EFFECTS OF SILICON ON SOIL CRUSTING AND SOIL QUALITY

By TAYLOR LYNN-MARIE BEARD

A thesis submitted in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE IN SOIL SCIENCE

WASHINGTON STATE UNIVERSITY Department of Crop and Soil Sciences

DECEMBER 2013

To the Faculty of Washington State University:

The members of the Committee appointed to examine the thesis of TAYLOR LYNN-MARIE BEARD find it satisfactory and recommend that it be accepted.

William L. Pan, Ph.D., Chair

Jeffery L. Smith, Ph.D.

James B. Harsh, Ph.D.

ACKNOWLEDGEMENTS

I would like to take this opportunity to express my appreciation to those who have contributed to this work:

First and foremost, Dr. Bill Pan, committee chair, for giving me the opportunity to explore new ideas throughout this graduate program;

Secondly, Dr. Jeff Smith, committee member, for his assistance with preparations, completion of lab work, and advice;

Thirdly, Dr. Jim Harsh, committee member, for his guidance and support throughout this project;

The Regional Approaches to Climate Change Project (REACCH) for providing the funding for this research;

Haley Saam, Clayton Waller, Mike Pecharko, Stephanie Jenck, Wendiam Sawadgo, Kyle Kammerrer, and Rodrigo Bonilla, undergraduate students, for their assistance in the field and laboratory stages of this research;

Tai Maaz, Kristy Ott, Lauren Young, Isaac Madsen, Jolene Mwengi, graduate students for their support, motivation, and interests in my research;

Ron Bolton, Margaret Davies, Debbie Bikfasy, Jeff Boyle, and Tammi Stubbs, Research Technicians, for their invaluable assistance throughout this entire graduate program;

The faculty and other graduate students in the Department of Crop and Soil Science for their academic support;

Finally, I would like to thank my family and friends for their continued support and confidence in my academic goals.

iii

EFFECTS OF SILICON ON SOIL CRUSTING AND SOIL QUALITY

Abstract

by Taylor Lynn-Marie Beard, M.S. Washington State University December 2013

Chair: Dr. William L. Pan

Silicon (Si) levels have a wide range of variation in plant and soil systems depending on abiotic and biotic factors. In the inland Pacific Northwest the predominant cropping system relies on wheat (a Si accumulator). Within this region, studies have shown high levels of total soil Si and evidence of Si compounds becoming potential cementing agents therefore degrading soil quality. The dependence of Si cycling on plant type, environmental factors, and agronomic inputs needs to be assessed in order to determine if introduction of canola (a non-accumulator of Si) could enhance soil quality by reducing the occurrence or severity of soil crusting in comparison to wheat-dominated systems. Both wheat and canola were grown in a greenhouse and upon harvest the wheat residue accumulated between 40–65% more Si than canola. This residue was then used in a laboratory incubation with soil pH as a variable. The results suggest that a higher pH, rather than residue type, was the primary factor positively affecting surface resistance, water soluble Si (Si_{ws}), and amorphous Si (Si_{am}). The greenhouse residues were also used in a decomposition study, which showed wheat had a slightly faster decomposition rate compared to canola, consistent with the lower starting C:N ratio of the wheat residue. An additional incubation with applications of amorphous silica (SiO₂) confirmed that such

iv

applications positively influence water loss, soil Si, surface resistance, and crust thickness. In addition to the silica treatments, soils from two cropping systems were used: one previously cropped in wheat and the other in canola. The soil previously cropped in wheat had higher soil Si_{am}, surface resistance, and crust thickness compared to the canola system demonstrating the influence crop rotation can have on Si related soil properties. A field survey of Si_{ws}, Si_{am}, and surface resistance showed little dependence across cropping systems. As shown from the experiments under controlled conditions, it can be concluded that Si cycling does affect important soil physical properties. The lack of confirmation in the field survey suggests that other factors influence the state of Si in active cropping systems and should be the focus of further research.

TABLE OF CONTENTS

ACKNOWLEDGEMENTS iii
ABSTRACTiv
LIST OF TABLES ix
LIST OF FIGURES xi
CHAPTER
1. INTRODUCTION
Research Goal
Objectives
Thesis Organization
Literature Cited
2. THE CYCLING AND ALLOCATION OF SILICON IN CANOLA AND WHEAT
2. THE CYCLING AND ALLOCATION OF SILICON IN CANOLA AND WHEAT GROWN WITH VARYING NITROGEN RATES
GROWN WITH VARYING NITROGEN RATES13
GROWN WITH VARYING NITROGEN RATES
GROWN WITH VARYING NITROGEN RATES 13 Summary 13 Introduction 14 Materials and Methods 16 Results 20 Discussion 31
GROWN WITH VARYING NITROGEN RATES13Summary13Introduction14Materials and Methods16Results20Discussion31Conclusions32

Introduction	39
Materials and Methods	40
Results	45
Discussion	57
Conclusions	58
Literature Cited	60
4. A COMPARISON OF TWO ROTATION HISTORIES	63
Summary	63
Introduction	64
Materials and Methods	66
Results	72
Discussion	84
Conclusions	85
Literature Cited	86
5. CONCLUSIONS	89
APENDIX I PILOT STUDY	91
Research Goal	91
Materials and Methods	91
Results	95
Literature Cited	100
APENDIX II SEM IMAGES	101
Introduction	101
Materials and Methods	101

Results and Discussion	
Conclusion	111
Literature Cited	

TABLE OF TABLES

CHAPTER

2. THE CYCLING AND ALLOCATION OF SILICON IN CANOLA AND WHEAT GROWN WITH VARYING NITROGEN RATES

2.1 Initial greenhouse soil data
2.2 Average yield per pot for each N fertilizer rate
2.3 Average total N per pot for each N fertilizer rate
2.4 Average residue C, N, and C:N per pot for each N fertilizer rate26
2.5 Average percent of N derived from fertilizer (%Ndff)26
2.6 Utilization of fertilizer N by plant organs and recovery efficiencies26
2.7 Average soluble components, hemicellulose, cellulose, lignin, ash, and Si in
wheat and canola straw27
2.8 Average amounts of Si found in the initial soil, post-harvest soil, plant
residue, roots, grain, and the difference or unaccounted for Si27
3. EFFECTS OF WHEAT AND CANOLA RESIDUE ON SOIL
3.1 Initial soil data45
3.2 Initial residue analysis45
3.3 Average values by treatment for pH, C:N, % ¹⁵ Ndfp (derived from labeled
plant residue), Si_{ws} , Si_{am} , surface strength, crust thickness, and plant %Si49
3.4 Average residue weight loss (g), concentration of Si (%), and g of Si over time
per treatment55

4. A COMPARISON OF TWO ROTATION HISTORIES

4.1 Initial soil data70
4.2 Average water loss, surface resistance, and crust thickness by treatment73
4.3 Mean Si _{ws} , and Si _{am} per treatment74
4.4 Penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil), and Si _{ws} (g Si/kg
soil) values for each crop rotation by depth located in Davenport, WA78
4.5 Penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil), and Si _{ws} (g Si/kg
soil) values for each crop rotation by depth located in Reardan, WA79
4.6 Penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil), and Si _{ws} (g Si/kg
soil) values for each crop rotation by depth located in Ralston, WA79
4.7 Penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil), and Si _{ws} (g Si/kg
soil) values for each crop rotation by depth located in LaCrosse, WA80
4.8 Irrigated Site #1 penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/ kg soil),
and Si_{ws} (g Si/kg soil) values for each crop rotation by depth located in
Odessa, WA80
4.9 Irrigated Site #2 penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil),
and Si_{ws} (g Si/kg soil) values for each crop rotation by depth located in
Odessa, WA81
APPENDIX I PILOT STUDY
A1-1 Initial Soil Data95
A1-2 Residue Analysis95
A1-3 Average water loss, pH, C:N, and surface resistance per treatment
A1-4 Average Si _{am} +Si _{ws} , Si _{am} , and Si _{ws} per treatment

TABLE OF FIGURES

2. THE CYCLING AND ALLOCATION OF SILICON IN CANOLA AND WHEAT GROWN WITH VARYING NITROGEN RATES

2.1 Average total N of aboveground biomass per fertilizer treatment for wheat and
canola
2.2 Average % nitrogen derived from fertilizer (Ndff) for wheat and canola
(straw+leaves) residue per fertilizer rate (mg/kg)28
2.3 Fiber concentrations in wheat residue per fertilizer treatment
2.4 Fiber concentrations in canola residue (straw+leaves) per fertilizer
treatment
2.5 Total Si concentration (%) for wheat and canola at each fertilizer rate30
2.6 Total accumulation of Si by treatment and crop type (%Si * yield)30

3. EFFECTS OF WHEAT AND CANOLA RESIDUE ON SOIL

3.1 Average water loss (g) per pot for each watering cycle during a two week	
period for each treatment	49
3.2 Average soil C:N over time per treatment	50
3.3 Average plant C:N over time per treatment	50
3.4 Average $\%^{15}$ Ndfp ($\%^{15}$ N found in the soil derived from labeled plant	
residue) over time per treatment	51
3.5 Average soil Si _{ws} (g/kg) over time per treatment	51
3.6 Average soil Si _{am} (g/kg) over time per treatment	52

3.7 Average soil surface resistance (g/cm ²) over time per treatment	52
3.8 Average soil crust thickness (mm) over time per treatment	;3
3.9 Average concentration (%) of plant Si over time per treatment	;3
3.10 Average residue weight (g/pot) over time per treatment	55
3.11 Average residue Si concentration (%) over time per treatment	6
3.12 Average grams of Si/pot over time per treatment	6
4. A COMPARISON OF TWO ROTATION HISTORIES	
4.1 General description of each site including: annual rainfall, soil type, years	
since introduction of canola, and the rotation sampled7	1
4.2 Penetrometer measurements in kPa per cm for each crop rotation at the	
Davenport research site8	31
4.3 Penetrometer measurements in kPa per cm for each crop rotation at the	
Reardan research site8	32
4.4 Penetrometer measurements in kPa per cm for each crop rotation at the	
Ralston research site8	32
4.5 Penetrometer measurements in kPa per cm for each crop rotation at the	
LaCrosse research site8	33
4.6 Penetrometer measurements in kPa per cm for each crop rotation at the	
Odessa research sites8	33
APPENDIX I PILOT STUDY	
A1-1 Average pH for each treatment over time9)6
A1-2 Average surface resistance for each treatment over time9)7
A1-3 Average Si _{am} and Si _{ws} for each treatment over time)9

APPENDIX II SEM IMAGES

A2-1 Pre-extraction SEM micrographs of residue comparison incubation at
~2Kx magnification105
A2-2 Post-extraction SEM micrographs of residue comparison incubation at
~2Kx magnification106
A2-3 Pre-extraction SEM micrographs of the cropping history comparison
incubation (previously cropped in wheat) at 2Kx magnification107
A2-4 Post-extraction SEM micrographs of the cropping history comparison
incubation (previously cropped in wheat) at 2Kx magnification108
A2-5 Pre-extraction SEM micrographs of the cropping history comparison
incubation (previously cropped in canola) at 2Kx magnification109
A2-6 Post-extraction SEM micrographs of the cropping history comparison
incubation (previously cropped in canola) at 2Kx magnification110

CHAPTER 1

INTRODUCTION

Soil Silica Cycling:

Silicon (Si) is the second most common element of the earth's crust with a mean content of 28.8 wt% (Hans Wedepohl, 1995). Although it is a common element, the understanding of Si pools and fluxes in terrestrial biogeosystems is lacking due to the complex weathering and neo-formation processes, which create a variety of Si phases within the soil. The solid phase of Si occurs in mineral soils developed from rocks or sediments and are mainly composed of primary crystalline silicates such as quartz, feldspars, mica, and secondary silicates, especially clay minerals (Sauer et al., 2006). These minerals can also contain cations such as iron, aluminum, copper, zinc, magnesium, and calcium (Farmer et al., 2005). Silicate minerals generally have a tetrahedral arrangement of Si surrounded by four oxygen atoms (Lindsay, 1979). The solubility of such minerals in terms of silicic acid (H₄SiO₄) is expected to range from 10^{-2.74} M (SiO_{2am}) to 10^{-4.0} M (quartz) with other silicate minerals having intermediate solubilities (Strober, 1967).

Most soils contain Si of biogenic origin (BSi), mainly in the form of phytoliths, and pedogenic amorphous silica (SiO_{2am}) (Drees et al., 1989). The solubility of such silica in soils is intermediate between quartz and SiO_{2am}. An example of an equilibrium reaction would be SiO₂(soil) + 2H₂O \rightleftharpoons H₄SiO₂⁰ (Lindsay, 1979). The chemistry of silica is very complex and equilibrium relationships are difficult to attain. This difficulty was demonstrated by Strober in 1967 when he measured the solubility of silicic acid in

aqueous suspensions containing different forms of silica and concluded that surface adsorption of silicic acid often prevents equilibrium relationships from being achieved.

In the liquid phase, dissolved silicic acid primarily occurs as monomeric silicic acid (H₄SiO₄) (Iler, 1979). It is within this phase that the readily available pool of Si can be found. Typical concentrations of Si in soil solutions fall between less than one and 65 mg Si/L; however, concentrations can differ widely in space and time depending on the particular soil minerals present and other abiotic and biotic factors (Sadiq et al., 1980; Wickramasinghe and Rowell, 2005). The dissociation of silicic acid and the polymerization of silicate species in solution also vary, however an example of an equilibrium reaction would be $H_4SiO_4^{0} \rightleftharpoons H_3SiO_4^{-} + H^+$ (Lindsay, 1979). Under the conditions that silicic acid in soil solution is controlled by soil SiO₂ levels, Lindsay (1979) found that only at pH values above 8.5 do the ionic silicates contribute significantly to the total SiO₂ in solution. In the normal pH range of soils, silicic acid comprises the major silicate species.

