



Effects of precipitated calcium carbonate from sugar purification on crusting soils
by John Matthew Inkret

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Land Rehabilitation

Montana State University

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Abstract:

Precipitated calcium carbonate (PCC), a by-product of the sugar industry, results from a sucrose juice purification technique. This material has several properties that distinguish it from geological calcite. These properties include an organic load, high water-holding capacity, small particle size, high surface area, and uniformity. A large stockpile of PCC is located at the Holly Sugar refinery in Sidney, Montana. This study was initiated at the request of Holly Sugar to investigate potential PCC application as a soil amendment to local agricultural soils that exhibit surface crusting.

Soil surface crusts, initiated by precipitation events, are common in Eastern Montana. These crusts can significantly inhibit crop seedling emergence. This study was designed to evaluate the potential of PCC to ameliorate crust formation in eastern Montana agricultural soils. Bulk soil samples from four different sites and stockpile PCC samples were collected for evaluation. Amendment (PCC) and the four soils were then physically and chemically characterized. Soil crust formation and strength of amended and control soils were evaluated in the laboratory and greenhouse by inducing crust formation and then measuring and analyzing specific soil crust properties and parameters. Soil areal extensibilities from saturated to dry moisture conditions were measured and evaluated. Outdoor trials evaluated seedling emergence success and surface crust strength. Greenhouse trials evaluated seedling emergence and the areal extent of soil surface fractures by a photographic quantification technique. The addition of PCC, at the rates tested, did not ameliorate the problematic physical characteristic of the soils. Soil crusting was not reduced by the amendments used in this study.

EFFECTS OF PRECIPITATED CALCIUM CARBONATE
FROM SUGAR PURIFICATION
ON CRUSTING SOILS

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John Matthew Inkret

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This thesis has been read by each member of the thesis committee and has been found to be satisfactory regarding content, English usage, format, citations, bibliographic style, and consistency, and is ready for submission to the College of Graduate Studies.

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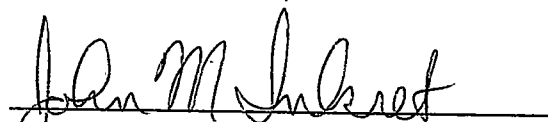
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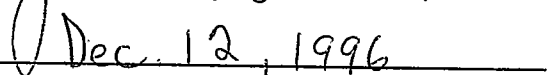
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ABSTRACT

Precipitated calcium carbonate (PCC), a by-product of the sugar industry, results from a sucrose juice purification technique. This material has several properties that distinguish it from geological calcite. These properties include an organic load, high water-holding capacity, small particle size, high surface area, and uniformity. A large stockpile of PCC is located at the Holly Sugar refinery in Sidney, Montana. This study was initiated at the request of Holly Sugar to investigate potential PCC application as a soil amendment to local agricultural soils that exhibit surface crusting.

Soil surface crusts, initiated by precipitation events, are common in Eastern Montana. These crusts can significantly inhibit crop seedling emergence. This study was designed to evaluate the potential of PCC to ameliorate crust formation in eastern Montana agricultural soils. Bulk soil samples from four different sites and stockpile PCC samples were collected for evaluation. Amendment (PCC) and the four soils were then physically and chemically characterized. Soil crust formation and strength of amended and control soils were evaluated in the laboratory and greenhouse by inducing crust formation and then measuring and analyzing specific soil crust properties and parameters. Soil areal extensibilities from saturated to dry moisture conditions were measured and evaluated. Outdoor trials evaluated seedling emergence success and surface crust strength. Greenhouse trials evaluated seedling emergence and the areal extent of soil surface fractures by a photographic quantification technique. The addition of PCC, at the rates tested, did not ameliorate the problematic physical characteristic of the soils. Soil crusting was not reduced by the amendments used in this study.

INTRODUCTION

Precipitated calcium carbonate (PCC) is a by-product of a sucrose purification process. This material results from the addition of calcium oxide (CaO) to juice extracted from sugarbeets (diffusion juice) in the presence of carbon dioxide (CO₂). Subsequent calcium carbonate (CaCO₃) precipitation and flocculation adsorbs non-sucrose impurities. This colloidal floc, or PCC, is then filtered out of the purified diffusion juice.

Juice purification processes within each refinery are generally consistent, producing a relatively homogeneous PCC material. Most sugar refineries utilize the same fundamental purification process, however PCC composition may vary between refineries in terms of calcium carbonate equivalence and organic matter content. The Holly Sugar refinery in Sidney, Montana has generated and stockpiled a significant amount of PCC.

As an agricultural liming agent, PCC has desirable chemical, physical, and biological properties: uniformity, small particle size and high surface area, high calcium carbonate equivalence, and organic nutrients. Diffusion juice impurities, mostly non-sucrose cytoplasmic constituents of sugarbeet root cells, constitute the organic portion of PCC.

This study was initiated at the request of Holly Sugar to investigate potential local PCC application as a soil amendment to crusting soils of irrigated agricultural lands along the lower Yellowstone River. The study objective concerned potential application of PCC

to four high pH soils to evaluate the effect of this material on soil surface crust formation and strength.

Soil samples were collected in August, 1994 from four cultivated and irrigated fields on the lower Yellowstone River near Sidney, Montana. All four soils exhibited surface crusting under certain conditions. The four soils will be referred to by the name of the farmers who own or cultivate the sites: Burbach 1, Burbach 2, Burbach 3, and Hardy. The four soils were chemically and physically characterized because soil crust formation and properties are affected by a host of soil characteristics. These problem soils were characterized in order to define which soil properties were adversely affecting plant growth (i.e. crusting mechanisms). Soil analyses included the following: pH, particle size distribution, electrical conductivity (EC), water-soluble and exchangeable cation concentrations, and organic matter content.

Effects of PCC on soil crust formation and strength were evaluated by areal extensibility measurements, penetrometer, and outdoor and greenhouse seedling emergence trials. Consistent observations of successful emergence only through proximal surface fractures prompted the development and implementation of soil surface image quantification. Effects of PCC treatment rates were then evaluated in terms of the extent of their impact on soil surface fractures as a percentage of total surface area.

LITERATURE REVIEW

Precipitated Calcium Carbonate Synthesis and Chemistry

Knowledge of PCC synthesis is essential to understanding the chemical, physical, and biological properties of PCC and its reactivity in soil. The sugar extraction and purification processes are outlined in the following text.

