

**THE ROLE OF NATURAL AND ANTHROPOGENIC FACTORS IN
SOIL ORGANIC MATTER QUALITY AND AGGREGATE STABILITY
OF COOL TEMPERATE SOILS**

BY

BRIAN H. WIEBE

A Thesis
Submitted to the Faculty of Graduate Studies
in Partial Fulfilment of the Requirements
for the Degree of

DOCTOR OF PHILOSOPHY

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University of Manitoba
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ABSTRACT

Wiebe, Brian Henry. Ph.D., The University of Manitoba, October 2002. The role of natural and anthropogenic factors in soil organic matter quality and aggregate stability of cool temperate soils. Major Professor; Dr. Tee Boon Goh. The breakdown of soil aggregates and subsequent dispersion of clay can reduce soil porosity resulting in restricted root growth, surface crusting, reduced infiltration rates, increased runoff, and increased soil erosion. This reduction in soil quality would eventually lead to decreased productivity. Soil aggregates from zero and conventional tillage treatments of an Osborne clay (Rego Humic Gleysol) and a Fortier silty clay (Gleyed Cumulic Regosol) as well as from three different slope positions along a native prairie catena (Black Chernozems) were suspended in water and subjected to varying intensities of ultrasonic energy. Soil organic C, total carbohydrate, diethyl ether extractable lipids (DEE-EL), chloroform extractable lipids (CHCl₃-EL), clay content, cation exchange capacity (CEC), and exchangeable Ca, Mg, Na, and K were determined. Within each site, differences in measured soil organic matter constituents between treatments or slope positions were small and generally not significant. Total soil organic C content was a poor predictor of clay dispersibility in all the soils studied. For the Fortier silty clay, multiple linear regression analyses indicated that clay dispersed more easily as exchangeable Mg relative to Ca increased and that CHCl₃-EL enhanced clay dispersibility, whereas DEE-EL reduced clay dispersibility. The regression models improved when the tillage treatments were considered separately indicating differences in the binding mechanisms between clay

particles under zero and conventional tillage. Different parameters were dominant at the different levels of applied energy indicating that the mechanisms responsible for weak bonds are different from those responsible for stronger bonds. Multiple linear regression analyses models for clay dispersion of the native prairie soils were much more consistent across energy levels than were those for the Fortier silty clay indicating less variation in bonding mechanisms in the prairie soils. The models showed that exchangeable Mg and CHCl_3 -EL enhanced clay dispersibility and that DEE-EL reduced clay dispersibility. CEC, most likely due to organic exchange sites, was also important in reducing clay dispersibility.

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I thank God for the many mysteries of nature that are worth studying and for the opportunity and ability to spend the last 10 years delving into some of them or as my nieces and nephews might say “playing in the dirt”.

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FOREWORD

This thesis has been written in manuscript style and contains two manuscripts. The manuscript entitled The effects of zero tillage management on soil organic matter composition and clay dispersion from soil aggregates of a Fortier silty clay and an Osborne clay will be submitted to the Canadian Journal of Soil Science. The manuscript entitled The effects of soil forming processes on soil organic matter composition and clay dispersion from soil aggregates along a native prairie catena will be submitted to Catena.

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LIST OF ABBREVIATIONS

ASF	aggregate size fraction
ASFs	aggregate size fractions
CEC	cation exchange capacity
CHCl ₃	chloroform
CHCl ₃ -EL	chloroform extractable lipids
D-1	shoulder slope position of the prairie catena
D-2	mid-slope position of the prairie catena
D-3	toe-slope position of the prairie catena
DEE	diethyl ether
DEE-EL	diethyl ether extractable lipids
EC	electrical conductivity
ESP	exchangeable sodium percentage
HOC	hydrophobic organic compound
HOCs	hydrophobic organic compounds
LCA	long chain aliphatic compound
LCAs	long chain aliphatic compounds
NMR	nuclear magnetic resonance spectroscopy
ODR	oxygen diffusion rate
RCB	randomized complete block design
SAR	sodium adsorption ration
TEL	total extractable lipids

1. INTRODUCTION

Soil aggregates are complex compound structures consisting of primary mineral particles with different mineralogies, organic molecules of varying sizes with different functional groups, living organisms or living components of organisms from single-celled bacteria to plant roots, the more or less intact residue of once living organisms, inorganic ions, and water molecules all interacting with one another. It is not surprising, therefore, that aggregates and their constituents are held together by many different types of bonds and that these bonds vary widely in strength. This range of bond strengths forms the basis for the concept of aggregate hierarchy in soils described by Oades and Waters (1991).

Attempts to unravel the mysteries of soil aggregates take many forms. The most common methods involve the breaking of bonds within soil aggregates following various pre-treatments. By understanding the effect of the pretreatment on the various components, deductions about the nature of the bonds can be made. The bonds under consideration can be those holding macroaggregates together and are generally considered broken when the aggregate fragments pass through a sieve of a specified size. Alternately the bonds of interest may be those binding the clay-sized particles together into soil microaggregates and these bonds are considered to be broken when the clay sized particle remains in suspension. In both cases the breaking of a bond requires a force to separate the particles or aggregate fractions from one another. The force for macroaggregate disruption is generally applied as moving water, either flowing across aggregates as in wet sieving or through impact as

with rain drop simulation. Dispersion of clay from microaggregates has been accomplished by chemical dispersion (generally by increasing the percentage of Na ions on the clay exchange sites through manipulating solution concentration or by chelating the polyvalent cations initially present on the exchange sites) or through physical dispersion. Physical dispersion is most commonly accomplished through shaking a soil-water suspension for a specified time in either an end over end or horizontal shaking device. Ultrasonic dispersion is another method of physical dispersion and involves the application of sonic energy to a soil-water suspension. The main advantage of ultrasonic dispersion is the ability to precisely control the amount of energy applied to the suspension. Ultrasonic dispersion will also apply energy more uniformly to the aggregate and be influenced less by aggregate shape. This has obvious advantages in the study of soil aggregates as it allows the dispersion of clay particles in well controlled steps based upon the strength of the clay-humus and clay-clay bonds, hence aggregate strength can be correlated to percent dispersed clay.

Clay dispersion is generally considered to be a two step process involving macroaggregate breakdown followed by dispersion of clay from microaggregates. Several soil organic matter fractions have been implicated in the binding of clay particles within soil aggregates. Soil polysaccharides, often estimated by hexose determination following acid hydrolysis, and soil lipids are among those most frequently mentioned. A role for soil humic substances is also often suggested. Exchangeable cations also affect the dispersibility of the clay particles. For example exchangeable Na has a dispersive effect on clay particles whereas exchangeable Ca reduces clay dispersion.

The cultivation of native prairie grasslands has a major impact on the quantity and composition of soil organic matter. The decrease in soil aggregate stability and the increase

in clay dispersibility under cultivation has been partially attributed to this reduction in soil organic matter. Crop rotations that add more residue to the soil and reduce the frequency of tillage, and zero tillage have been suggested as management techniques to improve soil organic matter quantity and quality.

Soils are formed through the modification of parent material as affected by living organisms, climate and relief acting over time. Differences in soil aggregate stability and clay dispersion between soil types can be attributed to any of these five factors or to a combination of them. Studying soil types within a toposequence along a catena largely removes the effects of differences in parent material, climate, and time. Relief and the consequent variability in soil moisture may be considered to be the main effect, affecting the degree of leaching, and the vegetation types and productivity. Restricting sampling to areas with native grassland reduces the effect of vegetation and focuses on the effects of leaching and vegetative productivity.

To test the hypothesis that soil organic matter quality, especially the content of hydrophobic compounds, is more important than soil organic matter quantity for the stable binding of clay particles within soil aggregates, two studies were initiated. The first study examined the effects of zero tillage on soil organic matter constituents and clay dispersion. The second examined the influence of slope position on soil organic matter and clay dispersion. Ultrasonic dispersion of clay in several energy steps was used to determine the dispersibility of clay from soil aggregates. Clay dispersion was then related to organic matter components such as carbohydrate and lipids. Two different cultivated soils, a Fortier silty clay and an Osborne clay, were selected to examine the effects of zero tillage on the composition of the soil organic matter and clay dispersibility. In the other study, three slope

positions along a catena in a native prairie grassland near Deloraine, Manitoba were selected to study the effect of microtopography and related effects on soil organic matter composition and clay dispersibility.

2. LITERATURE REVIEW

2.1 Introduction

A well developed soil structure provides a network of continuous larger pores that drain quickly after rain and provide the aeration necessary for root growth as well as a finer scale network of fine capillary pores which are able to retain water that is available to plants even during dry weather (Crompton 1967). The breakdown of good soil structure will, therefore, reduce the ability of a soil to provide a good environment for root and consequently plant growth and crop production. Aggregate breakdown followed by clay dispersion results in surface seal formation which reduces the infiltration rate of water (Shainberg et al. 1992) and inhibits soil aeration (Hillel 1980). When surface seals dry, they form soil crusts which may damage emerging seedlings (Hillel 1980). Stable soil structure is important for reducing soil erosion (Elliot et al. 1991), minimizing compaction (Hillel 1980; Cruse and Gupta 1991), and enhancing soil aeration, water infiltration, and water holding capacity and thereby improving plant growth (Dinel et al. 1990).

A better understanding of the processes involved in stabilizing soil aggregates and in binding clay within soil aggregates is essential for the sustainable management of our soils.

Soil structure results from the aggregation of primary soil particles (sand, silt, and clay) into compound particles which are separated from adjoining aggregates by surfaces of

weakness (Soil Classification Working Group 1998). Oades and Waters (1991) describe aggregation of surface soils in which structural stability is dominated by organic matter as follows. Aggregates less than 20 μm diameter are random mixtures of clay microstructures, biopolymers, and microorganisms. These aggregates form larger aggregates 20 to 250 μm diameter that are bound together largely by microorganisms and their metabolic products. Aggregates less than 250 μm diameter (microaggregates) are joined into aggregates greater than 250 μm diameter (macroaggregates) through physical entanglement by living roots and fungal hyphae. These three steps in aggregation also represent steps in the strength of bonds or surfaces of weakness and hence it is easier to break a macroaggregate into its constituent microaggregates than to disperse clay particles from within a microaggregate. Under field conditions aggregate breakdown precedes clay dispersion (Shainberg et al. 1992) and therefore the study of clay dispersion from soil aggregates must include both the study of the bonds stabilizing soil aggregates and the bonds between individual clay microstructures or particles.

2.2 Formation of Clay Quasi Crystals and Domains

In soils literature, the term clay or clay fraction denotes a textural class of minerals consisting of particles with an equivalent spherical diameter of less than 2 μm , some but not all of which may be crystalline (Theng 1979). The most common minerals in the clay fraction of temperate region soils are the layered aluminosilicate clays (Hillel 1980). Tetrahedra of four oxygen atoms surrounding a central cation (usually Si^{4+}) are the basic structural unit of the tetrahedral sheet whereas octahedron of six oxygen atoms surrounding

a larger cation of lesser valency (usually Al^{3+} or Mg^{2+}) form the basic structural unit of the octahedral sheet (Hillel 1980). The two main types of aluminosilicate clay minerals, depending upon the ratios of tetrahedral to octahedral sheets, are the 1:1 and 2:1 clay minerals. In 1:1 clay minerals, such as kaolinite, one tetrahedral sheet and one octahedral sheet are attached by sharing oxygen atoms. In 2:1 minerals, such as montmorillonite or illite, the octahedral sheet is attached to two tetrahedral sheets, one on either side (Hillel 1980). Isomorphous substitution by cations of similar size but lower valence for Si^{4+} and Al^{3+} during the formation of the tetrahedral and octahedral sheets produces clay minerals with a permanent negative charge (Theng 1979). Sposito (1989) describes three mechanisms by which the layer charge is balanced by cation adsorption to the clay surface. An inner sphere complex results if the cation loses its solvation shell upon adsorption to the surface functional group. An outer sphere complex forms if at least one water molecule is retained between the cation and the surface functional group. If the cation only neutralizes surface charge in a delocalized sense it is said to be adsorbed in the diffuse ion swarm. Inner sphere complexes are more stable than outer sphere complexes and therefore cations adsorbed via the inner sphere complex mechanism are not considered to be readily exchangeable (Sposito 1989).

Illite has a higher layer charge than montmorillonite and will form stacks of crystal units in which the layers are bonded to one another via inner sphere complexes with K^+ . These microstructures are called domains (Sposito 1989). Drying of soils containing montmorillonite will induce individual crystal structures of montmorillonite to become oriented and form packets of perfectly aligned sheets called tactoids (Emerson et al. 1986). Sposito (1989) prefers to call such parallel alignments of unit smectite layers quasi crystals.

The number of crystal units in a quasi crystal is determined by the bonding strength of the cations present with $\text{Ca}^{2+} > \text{Mg}^{2+} > \text{Cs}^+ > \text{K}^+ > \text{Na}^+ > \text{Li}^+$. If Li^+ is the only ion occupying exchange sites the crystal units do not form stacks. In a smectite which is saturated by bivalent cations the layers are aligned and bound due to outer sphere complexes of the bivalent cation with a pair of ditrigonal cavities of opposing siloxane surfaces in the tetrahedral sheets. The average diameter of a Ca montmorillonite quasi crystal has been reported to be 0.5 μm and consisted of about 20 individual crystal units (Emerson et al. 1986). This is less than 2 μm and clay quasi crystals would be included in a measurement of dispersed clay in suspension. The study of clay dispersion is therefore largely the study of the forces binding clay quasi crystals and domains to one another and to other soil particles.

2.3 Soil Aggregates

2.3.1 Formation of Aggregates

Oades (1984) suggested a model in which microaggregates form within the macroaggregates encasing decomposing roots and hyphae. In a chronosequence of tallgrass prairie, formation and stabilization of macroaggregates was most closely related to the effects of roots and external hyphae with organic C, carbohydrate, and microbial C playing a minor role (Jastrow et al. 1998). Golchin et al. (1994) described the model more fully. The organic material of root or litter origin is initially colonized by soil microbes and simultaneously adsorbs mineral particles. The mineral encrusted plant material is protected from rapid decomposition and forms the core of a water stable aggregate. During this initial

stage of decomposition, the microbes thrive on the carbohydrate rich plant material and produce mucilages and metabolites which interact with the mineral particles and stabilize the aggregate. As decomposition progresses, proteins, carbohydrates, and other labile components of the plant material are consumed and eventually join the soil organic pool as microbial products. The decomposition rate of the organic core slows as the residue becomes more recalcitrant or is used up and eventually the aggregate becomes unstable due to the reduction in the production of microbial metabolites. The macroaggregate then breaks apart and many microaggregates are produced. Net input rates of C for microaggregates is equal to the rates for small macroaggregates (Jastrow et al. 1996) which is consistent with microaggregates forming within macroaggregates. Jastrow et al. (1996) also found that the turnover time for C was almost three times longer in microaggregates than macroaggregates suggesting that once formed microaggregates are more stable than macroaggregates.

In a review of the biological bonding and binding mechanisms within soil macroaggregates, Degens (1997) divides the mechanisms into chemical bonding between soil particles by labile organic C and physical binding of particles by fungal hyphae and roots. Soil carbohydrate and aliphatic C were suggested as the main groups responsible for the chemical bonding. As a result of the types of binding and bonding present, macroaggregates are sensitive to the short term management of soils (Oades 1984).

Microaggregates are resistant to rapid wetting and are not easily destroyed by agricultural practices (Tisdall and Oades 1982). Clay quasi crystals and domains are held stabilized in microaggregates through polyvalent cation linkages to soil organic matter (Edwards and Bremner 1967), by persistent organic bonds and iron oxides (Tisdall and Oades 1982), by hydroxy-Al polyanions (Goh and Huang 1986; Gu and Doner 1993), by

association with biopolymers, microorganisms, and plant debris (Oades and Waters 1991).

Organic matter in mineral soils is generally intimately associated with inorganic particles. Without exception, it is impossible to separate all the organic matter from the clay, silt, and even sand particles by purely physical means. This alludes to the strong chemical and physical interaction between organic materials and the inorganic matrix.

I will focus the remainder of my discussion on the role of soil organic materials in the stabilization of clay within aggregates.

2.3.2 Bonding Mechanisms of Organic Functional Groups and Soil Clay

The organic C found in soils is in molecules which range in size from simple compounds released during decomposition processes to large polymers such as humic acid which may have molecular weights of 100,000 daltons or more (Paul and Clark 1996). This complex mixture of molecules interacts with soil minerals through a wide variety of mechanisms.

Sposito (1989) describes eight bonding mechanisms depending on the functional groups present on the organic molecule or polymer (Table 2.1). Cation exchange occurs when a protonated group, such as the -NH_3^+ of an amino group, replaces a monovalent cation on a clay exchange site. The protonation mechanism is most important when the acidity of the soil mineral surfaces is greatest such as at low pH or low soil water content. The bond forms between the organic functional group and a proton bound to the mineral surface or to a proton in an acidic water molecule in the hydration shell of an exchangeable metal cation. Anion exchange is the result of a carboxylate group (COO^-) replacing a monovalent exchangeable anion, such as Cl^- or NO_3^- , which was bound to a protonated

Table 2.1 Mechanisms of association between organic functional groups and soil minerals (from Sposito 1980)

Mechanism	Organic functional groups involved
Cation exchange	Amino, ring NH, heterocyclic N (aromatic ring)
Protonation	Amino, heterocyclic N, carbonyl, carboxylate
Anion exchange	Carboxylate
Water bridging	Amino, carboxylate, carbonyl, alcoholic OH
Cation bridging	Carboxylate, amines, carbonyl, alcoholic OH
Ligand exchange	Carboxylates
Hydrogen bonding	Amino, carbonyl, carboxyl, phenolic OH
van der Waals interaction	Uncharged organic units

surface hydroxyl ($-\text{OH}_2^+$). This is a weak bond but could be prominent in acid soils with a high content of metal oxides. Water bridging involves the complexation of anionic or polar functional groups (e.g., carboxylate or carbonyl) with a water molecule in the hydration shell of an exchangeable cation. Cation bridging occurs when the water molecule is displaced and the functional group bonds directly to the exchangeable cation. The ability of the functional group to displace the water molecule will determine whether water bridging or cation bridging occurs. Water bridging produces a weaker bond between the cation and the organic molecule than does cation bridging. Ligand exchange refers to a direct bond formation between an Al or Fe(III) in a soil mineral and a carboxylate group. The hydroxyl attached to the Al or Fe(III) is protonated in the first step than replaced by the carboxylate releasing a water molecule. As protonation favours the reaction, ligand exchange occurs more readily as pH decreases. Hydrogen bonding produces very weak bonds due to the low electronegativity of mineral surface oxygens and is therefore not an important mechanism of bonding between soil mineral and organic components. The van der Waals interactions

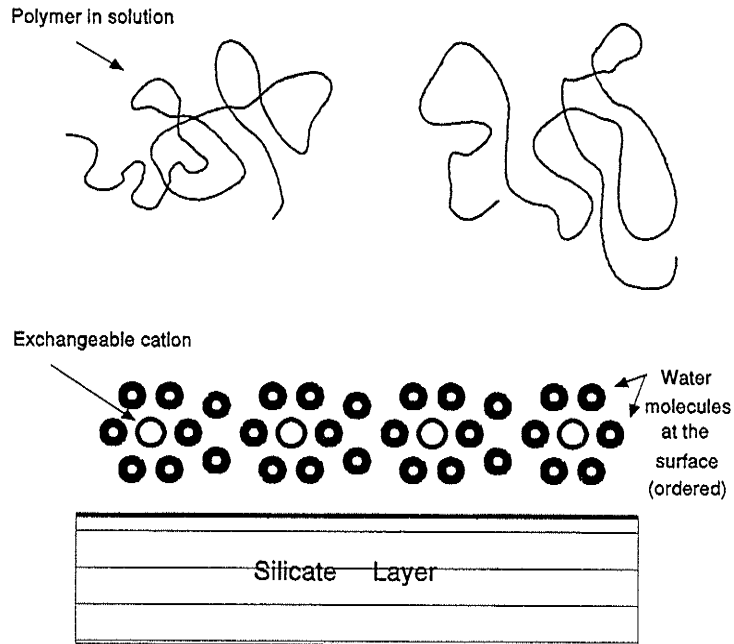
can be quite strong between uncharged portions of a polymer and a mineral surface. The effect is especially important when there is minimal ionization of acidic functional groups on the polymers.

These eight mechanisms are useful in understanding the interactions of individual organic micromolecules or functional groups with soil minerals but the interactions of organic macromolecules (polymers) with clays have some important differences (Theng 1979). Polymer chains are long, flexible and often polyfunctional. This allows the polymer to assume different conformations and to attach to the clay by many and varied polymer segment-surface bonds. The ions in solution and surface properties of the clay and polymer will determine whether a specific polymer will bind to clay particle and how strong that bond will be. The binding of charged and uncharged polymers will be discussed separately in the following sections.

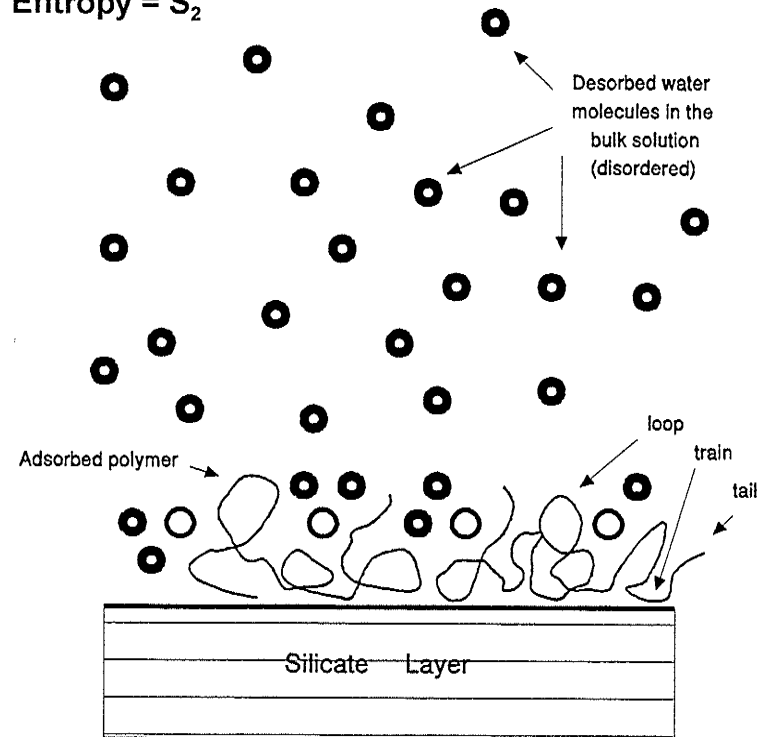
2.3.3 Adsorption of Uncharged Polymers

The mechanism whereby an uncharged polymer with polar functional groups will adsorb onto a clay surface in the presence of water is described by Theng (1979). Although the enthalpy change for the adsorption is virtually zero, a large number of water molecules will be desorbed from the exchangeable cations and clay surface to accommodate the polymer (Figure 2.1). The increase in entropy during adsorption is therefore the driving force for adsorption rather than the attraction between the polymer and the cations or clay surface. The polymer, which would be in a random coil in solution, would tend to collapse

Entropy = S_1



Entropy = S_2



For polymer adsorption, the entropy change ($\Delta S = S_2 - S_1$) is the sum of the conformational term (ΔS_c) and the "hydration" term (ΔS_H). Conformational entropy of the polymer decreases upon adsorption and will tend to inhibit adsorption. An increase in "hydration" entropy due to release of water from the clay and the polymer promotes adsorption. When the hydration term is dominant, $S_2 > S_1$ and polymer adsorption occurs.

Figure 2.1 Illustration of the desorption of numerous water molecules from the exchangeable cations at the clay surface during the adsorption of uncharged polymer molecules (after Theng 1979).

and spread out over the surface upon adsorption with trains involved in the adsorption and loops and tails extending out into the solution. Van der Waals forces between polymer hydroxyls and the O atoms of the siloxane surface will become increasingly significant as the clay-polymer complex dries and the polymer draws nearer to the surface. The type of cation present on the exchange sites will influence bonding in at least two ways. The polarizing power of the cation will determine the size of the hydration shell which will affect the interlayer spacing and hence the surface area accessible for adsorption. The polarizing power will also determine the number of water molecules in the hydration shell which can be displaced by the polymer, which in turn determines the amount of water released during adsorption and therefore influences the entropy gain during adsorption. Parfitt and Greenland (1970a) reported that the amount of water desorbed during adsorption of polyethylene glycol (molecular weight 300) on montmorillonite was influenced by the exchangeable cation in the order $\text{Na}^+ > \text{Ca}^{2+} > \text{Al}^{3+}$, with virtually no water desorbed in the case of the Al ion. Fuller (1993) found that in a suspension of dextran and montmorillonite, Ca-saturated montmorillonite had a much greater rate of flocculation than Mg-saturated montmorillonite. Though both are divalent the difference was attributed to the greater polarizing ability of Mg which would require the polymer to desorb water from the clay surface rather than from the hydration shell of the cation, encouraging a more complete collapse of the polymer onto the clay surface. With fewer loops and tails, there would be less interaction between particles and hence reduced flocculation. A secondary reason for reduced flocculation is the increase in thickness of the double layer caused by the larger hydrated diameter of the Mg cation. The resultant increase in distance between clay particles makes it more difficult for the polymers to bridge adjacent clay particles.

An hydrophobic polymer would adsorb to hydrophobic soil organic matter or to soil mineral surfaces (surface of -Si-O-Si- bonds) which are uncharged (Hassett and Banwart 1989). The driving force is again the entropy gain but now it is due to the destruction of the cavity occupied by the polymer in the soil solution and the partial destruction of the water shell surrounding the polymer. Sposito (1989) refers to this phenomena as the hydrophobic effect. Compounds bound through hydrophobic adsorption can be extracted by organic solvents such as CHCl_3 . Hydrophobic compounds which contain some polar functional groups can also bind to soil mineral surfaces via cation bridges and require treatment with Na-pyrophosphate or HCl-HF before extraction (Dinel et al. 1992).

2.3.4 Adsorption of Charged Polymers

Polymers with positively charged functional groups will displace exchangeable cations and tend to collapse very rapidly onto the negatively charged clay surface. The result is that most of the polymer is in trains with few loops and tails to form bridges to other particles (Theng 1979). Because the cationic polymer must replace cations on the exchange, the strength of the cation adsorption will affect polymer adsorption. Gu and Doner (1992) report that the ability of a cationic polymer to replace the exchangeable cations is in the order $\text{Na}^+ > \text{Ca}^{2+} > \text{hydroxy Al polycations}$.

Negatively charged polymers and negatively charged clay mineral surfaces repel one another. However bonding does occur and several possible mechanisms are described by Theng (1979). Cation bridging of the polymer with weakly hydrated or non-hydrated monovalent exchangeable cations or with polyvalent exchangeable cations in a dehydrated system can occur. In a hydrated system water bridging between the polymer and polyvalent

cations is possible. Clay crystal edge surfaces or adsorbed hydroxy compounds of iron and aluminum can participate in anion exchange or ligand exchange. Van der Waals force can become important between uncharged segments of the polymer chain and the clay surface upon drying. Also as with the adsorption of any polymer there will be the effect of the gain in entropy as water is displaced from the clay surface. The valency and hydrated diameter of the exchangeable cations present again have a large effect on adsorption. In this case the effect is largely due to the strength of the bridge formed and to the efficiency of the cations in shielding the charge on both clay and polymer, a necessary step in allowing the polymer to approach the surface near enough to adsorb. Divalent cations, Ca and Mg, were approximately 28 times more effective in increasing the sorption of an anionic polyacrylamide than monovalent cations, Na and K, due to their greater charge screening ability (Lu et al. 2002). The addition of anionic humic substances to a Na-montmorillonite suspension caused a reduction in viscosity due to the anionic polymer binding to positively charged clay edges and causing a charge reversal (Tarchitzky and Chen 2002). In the Ca-saturated system, the viscosity was much higher than in the Na-saturated system and was not affected by addition of the anionic humic substances. The negative charge of the montmorillonite and the humic substances was more effectively shielded by Ca than by Na and as a result the colloids interacted more strongly and viscosity was higher. Gu and Doner (1992) found that anionic polymer adsorption was in the order Al-polyanion > Ca > Na. Bridging by the cations was best if the cations were divalent or greater. Na did a poor job of masking the clay charge and therefore the anionic polymer could not approach the clay. Extracted soil polysaccharide reacted in a similar manner to the anionic guar polymer. The importance of the thickness of the diffuse double layer has been demonstrated by changing

the ionic strength. As the ionic strength is increased, the diffuse double layer becomes thinner and adsorption increases (Theng 1979; Gu and Doner 1992).

2.3.5 Role of Soil Carbohydrates in Aggregate Stability

2.3.5.1 Soil Carbohydrates. Complex carbohydrates result from the combination of simple carbohydrates such as the simple sugars pentose, hexose, and uronic acid to produce a wide range of compounds in plants, microorganisms, and soils (Paul and Clark 1996). Soil organic matter contains on average 10% carbohydrate much of which is of microbial origin (Cheshire 1977). The term polysaccharide is used to denote molecules consisting of 10 or more monosaccharide or sugar units, whereas the term carbohydrate refers to the entire group of monomeric, oligomeric, and polymeric species (Theng 1979).

Most plant or microbial polysaccharides, even when present in complex organic residues, are largely decomposed within a few weeks to a few months after addition to soil (Martin 1971). However, some polysaccharides do persist much longer which has been attributed to their unavailability to microorganisms or extracellular enzymes (Cheshire 1977). The polysaccharides would be protected in residual biological structures (Cheshire 1977), through encapsulation by clay particles (Cheshire and Mundie 1981; Oades 1984; Golchin et al. 1994a), and through adsorption on clay surfaces or by the formation of complexes with metals (Martin 1971; Cheshire 1977). Oades et al. (1988) attributes much of the stabilization of soil organic matter by clay to adsorption via cation bridges associated with carboxyl groups. As a result soil carbohydrate levels tend to be higher in soils with higher clay contents (Dalal and Henry 1988).

Soil carbohydrate levels reported for several mineral soils in Australia ranged from

0.38 to 4.43 g kg⁻¹ of soil (Oades 1971). Fuller and Goh (1992) reported substantial differences in total carbohydrate concentrations among three western Canadian soils under native vegetation. The highest was an Orthic Black Chernozem (6.3 g kg⁻¹ of soil) followed by an Orthic Dark Gray Chernozem (4.1 g kg⁻¹ of soil) and the lowest was an Orthic Gray Luvisol (1.9 g kg⁻¹ of soil). Upon examination of several soil types and cropping systems, Cheshire et al. (1984) found large differences in carbohydrate content among soil types and cropping systems within soils. A Luvisol (FAO) had carbohydrate concentrations of 17.0, 13.3, and 3.2 g kg⁻¹ of soil for grassland, arable ley, and continuous wheat respectively. A Cambisol (FAO) had similar carbohydrate levels under grass, but much higher carbohydrate content than the Luvisol under cultivated cereal (16.7 and 11.2 g kg⁻¹ soil for grass and arable barley respectively).

