2224 ± 284	2158 ± 363
P - F	2250 ± 291	1966 ± 522
M - F	2463 ± 302	2273 ± 344
H - F	2374 ± 144	2191 ± 722

[+] Mean ± standard deviation of 4 replicates per treatment.

Results for wheat yield in 1999 are shown in Table 5.2. The data indicate that there was no significant residual treatment effect on crop yield at both sites in year two. It therefore appears that the chemical structure of the amendment was no significantly altered following two growing seasons. Therefore. it was suspected that the leonardite remained insoluble throughout the two years of study. Due to flooding. no data was available for crop yield in year two on the Osborne C.

No residual treatment effect was seen in Waitville CL (1999). The inert properties of the material may have been a result of its hydrophobicity. This postulation brought about the initiation of studies using a modified soluble leonardite HA extract (P') as a soil amendment to increase soil aggregate stability and crop productivity (Chapters 6 and 7).

Table 5.2. Effect of leonardite on wheat yield[*] (kg/ha) for Waitville clay loam.

Treatment	Waitville C
C – F	1980 ± 360
P – F	2217 ± 342
M – F	2247 ± 428
H – F	2224 ± 266
C - F	1160 ± 349
P - F	1399 ± 155
M - F	1198 ± 178
H - F	1362 ±127

[*] Mean ± standard deviation of 4 replicates per treatment.

5.4. Conclusion

There was no leonardite treatment effect on the wheat (*Triticum aestivum* L.) yield

grown in Osborne C and Waitville CL in 1998. In 1999, the wheat yield was measured in an effort to determine if residual treatment effects were seen in the Waitville CL. Spring flooding prevented seeding of Osborne C in 1999. No residual treatment effects were found on crop yield grown in Waitville CL. The inert nature of the leonardite was attributed to its hydrophobic nature which reduce its contact with wheat roots.

6. MODIFIED SOLUBLE POWDER LEONARDITE ON AGGREGATE STABILITY AND WHEAT (*Triticum aestivum* L.) YIELD - GROWTH CHAMBER EXPERIMENT

Abstract

Incubation studies were conducted to determine if a soluble modified leonardite powder (P') could improve the productivity of AC Barrie wheat grown in Waitville CL Gray Luvisol. Effects of surface applied P' on aggregate stability and crop biomass were investigated for three time increments. Samples were taken immediately following amendment application (immediately following seeding), 6 weeks following seeding and at wheat maturity.

After 6 weeks of growth, soil chemical properties were investigated. Organic carbon (OC), electrical conductivity (EC) and pH were measured for all treatment application rates and 1.2 cm depth intervals.

Results showed there to be no significant treatment effect on aggregate stability or biomass for samples taken at all time intervals. Grain yield and harvest index measured at maturity showed no significant treatment effect. Chemical analysis revealed that as P' application increased, OC increased, while EC was unchanged. Results for pH were all similar except for the highest application rate, in which pH was increased.

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6.1. Introduction

All experiments described in previous chapters dealt with applying a raw leonardite material to the soil. This material is quite inexpensive due the fact that it is not processed. However, the previous chapters have shown that the material was relatively inert as it does not provide any beneficial effects on soil structure or wheat yield. Studies have shown that when they were modified (where in most cases hydrophilicity increased), HA had greater effects on soil physical properties than the unaltered material (Almendros 1994). As well, it was hypothesized that increased mobility associated with alteration of the leonardite in this experiment would result in increased root - HA interaction (Chapter 5).

Chapters 6 and 7 describe experiments where a chemically altered form of leonardite was used to amend the soil. This material is soluble in water (Appendix B) and has increased surface area compared to the raw material. Due to these changes in the characteristics of the leonardite, it was hypothesized that the material would be more physically active that its unaltered counterpart.

The purpose of the experiment was to determine if a modified leonardite powder would be more effective in improving aggregate stability and wheat (*Triticum aestivum*) productivity than products used in previous chapters. Incubation chambers were used in the pot study to minimize the error associated with field position. As well, five application rates were used in an attempt to determine the most beneficial amount of amendment application. The raw leonardite was highly hydrophobic as expected

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(Richardson and Wollenhaupt 1983). It was hypothesized that if the material could be altered to a hydrophilic (ie. soluble) powder material it would be more mobile in the soil.

6.2. Methods and Materials

The experiments selected in this study were conducted on incubated Waitville CL soils. The degraded Waitville clay loam soil was selected for the study as it was felt that this soil would be more responsive to the amendment addition than the Osborne C. Selected soil and amendment properties are shown in Appendix A and B, respectively. The chemically altered leonardite powder (P') that had been extracted using KOH was provided by the supplier. It was tested in the laboratory and determined to be soluble in water.

6.2.1. Incubation Conditions

A total of 54 pots were arranged in a randomized block design consisting of 3 replicates, with 6 application rates of soluble leonardite powder (P') and 3 sampling times. Pots were filled by weight: 5.5 kg of soil was added to 10 cm radius pots (soil depth was approximately 20 cm). AC Barrie wheat (*Triticum aestivum* L.) was seeded at a rate of 12 seeds per pot on June 1, 1999. Fertilizer was mixed in the top 10 cm of all pots with application rates of 75 kg/ha nitrogen (Urea), 40 kg/ha phosphorous (phosphoric acid) and 25 kg/ha potassium (potassium hydroxide). Leonardite was manually applied directly to the soil surface. No tilling or mixing of the soil followed

leonardite application. Rates of P' were 0. 75. 150. 300 and 500 kg/ha. Pots were watered to field capacity.

Soil was incubated at 60 % humidity and had 18 hours of daylight with daytime and nighttime air temperatures of 20°C and 13°C, respectively. Pots were rotated and soil was watered to field capacity weekly. At 8 and 14 days following seeding wheat emergence counts were recorded, and after 14 days wheat was reduced to 3 plants/pot. Soil samples were taken at seeding (T 0), 6 following seeding (T 6) and harvest (T H), for dry and wet sieving. After 6 weeks of growth (July 13) the wheat biomass was determined and wheat was harvested at maturity November 26. 1999 at which time yield and biomass were measured.

6.2.2. Aggregate Size Analysis

Undisturbed bulk samples were removed from pots. Samples were dried and broken into aggregates. Four, 50 g samples less than 9.5 mm in diameter were prepared; 2 samples were used each for wet (W) and dry (D) sieving. Aggregate stability was determined by comparing the characteristic mean diameters (CMD) obtained from D and W sieving as previously discussed in section 4.2.5.

6.2.3. Wheat Emergence, Biomass and Yield

Emergence counts in each pot were taken following 8 and 14 days of growth. Total biomass was measured following 6 weeks growth and at maturity. Shoots were cut approximately 1 cm above ground level, and transferred to pre-weighed paper bags.

Samples were stored in a drying room at room temperature for 5 days, re-weighed, and biomass was calculated.

Grain yield was measured at maturity. Grain was separated from the plant and its mass was calculated. Harvest Index (%), the ratio of grain to biomass multiplied by 100 %, was calculated.

6.2.4. Organic Matter, Electrical Conductivity and pH Measurements

Using a backsaver probe, soil from the 6 week incubation period was extracted and separated into 12.5 mm depth intervals. It was composited for each treatment at each depth for total carbon analysis using the Walkley-Black method (Allison 1983). Soil was sieved to <250 um and 0.5 grams was added to a 500 ml flask. Following this, 20 ml of 1.0 N $K_2Cr_2O_7$ and 20 ml of concentrated H_2SO_4 was added. After 30 minutes, 200 ml of distilled H_2O was added. An indicator (0-phenanthroline-ferrons complex) was added and the mixture was titrated with 0.5 N $FeSO_4$ until the mixture changed color.

A paste of soil obtained from 1.25 cm depth intervals and water was prepared for electrical conductivity and pH measurements. The paste contained 10 g of soil mixed with 50 ml of de-ionized water. Electrical conductivity was also determined for each 1.25 cm interval with an Orion model 160 conductivity meter and pH was determined using the glass electrode method with a Fisher Scientific Accumet 950 pH/ion meter.

6.2.5. Statistical Analyses

Analyses of variance (ANOVA) tests were performed using JMP IN software (SAS Institute 1997). The least significant difference method or Fisher's LSD test was used to

compare treatments found to be significantly different in ANOVA at the 95% level of significance.

6.3. Results and Discussion

6.3.1. Aggregate Stability

In an attempt to find the best agronomic application rate of leonardite. six different application rates of P' were used (including a control 0 kg/ha rate). It was expected that there would exist an optimum application rate which would exhibit the lowest CH in CMD value. Below and above this rate. it was hypothesized that CMD would be higher.

CMD values obtained from wet (W) and dry (D) sieving are shown in Figures 6.1. through 6.6. CMD obtained from W sieving represents aggregates that have undergone slaking. For the D sieving. CMD represents aggregates that were not subjected to water dispersion - thus the value of D is greater than that of W. The change (CH) in CMD is the difference between that found from W and D sieving (also given in Table 6.1.) is a measure of aggregate stability to water. Thus the smaller the CH value. the lower the amount of slaking that had occurred and the stronger the stability of the aggregate to water.

ANOVA was evaluated on CMD values for W, D. and CH. The results indicate that there is no significant treatment effect on CMD of W, D and CH for all sampling times and depths.