Sugarbeets entering the refinery are washed and sliced, emerging as thin-strip cossettes. Cossettes then enter a continuous diffuser for sugar extraction. The rotating diffuser consists of spirally arranged compartments containing solutions of decreasing sugar concentrations leaching countercurrent through the cossette mass.

Moderate heating of this solution, referred to as diffusion juice, denatures cell membrane proteins and allows cytoplasmic diffusion without disrupting the structural integrity of cell walls. This benign extraction system creates an osmotic gradient and is designed to extract maximum amounts of sucrose with minimal levels of impurities. Impurities are mostly the cytoplasmic constituents of root cells. Sugar enriched diffusion juice exits the lower end of the diffuser and, after screening and heating to 85° C, is ready for purification (McGinnis 1971).

Diffusion juice contains water, 10 to 15% sucrose, and ionic and colloidal impurities. Water is the solvent of diffusion juice and is a sufficiently strong acid to initiate dissociation of weak acids and bases through hydration reactions (McGinnis 1971).

Ionic impurities include organic and inorganic acids. Proteins, peptides, and pectins are present as colloids.

The concentrations of some acids in diffusion juice from the 1950 Sidney campaign as reported by Stark et al. (1950) are presented in Table 1. Acid anions were separated from diffusion juice using ion exchange resin columns and reported as a percentage of dry solids. These nitrogen-free acids occur in beets in small quantities, and more are produced as the products of decomposition of larger organic structures under the influence of microorganisms, enzymes, heat, and liming (McGinnis 1971). The sugar beet also contains 1% or more of nitrogen-containing compounds that will diffuse into the juice. These materials include amino acids, amides, ammonia, nitrates, proteins, peptides, and purines.

Table 1. Non-amino acids in diffusion juice.*

Acid anion	mg/L at 10% dry solids
Chloride	82
Sulfate	158
Phosphate	475
Oxalate	32
Citrate	655
Lactate	70
Malate	312

*From Stark et al. 1950.

The sorption of biological material to precipitating calcium carbonate particles in purification creates the organic fraction in the PCC by-product. The non-sucrose content of diffusion juice is a function of the quality of the beets and the conditions under which the sugar was extracted in the factory. Beet storage after harvesting and annual variations in growing conditions can also affect the quality of diffusion juice.

The juice purification process consists of five basic steps: preliming, liming, first carbonation, second carbonation, and filtration. In preliming, a small portion of liming agent is added to the diffusion juice. The liming agent is most often milk of lime, a suspension of mostly calcium hydroxide ($\text{Ca}(\text{OH})_2$) made by adding calcium oxide to sweetwater (dilute aqueous sucrose solution). Preliming encourages the formation of insoluble products from calcium hydroxide and non-sugars. A favorable effect is achieved in this step by maintaining low alkalinity in which calcium hydroxide forms only calcium ion. This progressive preliming condition is referred to as "stabilization" and has a favorable effect on the physical qualities of the precipitate formed. Stabilization promotes a smaller volume precipitate with more rapid sedimentation (McGinnis 1971). Preliming stabilization postpones the precipitation of proteins and other colloids by providing for ionic exchange into calcium forms. The colloidal floc of preliming is formed in the presence of a calcic mineral skeleton that enhances agglomeration into secondary larger particles in first carbonation (Dedek 1952).

Liming is the next step in purification and is simply a continuous addition of the liming agent to prelimed diffusion juice. Certain destruction reactions occur on liming.

These reactions include the saponification of amides into their corresponding ammonium salts, the decomposition of glucose and fructose into acid products, and protein dispersion (McGinnis 1971).

First carbonation immediately follows liming. Carbon dioxide is produced in the factory and applied to diffusion juice in the gaseous form. It must dissolve as carbonic acid (H_2CO_3) before it can react. Carbon dioxide gas bubbles are passed through the limed juice to precipitate the lime as very small insoluble calcium carbonate crystals (McGinnis 1971). Small bubbles and agitation of the liquid are favorable for better carbon dioxide absorption. Carbon dioxide solubility is a function of its own partial pressure in the gaseous phase, temperature, and the chemical composition of the liquid phase. Increasing ionic strength lowers carbon dioxide solubility (Dedek 1952).

In first carbonation, juices are purified by coagulation and precipitation of impurities and by adsorption on calcium carbonate crystal faces. Freshly precipitated calcium carbonate is often amorphous and is changed relatively slowly into a crystalline form (McGinnis 1971). The chemical purification is favored by high surface area of the precipitated calcium carbonate. Second carbonation precipitates most of the excess calcium hydroxide left in solution.

In the final step of filtration, the precipitated sludge is settled out in tray thickeners and separated by rotary vacuum filters. At this point, the filter cake contains 7 to 8% sugar. This is reduced to <1% by desweetening with hot water. The greatest change in the equilibrium between juice and precipitated sludge takes place at desweetening when juices are replaced by hot waters (McGinnis 1971). The PCC, "filter cake", or "lime cake", is

reslurried with water and discarded into percolation ponds. This creates the large stockpiles of PCC present at most western sugar beet processing facilities.

Hartmann (1971) reported that "lime cake" (PCC), in addition to containing about 75% calcium carbonate and adsorbed impurities from the juice, contains in the range of 6% organic nitrogen compounds, 4% of other mineral compounds, and a percentage of phosphoric acid equivalent. Filter aids such as diatomaceous earth may be present and will persist as siliceous material. Analyses of trace inorganic products present in "lime cake" as reported by Hartmann are summarized in Table 2.

Table 2. Inorganic products in lime cake (% dry basis).*

SiO ₃	Al ₂ O ₃	Fe ₂ O ₃	MgO	K ₂ O	Na ₂ O	SO ₃
3.06	2.44	0.54	1.62	0.08	0.18	0.16

*From Hartmann 1971.

Precipitated Calcium Carbonate As A Soil Amendment

The small and uniform particle size and high calcium carbonate equivalence of PCC are desirable properties for agricultural liming agents. Small particles mix readily with the soil, and high surface area allows for rapid and effective dissolution under acidic conditions. The application of calcium carbonate to soils is advocated for three primary purposes: to neutralize excess acidity, to improve soil structure, and to furnish calcium for plant nutrition. While neutralizing acidity by providing abundant hydroxyl ions (OH⁻), liming also adds calcium and can favorably affect other soil properties. For instance, soil

bacteria and actinomycetes have higher activities at pH values near neutral. Consequent bacterial polysaccharides produced through organic matter degradation are very effective as natural soil aggregating agents.