Tillage management, crop rotation, and soil moisture conditions can all influence soil carbohydrate levels. Upon cultivation, native prairie grassland soils show a decline in soil organic C (Dalal and Henry 1988; Blank and Fosberg 1989; Fuller et al. 1995; Six et al. 2000) and also a decline in soil carbohydrate (Dalal and Henry 1988; Fuller et al. 1995) probably as a result of the exposure of previously occluded soil organic matter (Golchin et al. 1994a; Golchin et al. 1994b). Zero tillage management has been shown to increase soil carbohydrate content more than blade cultivation after conversion from conventional tillage (Dormaar and Lindwall 1989). Carbohydrates were largely of plant origin under zero tillage and of microbial origin under blade cultivation indicating different organic matter decomposition dynamics. In a study of short term cropping effects, Angers and Mehuys (1990) found that after only two seasons total carbohydrate was higher under barley than alfalfa. The difference was attributed to higher root biomass production under the barley

as well as slower decomposition due to a less favourable C-to-N ratio. Both crops had higher levels of carbohydrate than did the bare fallow treatment. In contrast, Baldock et al. (1987) compared brome grass and continuous corn plots after 15 years and found no difference in soil carbohydrate levels. Conditions of low soil moisture during the growing season result in low levels of soil carbohydrate (Webber 1965; Angers and Mehuys 1989). The effect was attributed to a reduction in the activity of soil microorganisms at low soil moisture contents.

Soil carbohydrates are highly variable in composition and therefore cannot be expected to behave identically in the soil environment. Soil properties, especially clay content and moisture status, can have a profound impact on the quantity and quality of soil carbohydrates. Management practices can also affect both the concentration of soil carbohydrates and their composition.

2.3.5.2 Adsorption of Carbohydrates. Soil carbohydrates are complex mixtures of polar compounds that range in size from simple sugars to large polymers. Many are neutral under soil conditions but others, such as the polygalacturonic acids, are largely dissociated at pH 6 and above (Parfitt and Greenland 1970b).

Neutral carbohydrates would adsorb onto clay surfaces by cation or water bridge formation with polar groups and through van der Waals forces. The individual bonds would be weak but with large polymers the total number of bonds would make desorption difficult. Due to the displacement of many water molecules from cation hydration shells and the clay surface, the gain in entropy upon adsorption of a polymer is large and becomes the driving force for adsorption (Parfitt and Greenland 1970a; Theng 1979). The adsorption of a synthetic neutral polysaccharide onto illite was similar regardless of whether the

exchangeable cations were Na, Ca, or hydroxy-Al polycations (Gu and Doner 1992). Dextran adsorption onto montmorillonite was similar with Ca or Al on the exchange sites but was greatly enhanced when Na was the exchangeable cation (Parfitt and Greenland 1970b). They suggest that the large number of water molecules displaced from the hydration shell of Na was responsible for this difference, however Theng (1979) attributed at least part of the increase to clay swelling induced by the Na cations and hence there was an increase in clay surface area available for adsorption. Flocculation studies comparing the effect of exchangeable Ca and Mg on the adsorption of dextran onto montmorillonite have shown that dextran is a more effective flocculant in the Ca-montmorillonite system (Fuller 1993). The lack of flocculation of the Mg-saturated clay suspension was attributed to a greater collapse of the polymer onto the surface with shorter tails and fewer loops to interact with other particles. At the same time the larger hydrated diameter of the Mg ions would result in a thicker diffuse double layer and greater repulsive forces between clay particles which would inhibit flocculation.

Cationic carbohydrates have a strong attraction to the negatively charged clay surfaces and bind through cation exchange (Theng 1979). The strong attraction between polysaccharide and clay causes a high degree of collapse onto the clay and leave few loops or tails to interact with other clay particles. Metal cations are held on the soil exchange with different bond strengths and hence the ease of exchange or replacement of metal cations by the carbohydrate varies as a function of valence and ionic radius of the exchangeable cations present. The strength of the adsorption is in the order $Al^{3+} \gg Ca^{2+} > Mg^{2+} \gg NH_4^+ > K^+ > H^+ > Na^+$ (Hillel 1980). The application of this series to polysaccharide polymers has been demonstrated by Gu and Doner (1992). More of the cationic polysaccharide was adsorbed

on Na-illite than on Ca-illite and Al-polyanion-illite adsorbed the lowest amount of the polymer.

Negatively charged carbohydrates will be repelled by the negatively charged clay particles. However if polyvalent cations dominate the exchange sites the charges will be screened sufficiently so that the carboxyl groups on the uronic acid can approach the clay and form cation or water bridges to the clay (Theng 1979). Gu and Doner (1992) found that an anionic polysaccharide was adsorbed in the order of exchangeable cation valence (Al polyion > Ca > Na). Adsorption increased as pH decreased due to the decrease in the degree of the dissociation of the carboxyl groups which would reduce the repulsive forces. At low pH, the anionic polysaccharide became neutral and exhibited the same adsorption behaviour as the neutral polysaccharide. As with other polymers, the entropy effect and van der Waals forces would contribute to the strength of the clay-polymer bond (Theng 1979). Extracted soil polysaccharide exhibited adsorption behaviour most like that of the synthetic anionic polysaccharides (Gu and Doner 1992).

2.3.5.3 Carbohydrate and Soil Structure. The role of soil carbohydrates and more specifically soil polysaccharides in stabilizing soil structure has been the object of much research over the last half century (Martin 1945; Geoghegan and Brian 1946; Martin 1971; Oades 1978; Cheshire et al. 1983; Oades 1984; Angers and Mehuys 1989; Fuller et al. 1995).

Oades (1984) presented a model of soil aggregate formation based on clay bonding to mucilages. Mucilages are largely carbohydrates produced by plant roots, fungal hyphae, and soil microorganisms (Oades 1978). These mucilages, and especially those of microbial origin, play a fundamental role in the stabilization of the evolving aggregate as soil particles encrust decomposing organic material (Golchin et al. 1994b). Emerson et al. (1986) present

several ultrathin sections that show clay particles embedded in mucilage, clearly showing an interaction between the polysaccharide and the soil particles but not quantifying the effect on aggregate stability.

Several researchers have studied the addition of polymers to soil or clay samples and have added to our understanding of the effect of carbohydrates on aggregate stability. Polysaccharides increase the flocculation of suspensions of montmorillonite (Fuller 1993). Aggregate stability and hydraulic conductivity can also be influenced through the addition of polymers. Gu and Doner (1993) added a synthetic anionic polysaccharide, a soil humic acid extract, and hydroxy-Al polycations alone and in combination to Na-saturated soil or clay samples. The polysaccharide did not enhance flocculation of illitic clay or soil suspensions but greatly increased wet aggregate stability. The aggregates were air dried after addition of the polysaccharide and it was concluded that this allowed the polymer to approach and bond to the clay surface by van der Waals forces (and likely aided by the entropy effect) even though the exchange sites were dominated by Na. The presence of both polyvalent cations and anionic polysaccharides produced the greatest improvement in both aggregate stability and soil hydraulic conductivity. Swift (1990) found that incubation of natural soil aggregates with microbial exocellular polysaccharides (xanthan gum and alginate) enhanced wet aggregate stability. The effect, however, was transient due to decomposition of the added polysaccharides. Addition of humic acid also enhanced stability and the effect was more persistent than that of adding polysaccharide (Swift 1990; Piccolo and Mbagwu 1999).

Selective oxidation of carbohydrate (by sodium periodate, for example) has been used to demonstrate the stabilizing effect of soil carbohydrate on soil aggregates. Soil from

under long term grass was treated with 0.02 M sodium periodate for periods up to 1176 hours (Cheshire et al. 1983). Aggregate breakdown was greatly enhanced by oxidation of the soil carbohydrate. The experiment was repeated with several different soil types and cropping systems. Periodate oxidation of soil carbohydrate increased macroaggregate breakdown by 75 %. The aggregate stability was attributed to carbohydrate of microbial origin (Cheshire et al. 1984). Angers and Mehuys (1989) compared the effects of fallow, alfalfa, and barley on water-stable aggregation of a clay soil. After two seasons, barley and alfalfa produced more water stable aggregates than fallow. Periodate-borate treatment did not affect the quantity of water stable aggregates under fallow but did reduce the stability of the other treatments to the level of the fallow treatment. Corn and bromegrass treatments were compared after 15 seasons and although periodate treatment reduced the aggregate stability, the periodate sensitive material did not account for the differences in stability between cropping treatments (Baldock and Kay 1987).

Correlations of aggregate stability and measured soil carbohydrate levels have been used by several researchers to examine the stabilization of soil aggregates by carbohydrates. In the cropping study mentioned above, Angers and Mehuys (1989) report that water-stable aggregates were significantly correlated to soil carbohydrate extractable with 1.5 M H_2SO_4 ($r = 0.63$). Cultivation of a native prairie grassland in western Canada resulted in a decline in both soil carbohydrate levels and wet aggregate stability. The correlation was highly significant ($r = 0.89$) but several other measured parameters were also correlated with aggregate stability (Fuller et al. 1995). Fuller and Goh (1992) also found good correlations between aggregate stability and carbohydrate content in three soils under native vegetation. In a comparison of cover crops in orchards, the heavy fraction carbohydrate (contained in

soil particles with a density greater than 1.74 g mL^{-1}) was significantly correlated with soil structure whereas the remainder of the carbohydrate (light fraction) was not (Roberson et al. 1991). Total soil organic C and biomass C were also not correlated to structural stability. In a subsequent study (Roberson et al. 1995), moderate N addition increased the heavy fraction carbohydrate, and improved both slaking resistance and saturated hydraulic conductivity. The mineral-associated carbohydrates (heavy fraction) were estimated to be largely of microbial extracellular polysaccharide origin.

Polysaccharides or select fractions of the soil polysaccharides have been implicated as important stabilizers of soil structure in many soils. However others have found carbohydrates to be of little importance. Webber (1965) found that seasonal variation in soil polysaccharide levels did not correspond to seasonal variation in wet aggregate stability. Although soil organic materials were important in aggregate stabilization of an Alfisol and a Mollisol, oxides were the dominant factor determining soil aggregate stability in an Oxisol (Oades and Waters 1991). Total and extractable carbohydrate, microbial biomass C, and organic C content were all closely correlated to aggregate stability in a group of soils ranging in organic C content from 20 to 40 g C kg^{-1} (Haynes 2000). Monreal et al. (1995) examined a Chernozem (Mollisol) from western Canada and found good correlations between macroaggregate stability and lignin dimers ($r = 0.98$), sterols ($r = 0.94$), alkylaromatics ($r = 0.94$), and lipids ($r = 0.90$) but not for soil carbohydrate.

2.3.6 Role of Soil Lipids in Aggregate Stability

2.3.6.1 Lipids Defined. Unlike most of the other non-humic substances, lipids are not pure organic substances (Wilson 1987), that is, lipids are not defined based on their

chemical composition but on solubility criteria. Lehninger (1982) defines lipids as water-insoluble organic biomolecules that can be extracted from cells and tissues by non-polar solvents such as chloroform, ether, or benzene and he attributes lipid solubility and other distinctive properties to the hydrocarbon nature of a major portion of their structures. Because lipids are defined by solubility in water and a non-polar solvent, significant variation in chemical structure and physico-chemical properties among lipids is possible (Andreyev et. al. 1980).

Lipids can be divided into simple lipids, compound lipids, and derived lipids. Bohinski (1976) defines them as follows: Simple lipids include materials that are esters of fatty acids and glycerol (trihydroxyalcohol) or long chain monohydroxyalcohols. Examples of simple lipids are the acylglycerols and waxes. Compound lipids contain other substances in addition to the alcohol and fatty acids. Examples are sphingomyelins (sphingosine + fatty acid + HPO_4^{2-} + choline), cerebroside (sphingosine + fatty acid + simple sugar), and gangliosides (sphingosine + fatty acid + two to six simple sugars). Derived lipids are a highly variable group which do not fit into the simple or compound lipid categories but still meet the general definition of a lipid. Examples are steroids, carotenoids, and the water-insoluble vitamins. By definition alkanes, alkenes, fatty acids, and long chain monohydroxyalcohols are also classified as derived lipids. Most simple and compound lipids consist of fatty acids covalently bonded to a backbone structure and as a result their nomenclature is based upon the type of backbone structure present (Lehninger 1982). For example, glycerol forms the backbone of the acylglycerides, glycerol 3-phosphate forms the backbone of the phosphoglycerides, sphingosine forms the backbone of the sphingolipids, and high molecular weight nonpolar alcohols form the backbone of the waxes. This highly

variable group of compounds fulfils various roles in biological systems; lipids may be structural, energy and carbon storage units, transporters of nonpolar compounds, or function as vitamins and hormones (Bohinski 1976).

2.3.6.2 Soil Lipids. All living cells produce lipids (Bohinski 1976) and therefore all plants growing in soil and all organisms living in and on soil contribute to the highly variable material classified as soil lipids. The originating organisms have a large impact on the types of lipids deposited in the soil (Table 2.2) and upon the carbon chain length of the various lipids (Dinel et al. 1990). The effect of plant species on soil lipids has been shown (Wang et al. 1971; Perniola et al. 1981; McKeague et al. 1986). These effects have been attributed to differences in plant lipid content and plant influences upon the soil microbial population (Wang et al. 1971). The composition of the complex lipid fraction in soils depends upon the nature and amount of plant input and microbial synthesis as well as the rate of decomposition of lipids by soil organisms (Moucawi et al. 1981b). However, the ability of microorganisms to utilize and decompose these compounds is of primary importance in determining which lipids will persist or accumulate (Moucawi et al. 1981a, Moucawi et al. 1981b).

Andreyev et al. (1980) extracted lipids from various soils and found that the majority of the lipids were of microbial origin. Other workers have found lipids of plant origin to dominate (Fridland 1976; Fustec-Mathon et al. 1977; Moucawi et al. 1981b). Dinel et al. (1990) suggest that the majority of soil lipids originate from the surface lipids of plant and animal cells and have been modified by soil microorganisms during decomposition. Due to their original role as protectants against desiccation and disease, surface lipids possess

Table 2.2 Classes of compounds found in surface biolipids of various living organisms (Kolattukudy 1976).

Class of Compound	Animals	Insects	Plants	Algae	Fungi	Bacteria
1. Hydrocarbons						
<i>n</i> -Alkanes	X	X	X	X	X	X
branched alkanes	X	X	X	X	X	X
olefins	X	X	X	X		X
cyclic alkanes			X			
2. Ketones						
monoketones			X			X
beta diketones			X			
3. Secondary Alcohols		X	X			X
4. Alkane Diols (diesters)	X		X			
5. Free Fatty Acids	X		X			
6. Wax Esters						
primary alcohol esters	X	X	X	X	X	X
secondary alcohol esters		X				
diesters	X	X				
triesters	X	X	X			
7. Primary Alcohols	X	X	X	X		X
8. Aldehydes			X			
9. Terpenoids	X		X			

physical, chemical, and biological properties which make them more resistant than internal lipids to microbial breakdown (Dinel et al. 1990). Soil lipids include a variety of component structures, including alkanes, alkenes, fatty acids, alcohols, sterols, and various esters (Figure 2.2).

Lipid contents of several organic and mineral soil surface horizons were reported by Fridland (1976). The soils contained from 0.06% to 1.4% lipids; these values correspond to 1.9 to 16 percent of the total soil organic carbon. Dinel et al. (1991b) found that for an organic soil, the degree of decomposition had a major effect on the lipid content. Material which was only slightly altered (dominantly fibric) was dominated by carbohydrate (lipid:carbohydrate = 0.2) while highly decomposed material (dominantly humic) was predominantly lipid (lipid:carbohydrate = 1.4). For most soils (globally), four to eight percent of soil organic carbon is lipid (Dinel et al. 1990). The importance of carbohydrate in soil structural stability is well documented (Cheshire et al. 1984; Oades, 1984; Fuller et al. 1995) and the contribution of lipids, which occur in soils at about the same concentrations, should not be ignored.

Comparison between lipids and other soil non-humic organic matter components, such as carbohydrates and proteins, show that lipids in general have a much longer residence time in soil (Dinel et al. 1990). Lipids should then make up a greater proportion of the soil organic matter in biologically active soils. Preston et al. (1987) reported that drainage and cultivation of organic soils resulted in increased rates of decomposition for both carbohydrates and lipids. However, the effect was much less for lipid and as a result lipid levels increased proportionally. The seasonal fluctuation in lipid levels reported by Perniola et al. (1981) are likely due to seasonal fluctuation in soil biological activity.

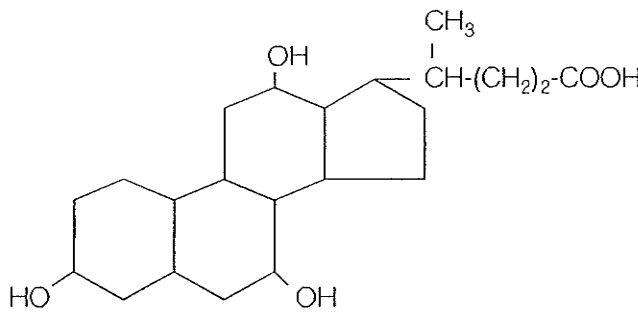
n-Alkanes	$\text{CH}_3-(\text{CH}_2)_n-\text{CH}_3$
Branched Alkanes	$\begin{array}{c} \text{CH}_3-\text{CH}-(\text{CH}_2)_n-\text{CH}_3 \\ \\ (\text{CH}_2)_n \\ \\ \text{CH}_3 \end{array}$
n-Alkenes	$\text{CH}_3-(\text{CH}_2)_n-\text{CH}=\text{CH}-(\text{CH}_2)_n-\text{CH}_3$
n-Fatty acids	$\text{CH}_3-(\text{CH}_2)_n-\text{COOH}$
n-Alcohols	$\text{CH}_3-(\text{CH}_2)_n-\text{CH}_2\text{OH}$
Diols	$\text{CH}_3-\text{CHOH}-(\text{CH}_2)_n-\text{CH}_2\text{OH}$
Sterols (eg. cholic acid)	 <p>The structure shows a steroid nucleus with three hydroxyl groups (HO- and -OH) and a side chain consisting of a methyl group (CH₃) attached to a CH group, which is further attached to a (CH₂)₂ group ending in a COOH group.</p>
Monoesters	$\begin{array}{c} \text{O} \\ \\ \text{R}_1-\text{CH}_2-\text{C}-\text{O}-(\text{CH}_2)_n-\text{R}_2 \end{array}$
Diesters	$\begin{array}{c} \text{CH}_3-\text{CH}-\text{CH}_2-\text{CH}-\text{CH}_3 \\ \quad \quad \\ \text{O} \quad \quad \text{O} \\ \quad \quad \\ \text{C}=\text{O} \quad \text{C}=\text{O} \\ \quad \quad \\ \text{R}_1 \quad \quad \text{R}_2 \end{array}$
Triesters	$\begin{array}{c} \text{CH}_3-\text{CH}-\text{CH}_2-\text{CH}-\text{CH}_2-\text{CH}-\text{CH}_3 \\ \quad \quad \quad \quad \\ \text{O} \quad \quad \text{O} \quad \quad \text{O} \\ \quad \quad \quad \quad \\ \text{C}=\text{O} \quad \text{C}=\text{O} \quad \text{C}=\text{O} \\ \quad \quad \quad \quad \\ \text{R}_1 \quad \quad \text{R}_2 \quad \quad \text{R}_3 \end{array}$

Figure 2.2 Chemical formulae of the major constituents of soil lipids (Dinel et al. 1990).

The highly complex and variable nature of soil lipids suggests that decomposition rates would also differ among lipids. Internal lipids are utilized rapidly as carbon sources by soil microbes (Dinel et al. 1990) and are generally found in significant quantities only in living cells. The degradation of the more recalcitrant external lipids is affected by many factors. A number of soil lipids have a toxic or inhibitory effect on the soil microflora (Fustec-Mathon et al. 1977) and can therefore be expected to be among the most resistant. Moucawi et al. (1981b) compared the decomposition rates of several lipids in four different soils, and they found that the unsaturated oleic acid was decomposed much more quickly than the saturated stearic acid at pH 4.1, while at near neutral pH the two rates were almost identical. Anaerobic conditions also favoured the decomposition of oleic acid over stearic acid, presumably as a result of yeast species requiring oleic acid for growth. Under aerobic conditions bacteria were dominant and no difference was observed. The decomposition of lipids of various carbon chain lengths and functional groups followed a similar pattern; near neutral, aerobic soil (high biological activity) resulted in very little difference in decomposition rate between compounds whereas acidic or anaerobic soils (relatively lower biological activity) showed lower rates with considerable variation between compounds. Dinel et al. (1990) and Fridland (1976) suggest that increased lipid content in drier soils is due to the reduction in microbial activity caused by water stress. Low temperature and very nutrient deficient soils also have low biological activity and show elevated lipid levels (Fridland 1976). Moucawi et al. (1981a) found that the addition of $\text{Fe}(\text{OH})_3$ to iron-poor soils enhanced the biodegradation of soil lipids. Paré et al. (1999) report that conventionally tilled treatments had 21% more total extractable lipid than did the zero tillage treatments. They attributed the difference to reduced decomposition of organic material under zero

tillage as a result of the reduced mixing of crop residue with the soil. The composition of the lipid fraction was also affected by tillage management as determined by sequential extraction with diethyl ether (DEE) and chloroform (CHCl_3). The ratio of diethyl ether-extractable lipid to CHCl_3 -extractable lipid was 40% higher under zero tillage. Dinel et al. (1996) used sequential extraction of lipids with DEE and CHCl_3 to estimate organic matter stability. They found that diethyl ether extractable lipids (DEE-EL) were relatively low-molecular weight straight-chain and branched alkanes, alkenes, and alkanolic acids with some phenolic and alkylaromatic compounds. CHCl_3 extractable lipids (CHCl_3 -EL) were relatively high-molecular weight paraffinic compounds with considerably fewer phenolic and aromatic compounds. DEE-EL are more easily biodegraded by soil microbes than CHCl_3 -EL most likely due to the greater hydrophobicity of the CHCl_3 -EL (Dinel et al. 1992). A higher DEE-EL to CHCl_3 -EL ratio would therefore indicate that decomposition is less advanced. A similar effect of zero tillage management on lipid composition was reported by Dinel et al. (1998). Monreal et al. (1995) found that, in comparison to a wheat and cultivated-fallow rotation, the macroaggregates from soil under continuous wheat contained higher levels of several organic matter constituents, including lipids.

2.3.6.3 Adsorption of Lipids in Soil. Soil lipids have low water solubility and a resulting reduced mobility in soil. Everett et al. (1995) found that the heat of burning forest slash caused hydrophobic compounds to volatilize, move downward, and recondense as deep as 7 cm below the surface. Schnitzer et al. (1988) suggest that in most soils the transport of hydrophobic compounds (lipids) is a result of water soluble fulvic acids mobilizing hydrophobic compounds and depositing them on clay surfaces where adsorption follows.

The adsorption of hydrophobic organic compounds (HOC) has been studied by

several workers. Suggested mechanisms of adsorption of non-polar HOCs include hydrophobic adsorption (Hassett and Banwart 1989), solvent partitioning (Chiou 1990), and the distributed reactivity model (Young and Weber 1995). Hydrophobic organic compounds with polar functional groups sorb to soil particles via polyvalent cation bridging (Dinel et al. 1992).

Chiou (1990) describes soil as a dual sorbent. Hydrophobic adsorption of HOCs occurs on soil mineral particles but the soil organic matter functions as a partition medium. Soil organic matter acts as a solvent for the HOCs which then partition between the water and the organic matter.

Young and Weber (1995) looked at systems with organic matter in various stages of humification over a wide range of HOC concentrations. They describe a sorption process which combines hydrophobic adsorption and partitioning into a single model. Sorption occurs in a combination of amorphous, condensed, and microcrystalline regions in natural organic matter, with the microcrystalline regions increasing in importance in older (more altered) organic matter fractions. The central concept of their "distributed reactivity model" is that sorption processes in soils range along a spectrum from simple phase partitioning to purely surface adsorption. Partitioning dominates in amorphous regions of the soil organic matter and adsorption becomes increasingly important as crystallinity of the soil organic matter increases.

Dinel et al. (1992) found that significant quantities of lipids added to soil could not be extracted from soil by organic solvents. Treatment with Na-pyrophosphate or HCl-HF (1.6:2.5M) aqueous solution allowed extraction of these "more reactive" compounds. They concluded that these compounds were not hydrophobically adsorbed or dissolved in soil

organic matter but rather lipid polar functional groups were binding to the soil colloids via polyvalent cation bridges.

2.3.6.4 Lipids and Soil Structure. Until recent years there has been little evidence for strong interactions between soil minerals and the lipid fraction (McKeague et al. 1986). Soil lipids have long been known to function as a C source for soil microbes (Dinel et al. 1990) or to act as inhibitors of seedlings (Everett et al. 1995) and soil microorganisms (Fustec-Mathon et al. 1977). This lack of attention to soil lipids in soil structure studies was probably due in part to the definition of lipids themselves. Only a part of the total hydrophobic aliphatic C in soil is extractable by organic solvents alone, which is the common definition of a lipid (Ziegler 1989). As a result many workers now use the terms total aliphatic C and long-chain aliphatic C so as to include both bound and free lipids (Schnitzer et al. 1988; Ziegler 1989; Dinel et al. 1992).

The influence of hydrophobic long-chain aliphatic compounds (LCAs) on soil physical properties has become easier to study with the improvement in analytical methods such as pyrolysis field ionization mass spectrometry (Golchin et al. 1995; Monreal et al. 1995) and both solution and solid state NMR (Wilson 1987; Wilson 1990; Dinel et al. 1992; Golchin et al. 1995). LCAs produce hydrophobicity in sandy soils under burned forest slash (Everett et al. 1995) and have caused hydrophobicity resulting in decreased production and increased erosion of sandy soils in many regions of southern Australia (Ma'shum et al. 1989). A role for LCAs in soil structure has also been suggested by several workers (Capriel et al. 1990; Dinel et al. 1991a; Dinel et al. 1991b; Monreal et al. 1995). The addition of a hydrophobic stearic acid and a hydrophobic humic acid resulted in more persistent increases in aggregate stability than did the addition of a hydrophilic polysaccharide gum (Piccolo and

Mbagwu 1999).

Capriel et al. (1990) examined the supercritical hexane extract from soils under various crop, manure/fertilizer, and cultivation treatments. Wet aggregate stability was highly correlated with the aliphatic hydrophobic fraction. The LCAs were easily biodegradable after extraction. Therefore, their persistence in soil was attributed to protection afforded through physical association with soil minerals. Increased aggregate stability was associated with increased LCA content. The various management systems did not affect the quality of the LCAs but only their quantity. In contrast, Diné and Nolin (2000) found that not only were total extractable lipids more abundant under perennial than annual crops but the portion that was extractable with diethyl ether was also higher under the perennial crops indicating a change in lipid quality with a change in management.

Diné et al. (1991b) amended soil with humic (abundant LCAs) and fibric (largely carbohydrate) peat materials. The humic materials had a greater effect than the less decomposed fibric material, but a longer incubation time was needed for the effect of the humic material to become apparent. Further studies using a beeswax extract as the amendment showed that all the LCA fractions increased wet aggregate stability (Diné et al. 1991a; Diné et al. 1992). However the LCAs which became "associated" with clay particles (extractable only after treatment with Na-pyrophosphate) had a much greater effect on aggregate stability than the chloroform extractable "free" LCAs. The bound or "associated" LCAs were rich in acid and polar functional groups whereas the free LCAs consisted of neutral hydrophobic aliphatic compounds. The free LCA effect was attributed to the hydrophobic LCAs protecting other water soluble binding agents (for example carbohydrates) within the aggregates (Diné et al. 1991a). The larger effect due to the

associated LCAs was suggested to be the result of the formation of polyvalent cation bridges between LCA functional groups and clay surfaces (Dinel et al. 1992). Polyvalent cation bridges form much stronger bonds than those formed due to hydrophobic bonding of the free LCAs. CO₂ evolution data and NMR analysis indicated that the LCAs were not of microbial origin but rather microbial modified LCAs from the beeswax. Soil microorganisms play an essential role in the modification of aliphatic compounds and their subsequent incorporation into soil aggregates, contributing to improved soil aggregate stability (Dinel et al. 1992).

Wet aggregate stability of soil aggregates can clearly be enhanced by lipid-soil colloid associations. Few workers have considered the effects of lipids or LCA on aggregate stability of soils in the field but correlation studies which have included lipid or LCA clearly show an association which warrants further study.

2.3.7 Measurement of Soil Aggregate Stability

Aggregate stability has been described as the resistance of the bonds within aggregates to external forces of impact, shearing, abrasion, or slaking (Angers and Mehuys 1993). Aggregate stability can be measured for both dry aggregates and wet aggregates.

Yoder (1936) described a method of wet sieving aggregates to determine their relative stability. Aggregates were placed on a sieve of a certain size, wetted, and then the sieve was lowered and raised in water at a specific rate for a specified length of time. The aggregates remaining on the sieve were weighed and the amount of water stable aggregates in the sample was known for that particular disruptive force. His method has formed the basis for many of the methods in use today. Variations on the wet sieving method include placing a whole soil sample on a nest of progressively finer sieves and then wet sieving to

determine the size distribution of water-stable aggregates, often expressed as the mean weight diameter or MWD (Angers and Mehuys 1993). Others have used various combinations of wetting speed and vacuum (Dickson et al. 1991) or pre-wetting with alcohol (Paré et al. 1999) to reduce the slaking of aggregates during wetting and to compare the stability of slaked and unslaked aggregates during wet sieving.

The effect of soil conditioners on aggregate stability and hence soil porosity has been studied indirectly through their influence on infiltration rates (Mustafa et al. 1988). Curtin et al. (1994b) measured soil hydraulic conductivity to determine the effects on soil structural stability of varying the Ca to Mg ratio of the soil solution. A drip-type rainfall simulator has been used to estimate aggregate resistance to water-droplet impact in a study of surface seal formation (Shainberg et al. 1992). One advantage of the rainfall simulator was the ability to vary the kinetic energy in the water droplets by known amounts. However, the drops were falling on a bed of soil so the energy was not evenly distributed among the aggregates. The method was useful to compare the effect of rainfall events on a soil surface and resultant infiltration rates but was not as useful for comparing the stability of the individual aggregates within the bed.

Clay dispersibility has also been used as a measure of aggregate stability. Dispersed clay is determined gravimetrically (removal of an aliquot of suspension by pipette and weighing the clay after drying) or by light transmission through the clay suspension at a depth calculated by Stokes' Law (Angers and Mehuys 1993). The dispersive force can vary from minimal shaking by hand to 16 hours of shaking by a mechanical shaker (Curtin et al. 1994c).