Analysis of soil obtained immediately following application of P' and watering to

field capacity revealed that all treatments had no effect on aggregate stability. This was anticipated since it was hypothesized that a longer period of time for mixing and reaction between the P and the soil may have been required.

In an attempt to determine the time period needed, soil was sampled after 6 weeks of growth and immediately following wheat harvest at maturity. Analysis of the aggregate stability obtained at the 6 week interval revealed no treatment effect. In addition, there was no effect of P treatment on aggregated stability of soil sampled following harvest.
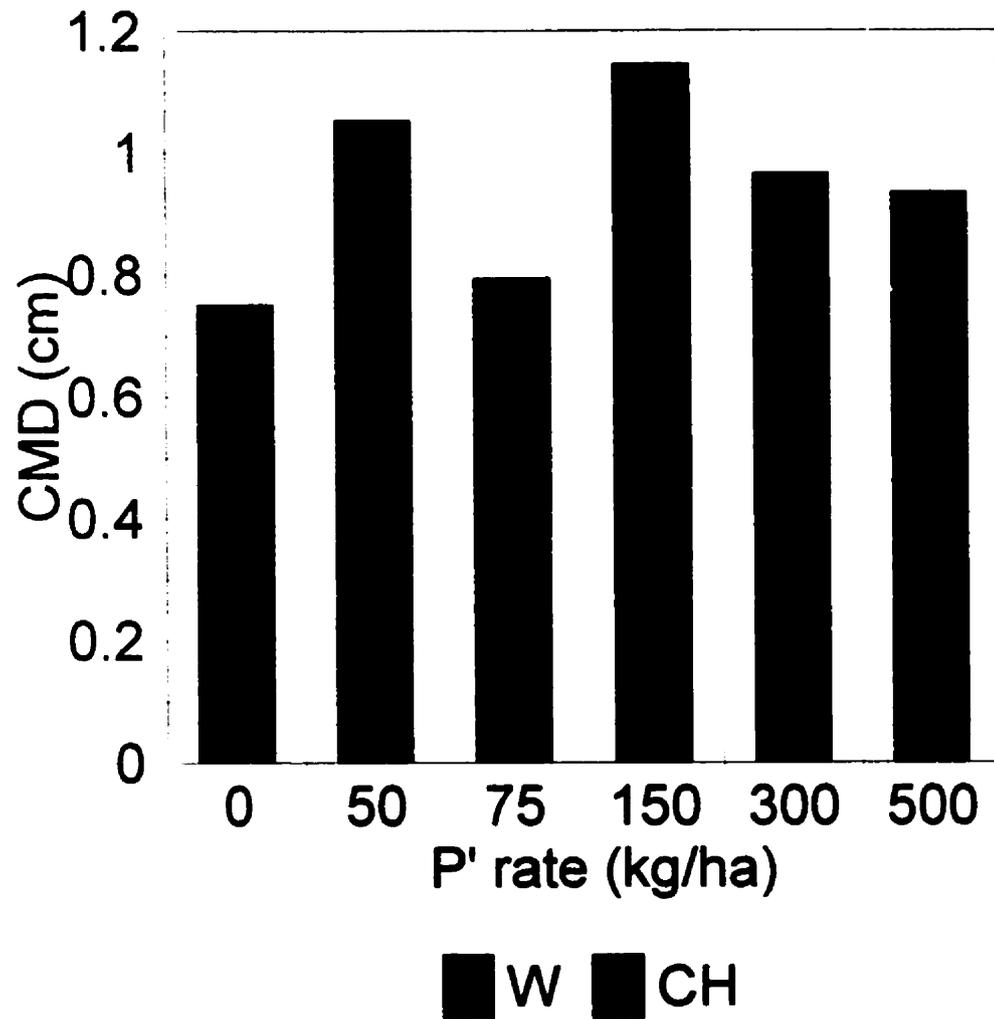
Figure 6.1. Characteristic Mean Diameters (CMD) of the 0-5 cm depth of Waitville clay loam obtained immediately following seeding (T 0), treated with different rates of a modified leonardite powder, 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

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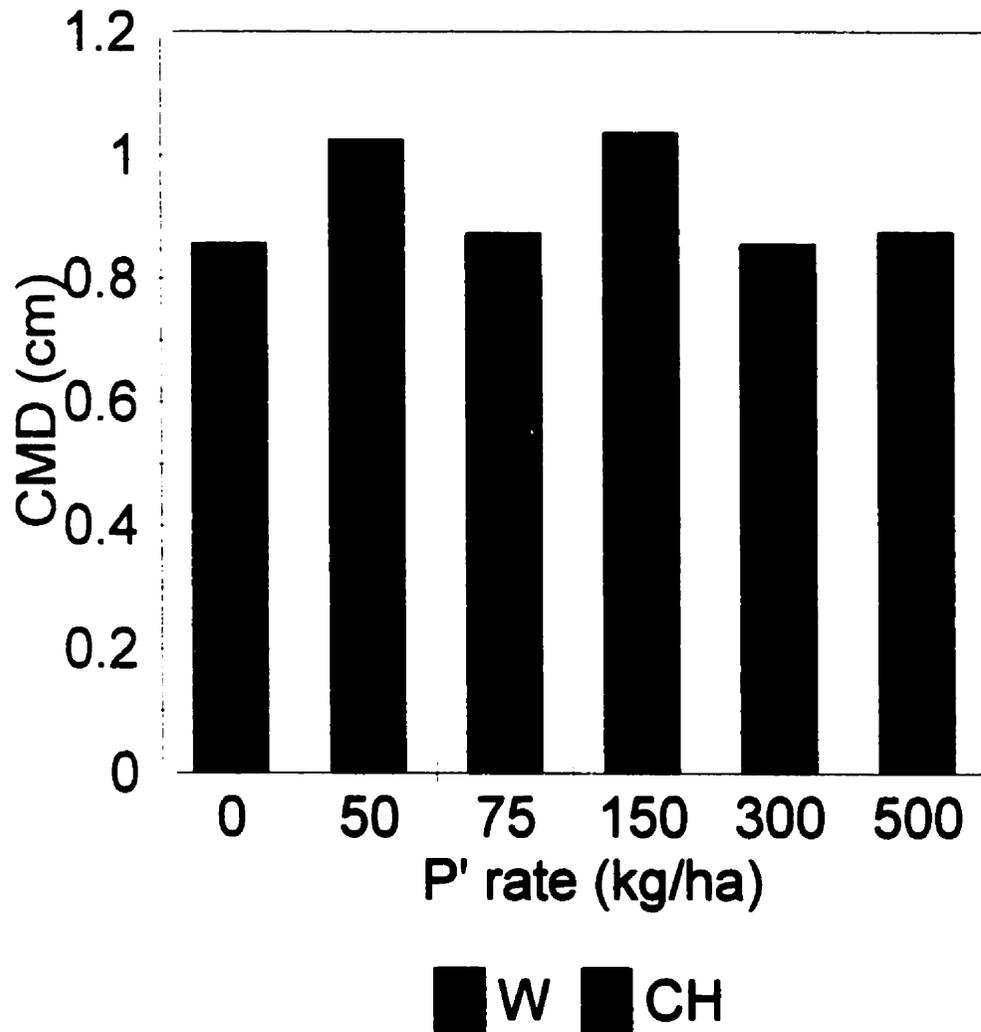
Figure 6.2. Characteristic Mean Diameters (CMD) of the 5-10 cm depth of Waitville clay loam obtained immediately following seeding (T 0), treated with different application rates of a modified leonardite powder (P'), 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