Nutrient bioavailability is also enhanced by liming acid soils. At near neutral pH levels, aluminum does not compete for exchange sites because of reduced solubility and calcium, magnesium, and potassium availability increase. Phosphorus bioavailability increases when soil pH increases from more acid values to about 6.5 (Lindsay 1979), as a result of decreased aluminum and iron solubility with increasing pH. Calcium cations can act as antagonists with respect to other cations and prevent excessive plant uptake of aluminum, hydrogen, and sodium.

Virtually all by-product filter cake (PCC) is reused as a soil amendment in sugarbeet growing areas in which the soils are predominantly acidic (e.g. California, Great Britain, Ukraine). As of 1984, the USSR was using in excess of three million metric tons of "lime cake" annually for remediating acid soils (Yagodin 1984).

In England, farm managers have reported that the "lime value" (neutralization potential) of "lime cake" may be more persistent than crushed geologic limestone. Also, the material appears to contain enough plant available phosphorus for one crop season and one half the necessary nitrogen for that season. Filho et al. (1991) reported no addition of phosphorus fertilizer necessary for sugar cane growth when 10 mt/ha of "lime cake" were added to acid soils in Brazil. Studies of the agricultural value of "filter cake" in Poland report variable calcium oxide content; an abundance of magnesium, phosphorus, and nitrogen; and low concentrations of metals (Gajek 1991).

Soil Crusting

Soil Crusting and Seedling Emergence

Soil crusting or sealing is common to agricultural soils worldwide and occurs over a range of climatic conditions (Holder and Brown 1974). Surface crusts are characterized by greater bulk density, higher shear strength, smaller pores, and lower hydraulic conductivity than the underlying soil (McIntyre 1958). Soil susceptibility to surface sealing is a function of soil physical, chemical, and biological processes.

Crusting and sealing can create crop production and irrigation management problems (Singer and Warrington 1992). The control of soil crusting is a problem encountered in securing adequate, uniform stands of sugarbeets on some soils (Yonts et al. 1983). A hard, dense layer at the soil surface affects aeration, reduces infiltration, and has a direct mechanical effect on plant growth (Lemos and Lutz 1957). Emergence of germinating seedlings is reduced by soil crusts (Hanks and Thorp 1956) and mechanical impedance is often the limiting factor in stand establishment (Arndt 1965). Seedlings emerging under crusted conditions are smaller and weaker than normal seedlings (Sale and Harrison 1964).

Seedling emergence is a function of crust strength and seedling emergence force (Arndt 1965). The magnitude of this effect is dependent on the interaction of two opposing forces: the energy development of the young plant and the degree of crust resistance (Lemos and Lutz 1957). Hanks and Thorpe (1956) showed a nearly linear decrease in the seedling emergence of wheat with an increase in crust strength. Joshi

(1987) also reported a negative linear relationship for millet emergence as crust strength increased. Seed weight and seedling emergence forces are closely correlated (Williams 1956) and frequent and wide cracking in the soil crust is often necessary for emergence of smaller seedlings (Sale and Harrison 1964). Horizontal growth and detouring of seedlings results in delayed and reduced emergence and root deformation (Arndt 1965).

Soil Crust Properties

The formation of surface crusts is the result of the reorganization of constituents within the crust as compared to the organization of constituents in the underlying soil (West et al. 1992). Various studies have revealed that soil crusting results from a combination of compaction, structure breakdown, and deposition of fine particles at the surface (Lemos and Lutz 1957). Different crusting soils vary widely in structural and textural characteristics and in the degree to which changes in structure may take place. In general, crusts have a greater bulk density, a higher percentage of finer grained particles, and a lower degree of aggregation than uncrusted soil. The most outstanding difference between a crust and the underlying soil is the bulk density value. Lemos and Lutz reported bulk density values ranging from 22 to 26% higher in the crusted portion of five soils.

Soil crust strength is also a function of water content. Using an emergent probe, Holder and Brown (1974) observed a negative linear relationship between crust strength and water content down to 2.2% moisture. At moisture contents below 2%, crust strength becomes highly variable as planes of weakness appear. Crusts tend to crack along planes

of the highest relative water content (Kemper et al. 1974) because of differential shrinkage. Holder and Brown (1974) noted a peak of impedance in the range between 2.8 and 2.2% moisture. A decrease in impedance, associated with crust cracking, was observed at moisture contents below this interval.

Soil Crusting Mechanisms - Mechanical

Weather plays an important role in structural crust formation. High-intensity rain followed by drying conditions is required for development of surface soil crusts (Miller and Radcliffe 1992). In sodic soils, the mechanical formation of crust is initiated by raindrop impact at the soil surface, with subsequent breakdown of soil aggregates. Dispersed clay suspended in the infiltrating water migrates into the soil and fills pores immediately beneath the surface (McIntyre 1958). Impacting raindrops also influence clay dispersion at the soil surface in non-sodic soils. In these materials, clay dispersion only occurs under the influence of mechanical energy from precipitation (Sumner and Stuart 1992). When aggregates are disrupted, dispersed and suspended clay particles are sorted by water and redeposited into the subsurface soil pores. On land covered by vegetation, raindrops lose kinetic energy as they are intercepted by foliage. Cultivated soils are vulnerable in two respects: they lack any vegetative overstory and many are structurally unstable.

Micromorphological investigations of crust structure (McIntyre 1958) have shown that surface crusts consist of two distinct layers. An upper skin is attributable to compaction by raindrop impact and overlies a "washed-in" zone of decreased porosity

attributed to the accumulation of small particles (Shainberg 1992). Subsequent crusts or seals will determine future allocation of precipitation to rain infiltration, runoff, and erosion (Sumner and Stuart 1992).

Crusts form on soils of almost any texture except sands with very low silt and clay contents (Lemos and Lutz 1957). Gerard (1965) noted stronger crusts with increasing silt, clay, and exchangeable sodium content. A broad range of particle size distributions have been reported for crusting soils, however silt is the predominant particle size in many soils that form crusts.