Ultrasonic dispersion of clay has been suggested as a more precise method of

applying energy to soil aggregates than the arbitrary disruptive energy applied during wet sieving or shaking (Fuller and Goh 1992). Gregorich et al. (1988) compared ultrasonic dispersion and wet sieving methods and found that ultrasonic dispersion was a useful method for separating microaggregates from soil for the study of the nature of the organic matter in the microaggregates. Microaggregate stability as determined by ultrasonic dispersion proved to be a sensitive indicator of soil susceptibility to seal formation, runoff, and erosion (Levy et al. 1993). Stepwise ultrasonic dispersion has also been used effectively to liberate and fractionate soil organic matter and study C distribution within aggregates (Roscoe et al. 2000) and has also been shown to release immobilized acid phosphatase from soil (De Cesare et al. 2000). The ability to apply precise amounts of energy throughout a soil suspension is a major benefit of using ultrasonic dispersion in the study of bonding between clay particles within soil aggregates. The ability to standardize energy inputs among laboratories is another advantage to the method of ultrasonic clay dispersion.

2.4 Summary

The soil organic material which we group as the carbohydrate and lipid fractions of soil organic matter consist of an extremely diverse collection of compounds. Soil carbohydrates may be neutral, positively charged, or negatively charged and can range in size from simple sugars to large polymers. Soil lipids also vary greatly in size and although all lipids are hydrophobic, there can be large variations in functional groups. Due to this great variability, the types and strengths of bonds will be variable within soil aggregates.

Work to date has demonstrated that soil aggregates are weakened by removal of

either carbohydrates or lipid and are affected by the type or types of exchangeable cations present. Correlation studies have also shown differences in aggregate stability can be associated with differences in the quantity of carbohydrate or lipid or other soil organic matter constituents. Separating carbohydrates into light and heavy fractions has improved the correlations by eliminating the organic material which is not bound to mineral particles from the analysis. Unfortunately the comparison of aggregate stability in most of these studies has been done at only a few levels of disruptive energy (generally only one) or the comparisons were based on the amount of energy required to disperse one-half of the clay.

The comparison of aggregate stability or dispersion of clay from within aggregates over several levels of disruptive energy, ranging from dispersion of only very weakly held clay to dispersion of all the clay, will provide useful information about the variety of bonds present in the aggregates. Correlations among soil carbohydrates, soil lipids, and exchangeable cations at each energy level would then help us understand the factors that are most likely responsible for the most stable aggregates and those associated with the weakest bonds.

3. THE EFFECTS OF ZERO TILLAGE MANAGEMENT ON SOIL ORGANIC MATTER COMPOSITION AND CLAY DISPERSION FROM SOIL AGGREGATES OF A FORTIER SILTY CLAY AND AN OSBORNE CLAY

3.1 Introduction

Conversion of native prairie to cultivated crop land results in time in a reduction in soil organic matter content (Balesdent et al. 1988; Fuller et al. 1995; Quideau and Bockheim 1996). With cultivation, soil aggregate stability decreases (Tisdall and Oades 1980) and clay dispersion occurs more readily (Tisdall and Oades 1980; Fuller et al. 1995). Tisdall and Oades (1980) attributed the decrease in water stable aggregates to enhanced oxidation of organic binding agents as a result of tillage. The oxidation of organic binding agents and the resultant production of fulvic acids both contributed to the significant increase in clay dispersion following tillage of grassland soils. As aggregates break down and clay is dispersed, the soil becomes more susceptible to crusting and seal formation leading to a reduced infiltration rate of water, resulting in increased runoff and increased soil erosion (Zhang and Miller 1996).

Several organic binding agents have been found to be positively correlated with soil aggregate stability. Cheshire et al. (1983) and Angers and Mehuys (1989) used periodate treatments to oxidize the carbohydrate in soil samples and showed a close relationship between soil carbohydrate and aggregate stability. Fuller and Goh (1992) also found a significant positive correlation between dispersion of clay by sonication and carbohydrate

but suggest that the correlation is highly variable and dependent on the soil being studied. Monreal et al. (1995) separated their study soil into macroaggregates (greater than 250 μm diameter) and microaggregates (less than 250 μm diameter). Wet aggregate stability of the macroaggregates was significantly positively correlated to lignin dimers, lipids, sterols, organic C, and organic N but not to soil carbohydrate content. For microaggregates, only organic C was significant but the correlation was negative. Diné et al. (1992) greatly reduced wet aggregate stability of a heavy clay by extraction of lipid compounds.

Cropping practices affect soil organic matter content and aggregate stability. Increasing perennial forages in a crop rotation will increase soil organic C (Haynes 1993; Haynes 1999). Emmond (1971) reported that reducing the frequency of bare tilled fallow in a cereal rotation had a positive impact on soil aggregation. Hunt et al. (1996) found that after nine years of conservation tillage, the organic C content of the surface 10 cm of a loamy sand soil had almost doubled in comparison to conventionally tilled plots. Baldock and Kay (1987) compared several bromegrass-corn rotations and found that, despite finding no significant increase in soil organic C, the size and stability of aggregates increased as bromegrass became more dominant in the rotation. An increase in soil aggregate stability has been reported with annual crops under zero tillage management (Weill et al. 1989; Dormaar and Lindwall 1989).

Crop rotation and management affect clay dispersion (Curtin et al. 1994a). Determination of and, more importantly, prediction of dispersible clay using existing databases has received more interest since dispersible clay became one of the parameters used in the estimation of interrill erodibility in the Water Erosion Prediction Project (WEPP) model (Brubaker et al. 1992).

Ultrasonic dispersion of clay was used as a measurement of dispersibility of clay for prediction of seal formation, runoff, and erosion in South Africa (Levy et al. 1993). Fuller et al. (1995) used an ultrasonic probe type homogenizer to apply a series of energy levels to disperse clay from soil aggregates collected from adjacent cultivated fields and native prairie. The clay in the cultivated aggregates was held less tightly indicating that these soils would be more susceptible to surface crusting and water erosion.

The purpose of this study was to examine the influence of zero tillage management on soil organic matter composition and how this might influence the dispersion of clay from within soil aggregates. The relative importance of the carbohydrate and lipid components of the soil organic matter as well as the soil exchangeable cations was estimated through correlation with clay dispersed by ultrasonic energy.

3.2 Materials and Methods

3.2.1 Site Characterization

Two ongoing zero tillage studies located on soils of different texture and internal drainage were chosen for soil aggregate sampling.

The first study was located near Brunkild, Manitoba (49°30' N, 97°34' W; legal description, NE 19-6-1W) on a heavy clay soil of the Osborne series, a poorly drained Rego Humic Gleysol (Ehrlich et al. 1953). The surface 10 cm of the Ap horizon of the soil had a pH of 7.8, an electrical conductivity of 0.6 dS m⁻¹, and an average particle size analysis of 8.5 % sand, 18.5% silt, and 73.0% clay (Fitzmaurice 1996). The plot area had been under zero tillage management, cropped to canola and cereal grains, from 1982 to 1990. In the fall

of 1990 the area was divided into zero tillage and conventionally tilled plots to produce a randomized complete block design with four replicates. That fall the conventionally tilled plots were cultivated to a depth of 12 cm. Cultivation was repeated on these plots in spring before planting (1991), after harvesting (1991) and prior to planting the following spring (1992). In the spring of 1992, the plots were sown to spring wheat (*Triticum aestivum* cv. Katepwa) with an Amazone zero tillage hoe drill. Due to highly variable crop emergence the plot area was resown to barley (*Hordeum vulgare* cv. Argyle) on June 9. In preparation for replanting both zero tilled and conventionally tilled plots were harrowed with diamond tooth harrows to break up clods formed during the initial seeding operation (Fitzmaurice 1996).

The second site was located at the University of Manitoba, Plant Science Department research plots near Portage la Prairie, Manitoba (49°58' N, 98°16' W). This location is now part of the Manitoba Crop Diversification Centre Portage Sub-station. The site was located on a silty clay soil of the Fortier series, a Gleyed Cumulic Regosol (Michalyna and Smith 1972). In 1979, a trial was established in a randomized complete block design with one conventional-, and seven zero-tillage treatments with four replicates. Several different crop rotations were used until 1984. From 1985 to 1992 the rotations were reduced to a cereal (either wheat or barley) and canola rotation, two of the replicates were sown to spring wheat and two to canola (*Brassica napus* L) each year. The pH of the 0-15 cm depth had been found to be in the 7.6 to 7.8 range for both treatments in each year since 1987. Over the same time period the electrical conductivity had varied from a high of 0.9 dS m⁻¹ in 1989 to a low of 0.2 dS m⁻¹ in 1991 with the variation being year to year and not between treatments (Alvin Iverson, personal communication). The particle size analysis was quite variable

across the site with an average value of 13 % sand, 47 % silt, and 40 % clay for the 0-10 cm depth of the Ap horizon (clay content ranged from a low of 37% in replicate one to a high of 53% in replicate two).

3.2.2 Soil Sampling and Analyses

In 1992, samples were taken from the 0-10 cm depth at mid-tillering (June 15 at Portage la Prairie and July 17 at Brunkild) and again in fall following harvest but prior to fall tillage (Sept. 12 at Portage la Prairie and Oct. 5 at Brunkild). Surface crop residue, which was often several cm thick on zero tillage plots at the Portage la Prairie site, was brushed aside prior to sampling. The soil samples were taken by inserting the blade of a small spade horizontally at the 10 cm depth and removing about 2 kg of soil which was placed in a plastic bag. The samples were transported back to the laboratory, and gently spread to fully air dry before dry sieving. At the Brunkild site, four 2 kg sub-samples were taken from near the edges of each treatment in each replicate to avoid areas sampled by other researchers. The Portage la Prairie samples consisted of three 2 kg sub-samples from each of the conventional tillage plots and also from an adjacent zero tillage plot in each replicate. Sub-samples from each treatment within each replicate were bulked after air drying. Four separate replicates were used for each treatment. At Portage la Prairie, the 0-5 cm and 5-10 cm depths were sampled separately due to an obvious visual difference in soil structure between the 0-5 cm and the 5-10 cm depths.

Large soil clods were gently broken apart by hand and the soil which was able to pass through a 9.5 mm sieve collected for further separation and analyses. A sub-sample was removed from the less than 9.5 mm aggregate size fraction (ASF) for particle size analysis

according to the pipette method.

The aggregates less than 9.5 mm in diameter were then further divided by dry sieving into five aggregate size fractions (ASFs): less than 0.25 mm, 0.25 to 0.85 mm, 0.85 to 2.00 mm, 2.00 to 4.75 mm, and 4.75 to 9.5 mm. A nest of sieves was placed on an automatic sieving device and air dry aggregates added at a loading rate of $\leq 0.6 \text{ g cm}^{-2}$. Sieving time was 15 seconds. Each ASF was then weighed and the distribution of ASFs calculated as a percentage by weight of the total sample (air dry basis).

The 0.25 to 0.85 mm ASF was then chosen for detailed exchangeable cation, organic matter, and clay dispersion analyses. This ASF contains important aeration and water conducting pores which are crucial for crop growth (Oades, 1984).

Exchangeable Ca, Mg, K, and Na were displaced from the soil exchange sites by saturation with Ba (unbuffered 0.25 M BaCl_2 solution) and measured by atomic absorption spectrometry. Cation exchange capacity (CEC) was then determined by measuring the Ba in solution following saturation with Mg (0.25 M MgCl_2 solution). Organic carbon was determined by a modified Walkley-Black Method (Yeomans and Bremner 1988). Soil carbohydrate was extracted from the soil samples by filtration following acid hydrolysis with 1.5 M H_2SO_4 at 85°C for 24 hours (Angers and Mehuys 1989). Hexose content of the extract was determined colorimetrically by the anthrone method of Brink et al. (1960) using glucose as the standard. The content of hexose equivalents was used as an estimate of soil carbohydrate.

Extractable lipids were determined by the method of Diné et al. (1996). A 10 g sample of the 0.25-0.85 mm ASF was sequentially extracted with diethyl ether (DEE) and chloroform (CHCl_3) in a Soxhlet apparatus. Each extract was dried and weighed. The

sequential extraction separates extractable lipid into DEE extractable lipids (DEE-EL) which are easily biodegraded by soil microbes and CHCl_3 extractable lipids (CHCl_3 -EL) which are more bioresistant (Dinel et al. 1992). Summing together DEE-EL and CHCl_3 -EL gives total extractable lipid (TEL).

Dispersible clay from within soil aggregates was determined by sonication. A probe-type ultrasonic homogenizer (Braun Sonic 2000) was used as per the method of Fuller and Goh (1992). Air dry aggregates (equivalent to 1.0 g oven dry) were placed in a 50 mL beaker to which 25.0 mL of distilled water was added slowly. The beaker was clamped at a 20° angle and the water added along the side of the beaker at a rate of less than 0.5 mL per minute so as to wet the aggregates from below and minimize aggregate disruption during wetting. After the aggregates were well covered with water, the rate was increased to 5 to 7 mL per minute. The beaker was then placed in an ice bath, the ultrasonic homogenizer probe inserted to a depth of 13 mm, and the specified energy applied. The different levels of applied energy were achieved by using a constant energy output and varying the length of time of sonication. Dispersed clay was determined by the pipette method. Each level of sonication was applied to freshly wetted aggregates in duplicate. For the Osborne clay, energy levels of 10, 25, 50, 100, 200, 300, 600 kJ L^{-1} were used to produce the clay dispersion curves. The Fortier silty clay had smaller amounts of weakly bound clay and was treated with energy levels of 50, 100, 200, 300, 400, 500, and 600 kJ L^{-1} to produce clay dispersion curves. The 1500 kJ L^{-1} energy level was used to determine the value for "maximum dispersible clay". Clay dispersion at each energy level was expressed as a decimal fraction of maximum dispersible clay.

Statistical analyses were performed using the General Analysis of Variance, Linear

Regression, and Forward Selecting Stepwise Linear Regression procedures of Statistix version 1.0 (Analytical Software 1996.). The effect of tillage on clay dispersion was evaluated by using analysis of variance to compare clay dispersion for the four field replicates at each energy level (RCB). Variation across energy levels was assessed using a split plot design with tillage treatment as main plot and energy level as subplot treatment. A square hyperbola equation was fitted to each clay dispersion curve to determine the amount of energy needed to disperse one-half of the clay. Correlations between soil parameters and clay dispersion were performed at each energy level for each tillage treatment for the Fortier silty clay (Portage la Prairie site).

3.3 Results and Discussion

3.3.1 Soil Aggregate Size Distribution

Regardless of season or tillage treatment, over 65% by weight of the Osborne clay was in aggregates or clods over 9.5 mm in diameter (Table 3.1). In contrast, an Osborne clay soil which had been in undisturbed grass for over 20 years had less than 25% of its aggregates larger than 9.5 mm and contained a high content of worm casts, as well as earthworms. Tillage did not have a large impact on aggregate size distribution for the Osborne clay but conventional tillage did result in 1-2% more of the total soil in the 0.25-0.85 mm ASF. The dominant soil structure type was subangular blocky and was similar in both zero and conventionally tilled plots.

In contrast, the Fortier silty clay showed different soil structure between tillage treatments and also between depths within a treatment. The reason for dividing the Fortier

silty clay samples into 0-5 and 5-10 cm depths was the initial observation in the field, which was corroborated by the dry sieving results, that about half of the soil in the 5-10 cm depth of the zero tillage treatments was made up of aggregates of 2.00-4.75 cm in diameter compared to only about a quarter of the soil falling into that ASF from the surface 5 cm. Tillage treatments also resulted in clear differences in soil structure. The dominant structure type in the zero tillage plots was angular blocky, whereas granular and subangular blocky structure dominated in the conventionally tilled plots.

Table 3.1 Percent of total aggregates within each aggregate size fraction for an Osborne clay and a Fortier silty clay under various management regimes.

Management type	Sample depth (cm)	Aggregate size fraction (mm)					
		<0.25	0.25-0.85	0.85-2.00	2.00-4.75	4.75-9.5	>9.5
Osborne clay - midtillering							
zero tillage	0-10	0.79	4.65	6.17	9.02	9.55	69.81
conventional tillage	0-10	0.91	6.65	8.31	9.56	8.15	66.41
grass - yard site	0-10	1.60	4.96	13.15	36.43	18.92	24.94
Osborne clay - fall							
zero tillage	0-10	0.43	2.76	4.20	6.11	6.82	79.67
conventional tillage	0-10	0.51	4.12	6.22	8.03	7.73	73.40
Fortier silty clay - midtillering							
zero tillage	0 to 5	2.77	11.61	19.38	23.84	11.40	31.00
	5 to 10	1.30	6.05	16.08	47.60	17.98	11.00
conventional tillage	0 to 5	9.61	33.94	20.07	11.14	6.05	19.20
	5 to 10	3.65	14.46	14.71	16.57	11.61	39.00
Fortier silty clay - fall							
zero tillage	0-5	1.09	5.61	11.05	15.99	11.88	54.38
	5-10	0.94	5.35	13.82	32.30	17.30	30.30
conventional tillage	0-5	5.82	26.19	20.04	13.12	10.36	24.47
	5-10	1.76	10.58	14.28	17.35	14.26	41.76
grassed alley	0-5	0.88	4.02	9.43	18.54	11.87	55.25
	5-10	0.75	3.28	6.21	14.56	11.85	63.34

For all tillage treatments at both depths the amount of soil in the two smallest ASFs decreased from mid-tillering to fall. In their model of aggregate hierarchy, Oades and Waters (1991) attribute the binding of microaggregates (ASF less than 0.25 mm) into macroaggregates (ASF greater than 0.25 mm) and the binding of macroaggregates into larger macroaggregates to the direct effects of roots and fungal hyphae. The proliferation of roots and hyphae between mid-tillering and harvest may well explain the decrease in the quantity of aggregates less than 0.85 mm in diameter by crop maturity. Following maturity roots would begin to die and decompose loosening their hold on soil aggregates. Tillage would then break some of the roots and hyphae resulting in an increase in the smaller ASFs again. Even without tillage, the action of freezing and thawing and the swelling and shrinking associated with seasonal soil moisture changes on clayey soils may disrupt some of these root and hyphal bonds and result in an increase in the smaller aggregate sizes by the following spring.

3.3.2 Soil Organic Matter and Tillage

3.3.2.1 Osborne Clay. Soils under conventional tillage contained 47 g of organic C kg⁻¹ soil in the surface 10 cm at both sampling times. The zero tillage plots showed an increase in organic C from 51 g kg⁻¹ at mid-tillering to almost 55 g kg⁻¹ of soil by the time of fall sampling. This change is equivalent to approximately 4 Mg C ha⁻¹ or about 10 Mg ha⁻¹ of soil organic matter which is not possible under these conditions and must be an artefact of sampling. The resultant statistically significant difference in organic C content under zero tillage and conventional tillage at the fall sampling (Table 3.2) must also be disregarded. At mid-tillering there was 20% more carbohydrate measured under zero tillage than with

conventional tillage. By fall there was a decrease in carbohydrate under zero tillage and a slight increase under conventional tillage with the result that the difference was no longer significant. DEE-EL and TEL were significantly higher under zero tillage at both sampling times, whereas, CHCl_3 -EL was not affected by tillage. In contrast, Paré et al (1999) found that zero tillage resulted in a lower concentration of all extractable lipid fractions and suggested that minimal incorporation of residue under zero tillage reduced the rate of residue decomposition and hence reduced total soil lipids. Osborne clay soils are by nature poorly drained (Ehrlich et al. 1953) and in both 1991 and 1992 parts of the plot area were inundated on several occasions. Fitzmaurice (1996) reported that during the 1992 growing season oxygen diffusion rates (ODR) below the 5 cm depth were less than the minimum needed for root growth and were often low enough in the surface 5 cm to negatively affect root growth. Due to high soil moisture and low soil oxygen levels, one could expect that surface residue would decompose more quickly than those incorporated during tillage, with the result that higher levels of extractable lipid were found under zero tillage.

3.3.2.2 Fortier Silty Clay. Conventionally tilled plots and zero tillage plots showed no significant differences in the contents of any organic fractions measured for the 0-5 cm depth at mid-tillering (Table 3.3). By the fall, carbohydrate had decreased by 45% under conventional tillage and by 42% under zero tillage. There was less variability at the time of fall sampling and zero tillage had significantly more carbohydrate in the surface 5 cm. None of the soil organic matter fractions measured were significantly affected by tillage at the 5-10 cm depth for either sampling time.

Table 3.2 Effect of tillage management on several soil organic matter fractions and exchangeable cations of the 0.25-0.85 mm ASF from the surface 10 cm of an Osborne clay, Brunkild, MB.

	tillage treatment means			
	at mid-tillering ^z		at fall	
	conventional	zero tillage	conventional	zero tillage
	g kg ⁻¹ soil		g kg ⁻¹ soil	
organic C	47.5 (0.9) ^y	51.2 (1.6) ^{ns}	47.0 (1.6)	54.6 (1.3) *
total hexose equivalents	8.56 (0.37)	10.41 (0.34) *	8.75 (0.43)	9.31 (0.63) ^{ns}
TEL	0.397 (0.004)	0.504 (0.018) *	0.424 (0.018)	0.565 (0.015) *
CHCl ₃ -EL	0.078 (0.003)	0.083 (0.011) ^{ns}	0.063 (0.007)	0.082 (0.004) ^{ns}
DEE-EL	0.319 (0.006)	0.421 (0.025) *	0.361 (0.015)	0.484 (0.011) *
DEE-EL : CHCl ₃ -EL	4.09 (0.21)	5.55 (1.19) ^{ns}	5.97 (0.78)	5.97 (0.22) ^{ns}
CHCl ₃ : TEL	0.20 (0.01)	0.17 (0.02) ^{ns}	0.15 (0.01)	0.14 (0.01) ^{ns}
	cmol kg ⁻¹ soil		cmol kg ⁻¹ soil	
CEC	47.1 (1.4)	48.0 (2.1) ^{ns}	44.2 (1.7)	45.4 (1.7) ^{ns}
Ca	17.3 (0.6)	17.4 (0.8) ^{ns}	16.5 (0.6)	15.9 (0.8) ^{ns}
Mg	8.41 (0.11)	8.11 (0.23) ^{ns}	7.11 (0.20)	7.01 (0.16) ^{ns}
K	3.76 (0.23)	3.97 (0.05) ^{ns}	3.51 (0.07)	4.01 (0.03) **
Na	0.36 (0.08)	0.22 (0.03) ^{ns}	0.22 (0.12)	0.08 (0.02) ^{ns}
ESP (%)	0.78 (0.18)	0.47 (0.09) ^{ns}	0.53 (0.29)	0.19 (0.06) ^{ns}

^z statistical comparisons are between tillage treatments within the same sampling time, ** zero tillage mean is significantly different from conventional tillage at the 1% level (LSD), * zero tillage mean is significantly different from conventional tillage at the 5% level (LSD), ^{ns} zero tillage mean is not significantly different from conventional tillage (LSD)

^y standard error of the mean

Table 3.3 Effect of conventional tillage (CT) and zero tillage (ZT) on several soil organic matter fractions and exchangeable cations of the 0.25-0.85 mm ASF of a Fortier silty clay, Portage la Prairie, MB.

	tillage treatment means							
	at mid-tillering ^z				at fall			
	0 - 5 cm depth		5 - 10 cm depth		0 - 5 cm depth		5 - 10 cm depth	
	CT	ZT	CT	ZT	CT	ZT	CT	ZT
	g kg ⁻¹ soil							
organic C	50.0 (0.8) ^y	50.3 (1.6) ^{ns}	46.3 (1.6)	43.8 (1.1) ^{ns}	51.0 (0.8)	52.6 (0.6) ^{ns}	48.2 (1.7)	44.8 (1.5) ^{ns}
hexose equivalents	8.33 (0.66)	9.09 (0.66) ^{ns}	7.04 (0.30)	7.65 (0.56) ^{ns}	4.58 (0.09)	5.25 (0.08) ^{**}	4.77 (0.26)	4.30 (0.10) ^{ns}
TEL	0.46 (0.02)	0.49 (0.03) ^{ns}	0.32 (0.01)	0.34 (0.02) ^{ns}	0.29 (0.19)	0.46 (0.12) ^{ns}	0.25 (0.02)	0.23 (0.02) ^{ns}
CHCl ₃ -EL	0.11 (0.01)	0.11 (0.01) ^{ns}	0.07 (0.002)	0.07 (0.01) ^{ns}	0.12 (0.08)	0.10 (0.06) ^{ns}	0.02 (0.01)	0.01 (0.003) ^{ns}
DEE-EL	0.36 (0.02)	0.38 (0.03) ^{ns}	0.25 (0.01)	0.27 (0.02) ^{ns}	0.23 (0.06)	0.35 (0.08) ^{ns}	0.23 (0.01)	0.22 (0.02) ^{ns}
DEE-EL : CHCl ₃ -EL	3.49 (0.30)	3.55 (0.52) ^{ns}	3.38 (0.19)	3.94 (0.68) ^{ns}	1.40 (0.09)	7.94 (4.79) ^{ns}	17.8 (6.0)	26.7 (7.8) ^{ns}
CHCl ₃ -EL : TEL	0.23 (0.01)	0.23 (0.03) ^{ns}	0.23 (0.01)	0.21 (0.03) ^{ns}	0.42 (0.02)	0.22 (0.07) ^{ns}	0.07 (0.02)	0.05 (0.02) ^{ns}
	cmol kg ⁻¹ soil							
CEC	35.2 (0.9)	33.7 (1.2) ^{ns}	38.6 (1.8)	36.3 (1.5) ^{ns}	-	-	-	-
Ca	12.5 (0.2)	12.8 (0.6) ^{ns}	12.5 (0.6)	12.1 (0.7) ^{ns}	-	-	-	-
Mg	5.49 (0.31)	5.17 (0.22) ^{ns}	5.62 (0.45)	5.23 (0.34) ^{ns}	-	-	-	-
K	1.93 (0.12)	2.18 (0.13) ^{ns}	1.46 (0.02)	1.26 (0.07) ^{ns}	-	-	-	-
Na	0.16 (0.01)	0.11 (0.01) ^{**}	0.15 (0.01)	0.12 (0.01) [*]	-	-	-	-
ESP (%)	0.46 (0.03)	0.32 (0.03) [*]	0.40 (0.03)	0.34 (0.04) ^{**}	-	-	-	-

^z statistical comparisons are between tillage treatments within the same sampling time and sampling depth, ^{**} zero tillage mean is significantly different from conventional tillage at the 1% level (LSD), ^{*} zero tillage mean is significantly different from conventional tillage at the 5% level (LSD), ^{ns} zero tillage mean is not significantly different from conventional tillage

^y standard error of the mean

Although in contradiction with the commonly held belief that zero tillage will increase soil organic C, the lack of a difference in organic C levels for the two tillage treatments is not unique to this study. Several researchers have found that a change from conventional to zero tillage had no effect on soil organic C. Campbell et al. (1999) found no increase in soil organic C after 12 years of zero tilled wheat on a semiarid silt loam (Brown Chernozem). In a moister location, Monreal et al. (1997) also found no effect of tillage on soil organic C content of the 0-8 cm depth of two Gray Brown Luvisol soils. In contrast, for a soil with very low initial organic C content, Hunt et al. (1996) reported that after 9 to 14 years the soil organic C content of a row cropped loamy sand soil was almost twice as high under zero tillage than under conventional tillage (1.2% and 0.72% respectively).

Soil hexose equivalents as determined by the anthrone method are a good estimate of relative levels of soil polysaccharides (Brink et al. 1960). Soil polysaccharides are derived from plant roots, plant debris, and the mucilages associated with roots or soil microbes (Oades 1984). The carbohydrate components would decompose more readily than the lipid compounds (Dinel et al. 1990) and therefore we would expect the carbohydrate levels to decrease by fall more so than the lipids.

3.3.3 Exchangeable Cations

Tillage treatment did not significantly affect the cation exchange capacity or the amounts of exchangeable Ca, Mg, or Na in the surface 10 cm of the Osborne clay. However zero tillage did result in a significant increase in exchangeable K (Table 3.2). Follet and Peterson (1988) attributed higher K levels in the surface 10 cm under zero tillage management to the accumulation of crop residue on the soil surface and higher soil organic

matter levels. As K is easily removed from cereal straw by leaching with water (Schomberg and Steiner 1999), the increase in exchangeable K could be attributed to crop uptake from soil below 10 cm, later leaching from crop residue and being held on the exchange sites near the surface. Fixation of this K by soil clay would be minimal as the clay minerals present in Osborne soils are mainly montmorillonite and illite with only small amounts of vermiculite present (Peter Haluschak, unpublished data). This process would have continued over the entire plot area while under zero tillage management (1982-1990). During cultivation, the surface residue and high K soil layer are mixed into the top 10-15 cm of the soil. Such mixing would both directly reduce exchangeable K concentration near the surface and increase the likelihood of K leaching below the sampling depth.

For the Fortier silty clay only exchangeable Na was significantly influenced by tillage (Table 3.3). Exchangeable Na and exchangeable Na percentage (ESP) were significantly reduced at both depths under zero tillage. Na is held less tightly than Ca, Mg, or K by the soil cation exchange sites leading to preferential leaching of Na. Hence, higher infiltration rates and increased moisture due to snow trap by standing stubble in the zero tillage plots could explain the reduction in Na under zero tillage. Although not directly measured at this site, higher infiltration rates would be expected under zero tillage. When compared to conventionally tilled treatments, higher infiltration rates in zero tilled treatments have been previously measured on a clay loam under zero tillage barley (Singh et al. 1996) and in silt loam and sandy loam soils (Azooz and Arshad 1996). Surface mulch, as is found in zero tillage fields, can increase the fraction of precipitation which is stored in a soil as well as the wetting depth following a precipitation event (Ji and Unger 2001) and, therefore, favours increased leaching.

3.3.4 Dispersion of Clay

The Osborne clay had over one half of its clay dispersed by ultrasonic energy of only 50 kJ L⁻¹ and as a result sonication levels of 10 and 25 kJ L⁻¹ were included (Figure 3.1). An output of 10 kJ L⁻¹ was as low as we were able to apply with our unit. At mid-tillering there was no significant effect of tillage on dispersion of clay from the 0.25 to 0.85 mm ASF. By fall, clay dispersion over the 100 to 200 kJ L⁻¹ range was significantly higher under conventional tillage (on average 4.3 % more clay was dispersed from the conventional tillage treatments, $p = 0.039$). Of the individual comparisons at each energy level, only the difference at 200 kJ L⁻¹ was significant (27% more clay was dispersed from the conventional tillage treatment, $p = 0.015$). Organic C, TEL, and DEE-EL were significantly higher under zero tillage at the fall sampling (Table 3.2) and a role for them might be suspected. However the tillage effects on TEL and DEE-EL were just as great at mid-tillering with no apparent effect on clay dispersion. Seasonal changes in clay dispersibility were also not significant, although there was a trend for zero tillage to be slightly less dispersible in fall while conventional tillage was slightly less dispersible at mid-tillering (Figure 3.2 and 3.3, Table 3.4).

Multiple regression analyses were performed on the organic and mineral components separately (Appendix III). The models with only mineral parameters were in general better than those containing only organic parameters but for the majority of energy levels, the models which included both organic and mineral parameters produced the best relationships (better adjusted r^2 values and better p -values). However, many of the best models were not significant ($p > 0.05$) and no pattern was apparent in the model parameters.