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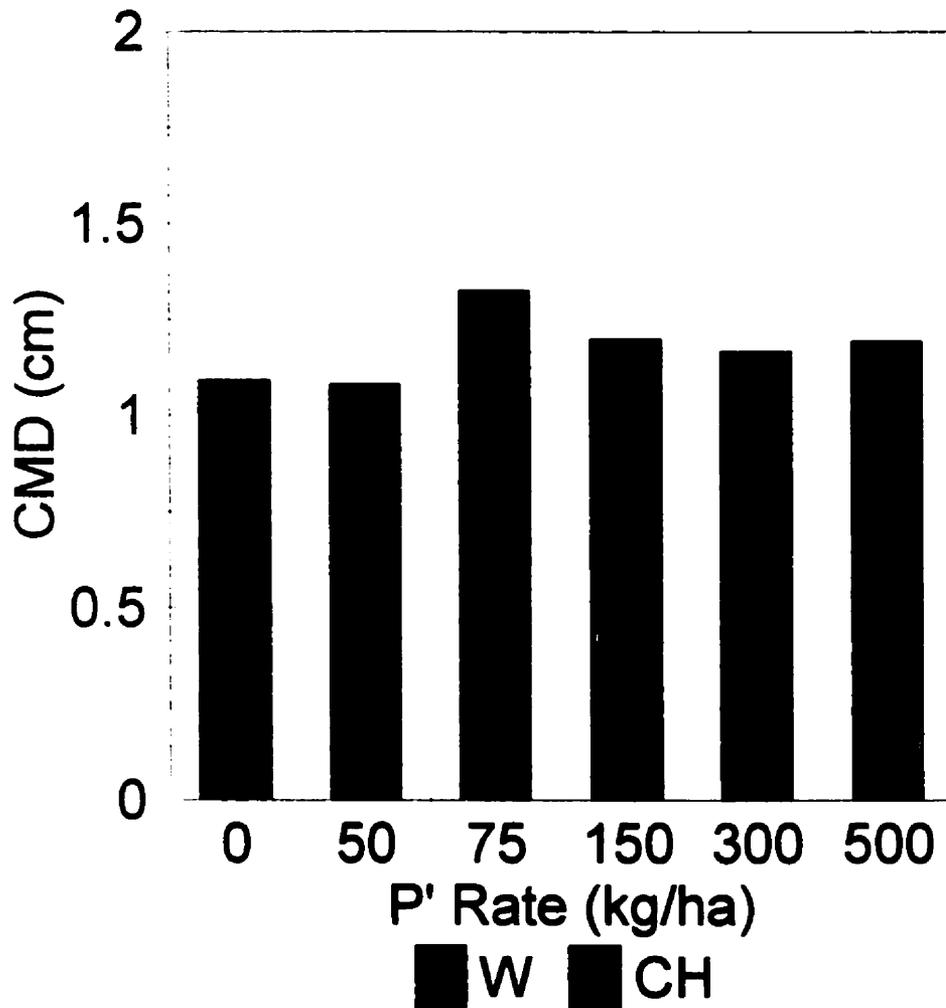
Figure 6.3. Characteristic Mean Diameters (CMD) of the 0-5 cm depth of Waitville clay loam obtained 6 weeks following seeding (T 6), treated with different application rates of a modified leonardite powder (P'), 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.
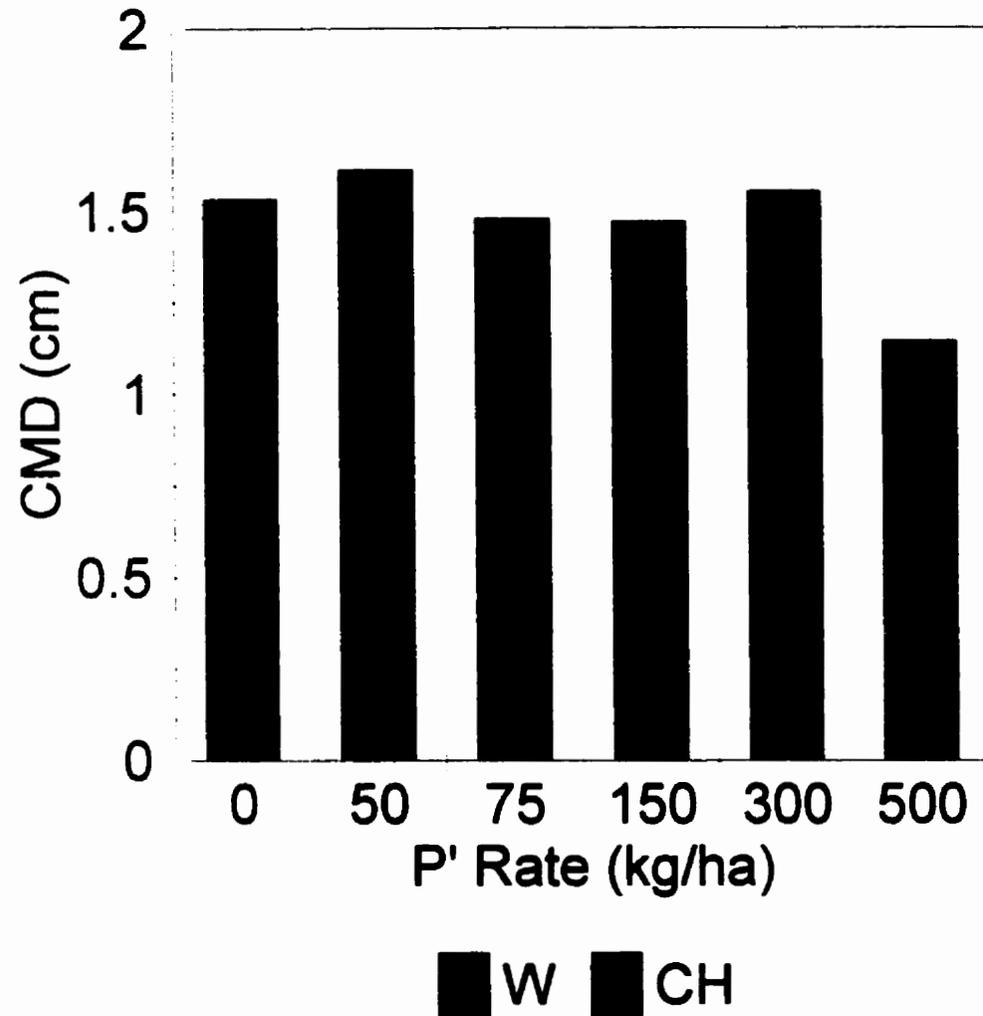
Figure 6.4. Characteristic Mean Diameters (CMD) of the 5-10 cm depth of Waitville
clay loam obtained 6 weeks following seeding (T 6), treated with different
application rates of a modified leonardite powder (P'), 1999. CMD values obtained
from wet sieving (W) and dry sieving are represented as the tops of the red and green
bars, respectively. The change (CH) in CMD is the difference between that found
from W and D sieving represented by the green area.

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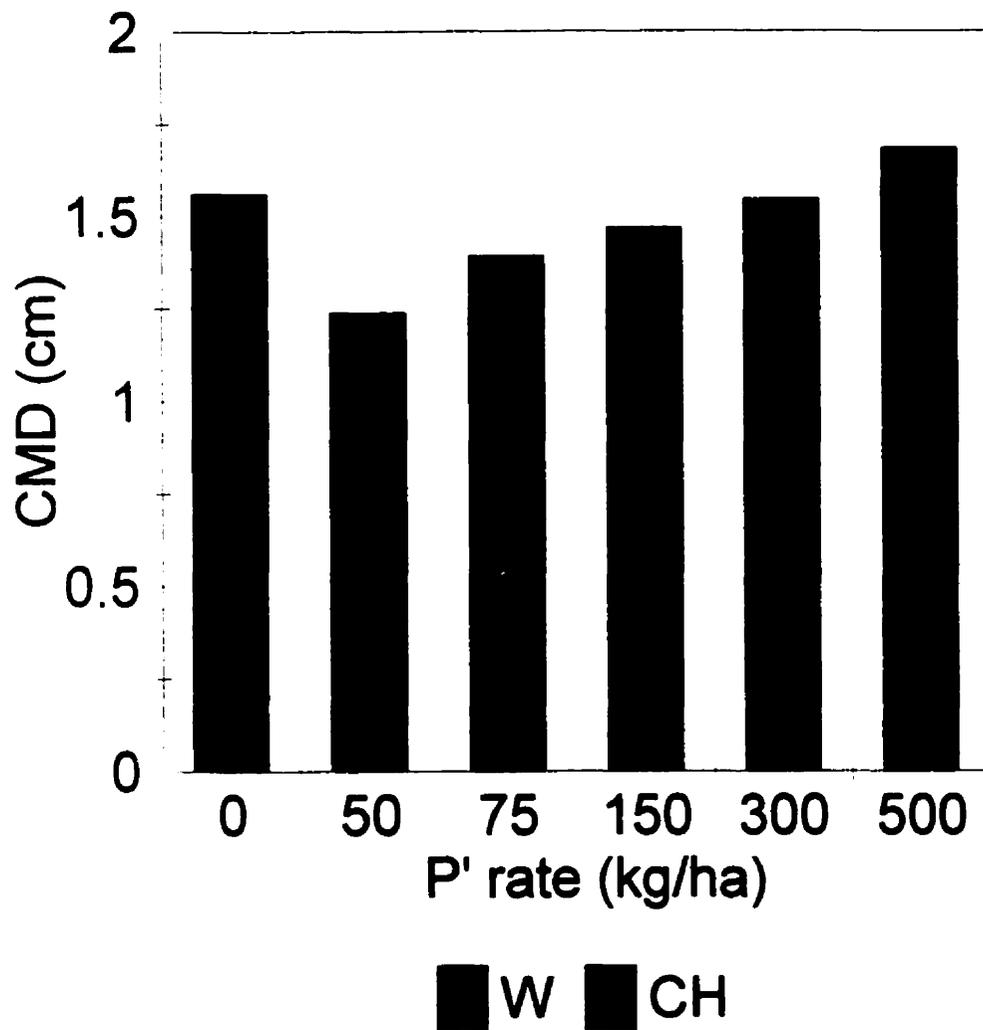
Figure 6.5. Characteristic Mean Diameters (CMD) of the 0-5 cm depth of Waitville clay loam obtained immediately following harvest (T H), treated with different application rates of a modified leonardite powder (P'), 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