Clays can also act as effective aggregating agents, especially when associated with calcium and potassium ions rather than sodium or magnesium (Klages 1965). Benefits of aggregating clay include increased surface cracking that disrupts surface crusts and allows plant emergence. Increasing the surface clay content can improve aggregation on some soils and clay accumulation in the subsoil is a common feature of soils in the western United States. Soil aggregation and crust reduction may be improved by plowing a few inches deeper in these soils.

Clay mineralogy also influences crust formation. Layered silicate clays having a 2:1 type lattice structure permit significant interlayer expansion and consequent shrinking and swelling of the clay. As clay minerals swell or disperse, they reduce pore diameter and water infiltration rate. The type and relative amounts of clay minerals and cations occupying exchange sites influence the likelihood of crust formation (Singer and Warrington 1992). Ben-Hur et al. (1985) suggested that soils with high shrink-swell 2:1 clays, such as montmorillonite, will crack upon drying. Soils with 1:1 vermiculite or illitic

clays will not crack as much. Similarly, Lemos and Lutz (1957) obtained high modulus of rupture values with soils containing 2:1 type clays.

In general mineralogical terms, arid region soils in the United States are likely to have a wide variety of clay types and are likely to have high activity clays. This should contribute to structural stability because cracking clays will disrupt a crust. Low organic matter, commonly described in arid and semi-arid region soils, and not clay mineralogy is a major cause of the poor structural condition of these soils (Ben-Hur et al. 1985).

Kemper and Koch (1966) showed that aggregate stability is a function of organic matter, clay, and oxide contents. As organic matter increased from <1% up to 2%, aggregate stability increased. Continuous cultivation of soils over decades can result in structural degradation stemming from the loss of this organic matter. The importance of organic matter to aggregate stability and soil crusting is not a simple relationship between total amount of organic carbon and stability, because not all the organic compounds in soils are responsible for aggregate stability (Oades 1984). The model of soil structure of Tisdall and Oades (1982) suggests that different kinds of organic matter are important for stabilizing aggregates of different sizes (Singer and Warrington 1992).

Soil Crusting Mechanisms - Chemical

Chemical dispersion and flocculation are governed by the attractive and repulsive forces present in the electrical double layer that results from the charged nature of soil colloids. On the basis of charge, divalent calcium ions are held more firmly to clay particles than monovalent sodium. Sodium ions also have larger hydration shells.

Consequently, because of its large size and small charge, the sodium ion creates a diffuse cloud of counter-ions around each clay particle that is much larger than that of a calcium system. When cation swarms overlap between clay particles, an ionic concentration forms midway between the two particles that is appreciably higher than the external concentration. Water moves into this diffuse double layer between the clay particles, causing them to swell. Compared to calcium, the larger ion cloud associated with sodic systems creates a larger diffuse double layer with a higher relative ion concentration between clay particles (Sumner 1992). As a result, large scale swelling and shrinking is common in sodic soils. Sodium induced swelling disperses soil aggregates into individual particles which destroys soil structure. Chemical formation of soil crusts involves aggregate disintegration and clay dispersion due to chemical forces in the diffuse double layer of clay micelles (Shainberg 1992).

Allison and Moore (1956) stated that factors other than exchangeable sodium and texture can be important in determining the crusting behavior of different soils. The range of particle size distribution, organic carbon content, and exchangeable sodium percentage (ESP) of some soils that have crusting problems illustrates why it is difficult to specify the precise value of each soil property that makes a soil susceptible to crust formation (Singer and Warrington 1992).

The importance of sodium in determining crust susceptibility cannot be overstated, but calcium and magnesium also play a role. There are calcareous crusts peculiar to certain environmental conditions (Lemos and Lutz 1957). Exchangeable magnesium can cause a deterioration in soil structure. In addition, magnesium enhances dispersion in

montmorillonite and illite compared to calcium (Bakker and Emerson 1973). Keren (1989) attributed lower infiltration rates of magnesium soils to the larger width of hydration shell of the adsorbed magnesium compared to that of the calcium ion. The width of hydration shell determines the strength of the link between the clay tactoids and therefore affects aggregate stability.

In summary, crust properties are affected by soil composition, structure, chemistry, mineralogy, rainfall characteristics, soil moisture, drying conditions, and organic matter content. Soils that have crusting problems must be characterized in order to define which soil properties are adversely affecting surface conditions. Soil chemical and physical data can then be used to determine the amendment type and rate.

Soil Crust Amendments

Strategies and techniques of chemical amelioration of acid or sodic crusting soils are based on the introduction of calcium into the soil adsorbing complex, thereby altering the composition of exchangeable cations. The choice of chemical amendments for the replacement of exchangeable sodium is directly related to the presence or absence of alkaline earth carbonates (e.g. calcite, dolomite) (USSLS 1969). The high pH of sodic soils (>8.5) imposes solubility constraints as to which calcium form to use as an amendment.

The beneficial effects of gypsum (calcium sulfate) on the physical condition of sodic soils are well documented. Gypsum is much more soluble than calcium carbonate at the higher pH conditions of sodic systems. Field observations of acid soils treated with

gypsum have shown reduced crust formation (Miller and Radcliffe 1992). Kazman et al. (1983) reported less chemical dispersion and increased permeability in alkaline soils after spreading phosphogypsum on the soil surface. The treatment was effective in soils of ESP 1.0, indicating that some chemical dispersion took place even at low ESP values, and possibly even in calcium-saturated soils.

Johnson and Law (1966) reported crust reduction and increased emergence on calcareous soils with topical application of concentrated sulfuric acid. The soil and acid combined to form a thin friable surface film of gypsum that prevented crust formation. Gypsum may also reduce crust strength of non-sodic, high magnesium soils by increasing the calcium to magnesium ratio.

Soil colloidal materials (clay particles and organic polymers such as humic substances and applied synthetic conditioners) cement primary particles into stable aggregates and improve soil structure. Organic amendments such as animal or green manures are always beneficial to soil structure. Many commercial synthetic soil conditioners are available.

METHODS AND PROCEDURES

Location of Study Sites

The Hardy soil is located on the north bank of the Yellowstone River floodplain approximately five miles upstream from the confluence of the Yellowstone and Missouri Rivers. Seasonal ice jams on the river cause local lowland flooding. This particular field had been flooded in March of 1994 with consequent saturation of the soil profile. Tillage produced hard, dense aggregates unfavorable for seed bed preparation. This irrigated field is used for sugarbeet production.