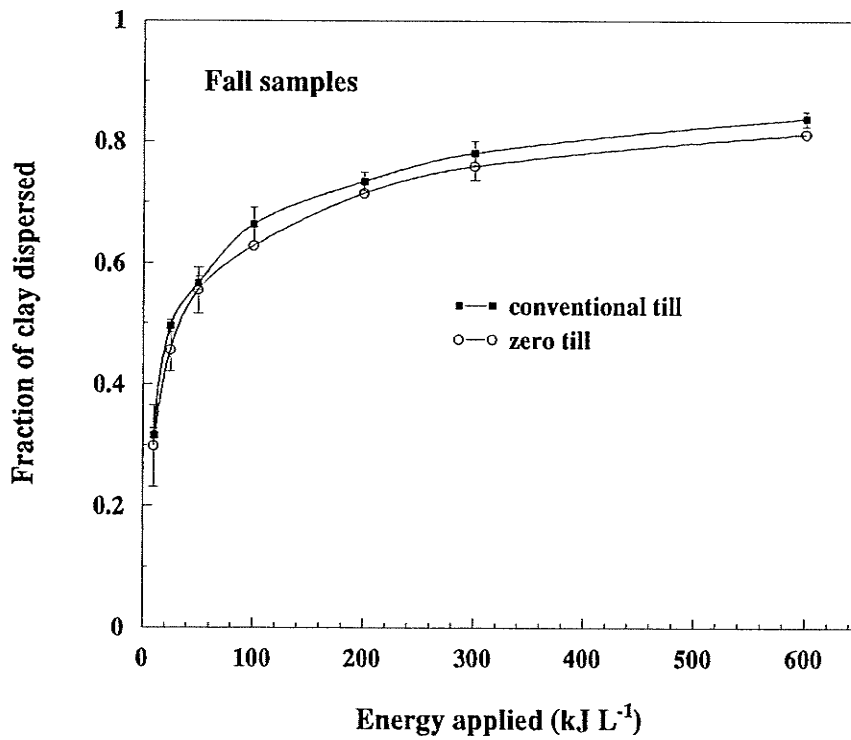
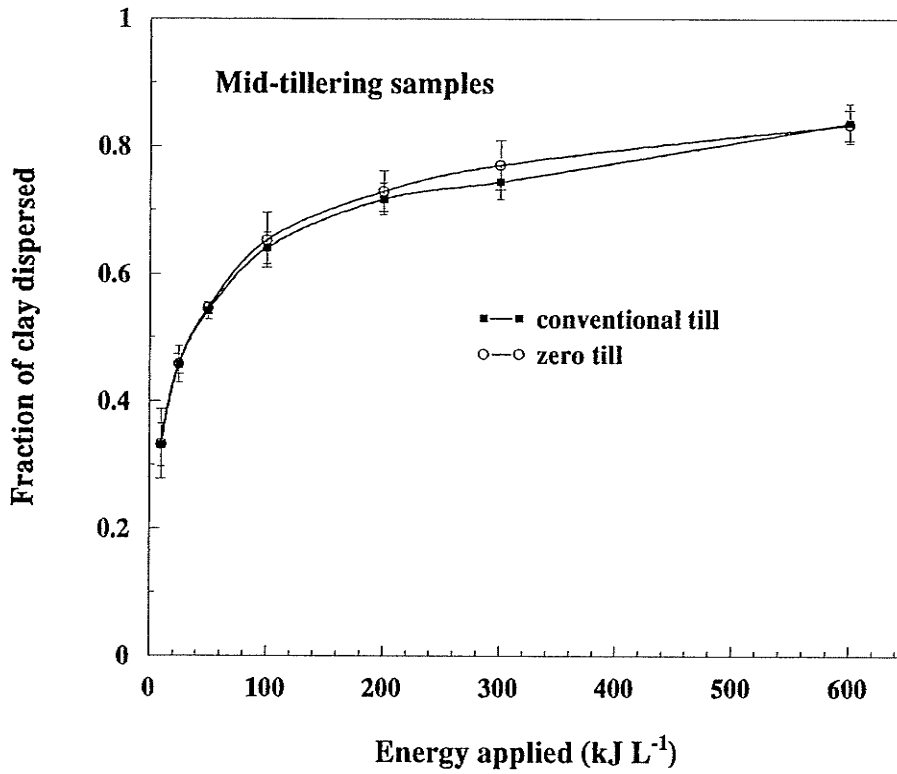


Figure 3.1 Dispersion of clay from within soil aggregates of the 0.25 to 0.85 mm ASF of an Osborne clay sampled at mid-tillering and in fall (error bars = 2 x standard error of the mean).

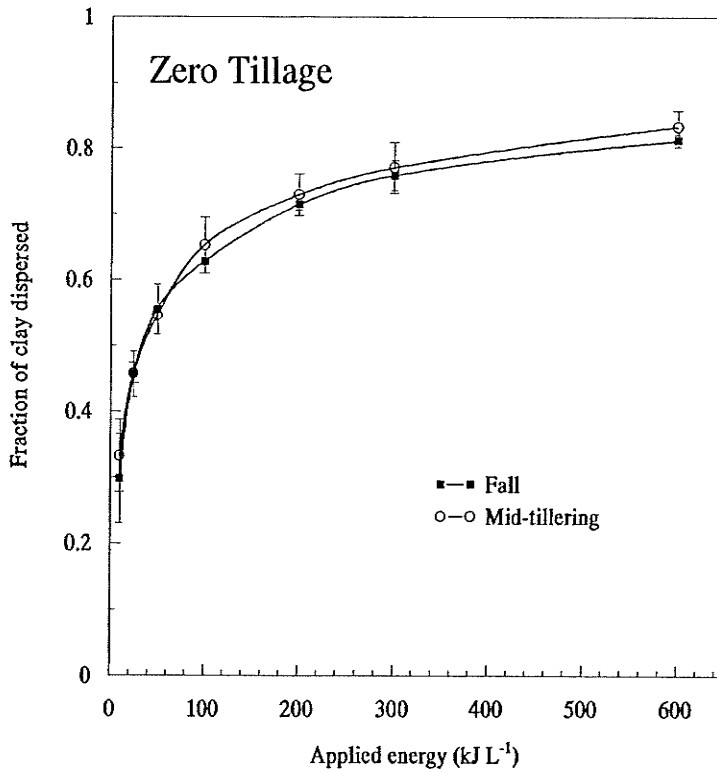
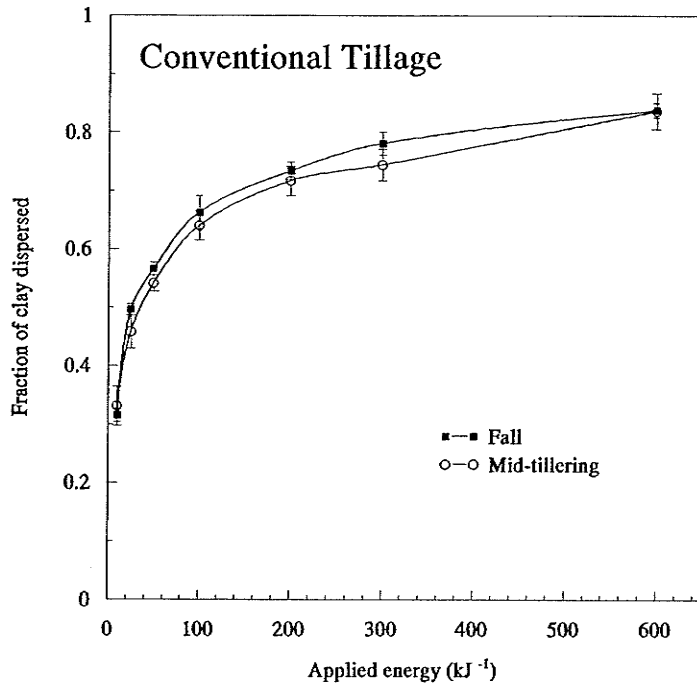


Figure 3.2 Effect of sampling time on the dispersibility of clay from aggregates of the 0-10 cm depth from an Osborne clay (error bars = 2 x standard error of the mean).

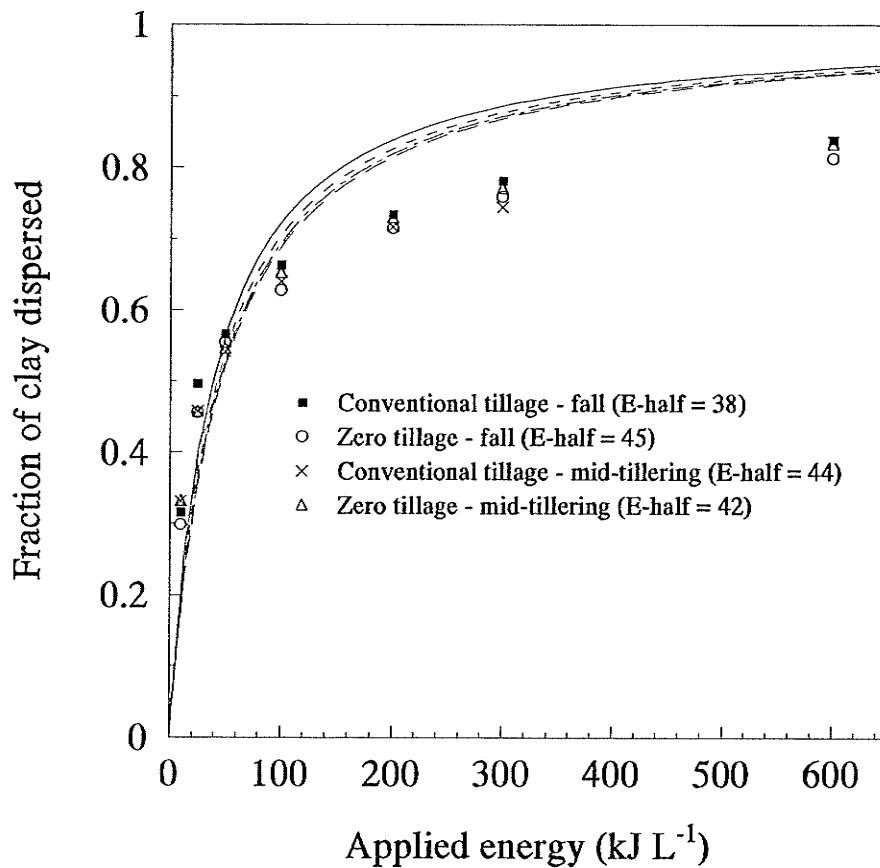


Figure 3.3 Fraction of clay dispersed at selected energy levels for the 0.25 to 0.85 mm ASF of the 0 to 10 cm depth of an Osborne clay. Plotted functions are for the best fitting square hyperbola equation for each treatment and sampling time. The E-half values represent the energy needed to disperse half of the clay and are in kJ L⁻¹. E-half values are determined from the equation of best fit.

Table 3.4 Values for E-half and the corresponding r^2 values determined by fitting the equation for a square hyperbola to clay dispersion curves from the 0.25 to 0.85 mm ASFs of an Osborne clay and a Fortier silty clay.

sample time and treatment	E-half (kJ L^{-1})	r^2 value
Osborne clay		
mid-tillering - conventional tillage	44	0.63
- zero tillage	41	0.69
fall - conventional tillage	38	0.71
- zero tillage	45	0.67
Fortier silty clay 0-5 cm depth		
mid-tillering - conventional tillage	116	0.99
- zero tillage	102	0.99
fall - conventional tillage	80	0.96
- zero tillage	91	0.98
Fortier silty clay 5-10 cm depth		
mid-tillering - conventional tillage	90	0.99
- zero tillage	79	0.99
fall - conventional tillage	56	0.90
- zero tillage	66	0.64

The study area had been managed under uniform zero tillage for many years and the tilled treatments had only been tilled four times since the beginning of the study. The wet spring and poor crop emergence necessitated re-seeding which required harrowing both conventional- and zero-tilled plots, and a second seeding operation with the hoe drill. The extra soil and residue disturbance resulted in little observable difference in residue cover among the two tillage treatments. Although some differences in soil organic matter fractions and clay dispersion had begun to develop by 1992, more time should be allowed to properly assess the effect of tillage at this site. At mid-tillering, the conventional tillage treatment in this study had an average soil organic C level of 47.5 g kg^{-1} of soil, a value midway between

the zero tillage treatment and an adjacent conventionally tilled field that had organic C contents of 51.2 and 43.8 g kg⁻¹ respectively.

The Fortier silty clay showed different results (Figures 3.4 and 3.5). At mid-tillering, the 0-5 cm depth of the conventional tillage treatment had significantly lower clay dispersion across the entire energy range ($p = 0.0142$) with a highly significant difference over the 200 to 400 kJ L⁻¹ range ($p = 0.009$). However at mid-tillering there was no tillage effect on any of the organic matter fractions measured (Table 3.3). By fall there was no longer any difference in clay dispersion between tillage treatments but differences in total carbohydrate and organic C were found.

The 5-10 cm depth displayed no effect of tillage on clay dispersion at mid-tillering (Figure 3.4). At the fall sampling, the zero tillage treatment did show lower clay dispersion values, though only those from the 300 and 400 kJ L⁻¹ levels were significantly different ($p = 0.0495$) (Figure 3.5). As was found for the mid-tillering surface samples, no significant differences were seen in the soil organic matter fractions measured (Table 3.3).

Missing data points for the lipid measurements and large variations in organic constituent levels within treatments contributed to the difficulty in assigning differences in clay dispersion to tillage effects on soil organic matter.

Further analysis, revealed a general pattern in clay dispersion in both the Osborne clay and Fortier silty clay sites. In the Osborne clay this presented itself as equivalent clay dispersion for both tillage treatments at mid-tillering. Yet by fall, conventional tillage has a greater clay dispersion which was significant over the 100 to 200 kJ L⁻¹ range (Figure 3.2).

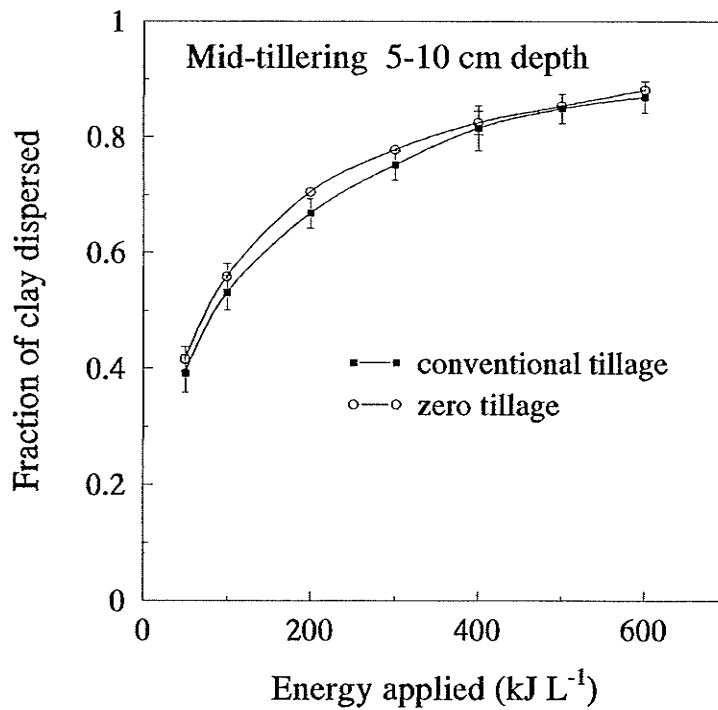
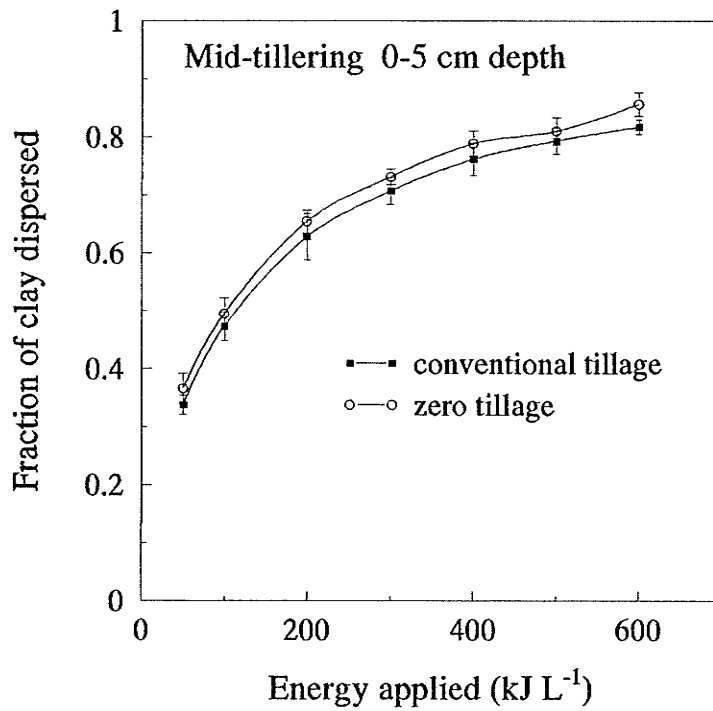


Figure 3.4 Dispersion of clay from soil aggregates of the 0.25 to 0.85 ASF of a Fortier silty clay sampled at mid-tillering (error bars = 2 x standard error of the mean).

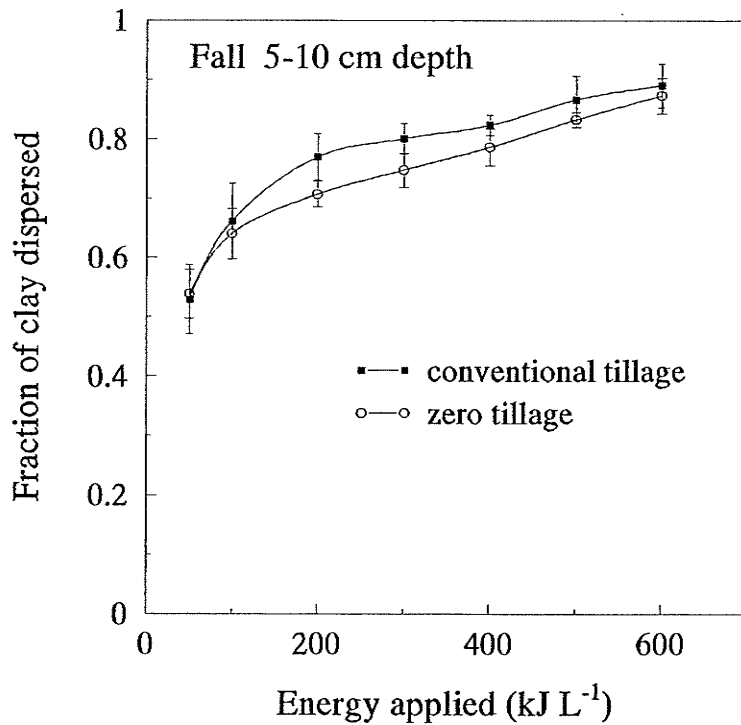
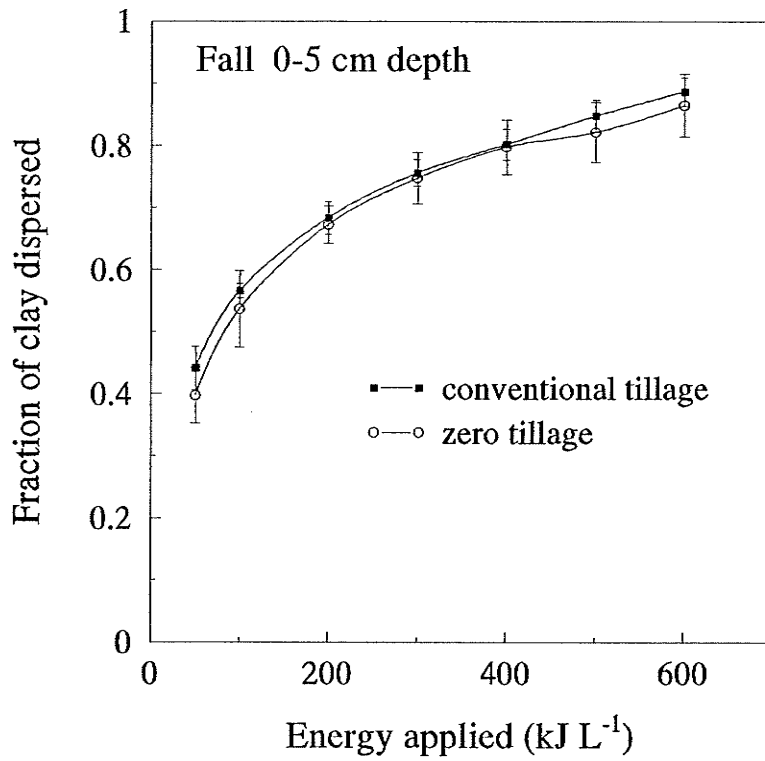


Figure 3.5 Dispersion of clay from soil aggregates of the 0.25 to 0.85 ASF of a Fortier silty clay sampled in fall (error bars = 2 x standard error of the mean).

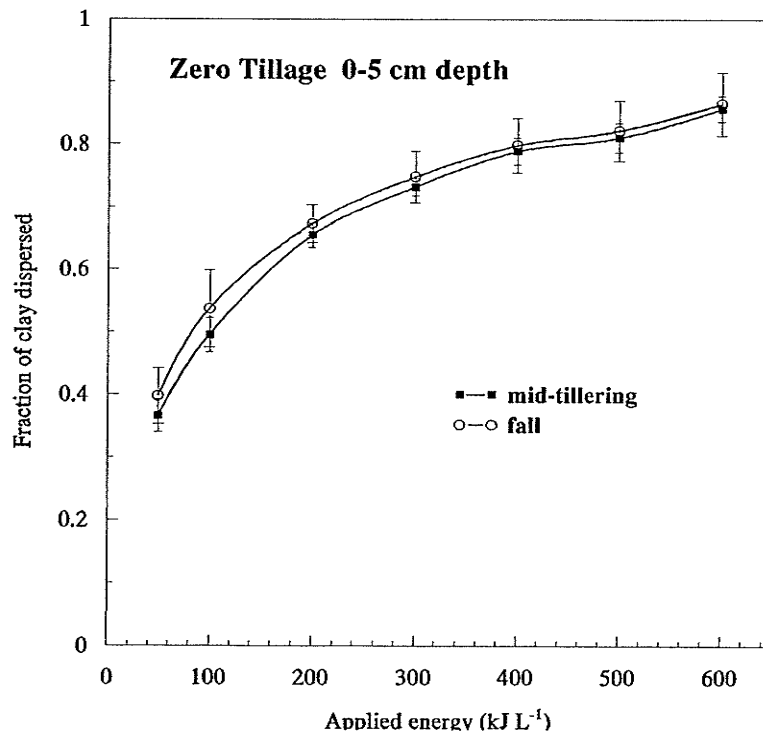
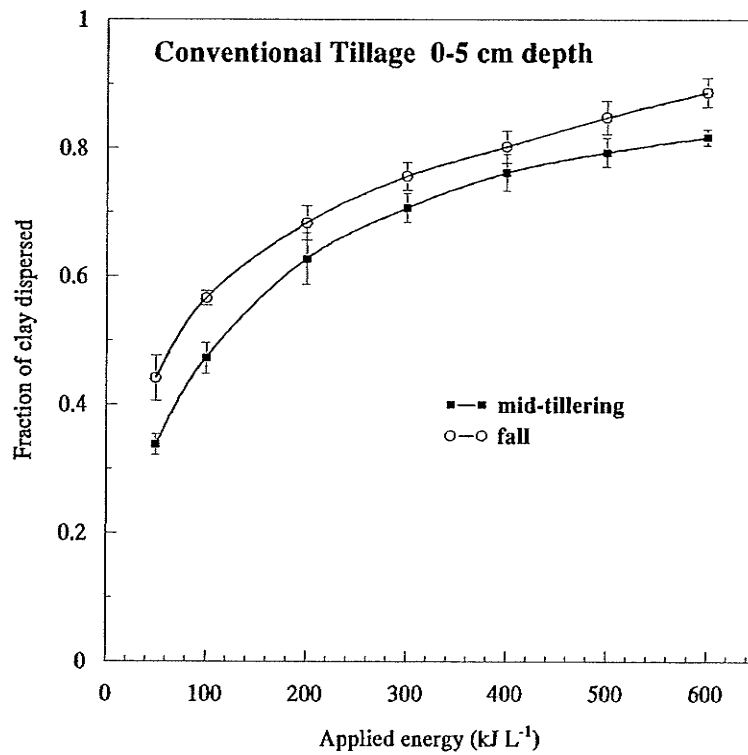


Figure 3.6 Effect of sampling time on the dispersibility of clay from aggregates of the 0-5 cm depth from a Fortier silty clay (error bars = 2 x standard error of the mean).

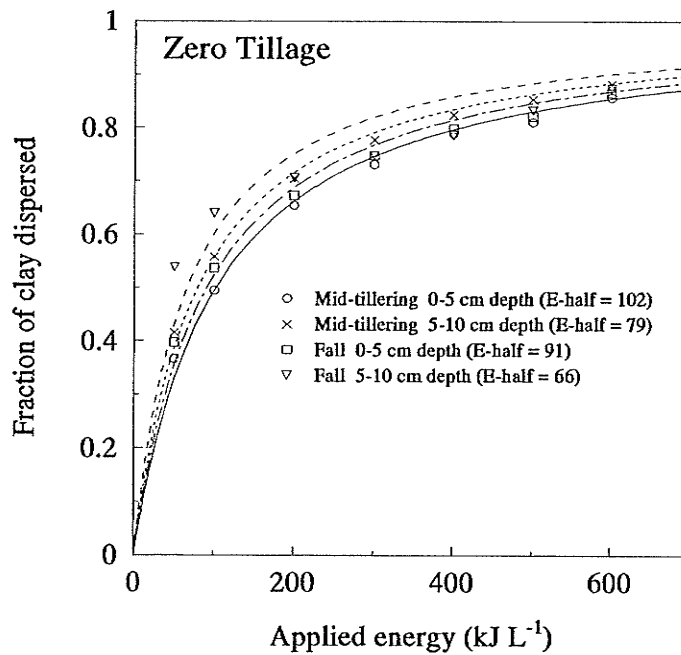
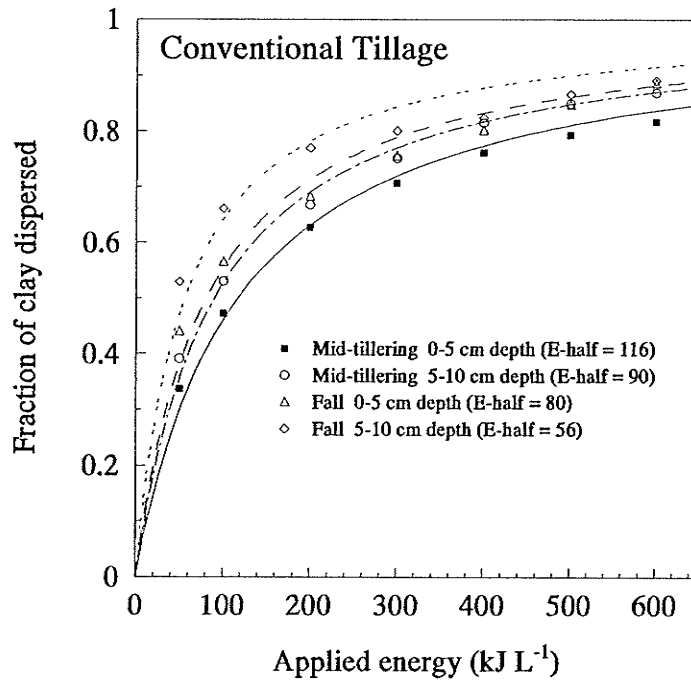


Figure 3.7 Fraction of clay dispersed at selected energy levels for the 0.25 to 0.85 mm ASF of the 0 to 10 cm depth of a Fortier silty clay. Plotted functions are for the best fitting square hyperbola equation for each treatment, depth, and sampling time. The E-half values represent the energy needed to disperse half of the clay and are in kJ L^{-1} . E-half values are determined from the equation of best fit.

At mid-tillering the 0-5 cm depth of the Fortier silty clay under zero tillage resulted in greater clay dispersion (Figure 3.4) but by fall the two tillage treatments had virtually the same dispersion curves (Figure 3.5). This pattern results from the greater seasonal variation in clay dispersion under conventional tillage. For both soils, conventional tillage shows a trend to an increase in clay dispersion from mid-tillering to fall (Figures 3.2 and 3.6). The difference was quite large for the Fortier silty clay conventional tillage treatment (average difference of 0.07 across all energy levels), whereas zero tillage shows a much smaller change (average difference of 0.02 across all energy levels). The E-half value for conventional tillage decreased by 36 kJ L⁻¹ (from 116 to 80 kJ L⁻¹) between mid-tillering and fall and for zero tillage decreased by only 11 kJ L⁻¹ (from 102 to 91 kJ L⁻¹) over the same time period (Table 3.4, Figure 3.7). Conventional tillage is characterized by a cycle of substantial mechanical disturbance prior to seeding, followed by three months of crop growth during which aggregate building can occur followed by mechanical disruption as soils are tilled following harvest. In a tillage cycle such as this, more seasonal variation in aggregation would be expected than in a zero tillage system which is based upon minimal mechanical disturbance of the soil. However, the aggregate size distribution under zero tillage also undergoes a large shift during the growing season (Table 3.1). For example, for the 0-5 cm depth of the conventional tilled soil, 63.6% and 52.1% of the soil was in aggregates less than 2.00 mm in diameter at mid-tillering and in fall respectively. Under zero tillage the difference was greater with 33.8% and 17.8% of the soil in aggregates less than 2.00 mm in diameter at mid-tillering and in fall respectively. Both treatments result in smaller aggregates being bound together into larger aggregates during the growing season. A change in aggregate size distribution cannot therefore explain the much greater seasonal

variation in clay dispersion under conventional tillage for the Fortier silty clay. At the very least however we are not looking at the identical aggregate pool in the 0.25-0.85 mm ASF in fall as at mid-tillering in either treatment.

Linear regression was used to more closely examine the effect of soil organic matter constituents and exchangeable cations on the dispersibility of the clay in the Fortier silty clay. Exchangeable cation and CEC values for the fall samples of the Fortier silty clay were not available and the mid-tillering values were used as the best approximation. All available data points for both treatments and both sampling times were pooled. Overall, total carbohydrate proved to correlate with clay dispersion better than any other single parameter (Table 3.5) and in all cases clay dispersion was reduced as carbohydrate concentration increased (coefficient less than 0). Only at the 200 kJ L⁻¹ level was total carbohydrate not the best correlation but it was a close second to organic C. Despite the correlation being significant at most energy levels, the highest r^2 was only 0.41 (at the 100 kJ L⁻¹ level; approximately half of the clay was dispersed). Fuller and Goh (1992) report a similar r^2 value ($r^2 = 0.50$) for a correlation of total carbohydrate with the energy needed to disperse one half of the clay in a given sample when including several different soil types in the regression. However, regressions were much better within the individual soils (r^2 values of 0.97 and 0.88), suggesting that carbohydrate has a significant role in clay binding but that the role might be different in different soils.