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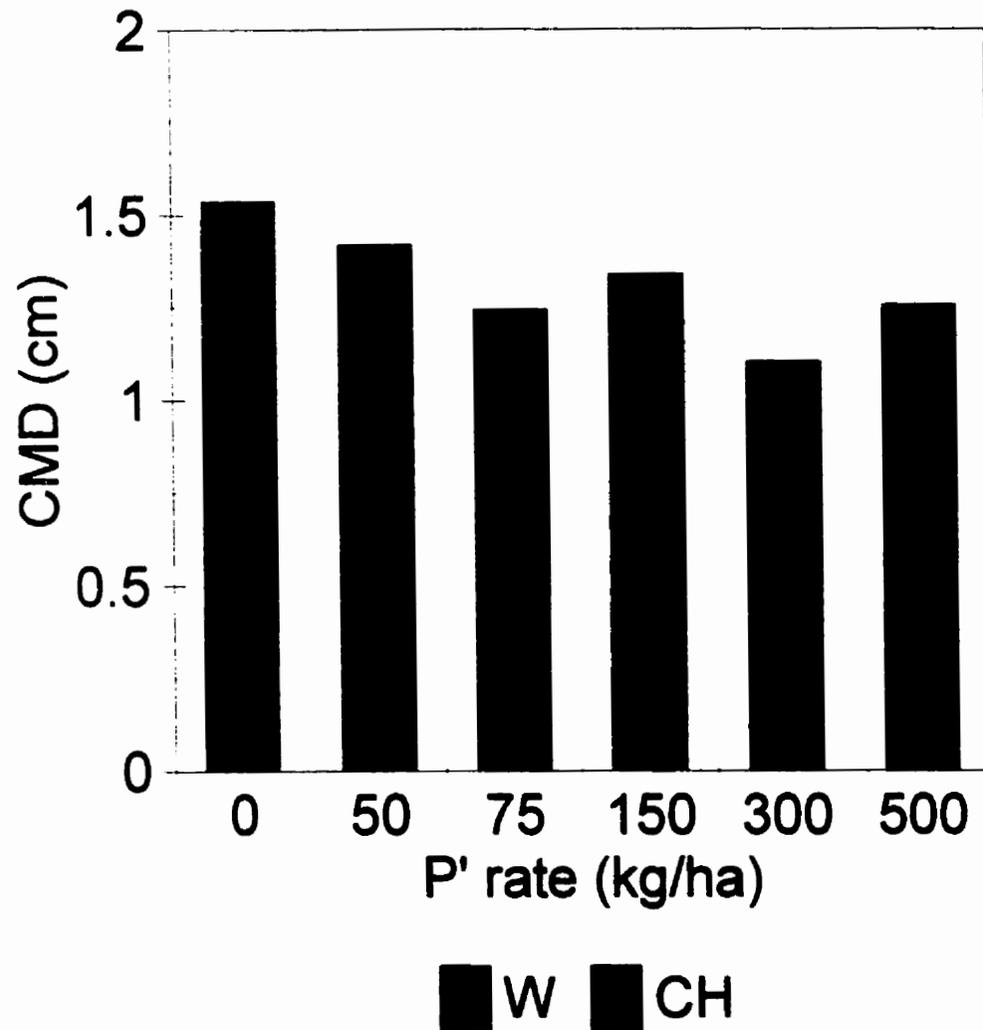
Figure 6.6. Characteristic Mean Diameters (CMD) of the 5-10 cm depth of Waitville clay loam obtained immediately following harvest (T H), treated with different application rates of a modified leonardite powder (P'), 1999. CMD values obtained from wet sieving (W) and dry sieving are represented as the tops of the red and green bars, respectively. The change (CH) in CMD is the difference between that found from W and D sieving represented by the green area.

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Table 6.1. Change in CMD[*] (mm) for 2 sampling depths of incubated Waitville CL sampled at 3 time periods following seeding: 0 weeks (T 0), 6 weeks (T 6wk) and maturity following harvest (T H).

Rate P' (kg/ha)	Soil depth (cm)	T 0	T 6 wk	T H
0	0-5	0.39 ± 0.07	0.69 ± 0.25	0.97 ± 0.32
0	5-10	0.47 ± 0.09	1.14 ± 0.33	1.09 ± 0.13
50	0-5	0.62 ± 0.39	0.63 ± 0.08	0.59 ± 0.35
50	5-10	0.61 ± 0.25	1.16 ± 0.18	1.01 ± 0.19
75	0-5	0.31 ± 0.05	0.92 ± 0.30	0.79 ± 0.30
75	5-10	0.42 ± 0.08	1.13 ± 0.26	0.76 ± 0.17
150	0-5	0.67 ± 0.24	0.75 ± 0.13	0.89 ± 0.07
150	5-10	0.66 ± 0.15	1.06 ± 0.11	0.94 ± 0.19
300	0-5	0.48 ± 0.07	0.67 ± 0.12	1.04 ± 0.10
300	5-10	0.48 ± 0.08	1.05 ± 0.63	0.69 ± 0.27
500	0-5	0.53 ± 0.23	0.70 ± 0.11	1.13 ± 0.08
500	5-10	0.52 ± 0.07	0.75 ± 0.13	0.70 ± 0.29

[*]Mean = standard deviation of 2 samples of each of 3 replicates per treatment.

A possible cause of the apparent non-effect on aggregate stability may have been related to the method of extraction. The HA from leonardite had been extracted using the base KOH. This may have contributed to ionic effects on cation bridging. Which could have influenced the aggregate stability. With the addition of the amendment, potassium, a monovalent ion with a low ionic potential was also added to the soil system. The affinity of humate for clay has been shown to decrease with ionic potential (Figure 1.1.. Theng 1979). Further, the type of bond involved depends on the valency of the cation on the exchange. Thus, addition of the potassium ion may have affected the adsorption of P'. and influenced the soil aggregation effect.

Following all studies. the possible reasons as to why the leonardite did not improve agronomic or structural conditions of the soil were discussed (Chapter 8). The

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main reason was though to be that there were not enough active functional groups on the leonardite surface.

6.2.2. Wheat Emergence, Biomass and Yield

Emergence counts are shown in Table 6.2. No treatment effect was found.

Table 6.2. Total wheat emergence counts of incubated clay measured at 2 time periods following seeding.

P' rate (kg/ha)	8 day	14 day
0	5.5 ± 3.5 *	10.2 ± 2.8
50	7.0 ± 1.6	9.2 ± 2.3
75	3.3 ± 2.1	8.3 ± 2.2
150	8.0 ± 3.3	10.6 ± 2.2
300	7.2 ± 4.1	10.8 ± 2.4
500	8.4 ± 2.7	11.6 ± 2.1

*Mean = standard deviation of 3 replicates per treatment.

There may be a number of reasons why there was no effect on seed emergence. The 14 day emergence counts indicated that the majority of seeds emerged, therefore, this study should have used a higher numbers of seeds per pot. There may have not been enough direct contact between the seeds and the P'. As well, if the P' had been adsorbed by the soil, it may have remained at the soil surface and never infiltrated to the seeding depth. Direct placement of P' with the seed may have given different results.

Table 6.3 shows 6 week biomass values for AC Barrie at different rates of P' amendment. There was no significant rate effect.

Table 6.3. Biomass/pot of AC Barrie wheat grown for 6 weeks in incubated Waitville clay loam soil amended with given rates of P'.

P' Rate (kg/ha)	Biomass/pot (g)
0	2.67 ± 0.29[*]
50	3.17 ± 0.30
75	2.49 ± 1.03
150	2.62 ± 1.03
300	3.18 ± 0.34
500	2.37 ± 1.07

[*]Mean = standard deviation of 3 replicates per treatment.

Results for biomass (B), grain mass (G) and harvest index (HI) are shown in Figure 6.7. The wheat was grown to maturity and harvested November 26, 1999. Actual values for B and G are given in Table 6.4. Grain yield was quite low. This was probably due to the low temperatures maintained in the incubation chamber. Biomass variance was very high. ANOVA ($\propto$=0.05) was performed on B, G, and HI. In all cases, there was no significant difference between the given rates of P'.

Reasons for the non-response of wheat biomass to the treatments may be similar to those related to emergence; there may not have been enough contact between the leonardite and the crop.