Silicon and its effects on plants:

Silicon is absorbed in the form of uncharged monomeric silicic acid, H₄SiO₄, by the plant root when the solution pH is below nine (Ma and Yamaji, 2006). Uptake can occur either passively, coinciding with the uptake of water, or as an active form of nutrient recruitment depending on the plant species (Prychid et al., 2004). Following uptake by the roots, Si is translocated to the shoots via the xylem. In the shoot, silicic acid is further concentrated through transpiration and is ultimately polymerized to Si_{am} (Richmond and Sussman, 2003). Deposition usually takes place in cell lumens, cell walls, and intercellular spaces or external layers (Casey et al., 2003). It is present in roots, leaves,

and inflorescence bracts of cereals (Epstein 1993). The incorporation of silicon into cell walls has at least two energetically positive effects. First, the role of Si is comparable to lignin in that it is a compression resistant structural component of cell walls (Epstein, 1993). However, the metabolic cost of synthesizing one silicon atom is only one adenosine triphosphate (ATP) while an equivalent amount of lignin costs about 27ATP (Van Soest 1996). Silica can therefore be considered an energetically inexpensive structural component of the cell wall. Second, the erect habit and disposition of the leaves of plants with high amounts of Si favor light interception and consequently photosynthesis (Epstein, 1993).

Of all the elements found in plants, Si has shown the greatest variation between plant parts, plant type, and species ranging from 0.1 to 10% on a dry weight basis (Epstein, 1993; Bilbro et al., 1991). The only significant trend is that monocots have the ability to accumulate more Si than dicots. Differences in Si uptake occur because the transport of Si in plants is much more complex than in other silicon-utilizing organisms. In plants, additional modes of transport are needed to enable long distance transport across specialized tissues and compartments from the roots to the stomata. Studies done by Mitani and Ma (2005) suggest that the density of the transporter that transports Si from the external solution to the cortical cells, and the occurrence of a transporter to transport Si from the cortical cells to the xylem, differs among plant species. In addition to the transport system a number of ecological factors including climate, soil characteristics, moisture, and plant maturity affect silica body development by influencing the amount of silica in the soil that is available to plants (Prychid et al., 2004).

Silica positively affects the growth and development of many plants by contributing to the mechanical strength of cell walls, keeping the plant erect, and helping in the positioning of leaves for light interception (Bilbro et al., 1991). Silica also helps protect against multiple abiotic and biotic stresses. An additional effect of Si is that numerous studies have shown it is effective in inhibiting diseases caused by both fungi and bacteria in different plant species. This is primarily due to the reinforcement of the cell wall by deposited, polymerized silica, which acts as a physical barrier (Epstein, 2009). The silica in trichhomes lends leaves the roughness and toughness that impede the penetration of herbivores and pathogens through the cell walls. Other benefits of Si include aiding in multiple biotic and abiotic stresses including toxic metals, high salinity, water deficits, and lodging in wheat (Euliss et al., 2005; Currie and Perry, 2007; Hashemi et al, 2010; Janislampi, 2012).

The dissolution of straw silica, which releases Si to the soil solution, depends not only on the purity of the silica, but also on the degree of exposure of the phytoliths. Surface dumbbell cells and protuberances are more exposed than granular amorphous silica enclosed in the plant matrix and are likely the source of the readily soluble fraction when straw is placed in solution or in the soil (Ma and Yamaji, 2006). Decomposition of the straw will in the long term expose more silica because phytoliths will be released from the plant matrix and dissolved. However, the rate of decomposition appears to be influenced by the concentrations of inherent silica and other structural components (hemicellulose, cellulose, and lignin) in the straw. When crop residues are incorporated into the soil, the ultimate concentration of Si in the soil solution is expected to depend on the rate of decomposition, dissolution rate of the phytolith-Si, adsorption of Si by the soil,

and other environmental factors (Brown and Mahler, 1987b). While there is no single indicator of the rate of residue decomposition in soil, positive correlations have been observed between high hemicellulose levels and decomposition rate (Voroney et al., 1989) while high lignin content, high C:N ratios, and low total N can lower decomposition rates (Soon and Arshad, 2002; Lupwayi et al., 2006; Stubbs et al., 2009). Therefore crop residue management may have an important impact on Si solubility and movement.

Why should we be concerned with Si levels?

With such variability of Si levels within plants, residue management has an important impact on Si solubility and movement. Studies have shown that Si levels can be linked to pan formation at deeper depths within the soil profile increasing mechanical resistance to roots, impairing drainage, and therefore may reduce plant growth and production (Brown and Mahler, 1987b; Gollany et al., 2006). While this relationship has been well researched, the relationship between Si levels and surface crusting has yet to be determined. Surface crusting is a common occurrence in many cultivated soils in arid and semi-arid regions. Important effects of a surface crust include reduction of infiltration rate (IR), enhancement of runoff, alteration of erosion, and most importantly interference with seed germination. The Pacific Northwest (PNW) has the deepest seeding depth in the world (up to 20 cm) for both canola and winter wheat in order to reach adequate water for germination. Due to this extreme seeding depth, it is not the coleoptile that emerges from the soil, but the first leaf. This can be an issue because the first leaf is thin, has weak structural support, lacks the emergence force or lifting capacity, and is therefore susceptible to kinking resulting in no emergence (Schillinger, 2011). Kinking generally

occurs when rainfall takes place after planting, but before emergence, causing the formation of a thin, fragile soil crust that the first leaf cannot penetrate.

Crusting formation:

The general sequence of events that leads to crust formation under rainfall conditions are as follows: (i) breakdown of soil aggregates caused by raindrop impact or slaking; (ii) movement of fine particles into the upper few millimeters of the soil, where they are deposited in the voids; and (iii) subsequent drying of the soil surface to form a thin film, which restricts further entry of water and movement of soil particles (Wakindiki and Ben-Hur, 2002). Two main types of soil crust, namely structural and depositional crusts, are generally recognized, according to their mechanisms of formation. Structural crusts are due mainly to water-drop impact, whereas depositional crusts are formed by translocation of fine particles and their deposition at some distance from their original location. The tendency of a soil to form a crust depends on aggregate stability. Aggregate stability has been found to increase with increasing clay content but, conversely, increasing clay content can also promote crust formation. Ben-Hur et al. (1985) explained this paradox by suggesting that in soils containing more than 20% clay, the clay fraction acts as a cementing material, stabilizing soil aggregates against the beating action of raindrops, and so preventing crust formation. On the other hand, in soils containing less than 20% clay, the clay acts as a substrate for crust formation, decreasing the steady-state hydraulic conductivity of the crust. Soils with high amounts of silt, low organic matter, and therefore low aggregate stability, such as those found in the PNW, have been identified as particularly susceptible to crusting.

Factors affecting crusting:

In addition to climate, there are multiple management factors that can influence the development of surface crusts however, for the purpose of this research the main focus will be on two factors: the effects of fertilizer use and the influence of cropping rotation. Increased use of ammonium (NH_4^+) based fertilizers in farm management systems during the last 70 years has affected the chemistry of surface soils in the Palouse area of northern Idaho and eastern Washington (Brown and Mahler, 1987a). The nitrification of NH_4^+ has acidified the top 25 cm of the soils and has resulted in reduced plant productivity and increased levels of soluble Si (Gollany et al., 2005). Variations in acidity influence reactions of Si, either causing adsorption (higher pH) or maintaining Si in the solution phase (lower pH). Additional research done by Gollany et al. (2006) examined the interactive effects of N fertilizer and crop residue management on water soluble Si (Si_{ws}) in a wheat-fallow cropping system on a Walla Walla silt loam. Nitrogen fertilizer application decreased Si_{ws} by 17% as a result of leaching. Amendments including larger quantities of crop residue increased the soil organic carbon (SOC) concentration and Si_{ws} by 10%. High SOC, reduced Si dissolution, illuviation, and deposition. Although the form of this Si_{ws} and SOC association was not determined, it is likely that the SOC reduced the siliceous surface available for dissolution. Gollany et al. (2006) suggested that the additions of sufficient crop residue might increase SOM and Si_{ws} interactions by forming phytoliths, Si_{am}, or Si complexes. Either one of these processes can account for reduced biodegradation and reduced siliceous surface area available for dissolution.

In arid and semi-arid areas, where soils are more susceptible to crusting, it may be beneficial to consider both the structural composition (specifically Si levels) of crops and

fertilizer use within the cropping system. Introduction of crops that accumulate less Si, such as canola or other dicots, may aid in decreasing the severity or occurrence of soil crusting therefore improving seedling emergence and subsequent crop productivity.

RESEARCH GOAL

The overall goal of this research was to determine if introducing canola into a crop rotation could reduce the occurrence or severity of soil crusting in comparison to wheat dominated systems.

Objectives:

The thesis objectives are to:

- 1) Determine the role and allocation of Si in both wheat and canola grown with varying fertilizer rates.
- Determine how various fertilizer rates influence the hemicellulose, cellulose, and lignin levels of both wheat and canola in order to suggest potential decomposition rates.
- Evaluate the decomposition and release of Si into the soil from wheat and canola residue and the effects on soil crusting.
- Determine how wheat and canola may affect soil crusting and silicon levels over a long period of time.

THESIS ORGANIZATION

Each chapter of this thesis is self-contained. Chapter 2 addresses objectives 1&2 through a greenhouse experiment. Chapter 3 contains two laboratory studies addressing objective 3. Chapter 4 introduces a laboratory/field study addressing objective 4 as well as additional questions raised by the experiments reported in chapter 3.

LITERATURE CITED

- Ben-Hur, M., R. Keren, I. Shainberg, and M. Gal. 1985. Effect of water quality and drying on soil crust properties. Soil Sci. Soc. Am. J. 49(1):191-196.
- Bilbro, J.D., D.J. Undersander, D.W. Fryrear, and C.M. Lester. 1991. A survey of lignin, cellulose, and acid detergent fiber ash contents of several plants and implications of wind erosion control. J. Soil Water Conserv. 46(4):314-316.
- Brown, T.H. and R.L. Mahler. 1987a. Effects of phosphorus and acidity on levels of silica extracted from a Palouse silt loam. Soil Sci. Soc. Am. J. 51(3): 674-677.
- Brown T.H. and R.L. Mahler. 1987b. Sorption of Silica in a northern Idaho Palouse silt loam. Soil Science 144(3):181-189.
- Casey, W.H., S.D. Kinrade, C.T.G. Knight, D.W. Rains, and E. Epstein. 2003. Aqueous silicate complexes in wheat, *Triticum aestivum* L. Plant, Cell Environ. 27:51-54.
- Currie, H.A., and C.C. Perry. 2007. Silica in plants: biological, biochemical, and chemical studies. Ann. Bot. 100(7):1383-1389.
- Drees, L.R., L.P. Wilding, N.E. Smeck, and A.L. Sankayi. 1989. Silica in soils: quartz and disordered silica polymorphs. p. 913-974. *In* J.B. Dixon and S.B. Weed (ed.)
 Minerals in Soil Environments. SSSA Book Series No. 1, Madison, WI.
- Epstein, E. 1993. The anomaly of silicon in plant biology. Proc. Natl. Acad. Sci. 91:11-17.
- Epstein, E. 2009. Silicon: its manifold roles in plants. Ann. Appl. Biol. 155(2):155-160.
- Euliss, K.W., B.L. Dorsey, K.C. Benke, M.K. Banks, and A.P. Schwab. 2005. The use of plant tissue silica content for estimating transpiration. Ecological Engineering 25(4):343-348.

- Farmer, V.C., E. Delbose, and J.D. Miller. 2005. The role of phytolith formation and dissolution in controlling concentrations of silica in soil solutions and streams. Geoderma 127(1):71-79.
- Gollany, H.T., R.R. Allmaras, S.M. Copeland, S.L. Abrecht, and C.L Douglas. 2005.Tillage and nitrogen fertilizer influence on carbon and soluble silica relations in aPacific Northwest mollisol. Soil Sci. Soc. Am. J. 69(4):1102-1109.
- Gollany, H.T., R.R. Allmaras, S.M. Copeland, S.L. Albrecht, and C.L. Douglas. 2006. Incorporated source carbon and nitrogen fertilization effects on carbon storage and soluble silica in a Haploxeroll. Soil Science 171(8):585-597.
- Hans Wedepohl, K. 1995. The composition of the continental crust. Geochimica Et Cosmochimica Acta 59(7):1217-1232.
- Hashemi, A., A. Abdolzadeh, and H.R., Sadeghipour. 2010. Beneficial effects of silicon nutrition in alleviating salinity stress in hydroponically grown canola (*Brassica napus*) L., plants. Soil Sci. Plant Nutr. 56(2):244-253.
- Janislampi, K.W., 2012. Effect of silicon on plant growth and drought stress tolerance [Online]. Available at <u>http://digitalcommons.usu.edu/etd/1360/</u>. All Graduate Theses and Dissertations. Paper 1360.

Lindsay, W.L. 1979. Chemical equilibria in soils. John Wiley and Sons Inc, New York.

- Lupwayi, N.Z., G.W. Clayton, J.T. O'Donovan, K.N. Harker, T.K. Turkington, and Y.K. Soon. 2006. Nitrogen release during decomposition of crop residues under conventional and zero tillage. Can. J. Soil Sci. 86:11-19.
- Ma, J.F., and N. Yamaji. 2006. Silicon uptake and accumulation in higher plants. Trends Plant Sci. 11(8):392-397.

- Mitani, N. and J.F. Ma. 2005. Uptake system of silicon in different plant species. J. EXP. Bot. 56:1255-1261.
- Prychid, C.J., P.J. Rudall, and M. Gregory. 2004. Systematics and biology of silica bodies in monocotyledons. The Botanical Review 69(4):377-440.
- Richmond, K.E. and M. Sussman. 2003. Got Silicon? The non-essential beneficial plant nutrient. Curr. Opin. Plant Biol. 6(3):268-272.
- Sadiq, M., B.E. Bledsoe, and C.G. Enfield. 1980. Solubility relationships of silica in soils. Commun. Soil Sci. Plant Anal. 28:1245-1252.
- Sauer, D., L. Saccone, D.J. Conley, L. Herrmann, and M. Sommer. 2006. Review of methodologies for extracting plant-available and amorphous Si from soils and aquatic sediments. Biogeochemistry 80(1):89-108.
- Schillinger, W.F. 2011. Rainfall impacts winter wheat seedling emergence from deep planting depths. Agron. J. 103(3):730-734.
- Soon, Y., and M. Arshad. 2002. Comparison of the decomposition and N and P mineralization of canola, pea and wheat residues. Biol. Fertil. Soils 36:10-17.
- Strober, W. 1967. Formation of silicic acid in aqueous suspensions of different silica modifications. *In* R.F. Gould, Ed. Equilibrium concepts in natural water systems. Adv. Chem. 67:161-182.
- Stubbs, T.L., A.C. Kennedy, P.E. Reisenauer, and J.W. Burns. 2009. Chemical composition of residue from cereal crops and cultivators in dryland ecosystems. Agron. J. 101:538-545.