Table 3.5 Regression analyses of factors affecting the dispersion of clay from 0.25 to 0.85 mm soil aggregates during sonication at selected levels of applied energy: Best simple linear regression models for pooled tillage treatments and seasons of the 0-5 cm depth of a Fortier silty clay (n=16).

applied energy (kJ L ⁻¹)	predictor variables	coefficient	variable p	model r ²	model p
50	constant	0.47864	0.0000	0.36	0.0140
	total carbohydrate	-0.01362	0.0140		
100	constant	0.61749	0.0000	0.41	0.0073
	total carbohydrate	-0.01463	0.0073		
200	constant	0.32994	0.1347	0.15	0.1348
	organic C	0.00646	0.1348		
300	constant	0.70212	0.0000	0.15	0.1354
	total carbohydrate	-0.00625	0.1354		
400	constant	0.78886	0.0000	0.30	0.0267
	total carbohydrate	-0.00784	0.0267		
500	constant	0.82256	0.0000	0.12	0.1873
	total carbohydrate	-0.00514	0.1873		
600	constant	0.86967	0.0000	0.21	0.083
	total carbohydrate	-0.00748	0.0830		
600	constant	0.91191	0.0000	0.22	0.0674
	total carbohydrate	-0.00815	0.0674		

Multiple regressions were employed to explore the possibility of a relationship between clay dispersion and total carbohydrate or some of the other parameters that we had measured. Forward selecting stepwise multiple regression was applied to find the best multiple linear regression models for the pooled data at each energy level (Table 3.6). Total carbohydrate was again conspicuous by its presence in each multiple regression model except for the 200 kJ L⁻¹ level where no multiple regression was better than the simple linear regressions shown in Table 3.5. Each multiple regression model also included at least one of the lipid fractions. TEL and CHCl₃-EL were consistently dispersive, that is their coefficient was positive indicating increased dispersion as their concentration increased. DEE-EL always had the opposite effect indicating a negative relationship with clay dispersion. Organic C was also in many of the models and was positively correlated with clay dispersion. The multiple correlations were an improvement over simple linear correlations at most energy levels producing r² values between 0.74 and 0.999 indicating that most of the variability was accounted for by these models. The 200 kJ L⁻¹ level was the exception with no significant models and an r² of only 0.15. Some of the models contained many factors (the 100 kJ L⁻¹ model included eight parameters, almost all of the parameters that we had measured), while others like the 50 kJ L⁻¹ level was well described by only three.

In a review of soil aggregation and bonding mechanisms, Degens (1997) suggested more meaningful correlations can be obtained by directly relating changes in concentration of soil organic C fractions to changes in aggregation within individual treatments over time rather than between treatments. Such a grouping would allow for the possibility of detecting that different organic fractions or other soil parameters are relatively more important under

Table 3.6 Regression analyses of factors affecting the dispersion of clay from 0.25 to 0.85 mm soil aggregates during sonication at selected levels of applied energy: Best multiple linear regression models for a Fortier silty clay for pooled tillage treatments and seasons for the 0-5 cm depth.

applied energy (kJ L ⁻¹)	predictor variables	coefficient	variable p	model r ²	model p
50	constant	-0.37502	0.1504	0.75	0.0098
	total carbohydrate	-0.01363	0.0359		
	organic C	0.01513	0.0104		
	CHCl ₃ -EL	0.83432	0.0263		
100	constant	-0.24414	0.0690	0.999	0.0177
	total carbohydrate	-0.01572	0.0089		
	organic C	0.01569	0.0165		
	Ca:Mg	0.04686	0.0585		
	K	-0.17627	0.0148		
	Na	-1.34366	0.0143		
	TEL	0.62326	0.0203		
	clay content	0.00658	0.0212		
	DEE-EL	-0.15604	0.0710		
200	constant	0.32994	0.1347	0.15	0.1348
	organic C	0.00646	0.1348		
300	constant	0.70212	0.0000	0.999	0.0006
	total carbohydrate	-0.00625	0.1354		
	total carbohydrate	0.51382	0.0002		
	TEL	-0.00937	0.0003		
	TEL	0.41991	0.0007		
	Na	-0.70828	0.0006		
	organic C	0.00785	0.0009		
	K	-0.06565	0.0011		
	DEE-EL	-0.15068	0.0036		
400	constant	0.66024	0.0155	0.91	0.0662
	total carbohydrate	-0.00626	0.0695		
	Na	-1.6455	0.0177		
	K	-0.22169	0.0191		
	organic C	0.00887	0.0594		
	CHCl ₃ -EL	0.19439	0.2054		
	TEL	0.32812	0.096		
	clay content	0.00546	0.0896		
	500	constant	0.87187		
total carbohydrate		-0.01203	0.0208		
TEL		0.87626	0.0182		
Ca:Mg		-0.10705	0.0310		
DEE-EL		-0.35666	0.1146		
600	constant	0.57614	0.0040	0.91	0.0064
	total carbohydrate	-0.00908	0.0119		
	Na	-1.27976	0.0040		
	organic C	0.01444	0.0033		
	K	-0.1234	0.0140		
	CHCl ₃ -EL	0.37558	0.03460		

different treatments. Forward selecting multiple regressions were conducted separately for the conventional tillage data (both sampling times included) and the zero tillage data (also both sampling times pooled). The result was an improvement in r^2 values or model probability or both at each energy level. The two best models at each energy level are shown in Table 3.7 (conventional tillage) and in Table 3.8 (zero tillage). Only one model is shown if only one model had a p value less than 0.05.

Within conventional tillage treatments, exchangeable Ca, exchangeable Mg, or the ratio of exchangeable Ca:Mg are important in the best model describing clay dispersion for each energy level except at the 300 kJ L^{-1} level. By the sign of the coefficients we see that exchangeable Ca reduces dispersion, exchangeable Mg increases dispersion, and not surprisingly as the exchangeable Ca:Mg ratio increases, clay dispersibility decreases. Total carbohydrate was a significant input in models for three energy levels (100, 300, and 400 kJ L^{-1}) and as in the pooled treatment results (Table 3.6), increases in total carbohydrate concentration were associated with reduced clay dispersion. One or two of the lipid fractions were also present in many of the models. CHCl_3 -EL and TEL were again associated with increased dispersion, while DEE-EL was associated with reduced dispersion. This opposing effect of CHCl_3 -EL and DEE-EL was also seen under native prairie grassland conditions (Chapter 4). Exchangeable K reduced dispersion at the 100 and 200 kJ L^{-1} energy levels.

Table 3.7 Regression analyses of factors affecting the dispersion of clay from 0.25 to 0.85 mm soil aggregates during sonication at selected levels of applied energy: Best multiple linear regression models for a Fortier silty clay for conventional tillage treatments over both sampling dates for the 0-5 cm depth.

applied energy kJ L ⁻¹	predictor variables	coefficient	variable p	model r ²	model p
50	constant	1.18982	0.1101	0.93	0.0346
	CHCl ₃ -EL	1.43709	0.0182		
	Ca	-0.07986	0.1472		
	constant	0.42387	0.0125	0.98	0.0094
	CHCl ₃ -EL	1.79797	0.0053		
	Ca:Mg	-0.11881	0.0379		
100	constant	0.65855	0.0001	0.98	0.003
	total carbohydrate	-0.02432	0.0001		
	Mg	0.01662	0.0399		
	K	-0.03795	0.0562	0.98	0.0004
	constant	0.67731	0.0002		
	total carbohydrate	-0.02441	0.0001		
	Ca:Mg	-0.02923	0.0998	0.73	0.0323
	ESP	0.14776	0.0728		
	constant	1.18114	0.0007		
200	K	-0.17176	0.0387	0.79	0.0562
	DEE-EL	-0.61429	0.0512		
	constant	1.73206	0.0219		
	K	-0.14016	0.0827	0.81	0.0024
	DEE-EL	-0.69683	0.0426		
	Ca	-0.046566	0.2416		
300	constant	0.81974	0.0000	0.99	0.0055
	total carbohydrate	-0.01368	0.0024		
	constant	0.45042	0.0033		
	TEL	1.02616	0.0034	0.82	0.015
	DEE-EL	-0.60896	0.005		
	constant	1.60879	0.0005		
400	CEC	-0.01972	0.0104	0.997	0.0335
	DEE-EL	-0.4165	0.039		
	constant	1.00602	0.0169		
	total carbohydrate	-0.02317	0.0215	0.996	0.0414
	ESP	0.32804	0.0432		
	Ca:Mg	-0.08716	0.0495		
500	constant	0.09583	0.2403	0.89	0.0169
	TEL	1.64952	0.0243		
	DEE-EL	-1.00512	0.0237		
	Mg	0.05358	0.042	0.999	0.0219
	constant	0.7237	0.0001		
	CHCl ₃ -EL	0.90647	0.0169		
600	constant	0.32449	0.0438	0.89	0.0169
	TEL	0.31936	0.0557		
	CHCl ₃ -EL	1.15809	0.0121		
	Mg	0.04074	0.0325	0.999	0.0219

Table 3.8 Regression analyses of factors affecting the dispersion of clay from 0.25 to 0.85 mm soil aggregates during sonication at selected levels of applied energy: Best multiple linear regression models for a Fortier silty clay for the zero tillage treatments over both sampling dates for the 0-5 cm depth.

applied energy kJ L ⁻¹	predictor variables	coefficient	variable p	model r ²	model p
50	constant	0.01954	0.6595	0.93	0.0001
	clay content	0.00885	0.0001		
	constant	-0.15441	0.1108	0.94	0.0020
	Mg	0.09761	0.0005		
total carbohydrate	-0.00861	0.0055			
	Ca:Mg	0.0376	0.0857		
100	constant	0.11062	0.3735	0.68	0.0122
	clay content	0.00989	0.0122		
	constant	0.0732	0.5959	0.69	0.0222
	Mg	0.10226	0.0108		
total carbohydrate	-0.01202	0.0537			
200	constant	0.33724	0.0124	0.96	0.0185
	organic C	0.00864	0.0094		
	TEL	-0.24359	0.0516	1.00	0.0042
	constant	0.49077	0.0052		
organic C	0.00697	0.0046			
	TEL	-0.24815	0.0051		
	Ca:Mg	-0.02715	0.0156		
300	constant	0.93511	0.0061	0.999	0.0223
	Ca:Mg	-0.08273	0.0162		
	clay content	0.00167	0.0301	0.999	0.0080
	DEE-EL	-0.17228	0.0322		
constant	0.99449	0.0029			
400	Mg	0.03235	0.0076	0.999	0.0080
	DEE-EL	-0.52278	0.0047		
	Ca:Mg	-0.07149	0.0088	0.999	0.0194
	constant	0.63102	0.0065		
TEL	-0.5019	0.0108			
	organic C	0.00848	0.012		
	DEE:CHCl ₃ -EL	-0.00743	0.0302		
500	constant	1.10441	0.0033	0.999	0.0118
	Ca:Mg	-0.10058	0.0076		
	DEE-EL	-0.12008	0.0561		
600	constant	0.59765	0.004	0.999	0.0085
	TEL	-0.51385	0.0055		
	organic C	0.00971	0.005	0.999	0.0085
	CHCl ₃ -EL	0.17802	0.0304		
constant	0.59765	0.004			
	organic C	0.00971	0.005		
	TEL	-0.33583	0.0162		
	DEE-EL	-0.17802	0.0304		

The zero tillage regression models produced very good r^2 values at all levels except the 100 kJ L^{-1} level (Table 3.8). As in the conventional tillage models, exchangeable Mg or exchangeable Ca:Mg are in many of the regression models. Again increases in exchangeable Mg are associated with increased dispersion and increases in the exchangeable Ca:Mg ratio result in reduced dispersion. Only the 600 kJ L^{-1} level does not have a model which includes Ca or Mg. Total carbohydrate appears as a significant controlling parameter of clay dispersion only in models for the 50 and 100 kJ L^{-1} levels, while the lipid fractions appear in all the models for 200 kJ L^{-1} and above. Again carbohydrate and DEE-EL reduce dispersion and CHCl_3 -EL are associated with increased dispersion. Unlike under conventional tillage, TEL is associated with reduced dispersion under zero tillage. TEL is the total lipid extracted and is the sum of DEE-EL and CHCl_3 -EL. The net influence of TEL on clay dispersion is determined by the combined influence of DEE-EL and CHCl_3 -EL. DEE-EL made up 85.3% of the TEL for the fall zero tillage treatment compared to 56.7% for the fall conventional tillage treatment. Therefore since DEE-EL were associated with reduced dispersion under zero tillage, it is not surprising that the TEL were also associated with reduced dispersion under zero tillage. The variation in clay content alone results in the best correlations with the variability in clay dispersion at the lowest two energy levels with dispersion increasing as clay content increases. Clay content had a dispersive effect in the model at the 300 kJ L^{-1} energy level as well. Organic C also appears in many of the models in a dispersive role .

Multiple regression analyses were performed on the organic and mineral components separately (Appendix IV). For the majority of energy levels within a tillage treatment (five out of seven), the models which included both organic and mineral parameters produced the

best relationships (better adjusted r^2 values and better p-values). This indicates that organic and mineral components of the soil are interacting to produce the bonds between clay particles and that their effects are not purely additive.

The presence of exchangeable Ca, exchangeable Mg, or the ratio of the two in almost all of the conventional and zero tillage models indicates that these cations are very important for explaining variations in clay dispersion within treatments. Exchangeable Na or the exchangeable sodium percentage (ESP) only appears twice. Exchangeable Na is more frequently mentioned than exchangeable Mg when considering exchangeable cations and clay dispersion. A sodic soil is defined as a soil with an ESP value greater than 15 and in these soils, clay-sized particles will tend to disperse and clog soil pores (Sposito 1989). The low levels of exchangeable Na in the Fortier silty clay (ESP less than 1) would account for its minor role in this case. Several workers have shown that exchangeable Mg can also have a dispersive effect on clay or stabilize suspensions of clay particles (Bakker et al. 1973; Tucker 1985; Fuller 1993, Heil and Sposito 1993) and as early as 1935 a magnesium solonetz was described in Manitoba (Ellis and Caldwell 1935). The relative abilities of some common exchangeable cations to disperse clay are in the order $\text{Na} > \text{K} > \text{Mg} > \text{Ca}$ (Dexter and Chan 1991). Due to lower charge per cation, the monovalent cations, Na and K, are less effective in neutralizing repulsive forces between clay particles than the divalent cations, Mg and Ca, and hence monovalent cations are more dispersive than divalent cations. For a given valence, the ionic radius determines the ionic potential of the ion and hence the hydrated diameter is greater for ions with smaller ionic radii. Na has a smaller ionic radius than K and therefore under similar conditions will have a larger hydrated diameter than K. The result is that Na is less effective than K in neutralizing repulsive forces between clay particles.

Similarly Mg has a smaller ionic radius than Ca, a larger hydrated diameter, and is less effective at neutralizing the repulsive forces between the charged clay particles. Therefore as Mg is more dispersive of clay than Ca, soil clays should disperse more easily as the Ca:Mg ratio decreases. In soil systems, an increase in exchangeable Mg relative to Ca will often result in higher levels of exchangeable Na due to the fact that Na is more competitive with Mg than Ca for exchange sites (Yousaf et al. 1987). Other workers (Tucker 1985; Curtin et al. 1994b) have shown that there is also a specific Mg effect. It is believed that the larger hydrated diameter of Mg compared to that of Ca results in weaker attractive forces between the Mg cations and the clay particles (Emerson and Chi 1977). It also increases the thickness of the diffuse double layer decreasing the attractive force between clay particles and hence the increase in clay dispersion (Bakker et al. 1973). The difference in the effects of Ca and Mg on flocculation of a clay suspension are much greater when soil organic matter is present (Heil and Sposito 1993a). Differences in affinity between the cations and the soil organic matter appeared to be the reason for the weaker cation bridges with Mg. Lyubartsev et al. (1998) compared the ability of Ca and Mg ions to induce polyelectrolyte associations of anionic rod-like viral particles. They found Ca induced associations at lower concentrations than did Mg (40 mM and 60 mM respectively) for high charge density particles. Their lower charge density particle (30% lower surface charge density than their high charge density particle) required a Ca concentration of 50 mM to induce associations. However, Mg was unable to induce particle associations for the low charge density particles (concentrations up to 1 M were tried). Fuller (1993) found that the flocculation of a montmorillonite-dextran suspension proceeded at a much slower rate when Mg was the exchangeable cation than if Ca occupied the exchange sites. This was attributed to a greater

collapse of the uncharged dextran polymer onto the clay surface in the presence of Mg, leaving fewer tails and loops to create bridges to other particles. In a study of cation selectivity, the exchange sites on smectitic clay particles had similar affinities for Ca and Mg ions, however soil organic matter had a strong preference for Ca (Curtin et al. 1998).

A major role for polysaccharides in soil aggregation has been suggested by several researchers (Cheshire et al. 1983; Oades 1984; Swift 1990; Fuller et al. 1995). In the Fortier silty clay, the greater prominence of soil carbohydrate in models for the pooled treatments rather than the individual treatments suggests that soil polysaccharides are more important for explaining variations between treatments rather than variation within treatments. However, we see that zero tillage has both higher clay dispersion (Figure 3.3) and higher carbohydrate levels (Table 3.3) at mid-tillering. The difference in carbohydrate is maintained in fall whereas the difference in clay dispersion is not (Figure 3.5). A more likely explanation is that the pooled treatments have combined the effects seen in the individual models where carbohydrate is important at low energy levels for zero tillage and for the mid and higher energy levels for conventional tillage. The presence of carbohydrate in the pooled model is probably reflecting the seasonal variation in carbohydrate and clay dispersion (Figure 3.6). The differences between the treatments would suggest that carbohydrate has a different role in stabilizing soil aggregates under the two tillage systems.

Two roles for lipids in enhancing soil aggregation have been suggested by Diné et al. (1991a). The unbound lipids, which would include DEE-EL and CHCl_3 -EL, are highly hydrophobic and could coat more water soluble binding agents, such as carbohydrate, and protect them within the soil aggregates thereby improving stability. The more polar lipids and those with acid functional groups would bind to clay surfaces through polyvalent cation

bridges. The cation bridge would be a much stronger bond and contribute directly to binding particles into stable aggregates.

DEE-EL are relatively low molecular weight straight-chain and branched alkanes, alkenes, and alkanolic acids with some phenolic and alkylaromatic compounds, whereas CHCl_3 -EL are relatively high molecular weight paraffinic compounds with considerably fewer phenolic and aromatic compounds (Dinel et al. 1996). If a soil sample were not extracted with DEE first, both groups of lipids would be extracted by CHCl_3 alone (Dinel, personal communication). The dispersive effect of the large CHCl_3 -EL molecules may be a result of their interference with binding of functional groups of other organic compounds to charged clay sites. Dinel et al. (1991a) suspected such a mechanism in some of their treatments. The smaller DEE-EL compounds would presumably interfere less and therefore be less dispersive. However, this would not explain the apparent reduction in clay dispersion due to DEE-EL. Perhaps due to their smaller size and greater concentration of functional groups, DEE-EL would be more effective at coating and protecting binding agents which would otherwise be easily solubilized and thereby reduce macroaggregate breakdown. Paré et al. (1999) compared soil aggregate stability before and after extraction with DEE and CHCl_3 , and concluded that the extracted lipids were responsible for over 50% of the wet aggregate stability in both conventional and zero tillage treatments.

Another reason for the presence of DEE-EL and CHCl_3 -EL in the regression models may be what they indicate rather than what they do. As the relative amounts of the two lipid fractions indicate the degree of decomposition of organic matter (Dinel et al. 1996), their appearance in the models may be linked to what they tell us about the state of the soil organic matter in the two systems. Following sequential extraction, Paré et al. (1999) used

DEE-EL as an indicator of the presence of easily biodegradable organic matter and CHCl_3 -EL as an indicator of more bioresistant organic matter. If one applies the same principle in this study, clay dispersion would seem to be inhibited by easily decomposed organic matter and enhanced by more well decomposed material. In the model of Tisdall and Oades (1982), the more easily decomposed components are described as transient and temporary binding agents and include the polysaccharides and gums associated with both roots and soil microbes in the rhizosphere as well as the direct binding of soil particles by roots and fungal hyphae. These binding agents are strongly influenced by the type of soil management and would also be expected to show greater seasonal variation. Swift (1990) has shown that the polysaccharide effect on aggregation is transient and declines as the polysaccharide decomposes. The large drop in DEE: CHCl_3 -EL under conventional tillage by fall suggests a great reduction in easily decomposable compounds which is paralleled by an increase in clay dispersion. One possible situation where this might occur has been described by Oades (1984). Clay dispersion from microaggregates is prevented by the binding action of polysaccharides, mainly mucilages, and also by plant roots and fungal hyphae which stabilize the macroaggregates. As these binding agents decompose, organic acids (especially fulvic acids produced during decomposition) are adsorbed onto clay surfaces, they increase the negative charge of the clay increasing disruptive forces. The result is a weakening of the bond and a simultaneous increase in the forces of repulsion between the clay particles. Humic acid (another decomposition product) has been shown to both produce stable persistent aggregates (Swift, 1990) and also to be an effective clay dispersant (Gu and Doner 1993). Theng (1979) describes how both changes in polymer concentration and electrolyte concentration can result in the same polymer causing either flocculation or stabilization of

a clay suspension. The observed association of organic C with enhanced clay dispersion may be the result of adsorption of the fulvic and humic acid fractions of the organic matter onto clay surfaces stabilizing them in suspension. Heil and Sposito (1993) found that soil suspensions were more stable prior to organic matter removal and suggested that soil organic matter would play a critical role in the mobility of soil colloids and adsorbed contaminants.

Increases in clay content were associated with increases in clay dispersibility at the lower energy levels of the zero tillage treatment. Clay dispersion is generally preceded by aggregate breakdown and aggregates with high clay content break down more easily when wetted (Shainberg et al. 1992). This is in agreement with Brubaker et al. (1992), who found that clay content was an excellent predictor of clay dispersibility over a wide range of soils. The same effect was seen on cultivated soils but not on virgin prairie soils due to the much greater slaking resistance found in the grassland aggregates (Curtin et al. 1994a). Gregorich et al. (1988) found that 500 kJ L^{-1} of ultrasonic energy disrupted most sand-sized (0.05 to 2.0 mm) and larger aggregates. It would then follow that clay content is an important predictor of clay dispersion at these lower energy levels due the effect of clay on aggregate stability.

3.4 Conclusions

The study of clay dispersion from field soils is a complex problem due to the involvement of two simultaneous processes, macroaggregate breakdown and dispersion of clay from microaggregates, and the wide range of bonding mechanisms that can occur among microaggregates and among the clay particles within microaggregates. The shape of the dispersion curves (Figures 3.1, 3.3, and 3.5) show nicely the progression in bond strength.

One-half of the clay at either site was easily dispersed, requiring about 50 kJ L^{-1} of sonic energy for the Osborne clay and around 100 kJ L^{-1} for the Fortier silty clay. The remaining half of the clay required progressively more energy for each consecutive percent of clay dispersed and 10 to 20% of the clay remained bound and undispersed even after 600 kJ L^{-1} of energy had been applied.

Tillage management affected both the kind and class of soil structure of the Fortier silty clay. The conventional tillage system produced a granular and subangular blocky structure with a greater percentage of smaller soil aggregates than under zero tillage. The most obvious effect of the change to zero tillage on the Fortier silty clay was the development of angular blocky soil structure. Both tillage treatments showed an increase in average aggregate size with depth and a seasonal shift in aggregate size distribution, forming more of the larger aggregates by fall.

Four tillage operations over two seasons were not sufficient to reduce the organic C levels of a zero tilled Osborne clay to the levels seen in conventionally tilled fields. Clay dispersion had not been affected significantly by the tillage operations but the stratification of K and therefore quite likely of other nutrients as well which had occurred under zero tillage has been eliminated by the four tillage operations.

The binding between clay particles of the Fortier silty clay was affected by tillage system. Regression analysis indicates that variability within treatments was due largely to the relative dispersive effect of exchangeable Mg compared to exchangeable Ca (and therefore the ratio of exchangeable Ca to Mg) and the binding effect of easily decomposed organic compounds, including but not limited to soil carbohydrates. The importance of the Ca to Mg ratio has special significance for southern Manitoba because the cation chemistry

of our clays is dominated by Ca and Mg. The role of lipids was less clear but a dispersive role for the larger molecular weight CHCl_3 -EL fraction and a stabilizing role for the smaller molecular weight DEE-EL fraction was indicated.

The roles of exchangeable Mg and exchangeable Ca in clay dispersion were masked when tillage treatments were pooled for regression analysis. Unbound lipids, carbohydrate, and other easily decomposed organic compounds, indicated by the relative amounts of DEE-EL and CHCl_3 -EL, were most important in explaining differences between treatments. The improvement in r^2 values upon analysing the treatments separately shows that the relative importance of the different parameters is changed by tillage system. For example, under zero tillage carbohydrate is more important at the lowest two energy levels than at other levels, whereas it is not in the model for the lowest energy level of the conventional tillage treatment but is present in the models for the intermediate levels. The much larger seasonal variation in clay dispersion with a similar change in carbohydrate level for the conventional tillage treatment supports the conclusion that there was a larger role for soil polysaccharides in aggregate stabilization under conventional tillage compared to zero tillage. Also the shift in model predictor variables with energy level suggests that different factors, not just a change in the quantity of a factor, are responsible for the differences in strength of bonding between clay particles and hence their increasing dispersibility as applied energy increases. Similarly variables such as exchangeable Ca and exchangeable Mg which are present in most of the models have an effect on all types of bonds.

The significance and contribution to knowledge of this chapter is to highlight the importance of exchangeable cations, especially the ratio of Ca to Mg in soils where they are dominant, whenever aggregate stability and clay dispersibility of soils are studied. The Ca

to Mg ratio influences bonds stabilizing macroaggregates as well as microaggregates. Their effect on the thickness of the diffuse double layer affects clay swelling and hence aggregate slaking but also affects the dispersion of individual clay particles. The effect on cation bridges between mineral and organic anions as well as the effect on uncharged polymer adsorption on clay surfaces would influence the strength of bonds holding clay in microaggregates. Further, the different effects of Mg and Ca on cation bridges between clay particles and organic polymers merits attention and should be investigated.

The dispersive effect of Mg on soil clay is of particular concern and should receive more attention by the Canadian agricultural community. The potentially deleterious effect of Mg will become increasingly important as irrigated acreage rises on the Canadian prairies and good quality irrigation water is supplemented or replaced by water of lower quality.

4. THE EFFECTS OF SOIL FORMING PROCESSES ON SOIL ORGANIC MATTER COMPOSITION AND CLAY DISPERSION FROM SOIL AGGREGATES ALONG A NATIVE PRAIRIE CATENA

4.1 Introduction

Soil aggregate breakdown and dispersion of clay at or near the soil surface, referred to as internal erosion, has long been a matter of concern in agricultural systems. The resultant pore constriction or blockage can result in reduced infiltration rates and increased runoff, which in turn can result in erosion of topsoil (Zhang and Miller 1996) as well reducing the available water for the crop. Dispersion of clay under native prairie has been of less concern than that which occurs under cultivation due to the larger economic impact of deterioration of tilled agricultural soils. As a result native prairie soils have been studied less than cultivated soils. Most studies looking at aggregate stability and clay dispersion under virgin soils focus on the effect of tillage and compare the soil from the virgin sites with nearby cultivated fields (Blank and Fosberg 1989; Loch 1994; Fuller et al. 1995). Fuller (1993) showed that clay binding within aggregates as determined by ultrasonic dispersion varied along a transect of virgin soils which included an Orthic Black Chernozem, an Orthic Dark Gray Chernozem, and an Orthic Gray Luvisol. Significant differences in clay dispersion and good correlations with carbohydrate were reported.

Compared to many other methods of clay dispersion, ultrasonic dispersion has the advantage that the researcher is able to apply specific amounts of energy to the submerged

aggregates. Ultrasonic dispersion also allows easy standardization between laboratories. Small differences in clay binding strength should be detectable with ultrasonic dispersion and thus add to our understanding of clay binding and aggregation.

The purpose of this study was to determine if smaller differences in soil development than those seen between great groups and orders by Fuller (1993) would still result in measurable differences in clay dispersibility. The relationships among dispersibility, soil organic matter constituents, and other measurable soil chemical and physical properties will be investigated.

4.2 Materials and Methods

4.2.1 Site Characterization

The study site was located under native prairie on the western edge of the Turtle Mountains near Deloraine, Manitoba, belonging to Cal and Julie Gervin (49°05' N, 100°29' W; legal description, SE 6-2-23 W1). According to the previous owner, Bill Poole, the area had never been tilled or pastured. The toposequence that we selected began near a knoll and sloped down to the WSW ending near a fence line and was bound on the north and south by small ravines. The area west of the fence line was more gently sloped and had a history of cultivation and use as pasture.

Southwestern Manitoba is the driest part of Manitoba, averaging 457 - 483 mm of precipitation per year (PFRA, 2001). The soils in the area of the study site have developed on deep, strongly calcareous, medium to moderately fine textured glacial till of mixed shale, limestone, and granitic rocks (Eilers et al. 1978).

The slope of the soil surface was steepest at the shoulder of the knoll and shallowest near the fence line (Figure 4.1). Three locations were selected along a 160 m transect of the catena to represent soils which had different horizons and morphology but were still Black Chernozems. The selection was based on the degree of leaching, estimated by the surface slope and vegetation, and corroborated by soil horizon development as seen in the soil profile. The driest location, on the shoulder of the knoll, was designated D-1, the intermediate position as D-2, and the most moist location, at the toe slope, as D-3. The slope was 14% between D-1 and D-2 and 10% between D-2 and D-3 as determined using a handheld clinometer. The transect was surveyed and a contour map produced (Figure 4.1). A soil pit was dug at each slope position and the horizon designations are shown in Table 4.1. Site selection and characterization occurred in the summer of 1992.

The dominant vegetation at D-1 was blue grama grass (*Bouteloua gracilis*) and *Stipa* species. Other grass and broadleaf plants including wild rose were also present. At D-2 and D-3 native bluegrass (*Poa* spp.) and western snowberry (*Sisyrinchium montanum*) dominated. Although D-2 and D-3 were populated by many of the same species, the growth was much taller and more dense at D-3.

4.2.2 Soil Sampling and Analyses

Two surface samples were taken at each of the three locations along the catena in October, 1993. One sample was taken approximately one meter to the north side of the pit location and the second, one meter to the south side. Samples were carefully cut out with a spade and removed as a block measuring approximately 30 cm square and 15 cm deep. The soil blocks were allowed to air dry fully prior to removal of the root mat. After removal

of the root mat, the bottom of each block was trimmed to leave a uniform thickness of 10 cm of soil. Because the solum at D-1 only extended to the 10 cm depth, the 0 to 10 cm depth was sampled at each slope position. The root mat and excess soil was discarded. The remaining soil block was gently broken apart by hand and the soil which was able to pass through a 9.5 mm sieve collected for further analyses. A sub-sample was removed from these samples and used for pH, electrical conductivity (EC), and particle size analyses (Table 4.2).

Table 4.1 Horizon designations of soil profiles at three sites along a native prairie catena.