As well a positive growth response to HA treatment may only be found in a soil low in organic matter or nutrient solution (Clapp *et al.* 1998 and references cited therein). As the Waitville CL used in this experiment contained both organic matter and fertilizer, there may have been a less dramatic effect seen than would be in a lower fertility soil.
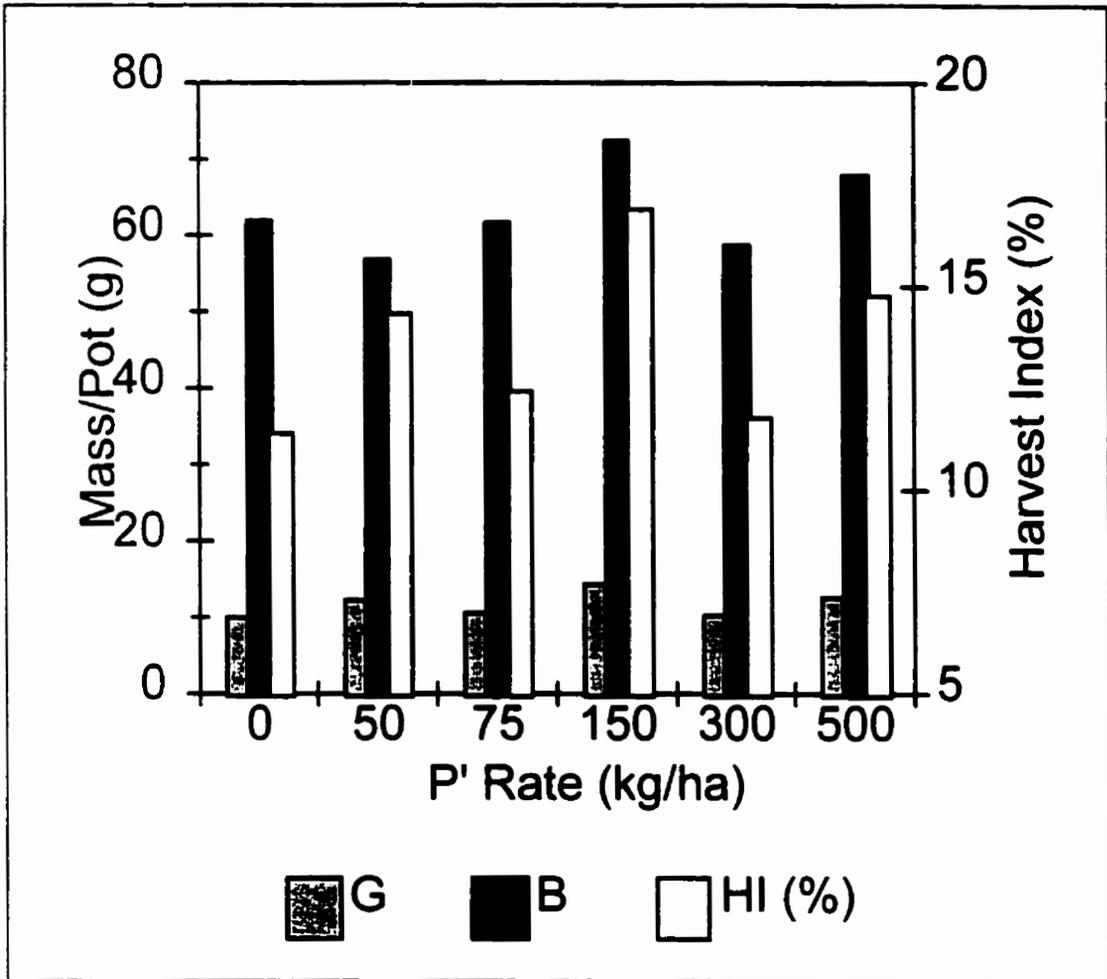
Figure 6.7. Effect of modified leonardite powder (P') application rate on wheat harvest
index % (HI) and mass (g) of total wheat biomass (B), and grain (G) incubated in
Waitville clay loam, 1999.

Table 6.4. Biomass and grain mass of wheat incubated with soil amended with various rates of modified leonardite powder (P').

P' Rate (kg/ha)	Biomass/pot (g)[*]	Grain mass/pot (g)[*]
0	61.9 ± 36.1	11.4 ± 2.5
50	56.9 ± 39.8	14.3 ± 3.7
75	61.7 ± 33.8	12.4 ± 3.8
150	72.5 ± 25.1	16.9 ± 1.4
300	58.8 ± 4.2	11.8 ± 7.3
500	68.0 ± 27.7	14.8 ± 3.3

[*]Average and standard deviation of 3 replicates per P' rate.

6.3.3. Organic Matter, Electrical Conductivity and pH Measurements

Table 6.5. shows the effect of different P' rates on soil organic carbon (OC) %, electrical conductivity (EC) and pH.

The results show that increasing P' rates have a positive effect on measured OC%. The control had an OC % value significantly lower than all treatments. Application rates of 50 and 75 kg/ha were statistically the same (α=0.05) and higher than the control. P' applied at rates of 75, 150 and 300 kg/ha were higher than the 50 kg/ha application rate, and all 3 were considered not to be statistically different. The 150.300 and 500 rates had a higher OC % than the lower rates of applied P'. These three rates were found to have a statistically similar effect on soil OC%. It appears that by adding P' it was possible to increase the overall OC content of the soil.

The P' added was in the form of a salt. Electrical conductivity was seen as a useful measurement of the salt content of the soil. There was a depth effect where EC was significantly higher in the top 1.2 cm of the soil compared to all other depths (α=0.05).

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This could be due to a number of reasons. The amendment was added to the surface of the soil, and if it was immobile, the EC would have been higher in the soil surface. Another cause could be evaporation. As the soil was near or at field capacity throughout the experiment, the roots would all have sufficient water for growth. Therefore there would be a great deal of evaporation, and as a result, salt would be transferred to and deposited at the soil surface.

As EC in the top 12.5 mm of the soil was approximately 3 times that of all other depths, the 0-12.5 cm depth was removed from statistical analysis. Table 6.5. shows that all rates of P' added caused no significant change in EC measured compared to that of the control. This showed that the addition of P' did not significantly change the total salt content of the soil.

The pH value of the 500 P' application rate was statistically higher than all other application rates and the control. All other values were statistically similar. The reason for the high pH associated with the highest application rate is unclear. As the amendment had a lower pH value than the soil a reverse relationship was expected.

Table 6.5. Application rate effect of modified leonardite powder (P') on mean[¥] organic carbon (OC), electrical conductivity (EC) and pH.

P' Rate (kg/ha)	OC %	EC [£] (mS/cm)	pH
0	1.524 ± 0.09 a[‡]	112 ± 22 a	6.09 ± 0.59 a
50	1.696 ± 0.10 b	152 ± 21 a	5.82 ± 0.28 a
75	1.764 ± 0.11 bc	167 ± 27 a	5.91 ± 0.23 a
150	1.807 ± 0.10 cd	133 ± 22 a	5.89 ± 0.19 a
300	1.780 ± 0.06 bcd	142 ± 22 a	5.80 ± 0.24 a
500	1.875 ● 0.14 d	100 ± 12 a	6.83 ● 0.54 b

¥ Mean of 10 samples composited at 0.5 inch intervals
‡ Means followed by the same letter within columns were not different (p,0.05)
£ 0-0.5 cm depth was excluded from analysis as it was a confounding variable

6.4. Conclusion

Soluble leonardite powder (P') had no significant effect on soil aggregate stability, wheat emergence, biomass or yield. Some chemical soil properties were changed. Organic carbon % was increased with increasing application of P'. Soil pH was unchanged as a result of P' addition in all treatments except in the 500 kg/ha rate. At this rate, application of P' increased soil pH. EC was unaffected at all P' application rates.

While physical and biological properties measured show that there was no treatment effect, the chemical properties show the amendment did change the soil. The increase of OC as a direct result of P' addition is of special interest.

7. INFLUENCE OF A SOLUBLE POWDER FORM OF LEONARDITE ON WHEAT SHOOT AND ROOT DENSITY GROWN IN AN INCUBATION CHAMBER

Abstract

In an effort to determine if a soluble modified leonardite powder (P') could improve the performance of AC Barrie wheat, incubation studies were conducted in 1999. P' was added to the surface of Almasippi SL at the time of seeding and following 6 weeks of growth, wheat shoot and root biomass, were measured.

No treatment effects were found. Emergence counts following 8 and 14 days growth as well as crop biomass revealed that leonardite had no positive or negative effect on wheat.

7.1. Introduction

Organic compounds may have a number of effects on plant growth (Tan 1993). A
number of studies have found that humic substances influence germination and
performance in a variety of crops. Humates have been shown to influence crops in a
number of ways (Lobartini *et al.* 1991; Burdick 1965). Stimulatory effects of humic
substances (derived from a number of sources including leonardite) on watercress.
tobacco. and barley have been documented (Ayuso *et al.* 1996).

Humic acid has also been shown to influence root growth in various species (Mylonas
and Mccants 1980) including wheat (Malik and Azam 1984) and corn (Lobartini *et al.*
1992). The increase in root growth is often greater than the increase in shoot growth
(Clapp *et al.* 1998). A possible auxin activity of humates found in leonardite has been
hypothesized to be a reason for increased root growth and abundance (O 'Donnel 1973).
Others have argued that stimulated root growth was due to increased micronutrient
availablity (Clapp *et al.* 1998). It was hypothesized that the magnitude of any leonardite
treatment effect would be greater in the root system than in the above ground biomass.
Therefore. both the above- and below-ground biomass was assessed. Although previous
chapters have shown that there was no treatment effect on crop yield. it was hypothesized
that leonardite could stimulate root growth.

This stimulation may reflect on soil macroaggregation, which is mainly influenced by
temporary binding agents including root exudates and fungal hyphae (Tisdall and Oades

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1982).

Preliminary trials have suggested a possible positive effect of a leonardite-derived

product on early shoot and root development during germination (L. Cramer, 2000,

personal communication).[6] Those trials used leonardite from the same supplier as used in

the following study.

The objective of this study was to determine if a soluble modified leonardite powder

soil amendment could stimulate seed germination and root and shoot growth.

7.2. Materials And Method

7.2.1. Incubation Conditions

A total of 10 pots were arranged in a complete randomized design consisting of 5

replicates, with 1 application rate (75 kg/ha) and 1 control. Pots were filled by weight:

5.5 kg of soil was added to 10 cm radius pots (soil depth was approximately 20 cm). AC

Barrie wheat (*Triticum aestivum* L.) was seeded at a rate of 12 seeds per pot on June 5,

1999 and grown Almasippi sandy loam (SL) (taken from TW11 R8 SE 17) in an

incubation chamber. Almasippi SL was chosen for the study rather than Waitville CL

because it was felt that separation of the roots from the soil would easier in a coarse

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textured soil. A soluble form of leonardite was applied immediately following seeding. Leonardite was manually applied directly to the soil surface. No tilling or mixing of the soil followed leonardite application. Fertilizer was mixed in the top 10 cm of all pots with application rates of 75 kg/ha nitrogen (Urea), 40 kg/ha phosphorous (phosphoric acid) and 25 kg/ha potassium (potassium hydroxide).