- Van Soest, P.J. 1996. Rice straw, the role of silica and treatments to improve quality.Werner and Ingbar's The Thyroid: A Fundamental and Clinical Text. 7th Ed.Philadelphia: Lippincott-Raven: 922–945.
- Voroney, R.P., E.A. Paul, and D.W. Anderson. 1989. Decomposition of wheat straw and stabilization of microbial products. Can. J. Soil Sci. 69:63-77.
- Wakindiki, I.I.C., and M. Ben-Hur. 2002. Soil mineralogy and texture effects on crust micromorphology, infiltration, and erosion. Soil Sci. Soc. Am. J. 66(3):897-905.
- Wickramasinghe, D.B. and D.L. Rowell. 2006. The release of silicon from amorphous silica and rice straw in Sri Lankan soils. Biol. Fertil. Soils 42(3):231-240.

CHAPTER 2

THE CYCLING AND ALLOCATION OF SILICON IN CANOLA AND WHEAT GROWN WITH VARYING NITROGEN RATES SUMMARY

Silicon (Si) content of crops can be an important factor to consider for residue management due to the fact that rate of decomposition can be affected by the inherent Si content and other structural components (hemicellulose, cellulose, and lignin) of the straw. In addition to decomposition rate, the cycling of Si back into the soil is also an important factor due to its potential as a cementing agent (Brown and Mahler, 1987). Therefore it may be beneficial to introduce crops into a rotation that have lower Si levels such as canola rather than Si accumulators such as wheat in order to assist with residue decomposition and lower soil crusting potential. Fertilizer use and its effect on soil pH is another influential management factor of soil Si levels. Variations in pH can influence reactions of Si, either causing sorption in calcareous soils or maintaining Si in the solution phase for plant uptake as seen in more acidic soils (Brown and Mahler, 1987). In order to further explore the role and allocation of Si and other structural components with varying fertilizer rates, spring wheat (Triticum aestivum 'Louise'), and spring canola (Brassica napus) were grown in a greenhouse experiment. Plants were grown in a 50/50 mix of Palouse silt loam and Sunshine Mix #2. Labeled fertilizer (¹⁵NH₄)₂SO₄ was applied at four different rates: 6, 60, 180 and 420 mg N kg⁻¹. Each treatment was split into three applications: emergence, tillering, and stem elongation. Once plants reached maturity, they were harvested and partitioned to separate grain, residue, roots, and soil components. Plant samples were analyzed for total C and N, atom %¹⁵N, fiber, and Si

content. Soil samples were analyzed for total C and N, NH_4^+ , NO_3 , pH, water soluble Si (Si_{ws}) , and amorphous Si (Si_{am}) . Canola produced more biomass with increasing fertilizer however; the recovery of fertilizer N in wheat biomass was greater. The Si and fiber analyses showed that the biochemistry differed between wheat and canola, but not significantly with fertilizer rate. Wheat had significantly more Si and hemicellulose compared to canola, which had significantly more lignin and cellulose than wheat. This suggests that canola may rely more on lignin for strength and protection while wheat relies on Si.

INTRODUCTION

Silicon (Si) is the second most common element of the earth's crust (Hans Wedepohl, 1995), however, the importance of Si in soil systems and therefore plant nutrition had not been brought to light until fairly recently. Emanuel Epstein (1967, 1994, 1999, 2009) has been the main advocate for recognizing Si as an essential element for plant nutrition due to the fact that multiple crops benefit from Si application. Although Si is a dominant element in most soils, weathering and neo-formation processes create a variety of Si phases and the understanding of the phases and fluxes is not completely understood. In addition to natural processes, agricultural management practices can also effect the Si cycle. The most influential management factor on soil Si adsorption is fertilizer use and its effect on soil pH. The soil solution pH influences reactions of Si in soil, either causing adsorption in calcareous soils or maintaining Si in the solution phase as seen in more acidic soils (Brown and Mahler, 1987). Increased use of ammonium (NH⁴⁺) based nitrogen (N) and phosphorus (P) fertilizers during the last 70 years has affected the chemistry of surface soils in the Palouse region (northern Idaho and eastern

Washington). The nitrification of NH_4^+ has acidified the top 25 cm of the soil and therefore increased the levels of soluble Si available for plant uptake (Gollany et al., 2005).

The Si content of crops can be an important factor to consider for residue management due to the fact that the rate of decomposition appears to be affected by the Si content and other structural components (hemicellulose, cellulose, and lignin) of the straw. With the increased adoption of no-till or reduced tillage management practices in the Palouse region, slowly decomposing residue can become an issue. Excessive residue can slow planting, reduce the rate of soil warming in the spring, reduce soil to seed contact, hinder seed germination, and inhibit seedling emergence (Stubbs et al., 2009). While there is no single indicator of straw quality that can predict residue decomposition in the soil, the structural components of residue and total C and N have shown correlations with decomposition rates (Baggie et al., 2004; Goh and Tutuna, 2004). High hemicellulose has generally been linked to rapid decomposition while high lignin content, high C:N ratios and low total N are associated with slower decomposition (Stubbs et al., 2009). However, composition and decomposition rates vary with crop type and even among cultivars. Therefore, knowledge of how fertilizer application effects residue composition may aid in designing rotations that incorporate more rapidly decomposing crops and cultivars while maintaining economic feasibility. Consequently, the objectives of this study are to 1) determine the role and allocation of Si in both wheat and canola grown with varying fertilizer rates in a greenhouse environment and 2) determine how varying fertilizer rates influence hemicellulose, cellulose, and lignin content of both wheat and canola in order to infer potential decomposition rates.

MATERIALS AND METHODS

Two plants were used in the experiment: wheat (Triticum aestivum 'Louise'), and canola (Brassica napus). The plants were grown in one kg oven dried 50/50 mixture of soil acquired from Palouse Conservation Field Station (Palouse fine-silty, mixed, superactive, mesic pachic ultic haploxerol) and Sunshine Mix #2, with a background N content of 20 mg N kg⁻¹ (see Table 2.1). Labeled fertilizer (¹⁵NH₄)₂SO₄ was applied at four different rates: 6 (control), 60 (low), 180 (medium) and 420 (high) mg N kg⁻¹. Fertilization was split into three applications: emergence, tillering, and stem elongation. There were five replications per treatment for a total of ten pots per fertilizer rate, with a total of 40 pots. Plants were thinned after emergence to attain three plants per pot. The bottom of each pot was sealed in order to try and eliminate leaching and therefore loss of ¹⁵N. Pots were randomly distributed within the greenhouse, and re-randomized and watered every two to three days with a measured amount of distilled water as needed. Pot weights and water amounts were recorded in order to maintain consistent moisture within treatments. Senesced canola leaves were collected throughout the course of the experiment and kept in a room temperature oven until analysis. Once plants reached maturity, they were harvested and partitioned to separate grain, residue, roots, and soil components in addition to the previously collected canola leaves. The plant materials were dried at 45°C for 48 hours and ground to a fine powder using a roller grinder. Plant samples were analyzed for total C and N, atom %¹⁵N, fiber, and silicon content. Soil subsamples were stored in a 0°C freezer until extraction for total C and N, NH₄⁺, NO₃⁻, pH, water soluble Si (Si_{ws}), and amorphous Si (Si_{am}) content.

Plant analyses

Total C and N were determined through combustion using a Truspec Carbon and Nitrogen Analyzer (LECO). Plant ¹⁵N atom % was determined using a coupled elemental combustion system (Costech Instruments) and Thermo Finnigan delta plus Advantage stable isotope ratio mass spectrometer (Thermo Fisher Scientific). Fiber analysis was conducted using a modified version of the VanSoest et al. (1991) procedure using the ANKOM automated system utilizing filter bags (ANKOM Technology Corp., Fairport, N.Y.). This includes a series of extractions to determine the fiber content of a plant sample. The neutral detergent fiber (NDF) solution removes soluble cell contents such as carbohydrates, lipids, pectin, starch, soluble proteins, and non-protein nitrogen. The fraction that is left contains hemicellulose, proteins bound to the cell walls, cellulose, lignin, and other recalcitrant materials such as Si. The acid detergent fiber (ADF) process removes hemicellulose and bound proteins. The fraction left behind contains cellulose, lignin, and recalcitrant materials. The final acid detergent lignin (ADL) process removes cellulose, leaving only lignin and other recalcitrant materials. After the ADL procedure, samples are ashed using a muffle furnace in order to determine the amount of recalcitrant materials. Plant silica was extracted and analyzed using methods modified from Van der Vorm (1987). A 0.1 g subsample of residue was ashed in a muffle furnace at 500°C for six hours. Forty mL of 0.08 M H_2SO_4 was used to rinse ash into polyethylene centrifuge tubes and 1.6 mL of 48-51% HF was then added to each sample. Tubes were shaken for one hour and allowed to sit over night. The solution was then adjusted to 50 mL with 0.08 M H₂SO₄ and three mL of 2.5% boric acid was added to neutralize the remaining

HF. Silica amounts were then determined by the colorimetric method outlined by Van der Vorm (1987).

<u>Soil analyses</u>

Ammonium and NO_3^{-} were measured calorimetrically with a Quickchem 8000 Series FIA+ system and AutoSampler (Lachat Instruments). Soil pH was measured using a 1:1 soil to water ratio. For each sample 10 g of soil and 10 g of water were placed into a polyethylene container and shaken vigorously for 30 seconds. Samples were allowed to settle for 15 minutes then agitated before the pH meter was submerged into the soil slurry. Water soluble silicon extraction methods from Albrecht et al. (2005) were followed. Five g of soil and 25 mL of distilled water were placed in polyethylene tubes, shaken for 30 minutes, allowed to settle overnight, centrifuged for 10 minutes, and then filtered with Whatman No. 42 filter paper. Amorphous soil silica is operationally defined as the Si extracted by a Na_2CO_3 solution (Follett et al., 1965). This method is used due to the fact that the solubility of Si_{am} strongly increases at higher pH levels. One g of soil and 25 mL of 0.5 M sodium carbonate were combined in a polyethylene tube, shaken in an 80°C water bath for 10 minutes at 100 RPM's. Samples were then allowed to cool and settle at room temperature and centrifuged at 2,500 RPM's for 10 minutes. Extracted solution was filtered with Whatman No. 42 filter paper and stored in a cool environment until analysis. Both Si_{ws} and Si_{am} solutions were analyzed using colorimetric procedures outlined by Van der Vorm (1987). One mL of extract was pipetted into small polyethylene container and diluted to three mL with DI water. Samples extracted with sodium carbonate had one mL HCl added and a few drops of potassium permanganate to adjust the color from a slightly yellow solution to a clear solution. One mL of 0.5 M

H₂SO₄ was added and samples were agitated and incubated at 40.2°C for 20 minutes.

One mL of 5% ammonium molybdate was added, agitated, and left to sit for five minutes. One mL of both 5% oxalic acid and 1.5% ascorbic acid were also added. Solution level was adjusted to 10 mL with DI water and agitated. Samples were left to sit for 20 minutes and then read with a spectrophotometer set at an absorbance of 700 nm. If color was too dark samples were diluted 10 times in order to reach an attainable reading.

<u>Statistical analyses</u>

All results were analyzed using a two-way ANOVA in the SAS system. To establish differences between treatments the Tukey method was used with a p-value of 0.05. The two factors analyzed were fertilizer treatment and crop type. Plant organs were analyzed separately.

Calculations

%Ndff = (atom % ¹⁵N plant – 0.3665)/(atom % ¹⁵N excess in fertilizer)

Where atom % 15 N excess in fertilizer = 4.6

Reference background atom % $^{15}N = 0.3665$

Utilization% = $(atom \% ^{15}N excess in plant * Dry yield of specific plant organ * Plant)$

organ N)/ (atom % ¹⁵N excess in fertilizer * Fertilizer application rate) * 100

Recovery % = (Fertilizer N in soil, roots, residue, grain)/(Applied fertilizer N) *100

 $\text{\%NDF} = ((\text{NDF dry wt.} - \text{bag wt.})/\text{sample wt.} @105^{\circ}\text{C})*100$

 $\text{\%ADF} = ((\text{ADF dry wt.} - \text{bag wt.})/\text{sample wt.} @105^{\circ}\text{C})*100$

 $\text{ADL} = ((\text{ADL dry wt.} - \text{bag wt.})/\text{sample wt.} @105^{\circ}\text{C})*100$

%Ash = ((Ash dry wt. - bag wt.)/(sample wt. @105°C*sample moisture))*100

Soluble cell components = ((100 - % NDF)* total residue dry wt)/100

Hemicellulose = ((% NDF - % ADF)* total residue dry wt)/100

Cellulose = ((%ADF - %ADL)* total residue dry wt)/100

Lignin (recalcitrant components) = ((% ADL - % Ash)* total residue dry wt)/100

Ash (including silica) = (% Ash * total residue dry wt)/100

g Si/100 g = ((*(1/1000)*(50/sample wt)*(1/1000))*100)*(28.0855/60.0855)

Si g/pot = (g Si/100 g * total residue dry wt)/100

Parameter	Initial Value
pH	7.1
Total N (mg N/kg soil)	20
Siws g Si/kg soil	0.4
Si _{am} g Si/kg soil	13.8
Atom % ¹⁵ N	0.37

 Table 2.1. Initial greenhouse soil data

RESULTS

Yield response to N rates

Both plant type (p-value < 0.001) and fertilizer treatment (p-value < 0.0001) proved to be significant factors for straw, grain, root, and total yield (Table 2.2). Wheat straw increased from an average of 12.7 g/pot to 22.5 g/pot with increasing N rates while canola straw increased from 9.5 g/pot to 23.8 g/pot. Both wheat and canola grain yield increased with increasing fertilizer rates, however wheat grain yields were much higher than canola. The wheat grain yields ranged from an average of 6.1 g/pot in the control treatment to 17.6 g/pot in the high treatment while canola grain yields ranged from 2.8 g/pot in the control treatment to 11.1 g/pot in the high treatment. The canola leaves were collected separately and also showed an increase in yield with increasing fertilizer (2.1 g/pot – 5.9 g/pot). The roots showed a slight response to increasing fertilizer rates, but the relationship was not as severe as the residue and grain yields. The high fertilizer wheat treatment resulted in the most roots (5.3 g/pot) followed by the medium (4.2 g/pot), control (3.0 g/pot), and the low fertilizer treatments (2.3 g/pot). The high fertilizer canola treatment produced less roots compared to wheat with a value of 4.0 g/pot followed by the medium (3.1 g/pot), low (2.2 g/pot), and control (2.1 g/pot) fertilizer rates. Canola generated the most total residue compared to wheat under the medium and high N fertilizer rates. Total canola yield ranged from 16.5 g/pot in the control to 44.8 g/pot in the high fertilizer treatment while total wheat yield ranged from 21.8 g/pot in the control to 35.4 g/pot in the high treatment.