The Burbach 1 and Burbach 2 soils are located approximately 25 miles west of Glendive, MT adjacent to the north bank of the Yellowstone River. These irrigated soils exhibit surface crust formation after precipitation events. Crusts formed over crop seeds inhibit seedling emergence. At the time of sampling, Burbach 1 was planted in field corn and Burbach 2 in sugarbeets.

The Burbach 3 soil is located approximately two miles upstream (southwest) of the Burbach 1 and 2 sites. This irrigated field was planted in sugarbeets at the time of sampling. The Burbach 3 soil is also prone to surface crusting after precipitation events.

Soil Analysis

Soil samples were oven dried at 55° C for 24 hours and passed through a #10 (2 mm) sieve. A glass combination pH electrode measured saturated paste extracts for determination of pH. Electrical conductivity of the extracts was measured with an EC bridge. Electrical conductivity is a common and reasonably precise measurement indicating the total concentration of the ionized constituents of soil solutions. It is related to the sum of the cations present in solution and usually correlates with the total dissolved solids (TDS). The property of conductivity is the reciprocal of the electrical resistivity expressed in reciprocal ohms per cm, or mmhos/cm (USSLS 1969). Saline soils are defined as those soils which exhibit saturated paste extract EC values greater than 4.0 mmhos/cm.

The same extract was used for determination of water soluble concentrations of calcium, magnesium, potassium, and sodium by atomic absorption spectroscopy. Extractable cations were determined in a neutral one normal ammonium acetate solution (Thomas 1982). Cation concentrations were measured by atomic absorption at the Montana State University soils analytical laboratory.

Particle size distribution was determined by the standard hydrometer technique (Gee and Bauder 1986). Saturation percentage is defined as the soil moisture content at saturation as a percentage of dry soil mass and is determined gravimetrically (USSLS 1969). The coefficient of linear extensibility (COLE) measurements followed methods outlined by Schafer and Singer (1976). Organic carbon content as a percentage was determined by the Walkley-Black Method (Nelson and Sommers 1982). This method is a

rapid dichromate oxidation technique with a correction factor of 1.32 to account for unrecovered carbon. A conversion factor of 2.0 was then applied to convert the organic carbon values to an estimate of total soil organic matter. Calcium carbonate equivalence, as a percentage, of the four soils was determined by measuring mass lost via carbon dioxide evolution. A strong acid (3M HCl) was added to an aliquot of soil and gravimetric weight loss was recorded. It was assumed that for every mole of carbon evolved, one mole of calcium carbonate was present in the soil (Nelson 1982).

Precipitated Calcium Carbonate Analysis

Precipitated calcium carbonate samples were taken at random from eight different locations in the stockpile in March, 1995. These samples were used for estimates of bulk density, water content, and organic matter. Bulk density of dry PCC was determined gravimetrically from known volumes of loosely packed material at stockpile moisture levels (USSLS 1969). This estimate provides information concerning the mass of dry material present in a given volume of stockpile. Moisture content was determined gravimetrically from stockpile moisture to oven dry (50° C to constant weight). This drying temperature is considerably less than the standard soil drying temperature (105° C). Excessive temperatures will drive off carbon dioxide from calcium carbonate and yield erroneously high values of mass lost as water. Accordingly, soils and materials high in carbonates should be dried at lower temperatures. Volumetric percentages were obtained by multiplying mass percentages by respective bulk densities. Organic matter content of

PCC was determined from four split samples. A standard pure calcite blank was analyzed to determine if the methodology (Nelson and Sommers 1982) distinguished inorganic and organic carbons in materials with high calcium carbonate contents. The neutralization potential of PCC was determined by potentiometric titration (Horwitz 1975). Mass lost via carbon dioxide evolution after an addition of strong acid (3M HCl) evaluated the calcium carbonate equivalence of PCC (Nelson 1982).

Effects of PCC On Soil Crust Formation And Strength

Bulk soil samples were collected from the four sites in March of 1995. Soils and PCC were air-dried and sifted through #10 (2mm) and #60 (.0098mm) sieves respectively. Soils were thoroughly mixed with PCC at designated treatment rates. Amended and control soils were evaluated with respect to crust formation and crust strength using the methods and procedures outlined in this section.

Areal Extensibility

Shrinkage of saturated soil material on drying was determined by measuring the dimensions of soil briquets before and after drying (USSLS 1969). This measurement provides information concerning the degree of dispersal (swell) and flocculation (shrink) of a soil. As an exploratory experiment, a range of PCC rates were evaluated for potential treatment effects on the areal extensibility of soil from saturated to oven-dry water contents (Table 3).

Saturated soil pastes were spread into precision brass briquet molds with inside dimensions of 35 x 70 x 9.45 mm (saturated surface area = 24.5 cm²). Measurements were recorded as coefficients calculated from the equation:

$$\frac{A_s - A_d}{A_d}$$

In this equation, the variable A_s is equal to the area at saturation and A_d represents the measured area after drying. Using this coefficient as a response variable, treatment effects were statistically analyzed for six rates over four soils by one and two way analyses of variance.

Table 3. PCC amendment rates for soil areal extensibility measurements.

Rate*	Tonnes/Hectare**
Control	0
1:32	69
1:24	92
1:16	138
1:8	275
1:4	550

*Ratio of air-dry PCC to air-dry soil by mass.

**Approximate metric tons (megagrams) PCC per hectare (15 cm furrow slice).

Outdoor Seedling Emergence

Sugarbeet seedling emergence through induced soil surface crusts was evaluated over a 35 day period. Control and treated soils (Table 4) were placed in 48 x 35 cm galvanized trays to a depth of 8 cm. Randomly arranged soil trays were placed outside on a flat level table in full sun. Twelve sugarbeet seeds were placed at a depth of 2.5 cm in each tray. Seeds were spaced 7.5 cm apart in two rows 18 cm apart.

After seeding, crust formation was initiated by evenly spraying water on all trays. Test tubes were placed upright around and between the trays to measure water applications and precipitation events. The irrigation and precipitation schedule are outlined in Table 5. Successful seedling emergence was recorded as a percentage for each tray (Appendix A, Table 22). Using this percentage as a response variable, treatment effects for four amendment rates were statistically analyzed over four soils using one and two way analyses of variance.

Table 4. PCC amendment rates: outdoor trials.