Location	Horizon	Depth (cm)	Classification
D-1	Ah	0-10	Rego Black Chernozem
	AC	10-20	
	Ck	20+	
D-2	Ah	0-12	Orthic Black Chernozem
	Bm	12-42	
	Ck	42+	
D-3	Ah	0-30	Orthic Black Chernozem
	Btj	30-60	
	Ck	60+	

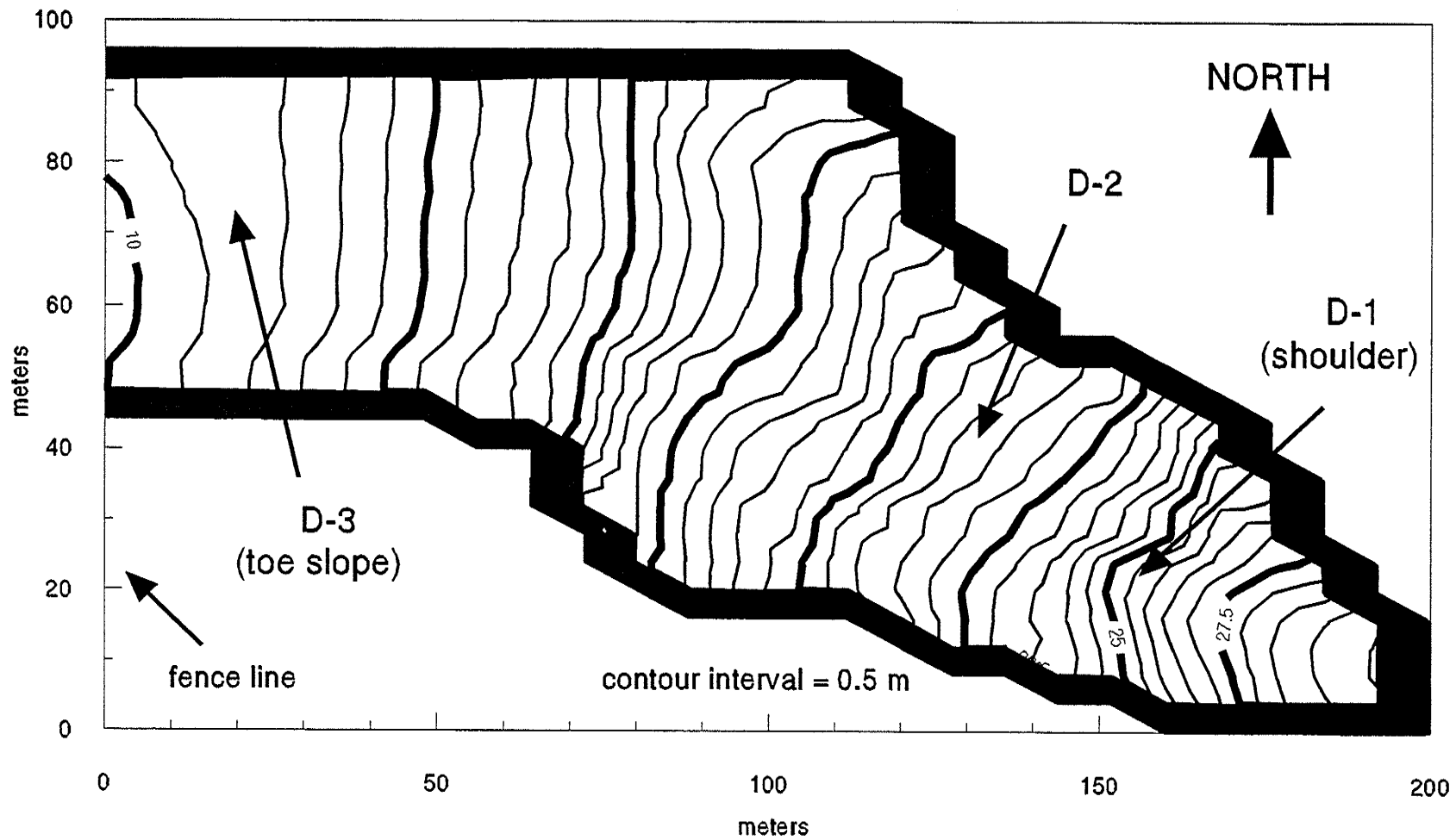


Figure 4.1 Contour map of a native prairie catena near Deloraine, MB.

Table 4.2 Soil characteristics for the surface 10 cm of three sites along a native prairie catena.

	D-1	D-2	D-3
Total soil			
Particle size analysis:			
Sand (%)	43.2	32.0	32.3
Silt (%)	26.8	28.8	30.9
Clay (%)	30.0	39.2	36.8
Exchangeable sodium percentage - ESP (%)	0.05	0.14	0.11
pH ^z	7.1	6.3	6.8
Electrical conductivity - EC (dS m ⁻¹) ^z	0.32	0.24	0.24
Aggregate size distribution (wt %)			
< 0.25 mm	18	9	7
0.25 - 0.85 mm	17	14	10
0.85 - 2.00 mm	9	12	14
2.00 - 4.75 mm	12	16	31
4.75 - 9.5 mm	15	19	20
> 9.5 mm	29	30	18
0.25 to 0.85 mm aggregate size fraction			
Clay content (%)	32.1 (0.01) ^y	39.7 (0.0001)	37.2 (0.002)
Cation exchange capacity - CEC (cmoles kg ⁻¹ soil)	27.9 (1.2)	27.8 (0.46)	26.7 (0.63)
Ca (cmoles kg ⁻¹ soil)	13.0 (0.46)	10.5 (0.38)	11.1 (0.94)
K (cmoles kg ⁻¹ soil)	1.69 (0.28)	2.80 (0.23)	2.55 (0.04)
Mg (cmoles kg ⁻¹ soil)	2.80 (0.036)	4.11 (0.33)	4.15 (0.16)
Na (cmoles kg ⁻¹ soil)	0.01 (0.002)	0.04 (0.009)	0.03 (0.012)

^z EC and pH measured in a 1:1 soil:deionized water paste

^y standard error of the mean (n = 2)

The aggregates less than 9.5 mm in diameter were further separated by dry sieving into 5 aggregate size fractions (ASFs): less than 0.25 mm, 0.25 to 0.85 mm, 0.85 to 2.00 mm, 2.00 to 4.75 mm, and 4.75 to 9.5 mm. The nest of sieves was placed on an automatic sieving device and air dry aggregates added at a loading rate of $\leq 0.6 \text{ g cm}^{-2}$. Sieving time was 15 seconds. Each aggregate size fraction (ASF) was weighed and expressed as a percentage by weight of the total aggregates.

The 0.25 to 0.85 mm ASF was chosen for detailed exchangeable cation, organic matter, and clay dispersion analyses as this ASF contains important aeration and water conducting pores (Oades 1984).

Exchangeable Ca, Mg, K, and Na were displaced from the soil exchange sites by saturation with Ba (unbuffered 0.25 M BaCl_2 solution) and measured by atomic absorption spectrometry. Cation exchange capacity (CEC) was then determined by measuring the Ba in solution following saturation with Mg (0.25 M MgCl_2 solution). Organic carbon was determined by a modified Walkley-Black method (Yeomans and Bremner 1988).

Extractable lipids were determined by the method of Diné et al. (1996). A 10 g sample of the 0.25-0.85 mm ASF was sequentially extracted with diethyl ether (DEE) and chloroform (CHCl_3) in a Soxhlet apparatus. Each extract was dried and weighed. The sequential extraction separates extractable lipid into the DEE extractable lipids (DEE-EL) which are easily biodegraded by soil microbes and CHCl_3 extractable lipids (CHCl_3 -EL) which are more bioresistant (Diné et al. 1992).

Clay from soil aggregates was dispersed by sonication, using a Braun Sonic 2000 probe-type ultrasonic homogenizer (Fuller and Goh 1992). One gram of air dry aggregates from the 0.25-0.85 mm ASF was placed in a 50 mL beaker. Deionized water was added

slowly along the side of the beaker so that the aggregates were wet by capillarity. Energy levels of 25, 50, 100, 200, 300, 400, and 500 kJ L⁻¹ were used to produce the dispersion curves. Each level of sonication was applied to freshly wetted aggregates on duplicate samples. The 1500 kJ L⁻¹ energy level was used to determine the value for “maximum dispersible clay”. Clay dispersion at each energy level was expressed as a decimal fraction of maximum dispersible clay. The remaining suspension from the 1500 kJ L⁻¹ treatment was centrifuged at 10 000 x g for 20 minutes and a 5 mL aliquot used for “solubilized carbohydrate” determination (Fuller and Goh, 1992). Solubilized carbohydrate was determined colorimetrically by the anthrone method of Brink et al. (1960) using glucose as the standard.

The parent material for these soils was highly calcareous, as evidenced by a strong effervescence in the C horizon when treated with a dilute HCl solution. Based on this fact, depth to carbonate was used as an indicator of the moisture and leaching regime during soil development. The effect of erosional loss from the soil surface on depth to carbonate was considered to be negligible. The soil has been under continuous prairie and minimal soil loss has been found under continuous grass cover (Gilley and Doran 1997).

Statistical analyses were performed using the Linear Regression and Forward Selecting Stepwise Linear Regression procedures of Statistix version 1.0 (Analytical Software 1996). The effect of soil forming processes on soil constituents and clay dispersion was evaluated by correlation with depth to carbonate using depth to carbonate as a proxy for moisture status and leaching regime. The square hyperbola equation was fitted to each clay dispersion curve to determine the energy needed to disperse one-half of the clay (E-half value). The effect of the various soil constituents on clay dispersion was examined by

regression analysis at each energy level.

4.3 Results and Discussion

4.3.1 Aggregate Size Distribution

The distributions of the various ASFs in the surface 10 cm was quite similar amongst the three slope positions (Table 4.2). D-1 contained a higher proportion of microaggregates (ASF less than 0.25 mm) and a higher content of aggregates in the 0.25 to 0.85 mm ASF than either of the other two slope positions. Oades and Waters (1991) found that for Mollisols and Alfisols with greater than 2% organic C, roots and fungal hyphae were the primary stabilizing agents binding microaggregates and small macroaggregates into larger aggregates. Roots and mycorrhizal fungi under native prairie also play a critical role in the stabilization of macroaggregates (Miller and Jastrow 1990). The more arid conditions at D-1 support much less vegetative growth than at D-2 or D-3 and as a result less root mass and we would therefore expect more microaggregates than at the other two positions.

4.3.2 Exchangeable Cations

Cation exchange capacity was similar for the 0 to 10 cm depth of all three slope positions however content of exchangeable cations was not (Table 4.2). At D-1 exchangeable Ca was higher while exchangeable K, Mg, and Na were lower than at D-2 or D-3. The contents of exchangeable cations were similar at D-2 and D-3. Linear regression analysis indicated that only exchangeable Mg and exchangeable Ca:Mg were significantly correlated with depth to carbonate (r^2 of 0.79 and 0.80 respectively, Table 4.3).

Table 4.3 Correlations between depth to carbonate, which was used as a proxy for soil moisture status, leaching regime, and productivity, and various soil parameters of the 0 to 10 cm depth under native prairie.

	r^2	coefficient ^z	p value
clay content (g clay/g soil)	0.61	.00111 ⁺	0.0660
exchangeable cations		cmol kg ⁻¹ soil	
Ca	0.50	-0.04155	0.1185
K	0.57	0.01876 ⁺	0.0815
Mg	0.79	0.0280 *	0.0176
Na	0.25	3.26x10 ⁻⁴	0.3075
Ca:Mg	0.80	-0.04135 *	0.0156
Ca + Mg	0.11	-0.01355	0.5144
Na:(Ca+Mg)	0.25	2.155x10 ⁻⁵	0.3167
CEC	0.20	-0.02116	0.3748
ESP	0.27	0.00123	0.2892
Soil organic matter fractions		g kg ⁻¹ soil	
organic C	0.48	-0.01131	0.1284
DEE extractable lipid (DEE)	0.35	1.85x10 ⁻⁴	0.2950
CHCl ₃ extractable lipid (CHCl ₃)	0.25	-3.26x10 ⁻⁵	0.3962
total extractable lipid (TEL)	0.32	1.52x10 ⁻⁴	0.3194
solubilized carbohydrate (SHex)	0.13	0.00159	0.4773
solubilized carbohydrate (g kg ⁻¹ clay)	0.45	-0.01393	0.1430
CHCl ₃ :organic C	0.15	-4.47x10 ⁻⁶	0.5131
CHCl ₃ : SHex	0.29	-2.19x10 ⁻⁵	0.3526
CHCl ₃ :TEL	0.33	-8.00x10 ⁻⁴	0.3127
DEE:CHCl ₃	0.29	0.04293	0.3606
DEE:OC	0.31	4.89 x10 ⁻⁵	0.3281
DEE:SHex	0.35	5.24x10 ⁻⁵	0.2926
organic C:clay	0.76	-0.08198 *	0.0236
organic C:SHex	0.13	0.00159	0.4773
TEL:organic C	0.29	4.43x10 ⁻⁵	0.3594
TEL:SHex	0.26	3.05x10 ⁻⁵	0.3766

^z * significant at the 5% level, ⁺ significant at the 10% level

4.3.3 Soil Carbohydrate and Lipid Fractions

The concentration of organic C in the 0 to 10 cm depth of the soil was greatest at D-1 and lowest at D-3 (Table 4.4). Dinel et al. (1998) similarly found higher organic C concentrations in the surface of the upper slope position of a catena located near Clinton Ontario. Due to the much deeper A horizon and the B horizon at D-3, it would be predicted that total soil organic C would be highest at D-3.

Solubilized carbohydrate levels (solubilized at 1500 kJ L^{-1}) were consistently measured at about 2 g kg^{-1} of soil throughout the catena (Table 4.4). Fuller and Goh (1992) found that 0.65 g of hexose equivalents kg^{-1} soil was solubilized from the Ah horizon of an Orthic Black Chernozem containing 5 g kg^{-1} hexose equivalents after application of 1000 kJ L^{-1} of ultrasonic energy. Samples taken from our catena in 1992 averaged about 11.5 g of total hexose equivalents kg^{-1} of soil. Assuming values were similar in 1993, the solubilized hexose equivalents were about 17% of total hexose equivalents compared to 13% reported by Fuller and Goh (1992). Considering the higher energy input used in this study, the value of 2 g kg^{-1} of soil is reasonable as more carbohydrate would be expected to be released at the higher level of applied energy.

The content of DEE-EL and CHCl_3 -EL in the 0 to 10 cm depth did vary somewhat among slope positions (Table 4.4), the difference being most noticeable in the ratio DEE: CHCl_3 extractable lipid. The difference did not translate into a significant linear correlation with moisture and leaching intensity as inferred from depth to carbonate (Table 4.3). Dinel et al. (1996) used sequential extraction of lipids with DEE and CHCl_3 to estimate organic matter stability. They found that diethyl ether extracted lipids which were relatively low-molecular weight straight-chain and branched alkanes, alkenes, and alkanic acids with

Table 4.4 Amounts of selected soil organic matter fractions in the 0.25 to 0.85 mm aggregate size fraction of the 0 to 10 cm depth at each slope position.

	D-1	D-2	D-3
organic C (g kg ⁻¹ soil)	56.7 (0.3) ^z	53.0 (0.1)	50.9 (0.3)
DEE-EL (g kg ⁻¹ soil)	0.051 (na)	0.059 (0.002)	0.060 (0.007)
CHCl ₃ -EL (g kg ⁻¹ soil)	0.0103 (na)	0.0073 (0.0001)	0.0085 (0.0008)
DEE:CHCl ₃ extractable lipid	4.93 (na)	8.10 (0.32)	7.24 (1.51)
TEL (g kg ⁻¹ soil)	0.061 (na)	0.066 (0.002)	0.069 (0.006)
solubilized hexose equivalents (g kg ⁻¹ soil)	1.97 (0.06)	2.02 (0.02)	2.07 (0.14)
solubilized hexose equivalents (g kg ⁻¹ clay)	6.06 (0.01)	5.13 (0.01)	5.44 (0.34)

^z standard error of the mean (n = 2)

some phenolic and alkylaromatic compounds. CHCl₃ extractable lipids (CHCl₃-EL) were relatively high-molecular weight paraffinic compounds with considerably fewer phenolic and aromatic compounds. DEE-EL were found to be more easily biodegraded by soil microbes than CHCl₃-EL most likely due to the greater hydrophobicity of the CHCl₃-EL (Dinel et al.1992). The lower ratio for DEE:CHCl₃ extractable lipid at D-1 compared to D-2 and D-3 would indicate greater biological recalcitrance of the soil organic matter at D-1 and would help account for the higher organic C levels at D-1.

4.3.4 Dispersion of Clay from Soil Aggregates

Clay dispersion increased as applied energy increased but with a diminishing return (less clay dispersed per unit of energy increase at the higher energy levels than at the lower energy levels) (Figure 4.2). However the rate of change was much slower for the prairie soils than the cultivated soils (Chapter 3), suggesting less variation in the clay bonds under native

prairie. The E-half values were also higher (Figure 4.3) indicating stronger bonds.

Linear regression analysis of clay dispersed by depth to carbonate was significant at each energy level (Table 4.5). The slope of the regression line was consistently positive (positive coefficient in the model, Table 4.5) indicating that the clay was more tightly held in the aggregates from D-1, which had the shallowest depth to carbonates. Simple linear regression of clay dispersed versus all measured soil characteristics and several ratios of measured soil characteristics were not helpful in explaining these differences (Table 4.6). The only significant correlation (at the 5% level) was with the ratio of exchangeable Ca to exchangeable Mg at the 300 and 400 kJ L⁻¹ levels ($r^2 = 0.67$ for both levels of applied energy).

Forward selecting stepwise multiple linear regression resulted in r^2 values of 0.99 to 1.00 at each energy level (Table 4.7). The best models at each energy level included the ratio of exchangeable Ca:Mg and the soil CEC. The majority also contained one of the lipid fractions or the ratio of the DEE:CHCl₃ extractable lipid. One model at 200 kJ L⁻¹ and one at 300 kJ L⁻¹ contained exchangeable Ca rather than a lipid component. The consistency in predictor variables across energy levels suggests much less variability in clay bonding under native prairie than was evident in tilled soils (Chapter 3). Most notable is the lack of a large pool of very weakly held clay in the native prairie soils which is probably the result of the ability of grassland soil aggregates to resist slaking. Curtin et al. (1994a) found much less dispersible clay in grassland soils than cultivated soils when tested with the rapid immersion method.

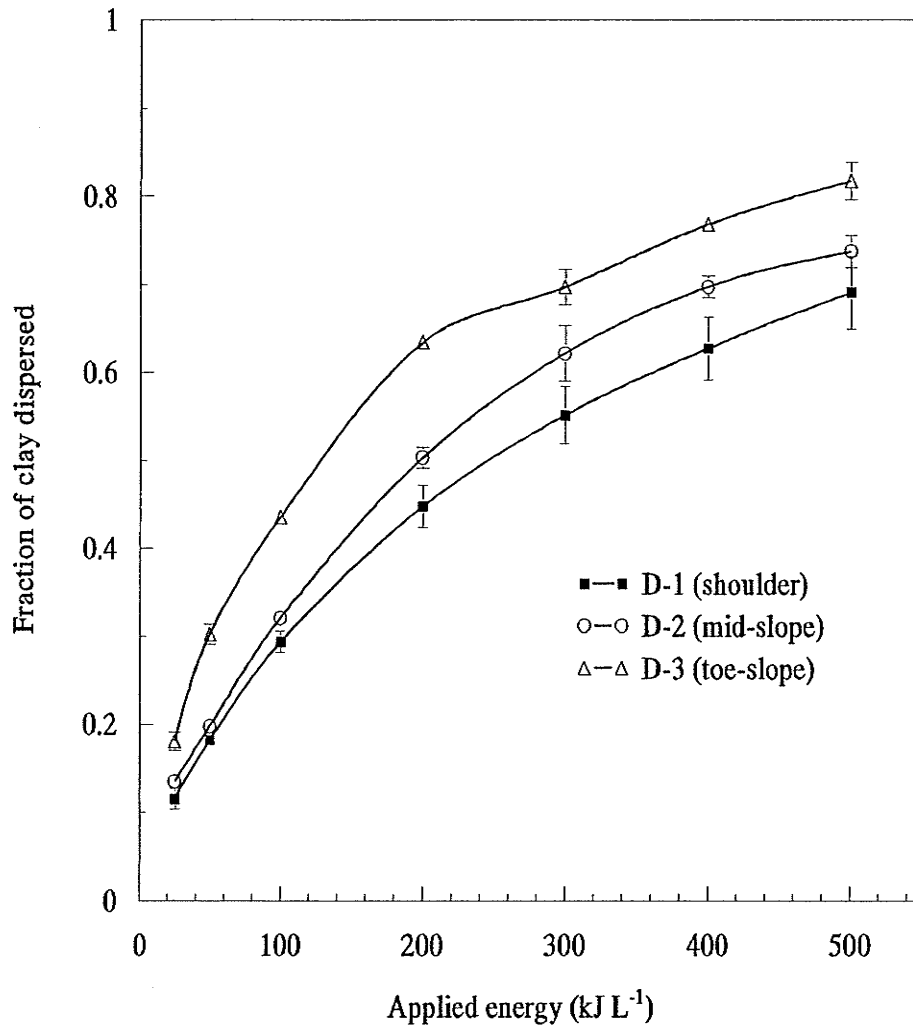


Figure 4.2 The dispersion of clay from soil aggregates of the 0.25-0.85 mm ASF of the 0 to 10 cm depth along a native prairie toposequence (error bars = 2 x standard error of the mean).

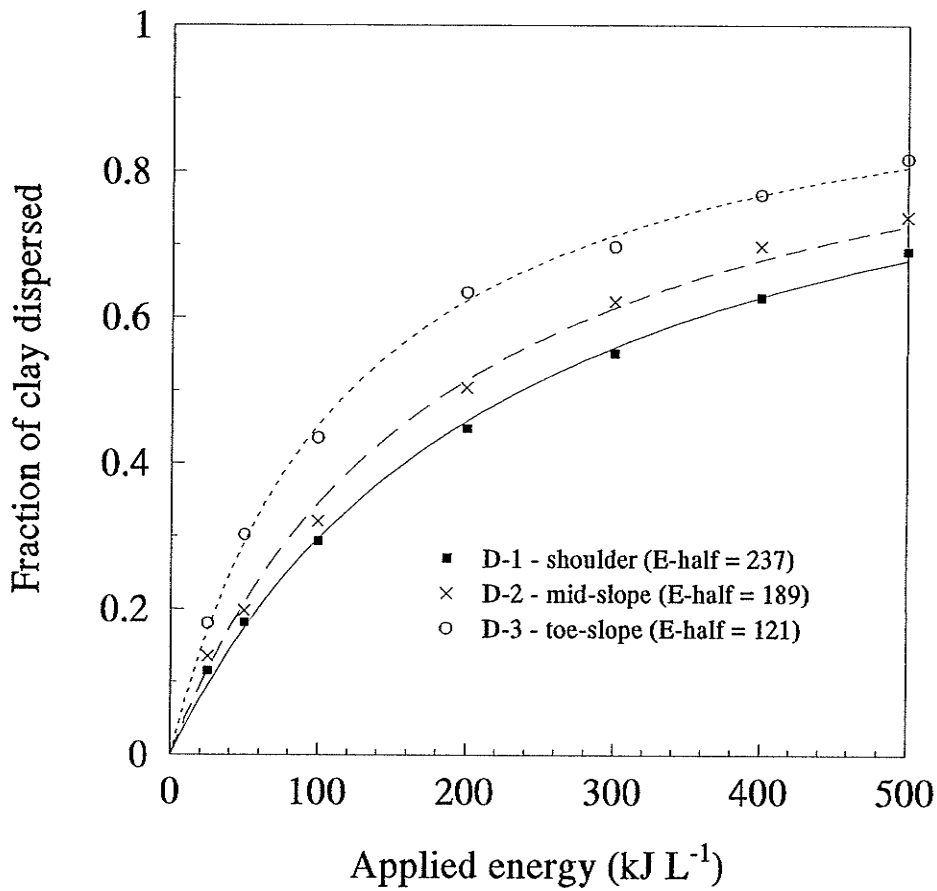


Figure 4.3 Fraction of clay dispersed at selected energy levels for the 0.25 to 0.85 mm ASF of the 0 to 10 cm depth of three slope positions under native prairie. Plotted functions are for the best fitting square hyperbola equation for each slope position and sampling time. The E-half values represent the energy needed to disperse half of the clay and are in kJ L^{-1} . E-half values are determined from the equation of best fit. All three equations had r^2 values greater than 0.99.

Table 4.5 Linear correlations between depth to carbonates and clay dispersed from 0.25 to 0.85 mm soil aggregates from the 0 to 10 cm depth of a native prairie at sonication levels of 25 to 500 kJ L⁻¹. Depth to carbonate was used as a proxy for soil moisture status, leaching regime, and productivity.

energy applied (kJ L ⁻¹)	r ²	coefficient	probability
25	0.77	0.00121	0.0214
50	0.72	0.00216	0.0338
100	0.77	0.00258	0.0221
200	0.83	0.0346	0.0110
300	0.79	0.00278	0.0180
400	0.85	0.00270	0.0086
500	0.70	0.00237	0.0374

Multiple regression analyses of dispersed clay was also examined separately for the organic and mineral components (Appendix V). No significant models were found indicating that both mineral and organic components are instrumental in clay bonding and also suggesting that their effects are not purely additive.

The importance of the exchangeable Ca:Mg ratio in the regression models shows Mg has a major influence on the strength of binding between clay particles in these soils. At each energy level, the value of the coefficient for exchangeable Ca:Mg was less than 0 indicating an increase in clay dispersion as Mg increased relative to Ca. The ratio ranged from a high of almost 5 at D-1 to between 2 and 3 for D-2 and D-3. In these soils, the simple linear regressions indicate that the specific Mg effect is more important than the increase in exchangeable Na that accompanies the increase in Mg on the soil exchange sites (Table 4.6). The r² values for exchangeable Mg ranged from 0.36 to 0.58 depending on the

Table 4.6 Correlation coefficients for linear regressions of clay dispersed versus various soil parameters during sonication at selected levels of applied energy - 0.28 to 0.85 mm aggregates from the 0 to 10 depth of a prairie catena (n = 6, except for correlations containing one or more of the lipid fractions where n = 5).

	level of dispersive energy (kJ L ⁻¹)						
	25	50	100	200	300	400	500
exchangeable Ca	0.08	0.08	0.11	0.17	0.14	0.21	0.07
exchangeable K	0.36	0.15	0.21	0.34	0.58	0.58	0.46
exchangeable Mg	0.45	0.36	0.42	0.46	0.48	0.58	0.43
exchangeable Na	0.08	0.03	0.04	0.04	0.10	0.12	0.08
exchangeable Ca:Mg	0.49	0.32	0.37	0.49	0.67*	0.67*	0.52
exchangeable Ca:Na	0.18	0.12	0.13	0.15	0.24	0.24	0.15
exchangeable (Ca + Mg)	0.01	0.00	0.00	0.01	0.00	0.01	0.02
exchangeable Na:(Ca + Mg)	0.05	0.02	0.03	0.03	0.08	0.10	0.05
CEC	0.05	0.23	0.19	0.14	0.01	0.03	0.00
ESP	0.09	0.04	0.05	0.05	0.11	0.13	0.08
clay content	0.26	0.12	0.18	0.28	0.41	0.47	0.31
CHCl ₃ -EL	0.03	0.00	0.01	0.05	0.11	0.18	0.08
DEE-EL	0.10	0.10	0.16	0.24	0.12	0.25	0.12
TEL	0.11	0.14	0.20	0.26	0.10	0.23	0.11
CHCl ₃ -EL : organic C	0.01	0.03	0.00	0.01	0.09	0.12	0.05
CHCl ₃ -EL : soluble carbohydrate (g kg ⁻¹ soil)	0.04	0.00	0.03	0.09	0.13	0.22	0.10
CHCl ₃ -EL : TEL	0.06	0.01	0.05	0.12	0.14	0.24	0.11
DEE-EL : CHCl ₃ -EL	0.04	0.01	0.03	0.08	0.10	0.19	0.07
DEE-EL : organic C	0.10	0.13	0.19	0.25	0.10	0.22	0.10
DEE-EL : soluble carbohydrate (g kg ⁻¹ soil)	0.08	0.07	0.13	0.18	0.09	0.23	0.10
organic C	0.13	0.25	0.26	0.27	0.11	0.17	0.06
organic C : clay content	0.29	0.25	0.30	0.39	0.38	0.46	0.26
organic C : soluble carbohydrate (g kg ⁻¹ soil)	0.07	0.07	0.11	0.19	0.10	0.18	0.10
soluble carbohydrate (g kg ⁻¹ clay)	0.23	0.10	0.12	0.16	0.35	0.32	0.25
soluble carbohydrate (g kg ⁻¹ soil)	0.07	0.07	0.11	0.19	0.10	0.18	0.10
TEL : organic C	0.10	0.16	0.21	0.26	0.08	0.19	0.09
TEL : soluble carbohydrate (g kg ⁻¹ soil)	0.08	0.13	0.18	0.17	0.04	0.15	0.07

* significant at the 5% level

Table 4.7 Best two regression models for each energy level from stepwise regression analyses for dispersed clay correlated with various soil properties - prairie catena 0.25 to 0.85 mm aggregates from the 0 to 10 cm depth (n = 5).

energy	predictor variables	adjusted r^2	standard deviation for the regression	probability
25 kJ L ⁻¹	exchangeable Ca:Mg; CEC; CHCl ₃ extractable lipid; constant	0.9970	0.00188	0.0348
	exchangeable Ca:Mg; CEC; DEE extractable lipid; constant	0.9892	0.00357	0.0660
50 kJ L ⁻¹	exchangeable Ca:Mg; CEC; DEE:CHCl ₃ extractable lipid; constant	0.9974	0.00314	0.0324
	exchangeable Ca:Mg; CEC; DEE extractable lipid; constant	0.9966	0.00414	0.0428
100 kJ L ⁻¹	exchangeable Ca:Mg; CEC; DEE extractable lipid; constant	0.9999	8.802x10 ⁻⁴	0.0078
	exchangeable Ca:Mg; CEC; DEE:CHCl ₃ extractable lipid; constant	0.9919	0.00648	0.0573
200 kJ L ⁻¹	exchangeable Ca:Mg; CEC; exchangeable Ca; constant	0.9997	0.00172	0.0118
	exchangeable Ca:Mg; CEC; CHCl ₃ extractable lipid; constant	0.9948	0.00670	0.0461
300 kJ L ⁻¹	exchangeable Ca:Mg; CEC; Ca; constant	0.9906	0.0.00753	0.0616
	exchangeable Ca:Mg; CHCl ₃ extractable lipid; organic C; constant	0.9900	0.00778	0.0636
400 kJ L ⁻¹	exchangeable Ca:Mg; CEC; DEE:CHCl ₃ extractable lipid; constant	1.0000	4.09x10 ⁻⁴	0.0036
	exchangeable Ca:Mg; CEC; CHCl ₃ extractable lipid; constant	0.9995	0.00167	0.0146
500 kJ L ⁻¹	exchangeable Ca:Mg; CEC; CHCl ₃ extractable lipid; constant	0.9998	9.62x10 ⁻⁴	0.0084
	exchangeable Ca:Mg; CEC; DEE:CHCl ₃ extractable lipid; constant	0.9991	0.00215	0.0189

energy level. The values for Na ranged from 0.03 to 0.12. The r^2 value for exchangeable Ca was also very low suggesting that total exchangeable Ca is not as important as the ratio of exchangeable Ca to exchangeable Mg.