Total N

Total N for both wheat and canola positively responded to N rates (Table 2.3). Compared to canola, wheat straw had the highest amount of N in the control, low, and high fertilizer treatments. The total N amounts for the wheat straw ranged from 54 mg/pot in the low treatment to 128 mg/pot in the high treatment while the total N concentrations for canola straw ranged from 39 mg/pot in the control to 102 mg/pot in the high treatment. The grain total N increased with increasing fertilizer rates and was generally higher in the wheat treatments. The total N amounts for the wheat grain ranged from 68 mg/pot in the control to 291 mg/pot in the high fertilizer treatment whereas the canola ranged from 65 mg/pot in the control to 296 mg/pot in the high fertilizer treatment. The canola leaves showed the same trend of increasing total N with increasing fertilizer levels (22 to 51 mg N/pot). When taking into account the total above-ground biomass (straw, grain, and leaves), total N was greatest for canola at the low, medium, and high rates mostly due to the accumulation of N in its leaves (Figure 2.1). In terms of below-ground N, wheat consistently had more N accumulated in roots at each rate of

fertilizer. The wheat root total N ranged from 17 mg/pot at the low fertilizer rate to 45 mg/pot at the high fertilizer rate while canola only ranged from 13 mg N/pot at the low fertilizer rate to 31 mg N/pot at the high fertilizer rate. Total N in the soil after harvest was not significantly different between treatment, however, there was slightly less N in the wheat treatments (ranging from 1843 mg N/pot to 1908 mg N/pot) compared to the canola treatments (ranging from 1804 mg N/pot to 1913 mg N/pot). The greater biomass production accounted for the increased N accumulation in canola and wheat straw, thus resulting in increased C accumulation as well. As a result, increasing fertilizer N rates did not lead to significantly lower C:N ratios of wheat and canola straw (Table 2.4).

¹⁵N enrichment

Canola and wheat exhibited similar patterns for the % N derived from fertilizer (%Ndff) among the different plant organs (Table 2.5). As fertilizer levels increased so did the %Ndff in the straw. Wheat had higher levels of %Ndff (5.2 - 57.1%) than canola (3.2 - 56.6%) at all fertilizer rates. Canola leaves also showed an increase in %Ndff with increasing fertilizer levels and was even slightly higher than amounts found in the canola straw ranging from 4.0 - 61.9%. Therefore if the leaves and canola straw were combined as would be in an agricultural field the canola would have higher %Ndff values than wheat (Figure 2.2). The %Ndff in the grain was very similar when comparing wheat and canola at each fertilizer rate. Greater proportions of grain N originated from fertilizer at higher rates, which ranged from about 22% in the low N rate to 70% at the highest rate. Incorporation of fertilizer N into soil N was minimal and could have been masked by high levels of soil organic N.

The recovery of fertilizer in the system ranged from 73% to 111% with canola having slightly higher values than wheat (Table 2.6). The various fates of fertilizer N included straw, senesced canola leaves, grain, roots, and soil. Increasing the level of N fertilizer generally resulted in a higher utilization of fertilizer N in grain, leaves, and residue. Additionally, less fertilizer N was recovered in the soil at higher rates. The partitioning of fertilizer N was similar in canola and wheat plants. At the highest N rate, about half of the fertilizer N was utilized in the canola and wheat grain. More fertilizer N remained in the total canola residue (including leaves) than wheat, while wheat roots generally contained more fertilizer N than canola. Interestingly, more fertilizer N remained in the soil after canola than wheat at the medium and high rates, perhaps due to loss of fibrous canola roots or a lower uptake efficiency.

Plant Hemicellulose, Cellulose, Lignin, and Silicon

Only the residue (straw and canola leaves) of both crops was analyzed for fiber and silicon components. Without taking yield into account, the fiber concentrations did not vary among fertilizer treatments, but they did vary between crop type (Figure 2.3 and 2.4). However, when multiplying the concentrations by the yield, the fiber concentrations increased with increasing biomass (Table 2.7). Canola accumulated higher amounts of soluble components, cellulose, and lignin than wheat. In contrast, wheat accumulated more hemicellulose and Si than canola. The amounts of ash were very minute and similar across all treatments. Without considering yield, the Si concentrations seemed to decrease with increasing fertilizer levels from approximately 5.5% to 3.5% in wheat and from 1.9% to 1.5% in canola (Figure 2.5). However, when multiplying this concentration by the plant yield, Si levels increased with increasing fertilizer rates (Figure 2.6). Silicon

amounts were significantly higher in wheat residue ranging from 0.65 g/pot to 0.79 g/pot compared to the amounts found in canola ranging from 0.21 g/pot to 0.48 g/pot (Table 2.7). From these results, it seems as though plant type has the greatest significance in allocation of fiber and silicon components rather than fertilizer levels. However, any effect N levels may have on these factors is obscured due to the increased residue levels with increasing N rates.

Si Cycle

In order to better understand the Si cycle a Si budget was completed on the greenhouse samples. The initial soil Si_{am} level for all treatments was approximately 13.8 g Si_{am}/kg soil. Soil samples taken after harvest indicated that plant type was the most significant factor in the amount of Si left over in the soil (p-value <0.001) with more Si remaining in the soil in which canola was grown. This may be due to the fact that canola takes up less Si compared to wheat as seen in Tables 2.7 and 2.8. The increase in soil Si_{am} content (10.4 g/kg soil to 12.9 g/kg soil) with increasing fertilizer as seen in the canola treatments may be due to the acidifying effect the fertilizer has on the soil. The pH for the canola treatments ranged from 7.3 in the control to 6.9 in the high N rate. The wheat treatments, however, showed the opposite trend, the pH ranged from 7.0 in the control to 7.2 in the high fertilizer treatment. Although this range is slight, the post-harvest soil samples reflected this effect of pH in the Si_{am} results ranging from 9.7 g/kg soil in the control to 9.5 g/kg soil in the high fertilizer treatment. With increasing fertilizer and therefore increased yields the straw, root, and grain Si increased. Wheat accumulated significantly more Si in both the straw (0.65 g/pot to 0.79 g/pot) and the roots (0.41 g/pot to 0.98 g/pot) compared to canola straw (0.21 g/pot to 0.48 g/pot) and roots (0.09 g/pot to

0.27 g/pot), especially at the higher N rates. The amount of Si found in the grain of both crops was minimal. Taking the post-harvest soil, residue, roots, and grain Si content into account the difference seen in the last column of Table 2.8 ranges from 0.1 g/pot to 3.1 g/pot or 0.8-22.5% of Si unaccounted for.

Fertilizer		Y	ields (g/pot)		
mg N/kg	Straw	Leaves	Grain	Roots	Total
		Wh	eat		
6	12.7 c		6.1 c	3.0 bdc	21.8 d
60	13.1 c		6.1 c	2.3 dc	21.5 d
180	17.6 b		12.4 b	4.2 ba	34.2 b
420	22.5 a		17.6 a	5.3 a	35.4 b
		Can	ola		
6	9.5 d	2.1 c	2.8 d	2.1 d	16.5 e
60	11.6 dc	3.4 c	3.4 d	2.2 d	20.6 d
180	17.6 b	4.4 b	6.3 c	3.1 bdc	31.4 cb
420	23.8 a	5.9 a	11.1 b	4.0 bac	44.8 a

Table 2.2. Average yield per pot for each N fertilizer rate

Table 2.3. Average tot	al N per pot for	each N fertilizer rate
------------------------	------------------	------------------------

Fertilizer		Total N (mg/pot)										
mg N/kg	Straw	Leaves	Grain	Roots	Soil							
		Whe	at									
6	61 bc		68 e	19 cb	1844 a							
60	54 c		78 ed	17 c	1908 a							
180	63 bc		168 b	31 b	1860 a							
420	128 a		291 a	45 a	1843 a							
		Cano	ola									
6	39 d	22 c	65 e	14 c	1911 a							
60	50 c	26 cb	84 c	13 c	1804 a							
180	69 b	33 b	154 b	20 cb	1913 a							
420	102 a	51 a	296 a	31 b	1902 a							

Fertilizer mg N/kg	g C/pot	g N/pot	C:N
	Whe	eat	
6	5.9 dc	0.06 bc	98.7 a
60	6.1 dc	0.05 c	113.1 a
180	6.2 dc	0.08 bc	80.8 a
420	10.6 b	0.13 bac	83.2 a
	Can	ola	
6	5.4 dc	0.06 c	89.4 a
60	6.7 c	0.07 bc	98.5 a
180	10.1 b	0.10 bc	100.1 a
420	13.7 a	0.15 ba	89.9 a

Table 2.4. Average residue C, N, and C:N per pot for each N fertilizer rate

Table 2.5. Average percent of N derived from fertilizer (%Ndff)

Fertilizer		%Ndff								
mg N/kg	Straw	Leaves	Grain	Roots	Soil					
		W	heat							
6	5.2 fe		3.8 f	2.4 gf	0.7 f					
60	18.2 c		21.8 e	13.8 def	2.8 d					
180	39.1 b		48.0 c	24.4 dc	3.6 cd					
420	57.1 a		70.0 a	40.3 ba	5.6 b					
		Ca	nola							
6	3.2 f	4.0 d	3.5 f	4.1 gf	1.1 fe					
60	16.3 dc	17.5 c	24.8 e	16.1 de	2.1 edf					
180	36.0 b	39.5 b	54.6 cb	28.8 bc	4.9 cb					
420	56.6 a	61.9 a	70.5 a	43.6 a	7.6 a					

Fertilizer		Utilization %						
mg N/pot	Residue	Leaves	Grain	Roots	Soil	Total		
			Wheat					
6	12.4 ba		10.1 cd	1.7 a	48.2 bdc	72.3 b		
60	12.3 ba		21.1 cb	2.7 a	66.7 ba	102.7 a		
180	12.3 ba		40.4 a	3.7 a	33.9 ed	90.3 ba		
420	16.7 a		46.4 a	4.1 a	23.5 e	90.7 ba		
			Canola					
6	5.0 b	3.4 b	8.7 d	3.1 a	80.8 a	101.0 ba		
60	10.2 ba	5.5 b	25.8 b	2.9 a	48.0 bdc	92.4 ba		
180	12.5 ba	6.5 b	42.2 a	3.0 a	47.2 bedc	111.3 a		
420	13.2 ba	7.3 a	47.4 a	3.0 a	32.7 ed	103.5 a		

Table 2.6. Utilization of fertilizer N by plant organs and recovery efficiencies

Fertilizer	Soluble	Hemicellulose	Cellulose	Lignin	Ash	Si	Total
mg N/kg	(g/pot)	(g/pot)	(g/pot)	(g/pot)	(g/pot)	(g/pot)	(g/pot)
			Wheat				
6	3.4 f	3.5 dc	4.3 edf	1.5 b	0.02 a	0.65 ba	13.4 de
60	3.5 fe	3.7 c	4.6 ed	1.2 b	0.01 a	0.66 ba	13.7 de
180	4.1 fed	5.0 b	7.3 cb	1.3 b	0.01 a	0.71 ba	18.4 c
420	5.0 ced	6.4 a	9.2 b	1.8 b	0.02 a	0.79 a	23.2 b
		Canola	(Shoots + L	leaves)			
6	4.7 cfed	1.5 fe	4.8 ed	1.5 b	0.02 a	0.23 de	12.8 de
60	5.8 cb	1.9 e	5.6 cd	1.7 b	0.02 a	0.21 de	15.2 dc
180	7.0 b	2.9 d	8.9 b	3.1 a	0.02 a	0.34 dc	22.3 b
420	10.0 a	3.9 c	12.2 a	3.6 a	0.02 a	0.48 bc	30.2 a

Table 2.7. Average soluble components, hemicellulose, cellulose, lignin, ash, and Si in wheat and canola straw

Table 2.8. Average amounts of Si found in the initial soil, post-harvest soil, plant residue, roots, grain, and the difference or unaccounted for Si

Fertilizer	pН	Initial Soil	Post-Harvest	Straw Si	Root Si	Grain Si	Difference
mg N/kg		Si (g/kg)	Soil Si (g/kg)	(g/pot)	(g/pot)	(g/pot)	(g/pot)
			Wheat				
6	6.96	13.8	9.7 c	0.65 ba	0.41 cb	0.010 a	3.0 ab
60	7.06	13.8	10.0 bc	0.66 ba	0.47 b	0.011 a	2.7 ab
180	7.14	13.8	9.6 c	0.71 ba	0.95 a	0.012 a	2.5 ab
420	7.22	13.8	9.5 dc	0.79 a	0.98 a	0.023 a	2.5 ab
			Canola				
6	7.34	13.8	10.4 bc	0.23 de	0.09 cb	0.005 a	3.1 ab
60	7.26	13.8	10.9 bac	0.21 de	0.13 cb	0.004 a	2.6 ab
180	7.24	13.8	12.2 ba	0.34 dc	0.26 cb	0.007 a	1.0 b
420	6.86	13.8	12.9 a	0.48 bc	0.27 cb	0.023 a	0.1 b

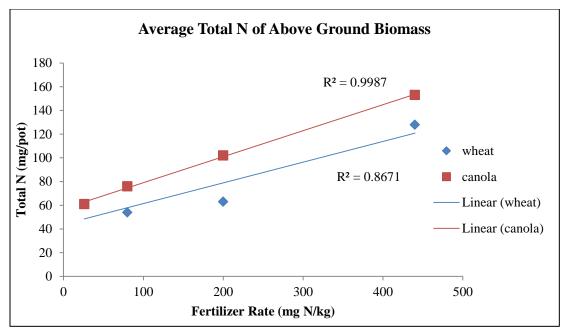


Figure 2.1. Average total N of above ground biomass per fertilizer treatment for wheat and canola