Rate*	Tonnes/Hectare**
Control	0
0.01	22
0.03	66
0.05	110

*Ratio of air-dry PCC to air-dry soil by mass.

**Approximate metric tons (megagrams) PCC per hectare (15 cm furrow slice).

Table 5. Irrigation and precipitation schedule for outdoor trial.

Day	Water (cm)	Comments
1	2.0	
3	0.5	
5	0.7*	*precipitation event
7	---	seedlings appeared
10	---	emergence recorded, penetrometer measurements taken

Penetrometer

A pocket penetrometer (SOILTEST CL-700) was used to measure unconfined crust strength. The principle underlying this instrument is that the release of the compressed spring sinks the plunger into the soil, measuring surface crust resistance independent of operator strength (Kullman et al. 1981). Twenty five measurements were recorded for the three Burbach soils over three treatments and a control. The Hardy soil was omitted from the penetrometer tests. An indistinct surface crust and subsurface soil aggregation prevented the plunger from breaking through and releasing. Means of soil crust resistance were statistically analyzed with two way analysis of variance. Extensive fractures interfered with instrument precision.

Greenhouse Seedling Emergence

Seedling emergence was measured in greenhouse trials. A randomized block design was used for this study. Four treatments of four soils using three replicates or

blocks yielded 48 experimental units. Treatment rates are the same as those used in the outdoor trial (Table 4). Control and treated soils were spread into 20 x 30 cm galvanized trays to a depth of 5 cm. Sugarbeet seeds were placed at a depth of 2.5 cm every 3.5 cm in three rows spaced 6 cm apart.

The irrigation schedule for the greenhouse trial is summarized in Table 6. Uniform aliquots of water were applied from a height of 0.5 m to create sufficient drop velocity to initiate crust formation. Seedling emergence as a percentage of each experimental unit (n = 18) was recorded and statistically analyzed with two way analysis of variance.

Table 6. Irrigation schedule for greenhouse trial.

Day	Water (ml)	Comments
1	400	
5	100	
7	50	seedlings appeared
10	---	seedling emergence recorded
12	---	soil surface photography

Soil Surface Image Digitization

A photographic quantification technique was designed to acquire data for comparisons of treatment effects on the extent of surface cracking. Using a 35 mm camera mounted on a copy stand apparatus, all 48 experimental unit surfaces were photographed at an F8 setting and a 1/15 second exposure using Kodak TMX 135-36 black and white film. A consistent focal length of 43 cm from soil surface to film was maintained. A

standard scale of length was photographed at this same focal length in order to develop the appropriate scale conversion factor for computer output data. Developed images were scanned into a black and white drawing format using a Hewlett Packard ScanJet IIcx scanner. This digitized data was analyzed using Delta-T-Scan Root Software and reported as surface fracture cover as a percentage of total area. Two way analysis of variance compared fracture cover percentage over four treatments and three blocks for each soil.

RESULTS AND DISCUSSION

Soil CharacterizationSoil pH and Electrical Conductivity

The EC and pH data for saturated paste extracts of the four soils are shown in Table 7. These data suggest calcareous systems.

Table 7. Soil pH and electrical conductivity (mean* \pm standard deviation).

Soil	pH	EC (mmhos/cm)
Burbach 1	8.3 \pm 0.03	1.7 \pm 0.02
Burbach 2	8.3 \pm 0.03	1.3 \pm 0.02
Burbach 3	7.6 \pm 0.04	4.3 \pm 0.03
Hardy	8.5 \pm 0.03	0.7 \pm 0.01

*n=6.

The pH of a solution in equilibrium with calcium carbonate and with the carbon dioxide level of the atmosphere is 8.3. Soils are naturally buffered in the alkaline pH range by precipitation of calcium carbonate which produces equilibrium soil pH's of 8.0 to 8.5 (Lindsay 1979). Field pH values of calcareous soils are less than 8.3 because of greater carbon dioxide concentrations in the soil gas phase than in the atmosphere. Soil pH's in excess of 8.5 are usually associated with sodium salts.

The Burbach 1 and Burbach 2 soils appear to be calcareous (pH=8.3) and non-saline. Using EC as the only criteria, Burbach 3 is slightly saline by definition (EC>4.0) with a lower pH (7.6) typical of saline soils (USSLS 1969). The Hardy soil has pH and EC values indicative of a calcareous and non-saline system.

Soil Calcium Carbonate Equivalence

Soil calcium carbonate equivalence as percentages of dry soil mass are presented in Table 8. Naturally occurring calcium carbonate is present in all four soils.

Table 8. Soil calcium carbonate equivalence (mean* \pm standard deviation).

Soil	CaCO ₃ Equivalence (%)
Burbach 1	11.56 \pm 0.61
Burbach 2	12.44 \pm 0.70
Burbach 3	13.60 \pm 0.65
Hardy	5.47 \pm 0.29

*n = 5.

In semi-arid regions, calcium carbonate accumulates in soils. Calcium from mineral weathering and exchangeable calcium ion are precipitated as calcite and occasionally as gypsum. The major variable is the partial pressure of carbon dioxide as it affects carbonate solubility and the reactions of carbon dioxide with water. When the mass of calcium carbonate in soils exceeds several percent, it controls both soil pH and soil solution calcium ion concentrations (Bohn et al.1985). Calcite at a designated partial pressure of carbon dioxide is the solid most likely to control the calcium ion activity in alkaline soils

(Lindsay 1979). The presence of calcium carbonate fixes the equilibrium activity of free calcium ion in solution with respect to pH.

The intensity factor of an element in a soil system is defined as the concentration of that element in the soil solution. The capacity factor is defined as the ability of solid phases in soils to replenish that element as it is depleted from solution (Lindsay 1979). In this study, soil calcium carbonate equivalence (Table 8) is the capacity factor and the water-soluble calcium ion concentration is the intensity factor. The intensity factor, or water-soluble concentration, of an element is maintained at a fixed equilibrium activity by the presence of solid phase material. Addition of soluble calcium-containing amendments to these soils may temporarily raise the intensity factor of calcium. However calcium carbonate precipitation, as calcite, will commence and the intensity factor (calcium ion concentration in soil solution) will eventually fall back to the original equilibrium activity controlled by calcite.

Soil Texture and Linear Extensibility

Particle size distributions and textural classes of the four soils are presented in Table 9. The three Burbach soils contain high silt and clay fractions. The Hardy soil is dominated by clay with no significant sand fraction. Soils such as these are vulnerable to structure loss as organic matter content decreases.