Pots were incubated at 60 % humidity and had an 18 hour day length with daytime and nighttime air temperatures of 20°C and 13°C respectively. During incubation, soil was watered to field capacity weekly.

7.2.2. Emergence and Harvest Activities

Emergence counts were taken at 8 and 14 days following seeding, and after 14 days wheat was reduced to 3 plants/pot. Following 6 weeks of growth, plants were harvested, washed and separated into shoots and roots for dry weight determinations. Both were oven dried at 60°C for 5 days. Oven dry plant biomass, root density and shoot to root gravimetric ratio were determined.

7.2.3. Statistical Analysis

Analysis of variance tests were performed using JMP IN software (SAS Institute 1997) at the 95% level of significance.

7.3. Results And Discussion

Wheat emergence counts for this study are displayed in Table 7.1. As shown, the 75

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kg/ha P' application rate did not have an effect on emergence compared to that of the control. ANOVA (α=0.05) confirmed this. Total wheat emergence was very good. More seeds per pot should have been added to see if the leonardite would provide an advantage to the crop grown.

Table 7.1. Effect of P' soil amendment on 8 day and 13 day wheat emergence.

P' Rate (kg/ha)	8 day	13 day
0	11.0 ± 1.00	11.6 ± 0.89
75	10.8 ± 1.30	11.6 ± 0.55

Mean ± standard deviation of 5 samples per treatment.

The effects that HAs have on germination and root growth should translate to high crop productivity. Therefore, the total above ground biomass and grain yield were studied to determine if the leonardite may have provided an advantage to the wheat grown in the amended soil. Table 7.2. shows the effect of P' (75 kg/ha application rate) on shoot and root biomass. Compared to the control (0 kg/ha rate) there was no effect of P' on biomass.

Root biomass showed a high degree of experimental error. This was due to small tears and possible loss of the roots during washing. Further studies should be conducted on crops grown in water alone.

Following all studies, the possible reasons as to why the leonardite did not improve agronomic or structural conditions of the soil were discussed (Chapter 8). The main reason was though to be that there were not enough active functional groups on the leonardite surface.

Table 7.2. Effect of P' on 6 week wheat shoot and root biomass.

P' Rate (kg/ha)	shoot biomass (g)	root biomass (g)
0	0.37 ± 0.03	0.62 ± 0.23
75	0.36 ± 0.04	0.56 ± 0.18

Mean ± standard deviation of 5 samples per treatment.

7.4. Conclusion

There was no significant treatment effect on crop emergence, or shoot and root

biomass. Variance of root biomass was probably due to experimental error. Separation

of small roots from the soil was difficult, and its biomass may have been underestimated.

The soil chosen for the study contained organic matter which may have provided

enough HA to have masked any effect on wheat productivity. There may have been some

adsorption of the amendment to the soil surface which would limit the direct contact

between the wheat and P'.

8. GENERAL SUMMARY AND DISCUSSION

Numerous studies have shown the effect of soil humic substances (HS) on soil

flocculation. coagulation and structure (Greenland 1971; Theng 1979; Visser and Caillier

1988: Kay 1997: Brandsma *et al.* 1999). These substances contained active functional

groups which reacted with the soil. Unfortunately, based on the lack of change in soil

physical properties with leonardite. it appears that the HS found in the leonardite used in

the experiment were not of the same form of active HSs as used in the previous studies.

8.1. Design of Field Study

Two soils were chosen for the field study: a Waitville Clay Loam Dark Gray Luvisol

and a Osborne Clay Humic Rego Gleysol. By adding organic carbon in the form of

leonardite to the soil. it was postulated that degree of granulation and structural stability

would be improved. as other studies have found positive relationships between the

organic carbon content on the degree of roundness of aggregates (Dexter 1985) and

structural stability (Kay 1998). This would have promoted crop production as a granular

structure is provides for good tilth and plant growth (Brady 1990). The magnitude of the

impact of leonardite on soil physical properties was thought to be greater in the luvisolic

soil due to its low clay and organic carbon content.

The soils were amended with 3 forms of leonardite. The raw leonardite was sieved to

give a material <2 mm (M). ground into a fine powder (P) and a prepared as liquid

suspension which contains humic acid (H).

A variety of tests were performed on the soil collected from the field study in order to assess its structure. Soil HS have been found to act as flocculation agents (Theng 1979). Flocculation, in turn, affects soil structure (Tisdall and Oades 1982). The structure influences a number soil properties including strength (Taylor and Gardner 1963), soil crusting (Hillel 1982), density (Hassett and Banwart 1992), and pore size distribution (Kay 1996). The sensitivity of each test used to assess these properties was different (Hillel 1982). While one test showed an amendment effect, others did not (Brandsma *et al.* 1999). Therefore it was necessary to perform a number of different tests.

8.2. Discussion of Results of Field Study

Following the field study, it was hypothesized that the main reason that there were no treatment effects of leonardite on either soil structure or crop production was that the material was insoluble. The mobility of the amendment in the soil and ability to coat and adsorb to the surface of the soil particles and aggregates would have been low as a result.

8.3. Design of Incubation Study

An incubation study was conducted. In this study, due to time constraints, only one soil could be used to test one physical property. The degraded Waitville clay loam was used as it was felt that this soil would be more responsive to the amendment addition.

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The stability of aggregates influences pore size distribution, soil strength and other physical properties. Therefore, it was felt that the best test for a treatment effect would be aggregate stability to water using wet and dry sieving. The incubation study also assessed the impact on leonardite on wheat emergence, above- and below-ground biomass and yield. For this portion of the incubation study Almasippi sandy loam was used as separation of the roots from the soil would be less difficult in a coarse textured soil.

Leonardite was applied as a chemically modified soluble powder in the incubation experiments. In modifying the raw material it was postulated that the mobility would increase as a direct result of increasing its solubility in water.

8.4. Discussion of Results of Incubation Study

Chemical testing showed that leonardite contained organic carbon and humic acids (HAs), but the degree of bonding was unknown. However, as leonardite was derived from a coal mine, it was inferred that the material has been exposed to high temperature and pressure conditions for an extended period of time. This lead to the postulation that there was a high degree of bonding between the carbon groups within the leonardite structure and it appears that the surface functional groups (carboxylic and phenolic acid) were not present. Although Table B.3. indicates that these functional groups were present, the method of extraction could have cleaved the macromolecule and released these groups from the structure of the molecule. That is, it is was thought that the

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carboxylic and phenolic groups are present, but are tied up in the macromolecular structure such that they are unable to react with the soil. Based on these assumptions, the pH dependent surface groups were not released from the lignite structure. The surface groups are responsible for adsorption, flocculation and coagulation, so in their absence, no effect on soil structure was observed.

It has been suggested that humic acids adsorb to clays via a water bridge (Stevenson 1982). Polyvalent cations in the soil are commonly surrounded by a hydration sphere. The humic substances replace water around the hydration sphere. The degree of replacement is related to the amount of active functional groups on the surface of the humic substance. An increase in entropy caused by the water desorption leads to an increase in free energy of adsorption, thus the humic substance is adsorbed.

Soil humic acids act to promote soil aggregation. Soil particles may be brought together by flocculation, and/or coagulation (Theng 1979). As well, the organic matter acting may to stabilize macropores and thereby stabilize macroaggregates (Tisdall 1996). Flocculation refers to aggregation caused by polymer bridging while coagulation involves processes where the primarily van der Walls forces drive the reaction (La Mer 1964).

According to Stevenson (1982) the conformational shape of humic substances in solution vary due to the degree of protonation and dissociation of their functional groups. When there is an abundance of functional groups, including COOH and phenolic-OH, on the surface of a polymer, it has the potential to have a high charge density. Under these conditions, the pH and ionic strength of the soil environment will greatly affect the shape of the molecule (Senesi et al. 1977). The ionic strength of the solution affects the activity

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of the molecule and the pka in relation to the pH of the environment effects the degree of dissociation of the humic substance. When the ionic strength of the soil solution is low, the polymers will be more dissociated and adopt a more stretched-out conformation, whereas at very high ionic strengths the chains begin to recoil (Theng 1979). The extension of the length of the molecule promotes particle bridging. When they are able to span the inter-particle distance this would lead to flocculation. Uncharged linear polymers are usually arranged in a spherical random coil and do not promote flocculation.

Soil humic substances contain pH dependent surface active functional groups that are play a role in the creation and stabilization of soil aggregates. It is postulated that the reason that leonardite did not promote soil aggregation was that there were not enough active functional groups on the surface to the macromolecular structure. It was found that the mere addition of organic carbon does not necessarily lead to an increase in aggregate stability. Rather, "it is the disposition rather than the type or amount of organic matter which is important" (Tisdall and Oades 1987).

The lack of crop response could also be due to a lack of surface active functional groups. The reasons postulated for the effect of humic substances on crops were due to their ability to provide a hormonal response (O'Donnel 1973), affect the solubility of micro-nutrients (Clapp et al. 1998). All experiments assessing the impact of leonardite on crop productivity showed there to be no treatment effect. It appears that the leonardite did not react with the root or the soil.

In summary, the experiments showed that there was no impact of leonardite on either

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the soil physical or agronomic properties. This was probably due to the lack of active functional groups.