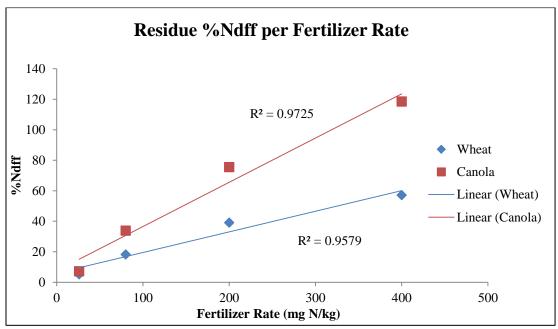


Figure 2.2. Average % nitrogen derived from fertilizer (Ndff) for wheat and canola (straw + leaves) residue per fertilizer rate (mg N/kg)

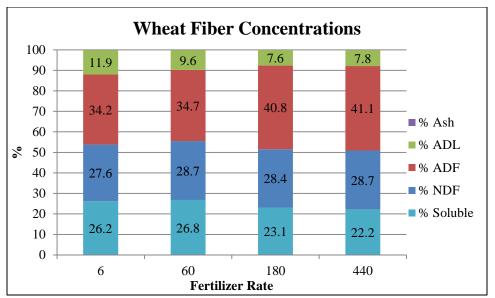


Figure 2.3. Fiber concentrations in wheat residue per fertilizer treatment

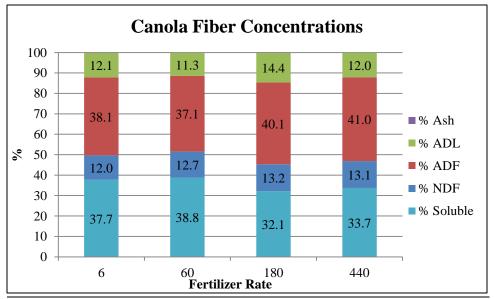


Figure 2.4. Fiber concentrations in canola residue (straw+leaves) per fertilizer treatment

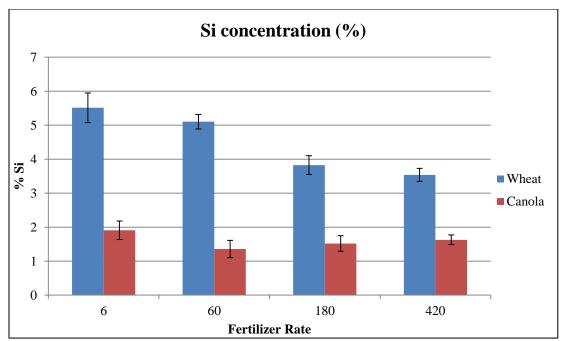


Figure 2.5. Total Si concentration (%) for wheat and canola at each fertilizer rate

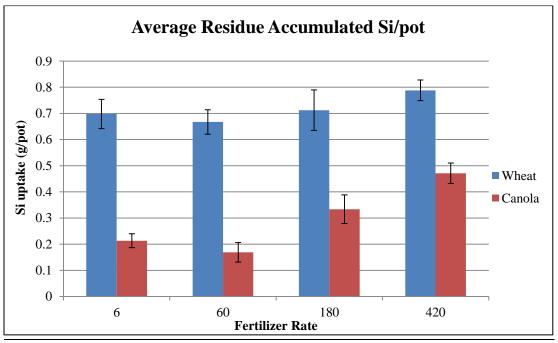


Figure 2.6. Average residue accumulation of Si/pot by treatment and crop type (%Si * yield)

DISCUSSION

As expected, crop yields, %Ndff, %N utilization, %N recovered, Total N and C generally increased with increasing fertilizer rates as seen in multiple other field and greenhouse studies (Hocking et al., 1997a; Hocking et al., 1997b; Cheema et al., 2001; Hocking et al., 2002; Malhi et al. 2006; Malhi and Lemke, 2007; Gan et al., 2010; Gombert et al., 2010). Increased yields and therefore increased leaf area with higher N rates resulted in a higher rate of photosynthesis causing an increase in total C and therefore no significant differences in C:N ratios were found between fertilizer rates. The results from the chemical composition analyses indicated that crop type is the distinguishing factor for fiber and silica allocation and accumulation due to physiological differences. When just considering concentrations of NDF, ADF, and ADL no significant differences were seen between fertilizer treatments suggesting that fertilizer rates did not have an effect on fiber concentrations. However, when the increasing yields with increasing fertilizer rates are taken into account the accumulation of these components does increase. These results are consistent with multiple studies exploring the effects of N rates on crop fiber content (Keady et al., 2000; Lemus et al., 2008; Guretzky et al., 2011). Si concentrations without factoring in yield appeared to decrease in both crops with increasing fertilizer rate. Early studies have obtained similar results (Lawes and Gilbert, 1884; Jones and Handreck, 1967) and determined that this could be a result of the dilution factor which is defined by Jarrell and Beverly (1981) as when the relative rate of dry matter accumulation increases more rapidly than the rate of nutrient accumulation, resulting in lower final concentrations in treated plants. An inverse relationship between silicon and lignin levels can be seen in these results suggesting that canola relies

primarily on lignin while wheat relies on Si for structural support. From this experiment, it seems that fertilizer levels did not have a significant effect on C:N, fiber, or Si levels, and therefore may not affect decomposition rates. Crop type consistently had the biggest effect on such factors. Future studies might include using such residue in incubations or decomposition studies in order to determine how the different crop types may affect fiber and Si release into the soil.

The Si mass balance showed that wheat accumulated more Si in both the residue and roots compared to canola resulting in less post-harvest Si_{am} levels in the soil. This is primarily due to the wheat roots ability to take up Si from the soil (Ma and Yamaji, 2006). For both crops, the most unaccounted Si was found in the control and low N rates suggesting that due to the lower yields the total Si uptake was also lower and therefore may have been lost from the system. Although the greenhouse experiment was considered a closed system due to taping the bottom of the pots, it is possible that some of the unaccounted Si was leached out of the soil. Another possibility is that some of the Si was transformed into a less labile or less soluble form.

CONCLUSION

In this experiment, it is clear that the characteristics of residue production varied among crops. Both crops produced more biomass with increasing fertilizer, but overall canola produced a higher quantity of residue than wheat. In terms of N accumulation, canola had a higher amount than wheat when considering both leaves and straw. However, neither fertilizer level nor crop type proved to establish significantly different C:N ratios. The recovery of fertilizer N in wheat biomass was greater than canola. Therefore, wheat was more efficient in taking up N from fertilizer, and produced more

grain per unit of N. The fiber analysis showed that the biochemistry differed between wheat and canola, but not significantly with fertilizer rate and did not show any evidence of a dilution effect. Crop type had the most significant effect on Si levels and an inverse relationship between ADL and Si was recognized suggesting that plants with lower Si content and high lignin may rely more on lignin for strength and protection. When looking at Si concentrations among the different fertilizer rates, a decrease with increasing fertilizer rates was shown. However, when taking into account the yield it is evident that this pattern was due to a dilution effect. These results suggest that fertilizer rate does not have a significant effect on fiber or Si accumulation.

The Si cycle analyses showed that the higher fertilizer treatments utilized the most Si. Wheat accumulated the most Si in both the roots and straw therefore leaving less Si_{am} remaining in the soil post-harvest. Higher amounts of unaccounted Si occurred at the lower fertilizer rates. Due to the lower yields and therefore lower Si requirements at these N levels the unaccounted amounts of Si could have been leached from the system. These results suggest that crop type does in fact influence fiber and Si uptake regardless of fertilizer level and therefore crop type should be an important consideration in systems wary of decomposition rates, fiber and Si release into the soil.

LITERATURE CITED

- Albrecht, S.L., H.T. Gollany, R.R. Allmaras, C.L. Douglas, and S.M. Copeland. 2005.Tillage and nitrogen fertilizer influence on carbon and soluble silica relations in a Pacific Northwest mollisol. Soil Sci. Soc. Am. J. 69(4):1102-1109.
- Baggie, I., D.L. Rowell, J.S. Robinson, and G.P. Warren. 2004. Decomposition and phosphorus release from organic residues as affected by residue quality and added inorganic phosphorus. Agrofor. Syst. 63:125-131.
- Brown, T.H. and R.L. Mahler. 1987. Effects of phosphorus and acidity on levels of silica extracted from a Palouse silt loam. Soil Sci. Soc. Am. J. 51(3):674-677.
- Cheema, M.A., M.A. Malik, A. Hussain, S.H. Shah, and S.M.A. Basra. 2001. Effects of time and rate of nitrogen and phosphorus application on the growth and the seed and oil yields of canola (*Brassica napus* L.). J. Agronomy and Crop Science 186:103-110.
- Epstein, E., and W.J. Grant. 1967. Soil losses and crust formation as related to some soil physical properties. Soil Sci. Soc. Am. J. 31(4):547-550.
- Epstein, E. 1994. The anomaly of silicon in plant biology. Proc. Natl. Acad. Sci. USA. 91(1):11-17.
- Epstein, E. 1999. Silicon. Annu. Rev. Plant Bio. 50(1):641-664.
- Epstein, E. 2009. Silicon: its manifold roles in plants. Ann. Appl. Biol. 155(2):155-160.
- Gan, Y., C.A. Campbell, H.H. Janzen, R.L. Lemke, P. Basnyat, and C.L. McDonald.
 2010. Nitrogen accumulation in plant tissues and roots and N mineralization under oilseeds, pulses, and spring wheat. Plant and Soil 332:451-461.

- Goh, K.M., and S.S. Tutuna. 2004. Effects of organic and plant residue quality and orchard management practices on decomposition rates of residues. Commun. Soil Sci. Plant Anal. 35:441-460.
- Gollany H.T., R.R. Allmaras, S.M. Copeland, S.L. Albrecht, and C.L. Douglas. 2005.Tillage and nitrogen fertilizer influence on carbon and soluble silica relations in aPacific Northwest mollisol. Soil Sci. Soc. Am. J. 69(4):1102-1109.
- Gombert, J., F. Le Dily, J. Lothier, P. Etienne, L. Rossato, J.M. Allirand, A. Jullien, A. Savin, and A. Ourry. 2010. Effect of nitrogen fertilization on nitrogen dynamics in oilseed rape using 15N-labeling field experiment. J. Plant Nutr. Soil Sci. 173(6):875-884.
- Guretzky, J.A., J.T. Biermacher, B.J. Cook, M.K. Kering, and J. Mosali. 2011. Switchgrass for forage and bioenergy: harvest and nitrogen rate effects on biomass yields and nutrient composition. Plant and Soil 339:69-81.
- Hans Wedepohl, K. 1995. The composition of the continental crust. Geochimica Et Cosmochimica Acta. 59(7):1217-1232.
- Hocking, P.L., J.A. Kirkegaard, J.F. Angus, A.H. Gibson, and E.A. Koetz. 1997a.
 Comparison of canola, Indian mustard, and Linola in two contrasting environments: I. effects of nitrogen fertilizer on dry matter production, seed yield and seed quality. Field Crop Research 49(2):107-125.
- Hocking, P.J., P.J. Randall, and D. DeMarco. 1997b. The response of dryland canola to nitrogen fertilizer: partitioning and mobilization of dry matter and nitrogen, and nitrogen effects on yield components. Field Crop Research 54:201-220.

Hocking, P.L., J.A. Kirkegaard, J.F. Angus, A. Bernardi, and L.M. Mason. 2002.
Comparison of canola, Indian mustard and Linola in two contrasting environments: III. Effects of nitrogen fertilizer on nitrogen uptake by plants and on soil nitrogen extraction. Field Crop Research 79(2):153-172.

- Jarrell, W.M., and R.B. Beverly. 1981. The dilution effect in plant nutrition studies. Adv. Agron. 34:197-222.
- Jones, L.H.P., and K.A. Handreck. 1967. Silica in soils, plants, and animals. Advances in Agronomy 19:107-145.
- Keady, T.W.J., C.S. Mayne, and D.A. Fitzpatrick. 2000. Prediction of silage feeding value from the analysis of the herbage at ensiling and effects of nitrogen fertilizer, date of harvest and additive treatment on grass silage composition. Journal of Agricultural Science 134:353-368.
- Lawes, J.B., and J.H. Gilbert. 1884. XLIII On the composition of the ash of wheat grain, and wheat straw, grown at Rothamsted, in different seasons, and by different manures. J. Chem. Soc., Trans. 45:305-407.
- Lemus, R., E.C. Brummer, C.L. Burras, K.J. Moore, M.F. Barker, and N.E. Molstad. 2008. Effects of nitrogen fertilization on biomass yield and quality in large fields of established switchgrass in southern Iowa, USA. Biomass and Bioenergy 32:1187-1194.
- Ma, J.F., and N.Yamaji. 2006. Silicon uptake and accumulation in higher plants. Trends in Plant Science 8(11):392-397.

- Malhi, S.S., R. Lemke, Z.H. Wang, and B.S. Chhabra. 2006. Tillage, nitrogen and crop residue effects on crop yield, nutrient uptake, soil quality, and greenhouse gas emissions. Soil and Tillage Research 90:171-183.
- Malhi, S.S., and R. Lemke. 2007. Tillage, crop residue and N fertilizer effects on crop yield, nutrient uptake, soil quality and nitrous oxide gas emissions in a second 4-yr rotation cycle. Soil and Tillage Research 96:269-283.
- Stubbs, T.L., A.C. Kennedy, P.E. Reisenauer, and J.W. Burns. 2009. Chemical composition of residue from cereal crops and cultivars in dryland ecosystems. Agron. J. 101:538-545.
- Van der Vorm, P.D.J., 1987. Dry ashing of plant-material and dissolution of the ash in HF for the colorimetric determination of silicon. Commun. Soil Sci. Plant Anal. 18(11):1181-1189.
- VanSoest, P.J., J.B. Robertson, and B.A. Lewis. 1991. Methods for dietary fiber, neutral detergent fiber, and nonstarch polysaccharides in relation to animal nutrition. J. Dairy Sci. 74:3583-3597.