The role of Na in clay dispersion has long been recognized. Exchangeable sodium percentage (ESP) has been used as a criteria to classify sodic soils and the sodium adsorption ratio (SAR) is used for evaluating the quality of irrigation water in regard to risk of clay dispersion and damage to soil structure. However the role of Mg is less well understood. Yousaf et al. (1987) reported increased clay dispersion in a Mg-Na compared to Ca-Na systems. The difference was attributed to higher levels of exchangeable Na and to a lowering of the solution electrolyte concentration in the Mg-Na system. Several others (Emerson and Chi 1977; Tucker 1985; and Curtin et al. 1994b) found specific Mg effects as well. Over a wide range of soils from the southern Canadian prairies, Mg appeared to be about 4 to 5% as dispersive as Na (Curtin et al. 1994b). The specific Mg effect was explained on the basis of the larger hydrated diameter of the Mg ion compared to the Ca ion. The larger hydrated diameter of Mg would result in stronger repulsive forces between clay particles in the Mg dominated system compared to a Ca dominated system (Emerson and Chi 1977). Fuller (1993) suggested that uncharged polymers, such as dextran, would collapse more completely on a Mg saturated clay than on a Ca saturated clay and would therefore be less likely to form bridges and would be less able to flocculate dispersed clay particles. The weaker electrostatic bonds formed by Mg should also result in weaker cation bridges between clay particles and organic binding agents. Lyubarstev et al. (1998) reported Mg ions to be ineffective in inducing associations between low charge density organic polyanions, whereas Ca ions were very effective.

An effect of CEC on clay dispersion has been reported over a range of soils (Coughlan and Loch 1984; Brubaker et al. 1992). The effect has been attributed to the relationship between CEC and clay mineralogy and the resultant increase in ease of aggregate slaking and hence clay dispersion as montmorillonite content and CEC increase (Coughlan and Loch 1984). In Gray Brown Podzolic soils which contain montmorillonite, the montmorillonite appears to be the mineral which translocates most readily (Cady 1967). Montmorillonite was dominant in horizons showing clay accumulation in a Miami silt loam which contained montmorillonite, vermiculite, illite, and kaolinite in all horizons (Thorp et al. 1967). The greater mobility of montmorillonite was attributed to montmorillonite's small particle size, physical and chemical reactivity, swelling properties, and ability to form complexes with organic compounds. However in the soils along the prairie toposequence, increases in CEC are associated with reduced dispersibility of clay indicating that the increase in CEC is not due to an increase in montmorillonite content. Within a relatively young catena such as this, clay mineralogy should be consistent due to uniform parent material and limited time for clay formation or degradation. Therefore, mineralogy differences could not account for the CEC effect. CEC could also simply indicate variation in clay content but this was not supported by linear regression of CEC and clay content ($r^2 = 0.004$). CEC did correlate well with soil organic C ($r^2 = 0.78$, $p = 0.020$), suggesting that variations in CEC within the catena were due to variations in organic matter, more specifically negatively charged organic matter. This would indicate a role for organic matter cation exchange sites in reducing clay dispersion, probably through the formation of polyvalent cation bridges between acidic functional groups on the organic matter and adjacent clay particles.

The consistently positive coefficient for CHCl_3 extractable lipid and the consistently negative coefficient for DEE extractable lipid and for the ratio of DEE: CHCl_3 extractable lipid suggest very different roles for the two lipid fractions.

Soil lipid content has been associated with increased wet aggregate stability (Capriel et al. 1990; Dinel et al. 1991a; Monreal et al. 1995). These hydrophobic (aliphatic) compounds contribute to stability directly by binding to soil inorganic constituents through polyvalent cation bridges (Dinel et al. 1992) or by producing a water repellent lattice around the soil aggregate as suggested by Capriel et al. (1990). Unbound lipids (DEE or CHCl_3 -EL) were found to be responsible for over 50% of the wet aggregate stability in both conventional and zero tillage treatments (Paré et al. 1999). Bound lipids (not extractable by DEE or CHCl_3) are involved in more permanent stability of soil aggregates (Dinel et al. 1992). Monreal et al. (1995) found that the effect of lipids did not extend to stabilizing microaggregates and therefore would be expected to have only a minor role in reducing clay dispersion once macroaggregates have been broken down.

DEE extractable lipids are relatively low molecular weight compounds with relatively more functional groups and are easily biodegradable whereas CHCl_3 extractable lipids are relatively high-molecular weight paraffinic compounds with fewer functional groups and change little during decomposition (Dinel et al. 1996). Both lipid types however are highly hydrophobic (extractable by organic solvents) and would be expected to bind by similar mechanisms to soil particles. DEE-EL are smaller than CHCl_3 -EL and have more functional groups and as a result could be expected to do a better job of coating and protecting other binding agents from dissolution by water. Large CHCl_3 -EL molecules could have the opposite effect if rather than protect binding agents, they bind to soil particles, and

due to their large size, block binding sites.

Another possible explanation of the apparent opposing effect of DEE-EL and CHCl_3 -EL on clay dispersion may be that the DEE: CHCl_3 extractable lipid ratio is indicating a difference in other soil organic matter components. Dinel et al. (1996) used DEE extractable lipid and DEE: CHCl_3 extractable lipid as indicators of the biodegradability of organic matter. Higher levels of DEE extractable lipid relative to CHCl_3 extractable lipid indicate that the organic matter is more easily biodegraded or is less decomposed. On this basis, the coefficients for both lipid fractions and the DEE: CHCl_3 extractable lipid ratio all indicate that clay dispersion increases as organic matter becomes more bioresistant or more decomposed.

The soil organic matter system is dynamic with addition of fresh organic materials, decomposition and humification occurring throughout the growing season. Under well drained conditions, decomposition rates will be lowest when either temperature or moisture or both are so low so as to virtually stop the activity of those organisms responsible. All of the slope positions were well drained and therefore one would expect to find the highest production of organic material and the fastest rate of decomposition at the moistest location, that is the toe-slope (D-3). If the initial addition of organic material has similar composition, it is the relative rates of addition and decomposition of organic material which determines whether or not the organic matter in one sample is more bioresistant or decomposed than another.

Along this catena, the mid-slope and toe-slope positions (D-2 and D-3) supported the same dominant species however the higher DEE to CHCl_3 extractable lipid ratio at D-2 indicates that the soil organic matter was on average more humified at D-3. Decomposition

byproducts undergo polymerization in the process of humification to form humus, which is resistant to decomposition (Paul and Clark 1996). The majority of soil humus consists of the large group of polyfunctional polymers known as humic and fulvic acids. The binding of these organic acids to clay particles via cation bridges could increase the dispersibility of the clay. Humic acid (a bioresistant decomposition product) has been shown to be an effective clay dispersant for Na-clay but reduced clay dispersion for the same clay following the addition of hydroxy Al polyvalent cations (Gu and Doner 1993). Heil and Sposito (1993a) found that suspensions of soil clay flocculated more easily following removal of the soil organic matter (treatment with H_2O_2). The increase in clay dispersion due to binding with humic acids could be a result of the increase in the negative charge and hence the repulsive forces between the clay-polymer particles as suggested by Oades (1984) or through steric hindrance of flocculation (Heil and Sposito 1993b). A role for organic acids such as humic and fulvic acids in facilitating the downward translocation of clay from eluvial horizons and the production of cutans in the Bt horizon has been indicated (Oades 1984; Gu and Doner 1993) especially if the soil exchange sites contain large percentages of monovalent cations such as Na.

Humic substances make up about 70-80% of the organic matter in most mineral soils (Schnitzer 1986) and can contribute substantially to the soil CEC. Clay minerals such as vermiculite or smectite can have a CEC of 70 to 250 $cmol\ kg^{-1}$ clay but humic substances may have 400 to 900 $cmol$ of charge kg^{-1} humic substance (Sposito 1989). In these soils, the CEC due to soil organic matter has been implicated in the reduction of clay dispersion and therefore soil humic substances would appear to have a net binding rather than a dispersive role in this case. Humic substances are a very diverse group and this does not preclude the

possibility that some humic or fulvic acids are enhancing the dispersion of clay particles (and hence clay translocation deeper into the soil profile) while others are involved in binding clay particles within soil aggregates.

The actual reason for the prominence of the lipid fractions in the regression models is most probably the direct effects of the DEE-EL and CHCl_3 -EL on clay dispersion. However the indication they give as to the degree of decomposition of other soil organic matter constituents cannot be completely ruled out.

4.4 Conclusions

The destabilizing role of exchangeable Mg is the main factor explaining differences in clay dispersion along the toposequence studied. Though not the main factor influencing clay dispersion, soil organic fractions did play a role in reducing dispersion. Unbound soil lipids have a role in both stabilizing the macroaggregates, probably through protection of water-soluble binding agents, as well as some soil lipids appear to aid dispersion of clay, possibly through adsorption to the clay particles blocking some of the binding sites. Variations in soil carbohydrate levels did not affect clay dispersibility. This does not rule out a role for soil polysaccharides but may indicate that when polysaccharides are at high levels such as in a native prairie soil, other factors will determine observed variations in clay dispersion.

The significance and contribution to knowledge of this chapter is to emphasize the critical role of exchangeable Mg and Ca in determining the strength of the bonds holding clay particles in soil aggregates. The dispersive effect of Mg ions in comparison to Ca ions

can be attributed in part to their effect on the diffuse double layer and clay swelling as well as a weaker attraction to clay surfaces. The weakening of clay-polymer bonds when Mg replaces Ca as the bridging cation also appears critical for explaining the increased clay dispersibility due to Mg. The basis for this effect is less clear. Further research into the different effects that Ca ions and Mg ions have on clay dispersibility is warranted.

5. SUMMARY AND CONCLUSIONS

In both agricultural and native prairie soils, there was a steady increase in the amount of clay dispersed from soil aggregates as the amount of ultrasonic energy applied to the soil:water suspensions was increased. The soils from each of the three sites produced characteristic clay dispersion curves (Figures 3.1, 3.4, 3.5, and 4.2). A large portion of the clay in the surface soil layer at the cultivated sites was easily dispersed. The Osborne clay soil had the highest percentage of weakly bound clay with an average E-half value of 42 kJ L⁻¹ (Table 3.4). The Fortier silty clay soil had E-half values ranging from 80 to 116 kJ L⁻¹ for the 0 to 5 cm depth (Table 3.4). In contrast, the shoulder and mid-slope positions of the native prairie catena had slightly less than 30 % of their clay dispersed when subjected to 100 kJ L⁻¹ of ultrasonic energy and E-half values of 189 and 237 respectively (Figure 4.3). Clay dispersion from the toe-slope position had an E-half value of 121 kJ L⁻¹ and was more similar to that of the cultivated soils than the to the other prairie soils.

Two seasons of conventional tillage following 10 years of zero tillage on an Osborne clay soil resulted in a small decrease in soil organic C in the surface 10 cm. Soil lipid levels were also influenced by tillage. DEE-EL and TEL values were higher under zero tillage than conventional tillage (33 % and 30 % higher respectively), however CHCl₃-EL was not influenced by tillage. Higher exchangeable K levels in the 0-10 cm depth under zero tillage indicates a different nutrient stratification under zero tillage in comparison to conventional tillage. Clay dispersibility was not significantly affected by tillage treatment or season. The

high rainfall and naturally poor drainage of the Osborne clay site interfered with planned tillage operations, reduced crop growth, and would have introduced extra variability that masks tillage effects. The treatments would need to be maintained for several more years at this site before the tillage systems could be compared with confidence.

Several differences between zero and conventional tillage were evident for the Fortier silty clay. Most obvious was the difference in aggregate kind and aggregate size distribution. Conventional tillage aggregates from the 0-5 cm depth were subangular blocky to granular whereas zero tillage resulted in angular blocky aggregates which on average were larger in size (Table 3.1). Average aggregate size increased with depth for both tillage treatments. Total hexose equivalents, and therefore soil polysaccharide levels, decreased markedly from mid-tillering to fall under both tillage treatments (45 % decrease under conventional tillage and a 42 % decrease under zero tillage) and in the fall 0 to 5 cm samples were higher under zero tillage than conventional tillage. In contrast soil organic C levels showed a slight increase by fall and total extractable soil lipid levels remained constant for both treatments. In general, soil lipids decompose more slowly than soil polysaccharides, therefore, it is not surprising that there would be more seasonal variation in carbohydrate than in soil extractable lipids. The conventional tillage treatment had lower clay dispersibility than the zero tillage treatment at mid-tillering but by fall there was no significant difference. Of note was the much greater seasonal variation in clay dispersibility under conventional tillage compared to zero tillage.

Multiple regression analyses proved useful for deducing an explanation of the variation in clay dispersion between samples and treatments. For the surface 5 cm of the Fortier silty clay, simple linear regressions were only able to explain 12% to 41% of the

observed variation for pooled data for both treatments and both sampling times (Table 3.5). The correlations were only significant at three of the seven energy levels. Carbohydrate gave the best (or tied for best) r^2 value at each energy level. The same data set analysed by stepwise multiple regression produced models with much better r^2 values and more of the models were statistically significant except for the 200 kJ L^{-1} energy level at which simple linear regression models were better than any multiple linear regression models (Table 3.6). Again carbohydrate appeared in the model for each energy level and was negatively correlated with clay dispersion. Extractable lipids appeared in most of the models with the two fractions exhibiting opposing effects. Increases in DEE-EL were associated with reduced clay dispersion and increases in CHCl_3 -EL were associated with increased clay dispersion. Total organic C was also positively correlated with clay dispersion. Exchangeable cations appeared in several models but not consistently. Some patterns were emerging but considerable variation was still not explained by the models.

Stepwise multiple regression analysis was repeated on the individual treatments. Although the reduced data set restricted the number of predictor variables that could be included in the final models, the models in general were much improved in either r^2 values or statistical significance or both. One of the clearest changes was the appearance in most of the models for either treatment of exchangeable Ca, exchangeable Mg, or the ratio of Ca to Mg ions on the exchange sites. Exchangeable Ca was associated with reduced dispersion, exchangeable Mg with increased dispersion, and clay dispersion decreased as the ratio of exchangeable Ca to Mg increased. Extractable lipids also figured prominently in models for both tillage treatments. As with the pooled data, increased levels of DEE-EL were associated with reduced clay dispersion and increased levels of CHCl_3 -EL were associated

with increased clay dispersion for both treatments. In contrast, differences in the role of carbohydrate were apparent between the treatments. Although associated with reduced dispersion in both treatments, carbohydrate is only important in models for the two lowest energy levels under zero tillage. In the conventional tillage treatment, carbohydrate appears in models for the mid-range of energy levels. Total soil organic carbon and clay content appear only in models for the zero tillage treatment. Clay content is associated with increased clay dispersion at the lower energy levels and increases in total soil organic carbon are associated with increased clay dispersion at the higher energy levels.

On the native prairie catena, unexpectedly, the most arid position, the shoulder of the knoll, had the highest soil organic C concentration (56.7 g kg^{-1} of soil in the surface 10 cm) and the toe-slope position, which was the most moist had the lowest concentration (50.9 g kg^{-1} of soil). However, the measured soil organic matter components did not follow the same pattern, and were consistent across slope positions. Due to differences in solum depth (Table 4.1), total organic C in the solum per unit surface area cannot be estimated. To correlate soil properties to soil forming processes, depth to carbonate was used as an indicator of degree of leaching and therefore of long term moisture status at each slope position. All three positions were well drained and no indications of impervious soil layers or perched water tables which could influence soil formation were evident. Of all the soil properties measured, only exchangeable Mg and hence the exchangeable Ca to Mg ratio were significantly correlated to depth to carbonate (r^2 of 0.79 and 0.80 respectively, $p = 0.05$). Clay dispersion curves indicate that on average the clay at the toe-slope position was less strongly held within aggregates than the clay at the shoulder of the knoll. This would suggest that clay dispersibility was reduced by increases in total soil organic C and increased by

higher levels of exchangeable Mg. Stepwise multiple regression analysis confirmed a role for exchangeable Mg (and the exchangeable Ca to Mg ratio) in explaining clay dispersibility (Table 4.7). CEC was prominent (in one or both models at each energy level). Linear regression analysis of CEC and other soil parameters suggests that the variation in CEC at this site is largely due to variation in the soil organic matter content and not due to variation in clay content. Therefore, the presence of CEC as a predictor variable in the model is most likely a result of the role of organic cation exchange sites and hence cation bridges in binding organic matter to clay particles. As in the Fortier silty clay, increases in DEE-EL levels were associated with reduced dispersion of clay and increases in CHCl_3 -EL were associated with increases in clay dispersion.

The Fortier silty clay tillage treatments and native prairie soils all showed a consistent association between exchangeable Mg and/or exchangeable Ca and dispersible clay across a wide range of applied energy levels. Clay was more easily dispersed as exchangeable Mg increased relative to exchangeable Ca. Exchangeable Na levels were too low to play a significant role in clay dispersion in these soils. Although both Mg and Ca ions are divalent, the Mg ion is more strongly polarizing, holds water molecules more tightly, and as a result has a larger hydrated diameter. As the content of exchangeable Mg relative to exchangeable Ca increases in a clay sample, the cation bridges between clay particles will be weaker, the diffuse double layer will be thicker, and the clays will swell more as water is added. As the content of Mg ions relative to Ca ions on exchange sites increases, the weaker bonds and increased repulsive forces which result upon wetting would result in aggregates which slake more easily and clay particles which disperse more readily. The bond strength and hence the preference for Ca ions rather than for Mg ions is even greater

on the exchange sites of soil organic matter. Clay particles which are held in microaggregates via cation bridges to soil organic matter exchange sites would therefore be held much more weakly if Mg was the bridging cation rather than Ca. Exchangeable Mg content will also affect the binding of organic molecules with few or no exchange sites. These compounds rely largely on the gain in entropy resulting from displacing bound water molecules from the hydration shells of exchangeable cations and clay surfaces as the driving force for binding to clay particles. They are less able to displace the water from around a Mg ion and would not be able to form strong bridges between clay particles at high Mg levels. Many soil polysaccharide molecules would fall into this category. It is evident that the exchangeable Ca to Mg ratio will have an effect on many of the different types of bonds which stabilize soil aggregates and hold clay particles in soil microaggregates. This would account for their presence in most of the models. The magnitude of the Mg effect changes with the bond type, however, and therefore variations in the effect at different energy levels and under different management systems (conventional tillage, zero tillage, and native prairie) would be expected.

Extractable lipids were also correlated with variability in clay dispersion for both the Fortier silty clay site and the native prairie catena at most energy levels. Increased DEE-EL was consistently associated with reduced clay dispersion and increased CHCl_3 -EL was consistently associated with increased clay dispersion. Removal of TEL has been shown to reduce aggregate stability but the effects of the two fractions were not considered separately (Dinel et al. 1992; Paré et al. 1999). The smaller DEE-EL have relatively more functional groups than CHCl_3 -EL and therefore could be more effective at coating and protecting more water soluble binding agents such as polysaccharides. Increased aggregate stability and

reduced clay dispersion would result. The larger CHCl_3 -EL would be less efficient protectors of other binding agents and due to their size could block binding sites and also create a steric hindrance to flocculation of clay particles. The ratio of DEE-EL to CHCl_3 -EL is also an indicator of the degree of decomposition of the soil organic matter. Relatively more DEE-EL indicates a higher content of easily decomposable organic matter. The opposing effects of DEE-EL and CHCl_3 -EL could be indicating that variations in transient binding agents such as root and microbial mucilages are major contributors to the measured variation in clay dispersibility. The dominance of carbohydrate in the simple linear regressions and the presence of carbohydrate in many of the multiple linear regression models for the Fortier silty clay supports this explanation. However the fact that selective removal of unbound lipids has been shown to reduce aggregate stability and also that the unbound lipids are in models for so many of the energy levels suggest a direct role as well.

Carbohydrate concentration was not associated with variation in clay dispersibility within the native prairie soils. This is not to say that soil polysaccharides did not have a role in binding clay, only that the variation seen in clay dispersion was not caused by variation in polysaccharide levels. Solubilized carbohydrate content was virtually identical among the three slope positions.

Soil polysaccharide did play a role in explaining the variation for the Fortier silty clay. Carbohydrate was more important in explaining variation for the conventional tillage treatment than for the zero tillage treatment. Carbohydrate decreased markedly from mid-tillering to fall for both tillage treatments, however conventional tillage showed a much greater increase in clay dispersion over the same time period. This suggests that carbohydrate and therefore soil polysaccharides play a greater role in reducing clay

dispersion under conventional tillage management.

At low levels of applied energy, clay content correlated well with clay dispersion for the zero tillage treatment. The correlation suggests that clay swelling was an important factor in the release of the most weakly held clay particles but was not important for more strongly bonded clay particles. Clay content did not appear in the models for the conventional tillage treatment or the native prairie soils.

Total soil organic C was a poor predictor of clay dispersibility. Soil organic C was present in models for many of the energy levels of the zero tillage treatment but was absent from both conventional tillage models and the native prairie soils. Where present, increases in soil organic C were associated with increased dispersion of clay. Most likely this was due to organic acids such as fulvic acid binding to clay particles and increasing the repulsive force between particles. The importance of organic matter CEC for reducing clay dispersion in the native prairie soils shows the opposite is also possible. When appropriate cations are present, exchange sites on organic matter can bind to clay particles via cation bridges and if the organic polymer is large enough and has enough exchange sites in can draw particles together and bind them into microaggregates. Both effects will occur in the same soil at the same time but which of the two dominates will depend on the composition of the soil organic matter and the relative proportions of the various cations. Trivalent cations will tend to form stronger bridges than divalent cations and of the divalent cations common in Manitoba, Ca will form stronger bridges than Mg. Monovalent cations will tend to form very weak bridges between organic matter and clay particles.

The distinctive shapes of the different clay dispersion curves show that not only do the bonds holding clay particles vary in strength within a soil but also that the relative

proportion of the clay that is weakly held differs between soils more so than between tillage treatments. The least arid slope position of the native prairie site had a dispersion curve shaped more like the Fortier silty clay than like the other prairie slope positions and had an E-half value very close to that of the 0 to 5 cm depth of the conventional tillage mid-tillering sample.

The native prairie soils produced dispersion curves with more gradual changes in slope than did the cultivated soils indicating more uniformity in clay binding under native prairie. This was corroborated by correlations of the clay dispersed with other soil properties at each of the energy levels. The predictor variables in the best multiple regression models were very similar across the range of applied energy levels indicating that bond strengths were influenced by the same variables throughout. In contrast, the predictor variables for both tillage treatments of the Fortier silty clay changed as applied energy increased suggesting a greater variability in bond types than under native prairie conditions. The predictor variables were also different between tillage treatments at the same levels of applied energy. Although the average bond strength between clay particles has not been greatly affected by tillage management, the types of bonding between clay particles has been affected by tillage. Polysaccharides have a greater role under conventional tillage than under zero tillage. Clay content and total organic C (or most probably components of organic C which were not specifically measured in our study) are more important under zero tillage.

Exchangeable cations, most notably the relative amounts of exchangeable Ca and Mg, are extremely important in determining clay dispersion. They directly affect attractive and repulsive forces between clay particles but also affect the strength of bonds between soil organic compounds and clay particles. Despite being present in equal

concentrations under both tillage treatments, the effect of exchangeable Ca and Mg was not apparent for the Fortier silty clay until the two tillage treatments were examined separately indicating that their effect was not the same in the two tillage systems. This further supports the conclusion that different bonding mechanisms and therefore different organic compounds were dominant in the bonds between clay particles under the two tillage management systems.

The importance of exchangeable cations, especially the ratio of Ca to Mg, suggests that the effects of exchangeable cations should be considered whenever aggregate stability and clay dispersibility of soils are studied.

Calcium to magnesium ratios should be considered in the evaluation of irrigation water in addition to the more traditional SAR. As irrigated acreage in Manitoba continues to expand there will be pressure to use lower quality water and care will be required to protect our soils from the negative effects of clay dispersion caused by the addition of water with poor Ca:Mg ratios.

The seasonal variation in clay dispersion is interesting and bears further study. Samples taken the following spring (immediately after spring thaw and again just before planting) would have added useful information on the effects of freeze-thaw cycles and would also have provided valuable information for determining when clay is most susceptible to dispersion.

The large seasonal variation in clay dispersibility under conventional tillage also has implications for other clay dispersion studies. To properly compare soils or treatments, samples should be taken as close as possible to the same time or crop stage. The degree of seasonal variation is also affected by treatment. In our study it was seen that treatments

which differed in clay dispersibility at one sampling time were not different at another. Therefore it would be best to not only sample at the same time but also to have more than one sampling time to properly compare treatments or soils.

6. CONTRIBUTION TO KNOWLEDGE

Regression analyses of clay dispersion data shows that the clay particles in soil aggregates under conventional and zero till management are not held by the same mechanisms. Pooling data from the different tillage treatments therefore masks the treatment effect on clay dispersibility and can lead to incorrect conclusions. Separate examination of the clay dispersion data for soil aggregates under conventional tillage, zero tillage, and native prairie grassland demonstrated that the content of extractable lipids and the relative proportion of exchangeable Mg to exchangeable Ca were important determinants of clay dispersibility in all three management systems.

Effects of total extractable lipids on soil aggregate stability have been reported, however the individual effects of the two extractable lipid fractions on clay dispersion have not previously been studied. DEE-EL and CHCl_3 -EL were found to have opposing effects on clay dispersibility under both zero and conventional tillage and under native prairie as well. DEE-EL was suspected to act through coating and protecting other more water soluble binding agents and thereby reducing clay dispersibility. The larger CHCl_3 -EL increased clay dispersibility possibly through blocking binding sites on the clay and soil organic matter or by creating steric hindrances to clay flocculation.

The Ca to Mg ratio influences bonds stabilizing macroaggregates as well as microaggregates. The dispersive effect of Mg ions in comparison to Ca ions can be attributed in part to their effect on the diffuse double layer and clay swelling and hence

reduced resistance to aggregate slaking as Mg content increases. Mg is held less tightly than Ca on clay exchange sites and therefore forms weaker bridges between individual clay particles and increases clay dispersibility. The effect of Mg on cation bridges between mineral and organic anions as well as the effect on uncharged polymer adsorption on clay surfaces reduces the strength of bonds holding clay in microaggregates in comparison to Ca. The weakening of clay-polymer bonds when Mg replaces Ca as the bridging cation appears critical for explaining the increased clay dispersibility due to Mg. It is evident that the exchangeable Ca to Mg ratio will have an effect on many of the different types of bonds which stabilize soil aggregates and hold clay particles in soil microaggregates. The magnitude of the Mg effect changes with the bond type, however, and therefore variations in the effect at different energy levels and under different management systems (conventional tillage, zero tillage, and native prairie) would be expected. This would explain why the Mg effect became more obvious when the conventional and zero tillage treatments were analysed separately. Exchangeable cations, especially the ratio of Ca to Mg where they are dominant, need to be taken into consideration whenever aggregate stability and clay dispersibility of soils is studied.

The dispersive effect of Mg on soil clay is of particular concern in the prairie provinces as Ca and Mg are the dominant exchangeable cations in many of our agricultural soils and should receive more attention by the Canadian agricultural community. The potentially deleterious effect of Mg will become increasingly important as irrigated acreage rises on the Canadian prairies and good quality irrigation water is supplemented or replaced by water of lower quality.

News Release

The breakdown of soil structure and subsequent dispersion of clay can cause reduced soil porosity and surface crusting. Poor crop emergence, restricted root growth, reduced infiltration of precipitation or irrigation water, increased runoff, and increased soil erosion can result and will all contribute to decreased agricultural production. Soils from zero- and conventional tillage plots near Portage la Prairie, MB, as well as soil from a hill slope under native prairie grassland near Deloraine, MB, were used in our study. The levels of the various soil organic matter components varied only slightly at each site and generally were not significantly affected by tillage or slope position .

Clay dispersion was not related to the total soil organic matter content but was affected by the quantity of hydrophobic (water repellent) organic compounds present in the soil organic matter. The smaller hydrophobic molecules (extracted by the organic solvent diethyl ether) helped prevent clay dispersion while the larger hydrophobic molecules (extracted by chloroform, another organic solvent) increased clay dispersion.

For both locations, the most important factor determining the resistance of the clay to dispersion was the relative quantities of exchangeable Ca and Mg ions present in the soil. Higher Mg and lower Ca content resulted in higher clay dispersion. This is of particular concern where irrigation water is applied. The deleterious effect of Na on soil structure has long been known and it is standard practice to assess the quality of irrigation water by its sodium adsorption ratio (SAR). Our research indicates that the ratio of Mg to Ca ions in the irrigation water is also critical and should be considered when assessing irrigation water quality.

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8. APPENDICES

Appendix I

Effect of tillage on earthworm populations for a Fortier silty clay as determined by a count of worms which surfaced following application of a formalin solution to the soil surface.

Tillage treatment	Replicate				Mean
	1	2	3	4	
large earthworms	worms/square meter ^z				
zero till ^y	26	38	22	10	24
short term conventional till	24	14	20	6	16
long term conventional till	8	18	4	2	8
small earthworms					
zero till	14	32	10	8	16
short term conventional till	10	12	10	6	9.5
long term conventional till	34	10	4	2	12.5
total earthworm numbers					
zero till	80	140	64	36	40
short term conventional till	68	52	60	24	25.5
long term conventional till	84	56	16	8	20.5

^z 25 mL of 37% formaldehyde in 4.5 L of water applied to 0.25 m² (50x50 cm) and count worms which surface during 10 minutes following application. Two quadrats sampled in each plot.

^y zero till = 13 years zero till

short term conventional till = 11 years zero till followed by 2 years conventional till

long term conventional till = 13 years conventional tillage

Appendix II

Specific surface area of clay isolates of a Fortier silty clay, Osborne clay, and three slope positions under native prairie as determined by EGME adsorption.

	specific surface area ^z	
	carbonates removed ^y	no pre-treatment to remove carbonates
	m ² g ⁻¹	
Fortier silty clay		
0-5 cm depth	369	478
5-10 cm depth	371	457
Osborne clay		
0-10 cm depth	320	399
Prairie toposequence		
D-1 (shoulder of knoll)		339
D-2 (mid-slope)		302
D-3 (toe-slope)		290

^z
^y all samples were treated with 30% H₂O₂ to oxidize soil organic matter treated with 1N NaOAc prior to soil organic matter oxidation

Appendix III

Regression analyses of factors affecting the dispersion of clay from 0.25 to 0.85 mm soil aggregates during sonication at selected levels of applied energy for the 0 to 10 cm depth of an Osborne clay: Best multiple linear regression models for organic and mineral parameters considered separately and combined.