8.5. Recommendations

A number of considerations should be made in future research of the impact of leonardite on soil physical properties. One of the major problems encountered in the study was that the amendment was applied according to possible agronomic application rates. These rates may not correspond with application rates required to condition the soil.

As well, economic considerations should be made. The application rate must be affordable such that the increase in the yield curve should be greater than the cost of purchasing and applying the soil amendment. The increase in the yield curve with application of the amendment would be higher in a degraded soil than a high quality soil. Future research should consider whether the physio-chemical properties of the soil can be altered in only one or two seasons. A longer study period may be required.

9. CONTRIBUTION TO KNOWLEDGE

The research provides a good understanding of the way in which organic matter influences soil physical properties. It shows that the mere physical addition of organic carbon to the soil does not necessarily translate to an improvement in soil physical properties. The interaction of humic substance with the soil is highly dependent on the composition of the humic substances.

While leonardite did not improve soil physical properties, it did not have any negative impact on the soils' physical and chemical properties or wheat growth in amended soils. However, leonardite did increase the organic carbon content of the soil. It appears that if one were able to chemically alter leonardite by cleaving the macromolecular structure, pH dependent functional groups may become exposed. These functional groups have a great deal of influence on soil physical properties.

A number of techniques were used to assess soil physical properties. It was found that soil physical properties are heterogenous and a large number of samples and various tests should be conducted to assess the impact of organic amendments on soil structure.

A combination of wet and dry sieving is a very good method to use to assess aggregate stability to water. By comparing the characteristic mean diameter of dry soil aggregates to those that have undergone wet sieving, it was possible to evaluate the degree of slaking or breakdown of aggregates under rapid wetting in the field.

Previous research has shown that the frequency distribution of logarithm of aggregate size follows a normal distribution. Thus, it was suggested that a plot of percent logarithm

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of oversize against a probability scale should produce a straight line (Gardner 1956). The soil was characterized by calculating the log mean geometric diameter or characteristic mean diameter. This was the diameter above which 50 percent of the aggregates fall. Tests conducted in this study confirmed this. The r^2 values of the slope of the line was very high (>95%) suggesting that this was a very good method to characterize the size of aggregates found in Manitoba soils. Further, in using this method, error due to overestimation of one aggregate size would be minimized compared to the mean weight diameter estimation. The mean weight diameter is calculated by obtaining the sum of the selected aggregate sizes multiplied by their weight and dividing the sum by the total soil mass.

Although the results of the study are negative, they are significant in demonstrating the probable ineffectiveness of raw leonardite soil amendments.

10. CONCLUSIONS

The effect of leonardite on soil physical and agronomic properties were assessed. There was no treatment response on any of the following physical properties: penetration resistance, soil moisture, bulk density, particle density, modulus of rupture, pore size distribution, aggregate stability. No treatment response was observed on the following agronomic properties of wheat: emergence, root mass, biomass and yield. However, tests showed that leonardite could supply organic carbon to the soil. The results show that the organic carbon contained in the leonardite was no the same as that contained in soil. The humic substances contained in leonardite are not humus.

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12. APPENDICES

11. APPENDICES

A. Description of Soil and Climate

Table A.1. Selected physical and chemical characteristics of Ap horizons of soils.

Location/ Soil Type	Depth (cm)	% S	% Si	% C	% Organic C	% Organic N
SW 2-17-17W Osborne C	0-12	6	37	57	3.2	0.3
NW 6-5-2W Waitville CL	0-14	26	39	35	2.1	0.2
SE 17-8-11W Almasippi SL		79	5	16		

Table A.2. Exchangeable Cations (milli equivalents) and Base Saturation of Waitville Soil (Ehrlich, Pratt and Poyser 1956).

Depth	Ca	Mg	K	Na	H	Total	Base saturation (%)
0-13	4.57	.90	43	-	0.98	6.88	85.76

Table A.3. Selected landscape properties of two soils.

Location/ Soil Type	Topography	Drainage	Parent Material
SW 2-17-17W Osborne C	Depressional	Poorly drained	fine textured lacustrine clay
NW 6-5-2W Waitville CL	Hummocky to rolling	Well drained	Boulder Till

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Table A.4. Climatic conditions of nearby[*] weatherstations including growing degree days above 5C (D) and accumulated growing season precipitation for wheat (A) (Ash 1991).

Weatherstation /condition		Mean	Standard deviation	Probability 10%	Probability 25%
Morris	D	175.92	64.96	92.46	132.08
	A	1777.68	168.70	1560.91	1663.81
Neepawa	D	212.38	81.58	107.55	157.32
	A	1590.98	110.79	1448.62	1516.20

[*]Osborne C is located near Morris and Waitville CL is located near Neepawa Manitoba.

Table A.5. Mineralogical composition[*] of clay fractions of the 0-13 cm depth of Waitville CL (Modified from Ehrlich. Pratt and Poyser 1956).

Mineral	Particle Size Fraction (μm)		
	0.2 - 2.0	0.1 - 0.2	<0.1
Montmorillonite	1	5	6
Hydromica	-	1	2
Vermiculite	-	-	1
Muscovite	2	1	1
Illite	3	2	1
Feldspars	1	2	2
Calcite	-	1	-
Quartz	6	1	<1
Kaolinite Group	1	-	-
Chlorites	-	-	1
Sepiolite	-	-	-
Talc	-	-	-
Others	Bayerite, Boehmite	Goethite, Bayerite	Hematite

[*] Results are in the scale of 1-20: 1 = 1-5 %: 2 = 6-10 % etc.

Table A.6. Profile description of Osborne C field plot (Goh 1998. personal
communication).

Date: May 21. 1998. 12:00 noon. clear. sunny. sun behind
Location: SW 2-17-17W
Soil name: Osborne clay
Topography: Flat
P.M: Alluvial clay
Described by: T.B. Goh
Condition of field: 20% cover by trash of previous crops
                       Volunteer weeds 15%

Ap 0-2.5 wavy to 8"   7.5 YR 2/1
                           Fine moderate to strong granular. Feel fine roots. Clay.
                           Occasional tonunges of a horizon probably in filling cracks
                           extending to 8". Indistinct wavy boundary to the transition
                           horizon below (AC). Hard to very hard when dry. Friable
                           when moist.
ACg 2.5-8.5 wavy to 10"   2.5 Y 3/1
                           At the boundary between the Ap and AC the structure is
                           very weak fine platy. The bulk of the AC horizon has a
                           weak fine to medium blocky structure. Clay skins are
                           common as are fine veins of the darker AC in this horizon.
                           Very fine roots are common (-). Clay to heavy clay. Sticky
                           when wet. Friable when moist. Hard when dry. Iron
                           mottles (freckles) are common and with diffuse boundaries.
                           No effervescence with HCl. Sharp wavy boundary to Ckg.
Ckg 8-10" to ÷   5 Y 3/1
                           Massive. Friable when moist. Sticky when wet. Heavy
                           clay. Very hard when dry. Limestone pebbles are rare.
                           ranging in size from 0.25 to 0.5" in diameter. Vigorous
                           effervescence.

Table A.7. Soil profile of a characteristic (uncultivated) Waitville clay loam (Modified from Ehrlich. Pratt and Poyser 1956).

LFH	2-0"	Reddish brown Partially decomposed leaf mat: slightly acid in reaction
A₁	very thin to absent	Very dark grey Mucky clay loam: finely granular: friable: neutral to slightly acid:
A₂	0-2 wavy to 4"	Pale brown Pale brown sandy loam to loam: weakly developed fine platy to crumb structure: moderately hard: coarse rock fragments are largely decomposed: slightly acid in reaction. Grades sharply into: -
B₁	2-4 to 6-10"	Brown Clay loam: fine to medium block aggregates: very hard when dry, moderately plastic when moist: aggregates have greyish coating near top and organic staining and darker color occurs with depth: slightly acid in reaction. Blends gradually into: -
B₂	6-10 to 13-15"	Dark Brown Clay loam: slightly stained with organic materials: fragmental structure; firm: contains some free lime carbonate. Grades sharply into: -
B₃	13-15 to 17-20"	Brown Clay loam: slightly stained with organic materials: fragmental structure: firm; contains some free lime carbonate. Grades sharply into: -
Cca	variable thickness	Light grey Calcium carbonate; clay loam; pseudo-crumb structure: friable when moist. weakly cemented when dry; contains some powdered iron concretions. Fades into: -
C		Light greyish brown Clay loam glacial till; pseudo-fragmental; hard; strongly calcareous: contains powdered iron concretions and glacial rock fragments.

Osborne Rego Humic Gleysol
Sample for Initial Characterization: taken within a radius of 1.5 m around sample point.
Sampled whole Ap, Followed by 10 cm of AC or C.

Figure A.1. Osborne C field plot design. Sub-plots were 3.2 by 5 meters. Leonardite treatment included a mine material < 2 mm (M), a finely ground powder (P), and a liquid humic acid extract (H). A control (C) subplot was also established. The depth of the Ap horizon (Table A.6.) was determined in subplots labeled 1-16.