CHAPTER 3

EFFECTS OF WHEAT AND CANOLA RESIDUE ON SOIL CRUSTING AND SILICON LEVELS

SUMMARY

Depending on the type of residue, large amounts of Si can be cycled through the plant and back into the soil. In the Palouse region (northern Idaho and eastern Washington) the dominant crop is wheat, which can have large amounts of Si compared to other crops such as canola (Casey et al., 2004). When crop residues are incorporated into the soil, the ultimate concentration of Si in soil solution is expected to depend on the rate of decomposition, dissolution rate and extent of phytolith-Si, adsorption of Si by the soil and other environmental factors (Wickramasinghe and Rowell, 2005). Once in solution or suspension, Si compounds are subject to leaching within the soil profile and possible deposition as potential cementing and blocking agents resulting in surface crusting (Brown and Mahler, 1987). In order to apprehend the relationship between the decomposition of Si from wheat and canola, a laboratory incubation and decomposition study was initiated. The laboratory incubation consisted of 100 g of Ritzville silt loam and 0.8 g of wheat or canola residue. Half of the samples received a surface application of 200 mg N/kg of soil. The incubation lasted 20 weeks and was kept at room temperature $(25^{\circ}C)$. The results from this incubation showed that pH rather than crop type was the dominant factor influencing the soil Si levels, crust thickness, and surface resistance. Samples that received fertilizer applications had lower pH, less soil Si_{am}, Si_{ws}, lower surface resistance, and reduced crust thickness. The decomposition study utilized six different residue types: field grown wheat fertilized 0, 50, and 100 lbs N/ac, field

grown canola fertilized at 0 lbs N/ac, and wheat and canola grown in a greenhouse fertilized with 440 mg ¹⁵N/kg. Hoagland's solution was applied and moisture was maintained within the storage container in order to enhance microbial activity and residue decomposition over a 12 week period. These results showed that residue weight decreased over time while %Si increased. The most dramatic differences occurred between weeks 0 and 8 when more labile components were decomposed leaving less labile Si and more recalcitrant organic materials behind for a much slower decomposition rate.

INTRODUCTION

Crop residue management may have an important impact on silicon (Si) solubility and movement. Depending on the type of residue, large amounts of Si can be cycled through the plant and back into the soil. In the Palouse region (northern Idaho and eastern Washington) the dominant crop is wheat, which can have large amounts of Si compared to other crops such as canola (Casey et al., 2004). The dissolution of straw Si depends not only on the purity of the Si, but also on the degree of exposure of the phytoliths. Surface dumbbell cells and protuberances are more exposed than granular amorphous Si (Si_{am}) enclosed in the plant matrix and are likely the source of the readily soluble fraction when straw is placed in solution or in the soil (Ma and Yamaji, 2006). Decomposition of the straw will in the long term expose more Si because phytoliths will be released from the plant matrix and dissolved. However, the presence of Si in the plant matrix appears to increase resistance to decomposition (Richmond and Sussman, 2003). When crop residues are incorporated into the soil, the ultimate concentration of Si in the soil solution is expected to depend on the rate of residue decomposition the rate and extent of

phytolith dissolution, adsorption of Si by the soil, and environmental factors such as temperature, moisture, and soil properties (Wickramasinghe and Rowell, 2005). One of the most important factors affecting the adsorption of Si is the soil pH. At higher pH levels adsorption occurs while at more acidic pH levels the Si is released into solution. Within the Palouse region, soil pH within the top 25 cm of the soil is primarily affected by fertilizer type and application rate (Gollany et al., 2006). Once in solution, Si compounds are subject to leaching within the soil profile or surface deposition as cementing and blocking agents resulting in surface crusting (Brown and Mahler, 1987).

Surface crusting is a common occurrence in many cultivated soils in arid and semi-arid regions. Important effects of a soil crust on surface and other phenomena include: reduction of infiltration rate (IR) (Dao, 1993; Zuzel and Pikul, 1994; Ben-Hur et al., 2004), enhancement of runoff (Clymans et al., 2011; Cornelis et al., 2011), alteration of erosion (Remley and Bradford, 1989; Feng et al. 2011), and interference with seed germination (Schillinger, 2011). In order to decrease the occurrence of surface crusting and therefore Si levels in the soil solution it may be beneficial to consider the addition of crops with lower Si levels, such as canola, into a cropping rotation. Therefore the objective of this research is to evaluate the decomposition and release of Si into the soil from wheat and canola residue and the effects on soil crusting through a laboratory incubation and a decomposition study.

MATERIALS AND METHODS

Laboratory Incubation

In order to determine the relationship between the release of Si from wheat and canola, an incubation was initiated for a period of 20 weeks. Each sample contained 100

g of Ritzville series silt loam acquired from Ralston, WA (See table 3.1). Soil was air dried and sieved through a two mm sieve prior to incubation. The wheat and canola residue was grown with 200 mg labeled ¹⁵N fertilizer per kg of soil in a greenhouse study. The residue was cut into small pieces ranging from two to five cm and 0.8 g was added to each 100 g soil sample and thoroughly mixed. A total of 120 samples of wheat and 120 samples of canola were created following this procedure. Half of the samples from each residue type received 200 mg N/kg of soil for a total of four treatments: canola with no fertilizer (CNF), canola with fertilizer (CF), wheat with no fertilizer (WNF), and wheat with fertilizer (WF). Samples were placed in polyethylene containers, arranged in a completely randomized design and stored in a room temperature oven (25° C). Every three days samples were brought up to field capacity ($\sim 30\%$) in order to simulate a wetting and drying cycle. Prior to watering, each sample was weighed to determine the amount of water loss between wetting cycles and to ensure samples were all receiving approximately the same amount of water. Destructive sampling occurred every two weeks and analyses included: pH, total C & N, ¹⁵N, soil water soluble Si (Si_{ws}), soil amorphous Si (Si_{am}), surface strength, crust thickness, and residue Si content. During the destructive sampling process, soil and residues were separated using a two mm sieve.

Decomposition Study

A 12-week soil incubation with six different residue treatments was conducted under laboratory conditions. An autoclaved, acid washed, coarse grain sand was used instead of soil. A subsample of 15 g was weighed into 50 ml polyethylene centrifuge tubes prior to amendment with residue. There were six residue treatments and one soil control, in quadruplicate for each week of sampling. Four of the residues were collected

from previous field fertility studies: spring wheat fertilized with 0 lbs N/ac, 50 lbs N/ac, and 100 lbs N/ac from Spillman Agronomy Farm in Pullman, WA, harvested in 2011, and spring canola fertilized with 0 lb N/ac from the Palouse Conservation Field Station (PCFS) in Pullman, WA, harvested in 2010. Two residue treatments were collected from a greenhouse study of wheat and canola fertilized with 440 mg ¹⁵N/kg. Residues were steamed prior to the addition to sand at a rate of 1:10 residue/sand mixture. To obtain a microbial inoculum containing a large number of different microorganisms with a wide range of decomposer abilities and land management histories, a mixture of four eastern Washington soil series were used including: Prosser, Ritzville, Broadax, and Palouse, each contributing equally to the mix. After incubation of the mixture for ten days at 50% water holding capacity, a soil:water suspension was prepared following the procedures of Marschner et al. (2010). The residues were inoculated with a total of 2.5 mL of microbial solution. Soils were extracted with a ratio of one g of soil with 10 ml of deionized water, and allowed to settle overnight. An additional 2.5 ml of a modified Hoagland's solution was added to each sample (providing 210 ppm N). The tubes were then placed in a sealed plastic container with an open water source to maintain constant moisture and prevent drying at 20°C. Tubes were destructively sampled for residue weight loss and Si levels after 0, 8, and 12 weeks. During the destructive sampling process, soil and residues were separated using a two mm sieve.

<u>Laboratory analyses</u>

Soil solution pH was represented by a 1:1 soil to water extract. For each sampling period 10 g of soil and 10 g of water were placed into a polyethylene container and shaken vigorously for 30 seconds. Samples were allowed to settle for 15 minutes then

agitated before the pH meter was submerged into the soil slurry. Total C and total N were determined through combustion using a Truspec Carbon and Nitrogen Analyzer (LECO). ¹⁵N atom % was determined using a coupled elemental combustion system (Costech Instruments) and Thermo Finnigan delta plus Advantage stable isotope ratio (Thermo Fisher Scientific).

Water soluble silicon extraction methods from Albrecht et al. (2005) were followed. Five g of soil and 25 mL of distilled water were placed in polyethylene tubes, shaken for 30 minutes, allowed to settle overnight, centrifuged for 10 minutes, and then filtered with Whatman No. 42 filter paper. Amorphous soil silica is operationally defined as the Si extracted by a Na_2CO_3 solution (Follett et al., 1965). This method is used due to the fact that the solubility of Si_{am} strongly increases at higher pH levels. One g of soil and 25 mL of 0.5 M sodium carbonate were combined in a polyethylene tube, shaken in an 80°C water bath for 10 minutes at 100 RPM. Samples were then allowed to cool and settle at room temperature and centrifuged at 2,500 RPM's for 10 minutes. Extracted solution was filtered with Whatman No. 42 filter paper and stored in a cool environment until analysis. Both Si_{ws} and Si_{am} solutions were analyzed using colorimetric procedures outlined by Van der Vorm (1987). One mL of extraction was pipetted into small polyethylene container and diluted to three mL with DI water. Samples extracted with sodium carbonate had one mL HCl added and a few drops of potassium permanganate to adjust the color from a slightly yellow solution to a clear solution. One mL of 0.5 M H_2SO_4 was added then samples were agitated and incubated at 40.2°C for 20 minutes. One mL of 5% ammonium molybdate was added, agitated, and left to sit for five minutes. One mL of both 5% oxalic acid and 1.5% ascorbic acid were also added. Solution level

was adjusted to 10 mL with DI water and agitated. Samples were left to sit for 20 minutes and then read with a spectrophotometer set at an absorbance of 700 nm. If color was too dark samples were diluted 10 times in order to reach an attainable reading.

In order to determine surface strength a Humboldt MFG. CO. pocket penetrometer was used. Measurements with the penetrometer always occurred at the end of each wetting and drying cycle in order to maintain consistency between measurements. Crust thickness was measured in mm using a caliper.

Residues were extracted and analyzed for Si using methods modified from Van der Vorm (1987). A 0.1 g subsample of residue was ashed in a muffle furnace at 500°C for six hours. Forty mL of 0.08 M H₂SO₄ was used to rinse ash into plastic tubes and 1.6 mL of 48-51% HF was then added to each sample. Tubes were shaken for one hour and allowed to set over night. The solution was then adjusted to 50 mL with 0.08 M H₂SO₄. Three mL of 2.5% boric acid was then added to neutralize remaining HF. Silica amounts were then determined by the colorimetric method outlined by Van der Vorm (1987). *Calculations*

 $\%^{15}$ Ndfp ($\%^{15}$ N in the soil derived from labeled plant residue) = (15 N atom% treated soil

-0.37) / ¹⁵N atom% plant residue - 0.37)

Value 0.37 is a standard value for natural ¹⁵N abundance in soil

Soil Si = Si ppm * (mL of extractant/g of soil) * (1 L/1000 mL) * (1000 g/1 kg) * 10 (if

diluted) = g of Si/kg of soil

Statistical analyses

Data was analyzed by the PROC GLM procedure in SAS 9.3 at a 95% confidence interval using Tukey's method of comparison. Three factors were used in the statistical analyses: crop type, fertilizer treatment, and time.

Table 3.1. Initial soil data

Soil Parameter	Initial Value
% Sand	18.6
% Silt	67.8
% Clay	13.6
% Moisture	16.5
рН	5.5
Total C %	0.99
Total N %	0.09
NO ₃ mg/kg	8.0
NH_4^+ mg/kg	1.0
Water soluble Si (mg/kg soil)	5.0
Amorphous Si (mg/kg soil)	1852
Bulk density (g/cm ³)	1.4

Table 3.2. Initial residue analysis

Residue	% ¹⁵ N	%NDF	%ADF	%ADL	%Ash	%Si	C:N
Wheat	2.2	28.4	40.8	7.6	0.07	0.38	80.7
Canola	2.0	13.2	40.1	14.4	0.10	0.15	100.1

RESULTS

Laboratory Incubation

Water Loss

Water loss stayed fairly consistent amongst treatments for each watering cycle

and showed no significant differences over time (see Figure 3.1).

The application of fertilizer was the determinant of soil pH (p-value <0.0001). On average, the application of fertilizer caused the soil pH to drop to approximately 4.6 while the treatments without added N had an average pH of 5.5. The WNF treatment had the highest average pH of 5.6 followed by CNF 5.5, CF 4.6, and WF 4.5 (Table 3.3). The effects of fertilizer application were rapid and pH levels were quite stable throughout the experiment.

Total C&N

The total soil C did not show any significant effects with crop type, fertilizer treatment, or time. Soil total N only showed significant differences with fertilizer treatment (p-value <0.0001). Fertilizer application for both wheat and canola residues resulted in higher amounts of soil N (0.099%) compared to control samples, which did not receive any fertilizer (0.085%). This effect on soil N also caused the soil C:N ratios to decrease in samples that had fertilizer application (see Table 3.3). Plant total C and total N were primarily affected by crop. Canola residue consistently had the most total C throughout the incubation with a value of 26.91% C compared to the wheat residue value of 22.81% C. The canola residue consistently had more total N (0.77%) compared to the wheat residue (0.70%). Taking these total C and N values into account the average C:N ratios after the initial two week period can be evaluated. Plant C:N ratio was significantly affected by crop type (p-value = 0.0085). The CNF had the highest C:N after two weeks with a value of 38.71 followed by CF 34.57, WNF 33.15, and WF 32.80 (see Table 3.3). When considering the initial residue C:N with canola at 100.1 and wheat with a value of

80.7 these ratios were almost halved by week two. This suggests that during this two week period, significant mineralization may have occurred.

^{%15}Ndfp

Over time, the level of N^{15} in the soil was expected to increase as the labeled plant residue was decomposed in the soil. However, the only significant difference was seen between crop types primarily due to different starting values of ¹⁵N as seen in Table 3.2. Throughout the entire incubation regardless of fertilizer application, wheat residue consistently contributed ~30% more ¹⁵N to the soil compared to canola. The WNF soil had the highest %¹⁵Ndfp (0.20%) followed by WF 0.19%, CF 0.14%, and CNF 0.14% (Table 3.3 & Figure 3.4).