Comparison of regression models for clay dispersion at selected energy levels containing organic parameters only, mineral parameters only, and both mineral and organic parameters: Pooled tillage treatments, Osborne clay soil - Brunkild site (n=16).

level of dispersive energy	Organic and Mineral Parameters included in Models						Organic Parameters Only						Mineral Parameters Only						
	model p value	r ²	adjusted r ² y	model parameters z	parameter coefficients	p value of parameter	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	
kJ L ⁻¹	10	0.1334	0.3614	0.2017	constant TEL K Mg	0.40533 -0.56606 0.10584 -0.02898	0.0544 0.0258 0.0612 0.1739	0.1334	0.3614	0.2017	constant TEL K Mg	0.40533 -0.56606 0.10584 -0.02898	0.0544 0.0258 0.0612 0.1739	0.6134	0.0169	-0.0533	constant K	0.23653 0.02178	0.1855 0.6314
	25	0.0635	0.5270	0.3550	constant Ca ESP OC Mg	0.80357 0.01322 -0.04461 -0.00667 -0.02643	0.0000 0.0468 0.0560 0.0115 0.0342	0.1119	0.2861	0.1762	constant DEE CHCl3/TEL	0.64197 -0.2544 -0.45231	0.0000 0.0488 0.0830	0.0856	0.1963	0.1389	constant Ca/Na	0.46 2.35E-05	0.0000 0.0856
	50	0.0485	0.2501	0.1966	constant Ca	0.68445 -0.00789	0.0000 0.0485	0.1309	0.095	0.1553	constant carb	0.62275 -0.00763	0.0000 0.1309	0.0327	0.2863	0.2353	constant Ca + Mg	0.7032 -0.00618	0.0000 0.0327
	100	0.0028	0.8481	0.7469	constant Ca Na K Mg clay carb	1.30584 0.01343 -0.10505 -0.07918 0.02802 -0.66433 -0.01848	0.0001 0.0156 0.0118 0.0027 0.0113 0.0030 0.0075	0.357	0.1465	0.0152	constant DEE OC	0.57488 -0.19814 0.00299	0.0000 0.1743 0.2396	0.0088	0.5168	0.4425	constant Ca/Na Ca	0.44195 3.18E-05 0.01159	0.0000 0.0117 0.0162
	200	0.0020	0.6970	0.6213	constant Ca clay DEE	1.05709 0.00575 -0.40706 -0.1512	0.0000 0.0656 0.0038 0.0473	0.1681	0.1311	0.069	constant carb	0.65861 0.00705	0.0000 0.1681	0.0012	0.7197	0.6496	constant Ca Ca/Na clay	0.7344 0.0097 1.62E-05 -0.196	0.0000 0.0023 0.0280 0.0332
	300	0.0054	0.6384	0.5480	constant clay CEC OC	1.33972 -0.61906 0.00374 -0.00372	0.0000 0.0019 0.0372 0.0567	0.3451	0.0638	-0.003	constant carb	0.70297 0.00657	0.0000 0.3451	0.0063	0.4236	0.3824	constant clay	1.17119 -0.44844	0.0000 0.0063
	600	0.0021	0.6115	0.5517	constant CEC OC	0.7472 0.00513 -0.00307	0.0000 0.0008 0.0104	0.2439	0.0956	0.031	constant TEL	0.87315 -0.09077	0.0000 0.2439	0.003	0.7419	0.6481	constant CEC Na/(Ca+Mg) Ca/Na K	0.35623 0.00364 4.52142 4.93E-05 0.06566	0.0032 0.0099 0.0023 0.0028 0.0144

^z Parameter abbreviations: CEC cation exchange capacity cmol/kg; Na exchangeable Na; Ca exchangeable Ca; Mg exchangeable Mg; ESP exchangeable Na percentage; SAR Na adsorption ratio; CHCl3 Chloroform extractable lipid (g/kg soil); carb total hexose equivalents extracted by acid hydrolysis (g/kg soil); DEE Diethyl ether extractable lipid (g/kg soil); TEL Total extractable lipid (DEE + CHCl3) (g/kg soil); OC Organic Carbon (g/kg soil); clay clay content as a percent of the mineral fraction

^y adjusted R²: A potential problem with R² is that it always increases as new independent variables are included in the model (RSS always decreases), even if they don't possess any relationship with the dependent variable. Adjusted R² is adjusted for the number of independent variables in the model to correct for this problem, and, therefore, will often be more interesting than the unadjusted R². Adjusted R² is computed as 1 - (n - 1)RMS/SS_T, where RMS is the residual mean square and n is the number of cases in the regression.

Comparison of regression models for clay dispersion at selected energy levels containing organic parameters only, mineral parameters only, and both mineral and organic parameters: Conventional tillage treatments for an Osborne clay soil - Brunkild site (n=8).

level of dispersive energy	Organic and Mineral Parameters included in Models						Organic Parameters Only											
	model p value	r ²	adjusted r ² ^y	model parameters ^z	parameter coefficients	p value of parameter	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter
kJ/L																		
10	0.0055	0.9978	0.9923	constant	-0.44546	0.0084	0.026	0.5900	0.5217	constant	-0.34702	0.1796	0.0006	0.9961	0.9909	constant	-0.88885	0.0014
				OC	0.01852	0.0021				OC	0.01419	0.0260				Ca	0.05978	0.0002
				CHCl3	-3.45045	0.0040										Ca/Na	4.64E-05	0.0009
				Mg	0.03724	0.0054										clay	0.37673	0.0062
				carb	-0.01918	0.0267										K	-0.04615	0.0087
				Na	0.05103	0.0311												
25	0.0243	0.5984	0.5314	constant	0.95266	0.0010	0.0279	0.5811	0.5113	constant	0.58797	0.0000	0.0816	0.633	0.4861	constant	0.36968	0.0044
				clay	-0.50959	0.0243				CHCl3/TEL	-0.64215	0.0279				Na	-0.07066	0.1305
																Ca/Mg	0.05842	0.1324
50	0.1392	0.3262	0.2139	constant	0.4442	0.0005	0.073	0.6489	0.5084	constant	0.81046	0.0003	0.1286	0.3409	0.231	constant	0.72447	0.0003
				DEE	0.32268	0.1392				CHCl3/TEL	-0.49719	0.0352				Ca+Mg	-0.00693	0.1286
										carb	-0.01972	0.0516						
100	0.0093	1.0000	0.9998	constant	1.8246	0.0030	0.3712	0.1347	-0.0095	constant	0.45051	0.0739	0.0027	0.9617	0.933	constant	1.2305	0.0002
				clay	-0.85536	0.0040				OC	0.00426	0.3712				clay	-0.56875	0.0010
				Ca/Mg	-0.08985	0.0096										Ca	-0.01453	0.0058
				carb	-0.01648	0.0119										CEC	0.00432	0.0084
				CHCl3	-0.74531	0.0210												
				Ca	-0.00333	0.0378												
				OC	0.00156	0.0460												
200	0.0047	0.9494	0.9115	constant	0.49355	0.0005	0.0633	0.4629	0.3734	constant	0.43751	0.0136	0.0295	0.5736	0.5025	constant	0.74825	0.0000
				ESP	-0.03689	0.0020				OC	0.0061	0.0633				ESP	-0.03445	0.0295
				DEE	0.34867	0.0113												
				Ca	0.00817	0.0200												
300	0.0006	0.9826	0.9695	constant	0.61103	0.0000	0.2477	0.2146	0.0837	constant	0.83243	0.0000	0.0189	0.629	0.5672	constant	0.7953	0.0000
				Na	-0.12622	0.0002				CHCl3/TEL	-0.40364	0.2477				ESP	-0.04977	0.0189
				TEL	0.52837	0.0016												
				CHCl3	-0.40247	0.0593												
600	0.0031	0.9988	0.9957	constant	-0.26615	0.0180	0.6324	0.0406	-0.1193	constant	0.88757	0.0001	0.0788	0.4271	0.3316	constant	0.68731	0.0001
				CEC	0.01219	0.0008				carb	-0.0058	0.6324				K	0.04127	0.0788
				DEE	0.6821	0.0017												
				clay	0.51148	0.0024												
				CHCl3	-0.84015	0.0043												
				Mg	-0.01322	0.0130												

^z Parameter abbreviations: CEC cation exchange capacity cmol/kg
 Na exchangeable Na
 Ca exchangeable Ca
 Mg exchangeable Mg
 ESP exchangeable Na percentage
 SAR Na adsorption ratio
 CHCl3 Chloroform extractable lipid (g/kg soil)
 carb total hexose equivalents extracted by acid hydrolysis (g/kg soil)
 DEE Diethyl ether extractable lipid (g/kg soil)
 TEL Total extractable lipid (DEE + CHCl3) (g/kg soil)
 OC Organic Carbon (g/kg soil)
 clay clay content as a percent of the mineral fraction

^y adjusted R²: A potential problem with R² is that it always increases as new independent variables are included in the model (RSS always decreases), even if they don't possess any relationship with the dependent variable. Adjusted R² is adjusted for the number of independent variables in the model to correct for this problem, and, therefore, will often be more interesting than the unadjusted R². Adjusted R² is computed as 1 - (n - 1)RMS/SS_T, where RMS is the residual mean square and n is the number of cases in the regression.

Comparison of regression models for clay dispersion at selected energy levels containing organic parameters only, mineral parameters only, and both mineral and organic parameters: Zero tillage treatments for an Osborne clay soil - Brunkild site (n=8).

level of dispersive energy	Organic and Mineral Parameters included in Models						Organic Parameters Only											
	model p value	r ²	adjusted r ² ^y	model parameters ^z	parameter coefficients	p value of parameter	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter
kJ L ⁻¹																		
10	0.0450	0.7107	0.5949	constant clay ESP	-1.63143 2.13672 0.18122	0.0503 0.0301 0.0538	0.1294	0.5587	0.3822	constant TEL carb	1.20651 -1.11657 -0.02985	0.0215 0.0569 0.1643	0.024	0.5999	0.5333	constant K	2.67596 -0.59148	0.0145 0.0240
25	0.1369	0.3293	0.2176	constant K	1.19111 -0.1839	0.0318 0.1369	0.3508	0.1457	0.0033	constant TEL	0.57157 -0.21397	0.0024 0.3508	0.194	0.4811	0.2735	constant clay Ca/Na	-0.21602 0.78327 -1.15E-04	0.5735 0.1146 0.1772
50	0.1533	0.3081	0.1928	constant Ca	0.69601 -0.00873	0.0002 0.1533	0.2119	0.2454	0.1196	constant carb	0.66456 -0.01157	0.0002 0.2119	0.1533	0.3081	0.1928	constant Ca	0.69601 -0.00873	0.0002 0.1533
100	0.0577	0.4775	0.3904	constant Mg	0.40482 0.03114	0.0070 0.0577	0.1304	0.3383	0.228	constant carb	0.47759 0.0165	0.0022 0.1304	0.0577	0.4775	0.3904	constant Mg	0.40482 0.03114	0.0070 0.0577
200	0.0132	0.8230	0.7523	constant clay CEC	1.14429 -0.66895 0.00361	0.0023 0.0245 0.0246	0.1345	0.3326	0.2214	constant carb	0.60333 0.01205	0.0001 0.1345	0.0621	0.4661	0.3771	constant clay	1.39006 -0.75619	0.0031 0.0621
300	0.0139	0.6631	0.6070	constant clay	1.80457 -1.1769	0.0010 0.0139	0.3815	0.1294	-0.0157	constant carb	0.66834 0.0098	0.3815 0.5354	0.0139	0.6631	0.607	constant clay	1.80457 -1.1769	0.0010 0.0139
600	0.0064	0.9408	0.8964	constant Mg K CHCl3	0.05555 0.02152 0.14362 0.38791	0.6944 0.0037 0.0109 0.0694	0.1904	0.2664	0.1441	constant carb	0.72998 0.00945	0.0000 0.1904	0.0096	0.9275	0.8731	constant CEC clay Ca/Mg	1.18819 0.00429 -0.4458 -0.07787	0.0008 0.0048 0.0278 0.0291

^z Parameter abbreviations: CEC cation exchange capacity cmol/kg; Na exchangeable Na; Ca exchangeable Ca; Mg exchangeable Mg; ESP exchangeable Na percentage; SAR Na adsorption ratio; CHCl3 Chloroform extractable lipid (g/kg soil); carb total hexose equivalents extracted by acid hydrolysis (g/kg soil); DEE Diethyl ether extractable lipid (g/kg soil); TEL Total extractable lipid (DEE + CHCl3) (g/kg soil); OC Organic Carbon (g/kg soil); clay clay content as a percent of the mineral fraction

^y adjusted R2: A potential problem with R2 is that it always increases as new independent variables are included in the model (RSS always decreases), even if they don't possess any relationship with the dependent variable. Adjusted R2 is adjusted for the number of independent variables in the model to correct for this problem, and, therefore, will often be more interesting than the unadjusted R2. Adjusted R2 is computed as $1 - (n - 1)RMS/SST$, where RMS is the residual mean square and n is the number of cases in the regression.

Appendix IV

Regression analyses of factors affecting the dispersion of clay from 0.25 to 0.85 mm soil aggregates during sonication at selected levels of applied energy for the 0 to 5 cm depth of a Fortier silty clay: Best multiple linear regression models for organic and mineral parameters considered separately and combined.

Comparison of regression models for clay dispersion at selected energy levels containing organic parameters only, mineral parameters only, and both mineral and organic parameters:
Pooled tillage treatments for a Fortier silty clay soil - Portage la Prairie site.

level of dispersive energy	Organic and Mineral Parameters Included in Models							Organic Parameters Only						Mineral Parameters Only										
	model p value	r ²	adjusted r ^{2y}	model parameters #	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N			
kJ L-1	50	0.0098	0.8315	0.7472	constant	-0.37502	0.1504	10	0.02	0.4523	0.3681	constant	0.11043	0.6872	16	0.1978	0.2207	0.1008	constant	0.22538	0.1068	16		
					carb	-0.01363	0.0359					carb	-0.01304	0.0154					clay	0.01027	0.1105			
					OC	0.01513	0.0104					OC	0.00714	0.1627					Mg	-0.05092	0.3073			
				CHCl3	0.83432	0.0263																		
	100	0.0183	0.9947	0.9763	constant	-0.36211	0.132	16	0.0063	0.5416	0.4711	constant	0.17959	0.4499	16	0.3143	0.0722	0.0059	constant	0.2679	0.2826	16		
					carb	-0.01607	0.0071					carb	-0.01394	0.0064					Ca + Mg	0.01389	0.3143			
					OC	0.01739	0.0169					OC	0.0085	0.0776										
					Ca/Mg	0.07236	0.072																	
K					-0.18838	0.0167																		
Na					-1.28329	0.0205																		
TEL					0.49332	0.029																		
clay	0.00648	0.0432																						
			200	0.1348	0.1525	0.092	constant	0.32994	0.1347	16	0.1348	0.1525	0.092	constant	0.32994	0.1347	16	0.3466	0.1504	0.0197	constant	0.70338	0.0004	16
				OC	0.00646	0.1348		OC	0.00646	0.1348					Mg	0.02916	0.1741		CEC	-0.00578	0.2708			
300	0.0006	0.9998	0.9992	constant	0.51382	0.0002	10	0.0471	0.8109	0.6596	constant	0.35883	0.072	10	0.3991	0.0513	-0.0165	constant	0.67965	0	16			
				carb	-0.00937	0.0003					carb	-0.00985	0.0233					K	0.02721	0.3991				
				TEL	0.41991	0.0007					OC	0.00445	0.1459											
				Na	-0.70828	0.0006					CHCl3/TEL	0.29634	0.0605											
				OC	0.00785	0.0009					DEE	0.42398	0.072											
				K	-0.06565	0.0011																		
				DEE	-0.15088	0.0036																		
				Mg	-0.00403	0.0752																		
400	0.0662	0.9806	0.9128	constant	0.66024	0.0155	10	0.1873	0.1207	0.0579	constant	0.82256	0	16	0.2351	0.089	0.0347	constant	0.95674	0	16			
				carb	-0.00626	0.0895					carb	-0.00514	0.1873					CEC	-0.00491	0.2351				
				Na	-1.6455	0.0177																		
				K	-0.22169	0.0191																		
				OC	0.00887	0.0594																		
				CHCl3	0.19439	0.2054																		
				TEL	0.32812	0.086																		
				clay	0.00546	0.0896																		
500	0.0413	0.8776	0.7552	constant	0.87187	0.0005	9	0.083	0.2134	0.1529	constant	0.86967	0	15	0.6369	0.0724	-0.0821	constant	0.61764	0.0143	15			
				carb	-0.01203	0.0208					carb	-0.00748	0.083					ESP	0.20241	0.355				
				TEL	0.87626	0.0182																		
				Ca/Mg	-0.10705	0.031																		
				DEE	-0.35666	0.1146																		
600	0.0159	0.9759	0.9276	constant	0.62696	0.0072	10	0.0674	0.2192	0.1634	constant	0.91191	0	16	0.3823	0.0549	-0.0126	constant	0.78562	0	16			
				carb	-0.01044	0.0164					carb	-0.00815	0.0674					K	0.03451	0.3823				
				Na	-1.37603	0.007																		
				OC	0.0115	0.0315																		
				K	-0.11709	0.0292																		
				CHCl3	0.42448	0.034																		
				Mg	0.01927	0.2685																		

* Parameter abbreviations: CEC cation exchange capacity cmol/kg
 Na exchangeable Na
 Ca exchangeable Ca
 Mg exchangeable Mg
 ESP exchangeable Na percentage
 SAR Na adsorption ratio
 CHCl3 Chloroform extractable lipid (g/kg soil)
 clay clay content
 DEE Diethyl ether extractable lipid (g/kg soil)
 TEL Total extractable lipid (DEE + CHCl3) (g/kg soil)
 OC Organic Carbon (g/kg soil)
 carb total hexose equivalents extracted by acid hydrolysis (g/kg soil)

^y adjusted R²: A potential problem with R² is that it always increases as new independent variables are included in the model (RSS always decreases), even if they don't possess any relationship with the dependent variable.
 Adjusted R² is adjusted for the number of independent variables in the model to correct for this problem, and, therefore, will often be more interesting than the unadjusted R². Adjusted R² is computed as 1 - (n - 1)RMS/SS_T, where RMS is the residual mean square and n is the number of cases in the regression.

Comparison of regression models for clay dispersion at selected energy levels containing organic parameters only, mineral parameters only, and both mineral and organic parameters. Conventional tillage treatment for a Fortier silty clay soil - Portage la Prairie site.

level of dispersive energy	Organic and Mineral Parameters Included in Models							Organic Parameters Only							Mineral Parameters Only							
	model p value	r ²	adjusted r ² ^y	model parameters ^z	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N	
KJ L-1																						
50	0.0094	0.9906	0.9811	constant CHCl3 Ca/Mg	0.42387 1.79797 -0.11881	0.0125 0.0053 0.0379	5	0.02	0.8731	0.8308	constant CHCl3	0.19537 1.38882	0.0169 0.02	5	0.452	0.0973	-0.0532	constant CEC	0.80831 -0.01189	0.1718 0.452	8	
100	0.0096	0.9999	0.9998	constant TEL CHCl3 Mg	-0.09336 0.62549 1.05038 0.03045	0.0713 0.0134 0.0063 0.0204	5	0.0001	0.9234	0.9106	constant carb	0.66943 -0.02321	0 0.0001	8	0.5041	0.0776	-0.0761	constant CEC	0.84264 -0.00918	0.1135 0.5041	8	
200	0.0323	0.8203	0.7305	constant K DEE	1.18114 -0.17176 -0.61429	0.0007 0.0387 0.0512	7	0.2543	0.3974	0.1965	constant CHCl3	0.5579 0.70182	0.0035 0.2543	5	0.2229	0.2354	0.108	constant Na/Ca+Mg	0.43014 25.4255	0.0416 0.2229	8	
300	0.0055	0.9945	0.9889	constant TEL DEE	0.45042 1.02616 -0.60896	0.0033 0.0034 0.005	5	0.0575	0.998	0.9918	constant DEE/CHCl3 TEL OC	-0.49087 -0.02663 1.07817 0.01583	0.2478 0.0386 0.0662 0.1225	5	0.3089	0.1708	0.0326	constant Mg	0.86287 -0.02394	0.0003 0.3009	8	
400	0.0335	0.9993	0.9972	constant carb ESP Ca/Mg	1.00602 -0.02317 0.32804 -0.08716	0.0169 0.0215 0.0432 0.0495	5	0.0764	0.4323	0.3377	constant carb	0.84606 -0.00994	0 0.0764	8	0.0483	0.5046	0.4221	constant CEC	1.29738 -0.01464	0.0008 0.0483	8	
500	0.0414	0.9989	0.9958	constant TEL DEE Mg	0.09583 1.64952 -1.00512 0.05358	0.2403 0.0243 0.0237 0.042	5	0.0727	0.7111	0.6148	constant CHCl3	0.7148 0.73029	0.0003 0.0727	5	0.1746	0.2832	0.1637	constant CEC	1.24943 -0.01218	0.0042 0.1746	8	
600	0.0219	0.9997	0.9988	constant TEL CHCl3 Mg	0.32449 0.31936 1.15809 0.04074	0.0438 0.0557 0.0121 0.0325	5	0.0042	0.7691	0.7306	constant carb	0.95879 -0.01654	0 0.0042	8	0.5232	0.0711	-0.0837	constant CEC	1.09351 -0.00686	0.022 0.5232	8	

^z Parameter abbreviations: CEC cation exchange capacity cmol/kg
 Na exchangeable Na
 Ca exchangeable Ca
 Mg exchangeable Mg
 ESP exchangeable Na percentage
 SAR Na adsorption ratio
 CHCl3 Chloroform extractable lipid (g/kg soil)
 DEE Diethyl ether extractable lipid (g/kg soil)
 TEL Total extractable lipid (DEE + CHCl3) (g/kg soil)
 OC Organic Carbon (g/kg soil)
 carb total hexose equivalents extracted by acid hydrolysis (g/kg soil)
 clay clay content

^y adjusted R²: A potential problem with R² is that it always increases as new independent variables are included in the model (RSS always decreases), even if they don't possess any relationship with the dependent variable. Adjusted R² is adjusted for the number of independent variables in the model to correct for this problem, and, therefore, will often be more interesting than the unadjusted R². Adjusted R² is computed as 1 - (n - 1)RMS/SST, where RMS is the residual mean square and n is the number of cases in the regression.

**Comparison of regression models for clay dispersion at selected energy levels containing organic parameters only, mineral parameters only, and both mineral and organic parameters:
Zero tillage treatment of a Fortier silty clay soil - Portage la Prairie site.**

level of dispersive energy	Organic and Mineral Parameters included in Models							Organic Parameters Only						Mineral Parameters Only								
	model p value	r ²	adjusted r ² y	model parameters z	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N	
kJ L ⁻¹																						
50	0.0001	0.9254	0.9129	constant clay	0.01954 0.00885	0.6595 0.0001	8	0.0583	0.476	0.3887	constant OC	-0.13822 0.0101	0.5582 0.0583	8	0.0001	0.9254	0.9129	constant clay	0.01954 0.00885	0.6595 0.0001	8	
100	0.0222	0.7819	0.6947	constant Mg carb	0.0732 0.10226 -0.01202	0.5959 0.0108 0.0537	8	0.0709	0.9291	0.8582	constant OC TEL	-0.06354 0.01732 -0.64035	0.717 0.0362 0.1097	5	0.0122	0.676	0.622	constant clay	0.11062 0.00989	0.3735 0.0122	8	
200	0.0042	1.0000	1.0000	constant OC TEL Ca/Mg	0.49077 0.00697 -0.24815 -0.02715	0.0052 0.0046 0.0051 0.0156	5	0.0185	0.9815	0.9631	constant OC TEL	0.33724 0.00864 -0.24359	0.0124 0.0094 0.0516	5	0.095	0.3953	0.2945	constant K	0.52031 0.06577	0.0004 0.095	8	
300	0.0223	0.9997	0.9988	constant Ca/Mg clay DEE	0.93511 -0.08273 0.00167 -0.17228	0.0061 0.0162 0.0301 0.0322	5	0.1062	0.6356	0.5142	constant DEE/CHCl3	0.69316 0.01122	0.0001 0.1062	5	0.0832	0.4179	0.3209	constant K	0.56754 0.07904	0.0005 0.0832	8	
400	0.008	1.0000	0.9998	constant Mg DEE Ca/Mg	0.99449 0.03235 -0.52278 -0.07149	0.0029 0.0076 0.0047 0.0088	5	0.0194	0.9998	0.9991	constant TEL OC DEE/CHCl3	0.63102 -0.5019 0.00848 -0.00743	0.0065 0.0108 0.012 0.0302	5	0.3751	0.1327	-0.0119	constant Ca/Mg	0.91974 -0.05106	0.0004 0.3751	8	
500	0.0118	0.9999	0.9996	constant Ca/Mg DEE	1.10441 -0.10058 -0.12008	0.0033 0.0076 0.0561	4	0.1248	0.9844	0.9532	constant carb CHCl3/TEL	0.95385 0.01032 -1.17097	0.0114 0.125 0.0853	4	0.1773	0.3301	0.1961	constant K	0.62224 0.0888	0.0043 0.1773	7	
600	0.0085	1.0000	0.9998	constant TEL OC CHCl3	0.59765 -0.51385 0.00971 0.1708	0.004 0.0055 0.005 0.0304	5	0.0085	1	0.9998	constant OC TEL CHCl3	0.59765 0.00971 -0.51385 0.17802	0.004 0.005 0.0055 0.0304	5	0.1657	0.2932	0.1754	constant K	0.68635 0.0802	0.0008 0.1657	8	

^z Parameter abbreviations: CEC cation exchange capacity cmol/kg; Na exchangeable Na; Ca exchangeable Ca; Mg exchangeable Mg; ESF exchangeable Na percentage; SAF Na adsorption ratio; CHC Chloroform extractable lipid (g/kg soil); DEE Diethyl ether extractable lipid (g/kg soil); TEL Total extractable lipid (DEE + CHCl3) (g/kg soil); OC Organic Carbon (g/kg soil); carb total hexose equivalents extracted by acid hydrolysis (g/kg soil); clay clay content

^y adjusted R²: A potential problem with R² is that it always increases as new independent variables are included in the model (RSS always decreases), even if they don't possess any relationship with the dependent variable. Adjusted R² is adjusted for the number of independent variables in the model to correct for this problem, and, therefore, will often be more interesting than the unadjusted R². Adjusted R² is computed as 1 - (n - 1)RMS/SST, where RMS is the residual mean square and n is the number of cases in the regression.

Appendix V

Regression analyses of factors affecting the dispersion of clay from 0.25 to 0.85 mm soil aggregates during sonication at selected levels of applied energy for the 0 to 10 cm depth of a native prairie toposequence: Best multiple linear regression models for organic and mineral parameters considered separately and combined.

level of dispersive energy kJ/L	Organic and Mineral Parameters included in Models							Organic Parameters Only							Mineral Parameters Only						
	model p value	r ²	adjusted r ²	model parameters ²	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N	model p value	r ²	adjusted r ²	model parameters	parameter coefficients	p value of parameter	N
25	0.0348	0.9993	0.997	constant CEC Ca/Mg CHCl3	0.73723 -0.022 -0.05563 21.1208	0.0319 0.0374 0.0178 0.031	5	0.5927	0.1061	-0.1918	constant TEL	0.0145 2.0043	0.9522 0.5927	5	0.2232	0.9775	0.8876	constant Ca/Mg Ca + Mg CEC Ca/Na	0.32471 -0.04632 0.02589 -0.01685 7.35E-05	0.2382 0.1171 0.1578 0.1947 0.2281	6
50	0.0324	0.9994	0.9974	constant CEC Ca/Mg DEE/CHCl3	2.85673 -0.07766 -0.09496 -0.03133	0.0165 0.0194 0.0173 0.0266	5	0.8067	0.0987	-0.2017	constant DEE	0.0645 2.96301	0.8437 0.6067	5	0.1763	0.8787	0.6968	constant Na Ca Mg	-0.6986 -4.81445 0.03454 0.1791	0.1691 0.1142 0.189 0.0684	6
100	0.0078	1	0.9999	constant CEC Ca/Mg DEE	4.80934 -0.12788 -0.12396 -10.3976	0.0045 0.0048 0.0043 0.0074	5	0.5012	0.1623	-0.1169	constant DEE	0.10186 4.44575	0.7832 0.5012	5	0.1185	0.9194	0.7984	constant Ca ESP Mg	-0.69414 0.03692 -1.55772 0.21016	0.1552 0.1549 0.081 0.0468	6
200	0.0118	0.9999	0.9997	constant CEC Ca Ca/Mg	3.16875 -0.10192 0.05002 -0.13756	0.0068 0.0081 0.0136 0.0063	5	0.4049	0.2376	-0.0166	constant DEE	0.14057 6.91061	0.7572 0.4049	5	0.0631	0.9575	0.8937	constant Ca + Mg ESP Mg	-0.70544 0.03969 -2.07876 0.22754	0.1356 0.1235 0.0422 0.0227	6
300	0.0616	0.9977	0.9906	constant Ca/Mg CEC Ca	1.93713 -0.11793 -0.05303 0.04427	0.049 0.0323 0.068 0.0673	5	0.5321	0.1044	-0.1194	constant Carb-sol	0.15388 0.23181	0.8341 0.5321	5	0.0954	0.9354	0.8384	constant Ca/Mg Ca + Mg Na	0.40272 -0.09082 0.03852 -2.39451	0.2081 0.0368 0.1338 0.1786	6
400	0.0036	1	1	constant CEC Ca/Mg DEE/CHCl3	2.71931 -0.05694 -0.10779 -0.01973	0.0023 0.0035 0.002 0.0055	5	0.3975	0.2422	-0.0077	constant CHCl3/TEL	0.86291 -1.2223853	0.0134 0.3975	5	0.1392	0.9049	0.7623	constant Ca/Mg Ca/Na Mg	0.46383 -0.05801 2.18E-04 0.08261	0.2528 0.2008 0.1595 0.2621	6
500	0.0084	1	0.9998	constant CEC Ca/Mg CHCl3	1.80344 -0.03774 -0.11479 38.6154	0.0067 0.0111 0.0044 0.0087	5	0.5713	0.1181	-0.1759	constant DEE	0.53113 3.81843	0.2263 0.5713	5	0.1847	0.8727	0.6819	constant Ca/Mg Ca/Na Ca + Mg	0.45261 -0.09146 1.37E-04 0.03429	0.2481 0.0865 0.2748 0.2195	6

* Parameter abbreviations: CEC cation exchange capacity cmol/kg
 Na exchangeable Na
 Ca exchangeable Ca
 Mg exchangeable Mg
 ESP exchangeable Na percentage
 SAR Na adsorption ratio
 CHCl3 Chloroform extractable lipid (g/kg soil)
 Carb-sol solubilized carbohydrate
 DEE Diethyl ether extractable lipid (g/kg soil)
 TEL Total extractable lipid (DEE + CHCl3) (g/kg soil)
 OC Organic Carbon (g/kg soil)

^y adjusted R2: A potential problem with R2 is that it always increases as new independent variables are included in the model (RSS always decreases), even if they don't possess any relationship with the dependent variable. Adjusted R2 is adjusted for the number of independent variables in the model to correct for this problem, and, therefore, will often be more interesting than the unadjusted R2. Adjusted R2 is computed as 1 - (n - 1)RMS/SS_T, where RMS is the residual mean square and n is the number of cases in the regression.