Table 9. Soil particle size distribution and texture.

Soil	Sand (%)	Silt (%)	Clay (%)	Texture
Burbach 1	23	42	35	Clay Loam
Burbach 2	26	40	34	Clay Loam
Burbach 3	28	43	29	Clay Loam
Hardy	2	27	71	Clay

The coefficient of linear extensibility (COLE) is a measure of the shrink-swell capacity of soil and is reported as a ratio of dry soil length (Grossman 1968). Coefficients of linear soil extensibility are listed in Table 10. Values exceeding 0.03 generally indicate the presence of montmorillonite (2:1) clays. Values greater than 0.09 usually indicate sufficient montmorillonite to noticeably alter the shrink/swell properties of a soil (Schafer and Singer 1976).

Table 10. COLE and soil saturation percentage (mean \pm standard deviation).

Soil	COLE*	Saturation Percentage**
Burbach 1	0.065 \pm 0.005	39.3 \pm 0.78
Burbach 2	0.071 \pm 0.004	40.8 \pm 0.94
Burbach 3	0.067 \pm 0.005	28.7 \pm 0.81
Hardy	0.167 \pm 0.011	60.4 \pm 1.96

*n = 6.

**n = 10.

The Hardy soil had a large clay fraction (71%) with a large percentage of swelling clays. When a soil of this nature is water saturated, clay dispersal destroys aggregate structure. The three Burbach soils exhibited some shrink-swell tendencies.

The saturation percentage is calculated from the mass of oven-dry soil and the volume of water added to saturation (USSLs 1969). Soils with significant amounts of 2:1 clays have high saturation percentages due to interlayer expansion of those clays. This swelling of 2:1 clays results in high water-holding capacity and low hydraulic conductivity. Saturation percentage values (Table 10) correlate with COLE measurements.

Water-Soluble Cations and Sodium Adsorption Ratios

Analyses of soluble cations provide accurate determinations of total salt contents of soil solutions. Relative concentrations of cations in soil-water extracts correlate with the composition of the exchangeable cations in the soil. Calcium, magnesium, potassium, and sodium predominate in semi-arid region soils. The water-soluble concentrations of those cations in study site soils are displayed in Table 11.

Table 11. Water-soluble cation concentrations (meq/L).

Soil	[Ca]	[K]	[Mg]	[Na]
Burbach 1	11.80	0.77	5.90	7.10
Burbach 2	9.30	0.67	3.70	5.30
Burbach 3	31.10	2.40	16.10	3.70
Hardy	3.93	0.69	1.47	4.23

Calcium is abundant in all four soil solutions but relatively higher in the Burbach 3 soil solution. The concentration of water-soluble calcium in the Burbach 3 soil (31.10 meq/L) suggests the presence of gypsum (Drever 1982). Calcium sulfate may control calcium solubility in this soil with consequent higher concentrations of water-soluble calcium. This soil also has significant amounts of water-soluble magnesium present with potential Mg-clay dispersion effects. Magnesium and calcium sulfates may be responsible for the salinity of this soil.

Water-soluble data can be used to calculate the sodium adsorption ratio (SAR) of a soil. The SAR is a ratio, for soil extracts, used to express the relative concentration of sodium ions in exchange reactions of the soil (USSLS 1969). Ionic concentrations are expressed in milliequivalents per liter of soil solution and the ratio is determined by the equation:

$$SAR = \frac{[Na]}{\sqrt{\frac{[Ca] + [Mg]}{2}}}$$

The sodium status is well below levels inhibitory to plant growth ($SAR < 15$) for all four soils (Table 12). Sodium adsorption ratio values indicate no sodicity hazard in study site soils (USSLS 1969).

Table 12. Sodium adsorption ratios (SAR).

Soil	SAR
Burbach 1	2.39
Burbach 2	2.08
Burbach 3	0.76
Hardy	2.57

Exchangeable Cations

The "exchange complex" refers to the collective surface-active constituents of the soil. The cations that dominate the exchange complex have a marked influence on soil properties. Subtracting water-soluble ion concentrations from the ammonium acetate extractable concentrations provides exchangeable ion data. The concentrations of four exchangeable cations for two soils, Burbach 2 and Hardy, are listed in Table 13.

Table 13. Exchangeable cation concentrations (meq/100g).

Soil	[Ca]	[K]	[Mg]	[Na]
Burbach 2	33.90	0.57	4.86	0.38
Hardy	38.10	0.13	0.45	0.87

As pH measurements and water-soluble data suggested, exchange sites are dominated by calcium. Calcium carbonate and calcium sulfate dissolution in the extracting solution can lead to erroneously high values of extractable calcium but in view of the

water-soluble data (Table 11), a calcium dominated exchange complex would be expected in all four soils.

The data in Table 13 were used to calculate the soil ESP. These percentages should generally correspond to respective SAR values. The ESP is an expression of the relative amounts of exchangeable sodium as a percentage of the cation exchange capacity (CEC). It is the degree of saturation of the soil exchange complex with sodium and is determined by the equation:

$$ESP = \frac{\text{Exchangeable [Na]} (meq/100g)}{[CEC] (meq/100g)} \times 100$$

The degree of soil sodicity is often described by the ESP. However, determination of this value is tedious and there is a generally good relationship between SAR of the soil solution and ESP of the soil. The SAR is a satisfactory index to the exchangeable sodium status of most soils (USSLS 1969). Exchangeable sodium percentage and SAR values typically show stronger correlation as values increase. Values given in Table 14 correlate well given the low percentages and ratios.

Exchangeable sodium data indicate no sodicity hazard in the study soils. By definition (USSLS 1969), the Burbach 1, Burbach 2, Burbach 3, and Hardy soil samples are not sodic. High exchangeable sodium will cause significant surface crusting and these soils form strong crusts yet they reveal low exchangeable sodium. Surface crusting of these particular soils is probably a function of soil physical and biological properties, not exchangeable sodium.

Table 14. ESP and SAR comparisons.

Soil	ESP	SAR
Burbach 2	0.96	2.08
Hardy	2.20	2.57

Soil Organic Matter

The organic matter content of a soil influences many of its properties. These include the capacity of a soil to supply N, P, S, and trace metals to plants; infiltration and retention of water; degree of aggregation and overall structure; and cation exchange capacity (Nelson and Sommers 1982). The organic matter contents for the four soils are shown in Table 15.