Soil Siws & Siam

Water soluble Si showed the biggest differences not between crop type, but between fertilizer treatments (p-value <0.0001). Throughout the course of the incubation, the WNF treatment had the highest average amount of Si_{ws} (0.86 g Si/kg soil) followed by CNF (0.81 g Si/kg soil), CF (0.59 g Si/kg soil), and WF (0.55 g Si/kg soil) (Table 3.3). In addition to the differences seen between treatments, time also showed significance (pvalue >0.0001). From week two to week six the values were fairly consistent. However, starting at week eight and lasting until week fourteen a peak in all treatments values for Si_{ws} was seen (see Figure 3.5). Week sixteen through twenty the values decreased back to values similar to those seen in the beginning of the incubation.

The fertilizer treatments had the most significant effect on Si_{am} (p-value = 0.04) as seen in the Si_{ws} results. Throughout the course of the incubation, the WNF treatment had the highest average amount of Si_{am} (37.6 g Si/kg soil) followed by CNF (37.2 g Si/kg

soil), WF (31.6 g Si/kg soil), and CF (28.8 g Si/kg soil) (Table 3.3). These results suggest that with higher pH more Si_{ws} and Si_{am} is being adsorbed to the soil particles within the upper few mm of the soil. Time also showed significance (p-value >0.001). Weeks two through six were variable however, a peak in Si_{am} levels starting from week six lasting until week twelve was seen (see Figure 3.6). From week twelve to the end of the experiment the Si_{am} values stayed fairly consistent between treatments.

Surface Strength

The biggest factor in determining surface strength, and therefore the strength or presence of the crust, was the application of fertilizer (p-value <0.0001). The WNF treatment consistently showed the strongest surface strength throughout the course of the incubation with an average value of 62.7 g/cm² followed by CNF 51.0 g/cm², CF 42.5 g/cm², and WF 34.0 g/cm² (Table 3.3). Although treatments had a significant effect on surface strength, an increase in strength over time was not seen (see Figure 3.7).

Crust Thickness

Fertilizer treatments had the most significant effect on crust thickness (p-value <0.0001). The overall average by treatment showed WNF had the thickest crust 23.1 mm followed by CNF 22.0 mm, WF 19.0 mm, and CF 18.7 mm (Table 3.3). Over time, the crust thickness for all treatments steadily increased from approximately 16.5 mm to 24.7 mm (p-value <0.0001)(see Figure 3.8).

Plant Si

The samples gathered for plant Si throughout the experiment showed significant differences between plant type (p-value <0.0001), fertilizer treatment (p-value = 0.009), and time (p-value <0.001). As expected, wheat had the highest average amount of Si with

a value of 0.51% while canola was only comprised of 0.31% Si. The average individual treatment values are as follows: WNF 0.53 % Si, WF 0.49 % Si, CNF 0.34 % Si, and CF 0.28 % Si (Table 3.3). These values suggest that the application of fertilizer enhances residue decomposition therefore decreasing the concentration of Si within the WF and CF treatments. The percent Si for all treatments was fairly consistent from weeks two to ten then a peak in %Si was seen beginning from week ten and lasting throughout the rest of the experiment (see Figure 3.9).

Table 3.3. Average values by treatment for pH, C:N, %¹⁵Ndfp (derived from labeled plant residue), Si_{ws}, Si_{am}, surface strength, crust thickness, and plant %Si

Treatment	рН	C:N Soil	C:N Crop	% ¹⁵ N dfp	Si _{ws} g/kg soil	Si _{am} g/kg soil	Surface Strength g/cm ²	Crust Thickness (mm)	Plant %Si
Wheat no	5.6	11.94	33.15	0.20	0.86	37.6	62.4	23.1	0.53
fert (WNF)	а	а	b	а	а	а	а	а	a
Wheat fert	4.5	10.27	32.80	0.19	0.55	31.6	34.0	19.0	0.49
(WF)	b	b	b	а	b	b	b	b	а
Canola no	5.5	12.22	38.71	0.14	0.81	37.2	51.0	22.0	0.34
fert (CNF)	а	а	а	b	a	а	a	а	b
Canola fert	4.6	10.24	34.57	0.14	0.59	28.8	42.5	18.7	0.28
(CF)	b	b	ba	b	b	b	b	b	b

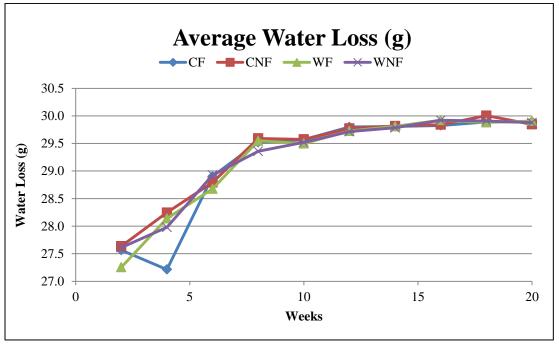


Figure 3.1. Average water loss (g) per pot for each watering cycle during a two week period for each treatment

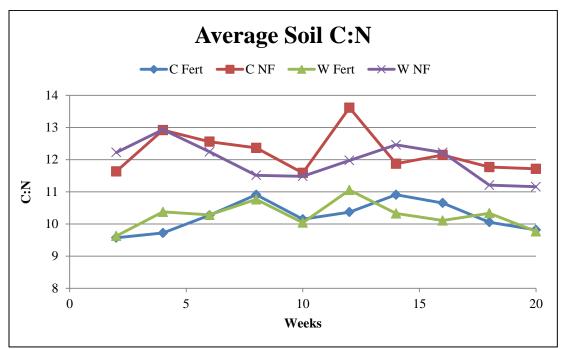


Figure 3.2. Average soil C:N over time per treatment

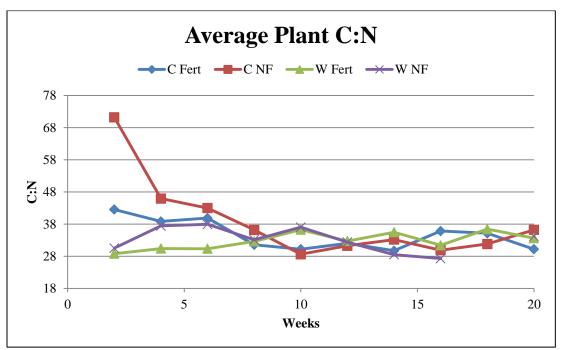


Figure 3.3. Average plant C:N over time per treatment

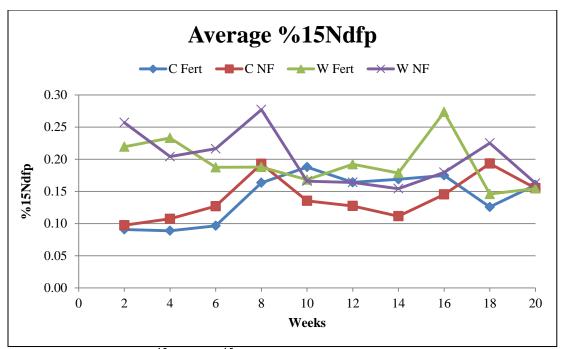


Figure 3.4. Average %¹⁵Ndfp (%¹⁵N found in the soil derived from labeled plant residue) over time per treatment

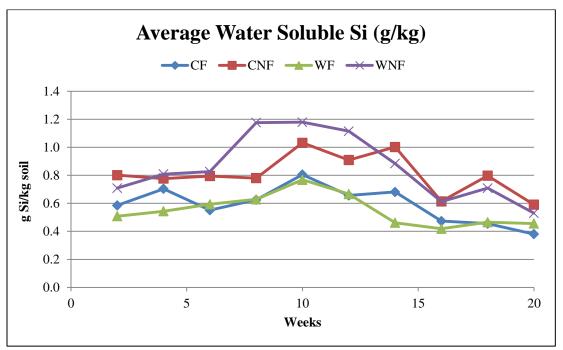


Figure 3.5. Average soil Si_{ws} (g/kg soil) over time per treatment

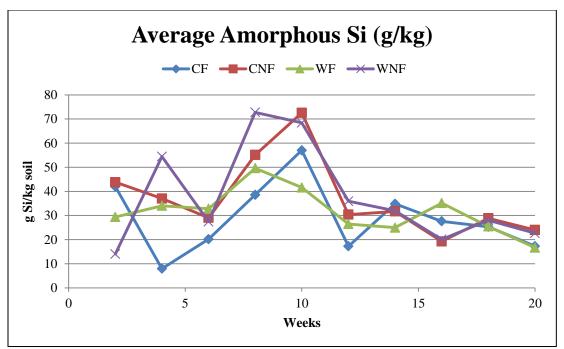


Figure 3.6. Average soil Si_{am} (g/kg soil) over time per treatment

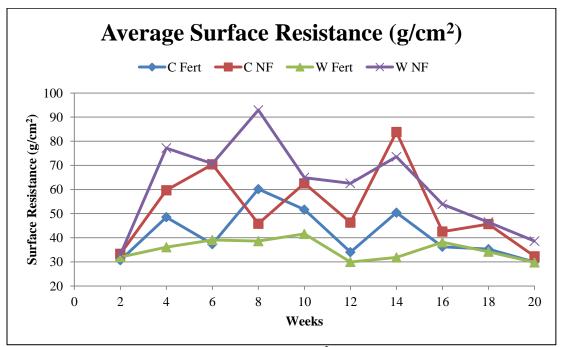


Figure 3.7. Average soil surface resistance (g/cm²) over time per treatment

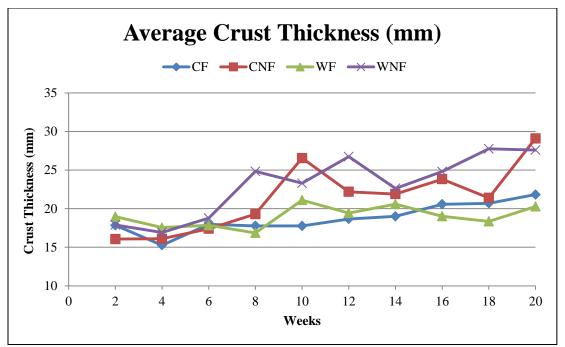


Figure 3.8. Average soil crust thickness (mm) over time per treatment

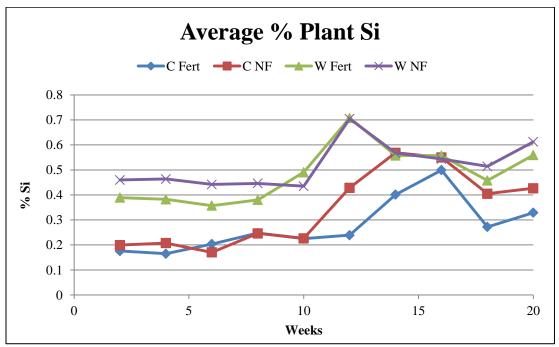


Figure 3.9. Average concentration (%) of plant Si over time per treatment

Decomposition Study

Weight Loss

Both crop type (p-value = 0.0009) and time (p-value <0.0001) significantly effected weight loss. Wheat had a higher rate of weight loss starting at 1.5 g/pot at week zero and ending at approximately 0.8 g/pot at week twelve. Canola started at 1.5 g/pot and by week twelve the residue weight was approximately 1.0 g/pot (see Table 3.4 and Figure 3.10).

Plant Si

Silicon concentrations were affected by crop type (p-value <0.0001), fertilizer level (p-value<0.0001), and week (p-value = 0.0052). Wheat consistently had higher Si concentrations than canola as expected due to wheat being a Si accumulator. Throughout the duration of the experiment, the wheat residue that was grown under the 100 lbs N rate had the highest Si concentration followed by wheat 0 lbs N, wheat 50 lbs N & wheat GH, canola 0 lbs N, and canola GH. Over time, the wheat Si concentrations increased while the canola Si concentrations slightly decreased (Figure 3.11). By multiplying the Si concentration by the residue weights at each time point the g of Si per sample was determined. These values were also significantly affected by crop (p-value <0.0001), fertilizer rate (p-value <0.0001), and week (p-value <0.0001). Over the 12 week period all residue treatments decreased in the amount of g of Si due to the overall weight loss of the residue (see figure 3.12). This suggests that other soluble components are being decomposed first including some soluble or labile Si leaving behind the more recalcitrant pool to be decomposed later on.

Week	Canola 0	Canola	Wheat 0	Wheat 50	Wheat	Wheat						
Week	lbs N	GH	lbs N	lbs N	100 lbs N	GH						
Weight (g/pot)												
0	1.49 a	1.49 a	1.49 a	1.49 a	1.50 a	1.49 a						
8	1.08 b	1.12 b	0.96 cb	1.08 b	1.09 b	0.96 cb						
12	0.97 cb	1.02 cb	0.76 c	0.79 cb	0.88 cb	0.79 cb						
	I	I	% Si	I	I							
0	0.31 fe	0.21 fe	1.24 bc	0.81 dc	1.59 ba	0.61 de						
8	0.28 fe	0.15 fe	1.65 ba	0.87 dc	1.80 a	0.97 dc						
12	0.19 fe	0.13 f	1.62 ba	1.03 dc	1.79 a	0.99 dc						
	I	I	g of Si/pot	I	I							
0	0.005 hgf	0.003 hgf	0.019 bac	0.012 edc	0.024 a	0.009 edf						
8	0.003 hgf	0.002 hg	0.016 bdc	0.009 edf	0.020 ba	0.009 edf						
12	0.002 hg	0.001 h	0.012 edc	0.008 egf	0.016 bdc	0.008 ehgf						

Table 3.4. Average residue weight loss (g), concentration of Si (%), and g of Si over time per treatment

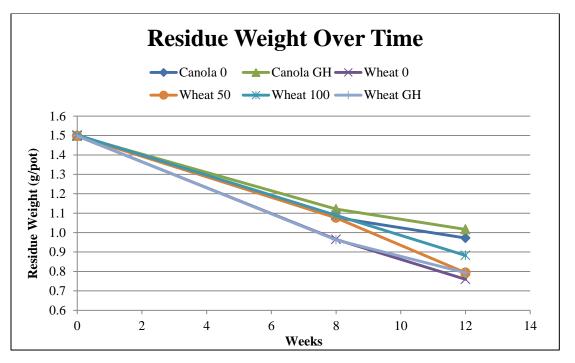


Figure 3.10. Average residue weight (g/pot) over time per treatment.