Waitville Gray Luvisol
Sample for Initial Characterization: taken within a radius of 1.5 m around sample point.
Sampled whole Ap, Followed by 10 cm of AB or B in some instances.
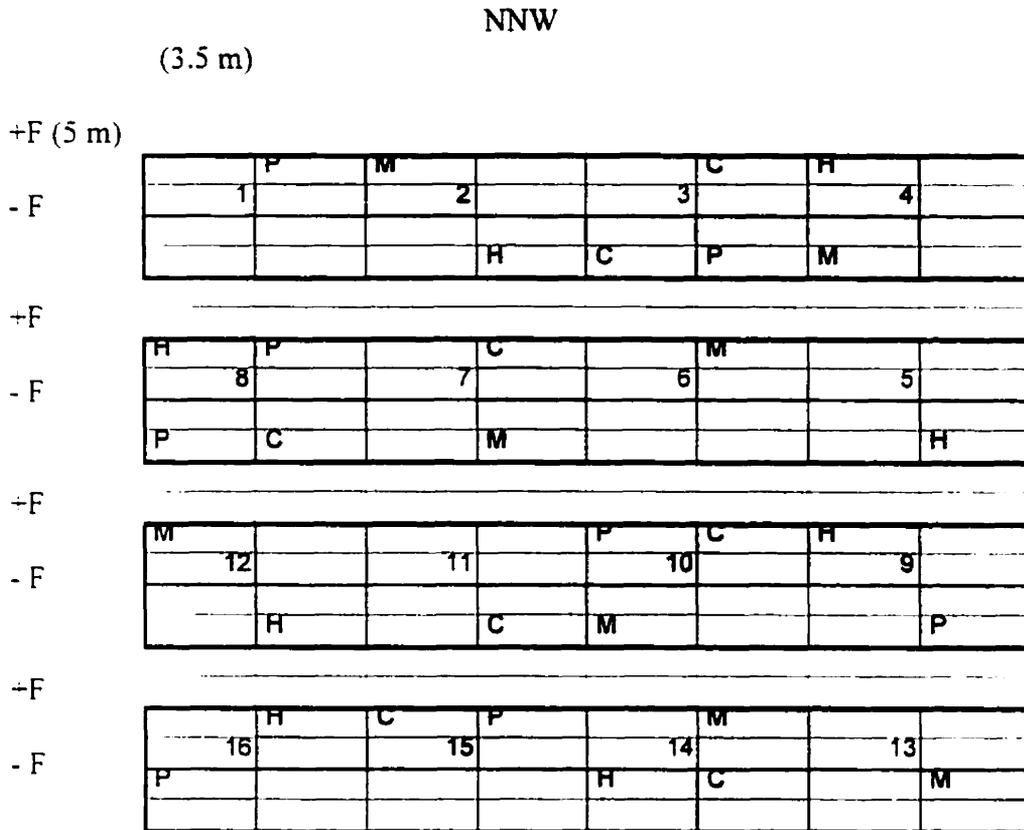
NNW

(3.5 m)

+F (5 m)

- F

Figure A.2. Waitville CL field plot design. Sub-plots were 3.2 by 5 meters. Leonardite treatment included a mine material < 2 mm (M), a finely ground powder (P), and a liquid humic acid extract (H). A control (C) subplot was also established. The depth of the Ap horizon (Table A.6.) was determined in subplots labeled 1-16.

Table A.8. Depth[*] of Ap horizons of Osborne C and Waitville CL at sampling points shown in Figures A.3. and A.4.. respectively.

Replicate	Sampling Point	Ap Horizon Depth (cm)	
		Osborne C	Waitville CL
1	1	7.5	12
	2	6	12.3
	3	7.2	12.7
	4	4.8	13
2	5	9.5	13
	6	6.8	13.7
	7	6.3	13.3
	8	7.3	14.3
3	9	8.3	13.8
	10	9	15.9
	11	10.3	15.2
	12	10.5	14.4
4	13	11	14.7
	14	11.7	18
	15	10.3	18.3
	16	12.5	17.8

[*] Average of 3 cores per sampling point.

Table A.9. Effect of leonardite on emergence of canola (plants/m^2)(S. Dilk. 2000[1]).

Treatment	Soil Type and Year			
	Luvisol		Chernozem	
	1998	1999	1998	1999
Control	117	72	98	97
50kg ha^{-1} P	126	66	89	117
150kg ha^{-1} P	128	74	76	94
500kg ha^{-1} P	119	72	98	111
1000kg ha^{-1} P	126	72	74	133
5000kg ha^{-1} P	115	67	96	115
50kg ha^{-1} M	135	66	87	155
150kg ha^{-1} M	119	60	90	118
500kg ha^{-1} M	122	66	90	131
1000kg ha^{-1} M	141	56	108	115
5000kg ha^{-1} M	124	74	103	90
50 L ha^{-1}	128	73	94	159
100 L ha^{-1}	111	74	103	131
300 L ha^{-1}	146	80	111	105

Type of Leonardite, P = Powder, M = Mine Material, L = Liquid

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[1]Personal communication with S. Dilk, graduate student, University of Manitoba.

B. Description of Leonardite Forms

Leonardite used in the proceeding chapters was provided by Luscar Ltd. Selected physical and chemical characteristics of the following products have been included:
L- 11 HA LIQUID (H)
L- 31 DRY POWDER (P)
L- 48 MODIFIED SOLUBLE POWDER (P')

Table B.1. Selected chemical and physical characteristics[‡] of leonardite materials used.

| Leonardite Form | OC[φ]% (w/w) | pH[γ] | | | Bulk Density[ς] (g/cm³) | Moisture Content[δ] % (w/w) |
		deionized H₂0	0.01M CaCl₂	alkaline extract		
H	na	na	na	8.0	na	na
P	58.5	5.8	4.2	3.6	0.68	18.0

[‡]Laboratory examination preformed by Dr. S. M. Lee.
[γ] Glass electrode method (10g sample / 50ml deionized water or 0.01M CaCl₂)
[ς] Bulk Density (mass of oven-dry leonardite/total vol soil)
[δ] Gravitation method (105C oven, 24 hr)
[φ] Walkley-Black method

Table B.2. Humic acid (HA) and fulvic acid (FA) contents[‡]: SSSA method (solid sample 100 g or liquid sample 500 ml)

Sample	Yield (HA + Ash)	Ash in HA	Yield (HA only)*	Yield (FA + Ash)	FA (with ash) in sample
H	42.3	8.0	38.9	5.5	5.5
P	27.2	9.7	24.6	3.0	3.0

* Yield (HA only) = Yield (HA + Ash) - (Yield (HA + Ash)(Ash in HA)
[‡]Laboratory examination preformed by Dr. S. M. Lee.

Table B.3. Characteristics[‡] of Humic acid (HA). (All measurements given in meq/g HA).

Sample	Total acidity	Carboxyl (COOH)	Phenolic OH
H	7.7	3.6	4.1
P	8.3	4.0	4.3

[‡]Laboratory examination preformed by Dr. S. M. Lee.

The liquid leonardite material was chemically altered to increase it's solubility in water. The method for this is as follows[2]:

Formulation

Water	80 L
Potassium Hydroxide (90% Purity. Flakes)	0.499 kg
Leonardite (L31/Mont)	8.0 kg
Potassium sulphate (99.8 % purity)(adj)	1.395 kg

Procedure

1. Add water to mixer
2. Add KOH to tank and mix for 30 minutes
3. Sample solution for pH (should be 13.0)
4. Add leonardite in four equal quantities. 10 minutes apart
5. Mix solution for 1 hour
6. Add K_2SO_4 in four equal qualitites. 10 minutes apart
7. Mix solution for 4 hours
8. Pump solution to floor for drying
9. Let solution dry for a minimum of 90 hours
10. Check moisture content as a liquid and the powder after drying

Table B.4. Selected properties[*] of P'

HA content	pH	EC (dS/m)	Humic Acid	Potassium	Sulphur	Solubility in water
9.45 %	5.9	17.0	9.5%	6.0%	2.8%	S (above 50%)

[*]Laboratory examination preformed by Dr. S. M. Lee.

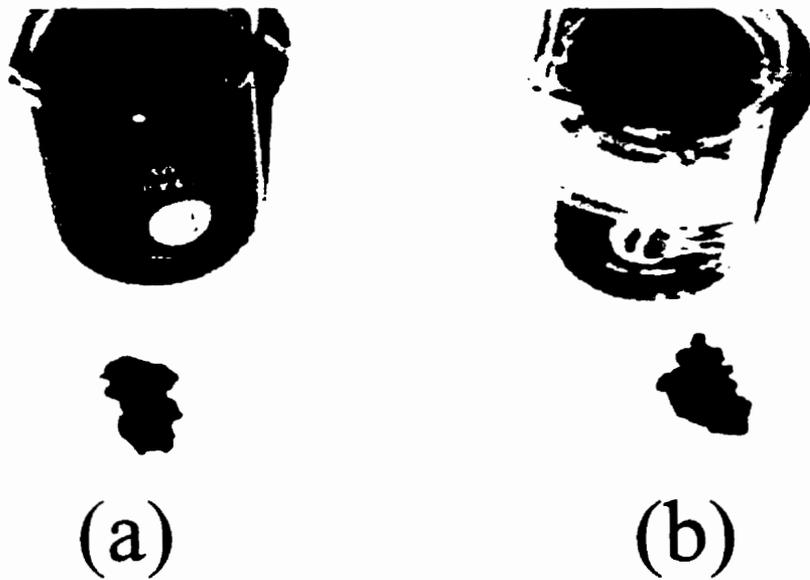[2]Personal communication with Dr. S.M. Lee (1999).

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Figure B.1. Solubility of the modified leonardite powder (a) and the unaltered leonardite powder (b) in water.