Table 15. Soil organic matter content (mean* \pm standard deviation).

Soil	Organic Matter (%)
Burbach 1	1.35 \pm 0.05
Burbach 2	1.28 \pm 0.01
Burbach 3	1.68 \pm 0.01
Hardy	2.55 \pm 0.09

*n = 2.

Soil organic matter levels below 1.5% are low for semi-arid region soils that generally exhibit median levels of approximately 1.5 to 2.5% (Munshower 1994). The

Hardy soil has the highest organic matter content. Soils very high in clays, through organomineral complexation, have slower rates of organic matter degradation. Increasing the organic matter content of these soils would likely improve soil structure and aggregation and reduce surface crusting.

Precipitated Calcium Carbonate

Properties At Stockpile Conditions

The bulk density of PCC is typical of a small particle size and high surface area material (Table 16). Stockpile moisture is fairly consistent at approximately thirty percent by mass. This material is stable with respect to wind erosion at stockpile moisture conditions, however PCC is extremely dusty after oven-drying. Therefore some moisture content is necessary for the material stability required in standard large-scale handling procedures.

Table 16. Bulk density, water and organic matter contents of precipitated calcium carbonate at stockpile conditions (mean \pm standard deviation).

	Bulk Density* (dry g/cm ³)	Water Content*		Organic Matter** (%)
		mass(%)	volume(%)	
Mean	0.72 \pm 0.07	31.8 \pm 3.8	23.1 \pm 3.8	4.16 \pm 0.10

*n = 8.

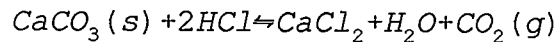
**n = 4.

The organic matter content was indirectly estimated by multiplying the organic carbon concentration as determined by the Walkley-Black method by two (2). This

number is the ratio of organic matter to organic carbon commonly found in soils (Nelson and Sommers 1982). Carbon comprises 48 to 58% of the total mass of soil organic matter. Evidence has been presented that suggests factors between 1.9 and 2.5 for conversion of surface and subsoil carbon to organic matter respectively (Broadbent 1953).

The short-chain carbon organic matter of PCC may require a higher factor, which would yield slightly higher organic matter content than those presented in Table 16. In other words, carbon may comprise less than 48% of the organic acids and peptides of PCC. The organic load present in PCC is of a distinctly different origin than the humic or fulvic materials present in soils.

The determination of calcium carbonate by the mass of carbon dioxide evolved after addition of 3M HCl utilizes the following reaction:



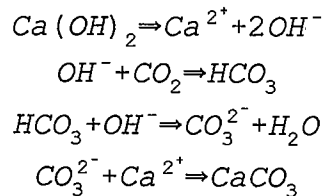
For every mole of carbon dioxide lost, one mole of calcium carbonate is assumed present in the original material. Two sources of error are: incomplete decomposition of the calcium carbonate and loss of water vapor. This method will not measure any calcium hydroxide or calcium oxide present. In contrast, neutralization potential determined by potentiometric titration measures the total alkalinity (calcium oxide, calcium hydroxide, and calcium carbonate) of a liming agent. Neutralization potential and calcium carbonate equivalence data are summarized in Table 17.

Table 17. Calcium carbonate equivalence of PCC (mean* \pm standard deviation).

	Mass Lost	Titration
CaCO ₃ Equivalence	85.60 \pm 2.11	92.90 \pm 0.42

*n = 5.

The data in Table 17 suggest that calcium hydroxide and/or calcium oxide may be present. Calcium hydroxide in the presence of water and carbon dioxide will form calcium carbonate as illustrated by the following reactions:



In the absence of inhibitors, the rate of calcite precipitation at pH values greater than 7.5 is fast, and calcium ion activity values generally reach equilibrium within several hours (Inskeep and Bloom 1985). However, several ions and compounds such as magnesium, phosphate ion, and organic ligands can inhibit calcium carbonate precipitation. The mechanism of inhibition for these ions is adsorption of the ion on the calcite crystal surface and subsequent blockage of crystal growth sites (Inskeep and Bloom 1986). Given the presence of calcite precipitation inhibitors in diffusion juice (e.g. organic ligands, magnesium, and phosphate ion), the rapid flocculation, and the relatively brief period of time in solution; it is logical to assume that the entire stockpile system has not proceeded to equilibrium. That is, calcium oxide and hydroxide are probably present in stockpiled PCC.

Effects of PCC on Soil Crust Formation And Strength

Areal Extensibility

Areal extensibility means calculated from six replicates for each of four soils over five treatments and a control are illustrated in Figure 1. Two way analysis of variance indicated that a significant difference existed between soils (Table 18). One way analysis of variance evaluated means across treatments for each soil. Figure 1 illustrates the coefficient of areal extensibility for the four soils across five PCC rates. Statistical analyses may be found in Appendix B (Tables 30 through 34).

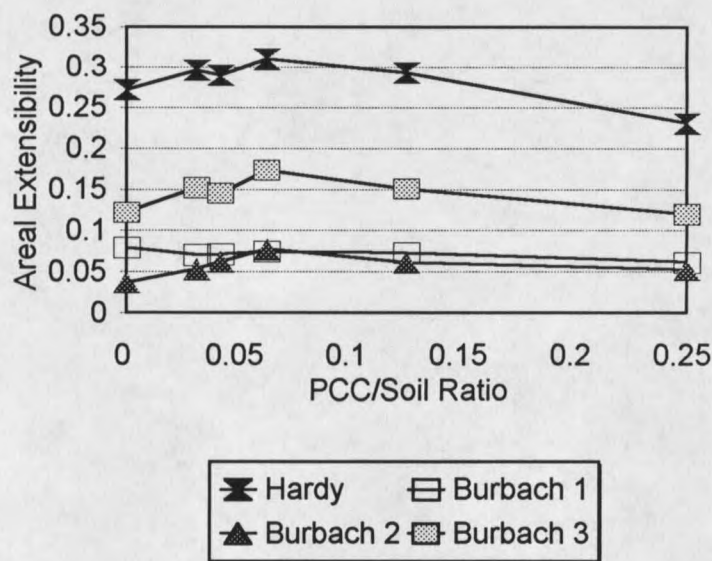


Figure 1. Soil areal extensibility (n = 6).