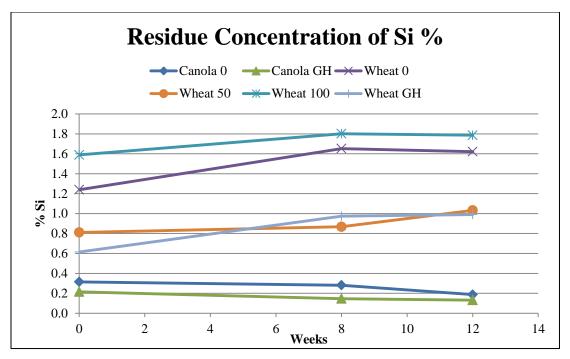


Figure 3.11. Average residue Si concentration (%) over time per treatment.

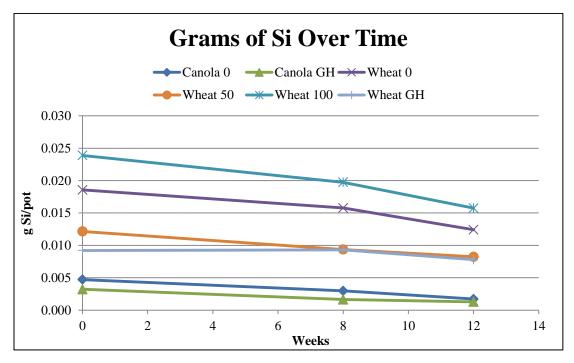


Figure 3.12. Average grams of Si/pot over time per treatment

DISCUSSION

Plant Si concentration differences were seen primarily between crop types. The treatments containing wheat residue were expected to have higher soil Si_{ws} and Si_{am} levels compared to canola due to the higher amount of Si present in wheat. However, crop type was not as significant as expected within this parameter. The fertilizer treatments had the biggest effect by decreasing the pH and apparently enhancing Si release into the soil solution. This release could have led to leaching of the silicon towards the bottom of the container as seen in field studies conducted by Brown and Mahler (1987). Since samples were only collected from the surface crust in this experiment it would be interesting for future experiments to collect samples from both the crust and the bottom of the container to obtain a complete mass balance.

Since the residue was mixed into the soil it was impossible to retrieve all residue during destructive sampling. This prevented weighing the residue to determine weight loss and therefore total residue weight at each sampling time point. This is why the plant Si values can only be expressed as a concentration. Such obstacles lead to the initiation of the decomposition study.

The decomposition study showed that most of the decomposition took place during the first eight weeks. With a decrease in more labile components of the residue, the Si concentration was slightly higher at week eight than at time zero. The wheat 0 lbs N/ac treatment was expected to have the lowest concentrations of Si within the wheat treatments. However, this was not the case as seen in Figure 3.11. The reason for the much higher concentrations of Si within this treatment is unknown, but could be due to different environmental factors (Epstein, 2009). When considering the weight loss, loss

of Si in g was much less than the residue as a whole confirming that the Si concentration does in fact increase over time (Daughtry et al., 2010; Kriauciuniene et al., 2012). Comparing the Si concentrations from the laboratory incubation and the decomposition study, the decomposition study seemed to have a faster rate of decomposition. The laboratory incubation did not show an increase in Si concentration until week twelve rather than week eight as seen in the decomposition study. Having the weight loss rates from the decomposition study, the hypothesis that the concentration of Si increased as decomposition increased in the laboratory incubation cannot be rejected.

CONCLUSIONS

Although the laboratory incubation showed no drastic differences between soil parameters over time, some important interactions were observed between treatments. The samples that did not receive fertilizer treatments had higher pH, Si_{am}, Si_{ws}, and surface strength values compared to the treatments that did receive a fertilizer application. This suggests that pH is the primary factor influencing soil Si levels and therefore surface strength. The wheat treatments contributed more ¹⁵N to the soil and had higher Si concentrations compared to the treatments containing canola residue. However, these results are primarily due to the physical aspects of the crop and the conditions in which the crop was grown rather than any affects from the incubation. As seen in Table 3.2, the wheat residue started out with a lower C:N ratio and higher %Si therefore the wheat residue was able to decompose slightly faster releasing more ¹⁵N into the soil and maintained higher Si levels in the residue compared to the canola treatments. When considering the original C:N ratios of the residue, they were almost halved by week two suggesting that a large amount of C and N was lost from the residue during this period.

The decomposition study showed that decomposition occurred most rapidly between week zero and week eight then slowed down from week eight to week twelve. Although the residue weights decreased, %Si increased with time as seen in the laboratory incubation. When looking at the amount of Si in g lost over time, the greenhouse residues lost more between week 0-8 and slightly less between 8-12 weeks. Like the laboratory incubation, this suggests that less Si is being recycled back into the soil. While both studies showed distinct differences between Si levels in crop type, such levels did not seem to have an effect on the other soil parameters analyzed. The most important factor that effected soil Si levels, crust thickness, and surface resistance was pH. Future studies are needed in order to learn more about the effects of Si cycling with different crops. Although this work did not confirm the hypothesis that by introducing canola into a cropping system soil crusting would become less of an issue, it did confirm that fertilizer management plays a significant role in Si cycling within the soil and therefore may contribute to soil crusting, surface strength, and crust thickness.

LITERATURE CITED

- Albrecht, S.L., H.T. Gollany, R.R. Allmaras, C.L. Douglas, and S.M. Copeland. 2005.
 Tillage and nitrogen fertilizer influence on carbon and soluble silica relations in a Pacific Northwest mollisol. Soil Sci. Soc. Am. J. 69(4):1102-1109.
- Ben-Hur, M., A. Paz, and M. Lado. 2004. Organic matter and aggregate size interactions in infiltration, seal formation, and soil loss. Soil Sci. Soc. Am. J. 68(3):935-942.
- Bertrand, I., B. Chabbert, B. Kurek, and S. Recous. 2006. Can the biochemical features and histology of wheat residues explain their decomposition in soil?. Plant and Soil 281:291-307.
- Bissonnais, Y.L., and M.J. Singer 1992. Crusting, runoff, and erosion response to soil water content and successive rainfalls. Soil Sci. Soc. Am. J. 56:1898-1903.
- Brown, T.H., and R.L. Mahler. 1987. Effects of phosphorus and acidity on levels of silica extracted from a Palouse silt loam. Soil Sci. Soc. Am. J. 51(3):674-677.
- Casey, W.H., S.D. Kinrade, C.T.G. Knight, D.W. Rains, and E. Epstein. Aqueous silicate complexes in wheat, *Triticum aestivum* L. Plant, Cell Environ. 27(1):51-54.
- Clymans, W., G. Govers, B. Van Wesemael, P. Meire, and E. Struyf. 2011. Amorphous silica analysis in terrestrial runoff samples. Geoderma 167:228-235.
- Cornelis, J.T., H. Titeux, J. Ranger, and B. Delvaux. Identification and distribution of the readily soluble silicon pool in a temperate forest soil below three distinct tree species. Plant and Soil 342:369-378.
- Dao, T.H. 1993. Tillage and winter wheat residue management effects on water infiltration and storage. Soil Sci. Soc. Am. J. 57(6):1586-1595.

- Daughtry, C.S.T, G. Serbin, J.B. Reeves III, P.C. Doraiswamy, and E.R. Hunt Jr. 2010. Spectral reflectance of wheat residue during decomposition and remotely sensed estimates of residue cover. Remote Sens. 2:416-431.
- Epstein, E. 2009. Silicon: its manifold roles in plants. Annals of Applied Biology 155:155-160.
- Feng, G., B. Sharratt, and F. Young. 2011. Soil properties governing soil erosion affected by cropping systems in the US Pacific Northwest. Soil Tillage Res. 111(2);168-174.
- Follett, E.A.C., W.J. McHardy, B.D. Mitchell, and B.F.L. Smith. 1965. Chemical dissolution techniques in the study of soil clays: Part1. Clay Minerals 6:23-34.
- Gicheru, P., C. Gachene, J. Mbuvi, and E. Mare. 2004. Effects of soil management practices and tillage systems on surface soil water conservation and crust formation on a sandy loam in semi-arid Kenya. Soil and Tillage Research 75:173-184.
- Gollany, H.T, R.R. Allmaras, S.M. Copeland, S.L. Albrecht, and C.L. Douglas. 2006. Incorporated source carbon and nitrogen fertilization effects on carbon storage and soluble silica in a haploxeroll. Soil Sci. 171(8):585-597.
- Kriauciuniene, Z., Velicka, R., Raudonius, S. 2012. The influence of crop residues type on their decomposition rate on the soil: a litter bag study.Zemdirbyste=Agriculture 99: 227-236.
- Ma, J.F., and N. Yamaji. 2006. Silicon uptake and accumulation in higher plants. Trends Plant Sci. 11(8):392-397.

- Marschner, P., S. Umar, and K. Baumann. 2010. The microbial community composition changes rapidly in the early stages of decomposition of wheat residue. Soil Bio. Biochem. 43:445-451.
- Remley, P.A., and J.M. Bradford. 1989. Relationship of soil crust morphology to inter-rill erosion parameters. Soil Sci. Soc. Am. J. 53(4):1215-1221.
- Richmond, K.E., and M. Sussman. 2003. Got silicon? The non-essential beneficial plant nutrient. Curr. Opin. Plant Biol. 6(3):268-272.
- Schillinger, W.F. 2011. Rainfall impacts winter wheat seedling emergence from deep planting depths. Agron. J. 103(3):730-734.
- Van der Vorm, P.D.J., 1987. Dry ashing of plant-material and dissolution of the ash in HF for the colorimetric determination of silicon. Commun. Soil Sci. Plant Anal. 18(11):1181-1189.
- Wickramasinghe, D.B., and D.L. Rowell. 2005. The release of silicon from amorphous silica and rice straw in Sri Lankan soils. Biol. Fertil. Soils. 42(3):231-240.
- Zuzel, J.F., and J.L. Pikul. 1994. Soil crusting and water infiltration affected by long-term tillage and residue management. Soil Sci. Soc. Am. J. 58(2):1524-1530.

CHAPTER 4

A COMPARISON OF TWO ROTATION HISTORIES SUMMARY

Plants greatly differ in their ability to accumulate Si, a non-essential, but beneficial element and therefore, the amount of silicon cycled is highly dependent on the crops occurring in the system (Richmond and Sussman, 2003; Ma and Yamaji, 2006). In order to determine the effects of the decomposition of Si from wheat and canola on soil crusting, an incubation and field survey was initiated. Each sample contained 250 g of Ritzville series silt loam acquired from Ralston, WA. The soil was harvested from two fields. The first field was previously cropped in wheat and the second field was previously cropped in canola. Four levels of a monomeric silicic acid solution (H_4SiO_4) solution was randomly applied to 21 samples of each soil type: 0 g/pot (control), 0.105 g/pot (low), 1.05 g/pot (medium), and 10.5 g/pot (high). Samples were destructively sampled every 1, 3, 7, 10, 14, 21, and 28 days. Analyses included: surface resistance, moisture, water soluble silica (Si_{ws}), amorphous silica (Si_{am}), crust thickness, and scanning electron microscopy (SEM). The results confirmed that application of H_4SiO_4 increased water loss, soil Si, surface resistance, and crust thickness. Significant differences were not seen with increasing H_4SiO_4 levels, just between the control and the treatments. The soil that was previously cropped in wheat had higher soil Si, surface resistance, and crust thickness compared to the canola soil. A field survey was conducted in order to further determine the long-term influence canola might have on a rotation in a field setting. Six research locations were chosen throughout eastern Washington: Davenport, Reardan, Ralston, LaCrosse, Odessa Irrigation Site #1, and Odessa Irrigation

Site #2. Each field was sampled 10 times by a field penetrometer and three one-foot soil cores were taken. Each soil core was separated into 0-10 cm, 10-20 cm, and 20-30 cm depth increments. Each core and respective depth increment was sampled for moisture, Si_{ws}, Si_{am}, and pH. The field survey did not show much significance with crop rotations and these parameters. Penetration resistance, soil Si levels, moisture, and pH all varied with location and soil depth suggesting that the climatic differences were too great for relationships to be seen across all sites.

INTRODUCTION

Terrestrial ecosystems recycle large quantities of silicon (Si) through the Si uptake by crops, grasslands, and forest vegetation (Bartoli, 1983; Alexandre et al., 1997; Epstein, 1999; Meunier et al. 1999; Blecker et al., 2006). Plants greatly differ in their ability to accumulate Si, a non-essential, but beneficial element and therefore, the amount of silicon cycle is highly dependent on the types of plants occurring in the system (Richmond and Sussman, 2003; Ma and Yamaji, 2006). Most studies of Si uptake in higher plants have been focused on monocots, which are typical Si-accumulators. Silicon is taken up as aqueous monosilicic acid (H_4SiO_4) and translocated to transpiration sites within the plant where it polymerizes as phytoliths, which consists of amorphous biogenic opal. Phytoliths return to the soil within organic residues (Cornelis et al. 2010) so that this biogenic silicon (BSi) is distributed between plant and soil in terrestrial ecosystems. Research on forested ecosystems demonstrates a prominent biologic role in silica storage and export from terrestrial environments. The annual Si uptake in forested systems has been linked to transpiration and thus depends on tree species (Cornelis et al., 2011). Bartoli (1983) found that the biological cycle of silicon is fast in the deciduous

forest ecosystem (26 kg/ha/yr absorbed) and, in contrast, slow in the coniferous system (8 kg/ha/yr absorbed). These different rates of Si recycling strongly influence the rate of BSi restitution to the topsoil, given the high mobility of Si in soil-plant systems.

Recent papers have demonstrated that land use changes can have significant effects on Si mobilization. A study performed by Clymans et al. (2011) compared silica (SiO₂) pools within soil profiles under four different land use types in southern Sweden: continuous forest, grazed forest, pasture, and arable land. The continuous forest area had the highest amount of amorphous SiO₂ (66,900 kg/ha) and water soluble SiO₂ (952 kg/ha). The arable land had the second highest values with an amorphous SiO₂ content of 28,800 kg/ha and water soluble SiO₂ at 239 kg/ha. Pasture had slightly lower amorphous SiO₂ (27,300 kg/ha) compared to the arable land, but had higher water soluble SiO₂ levels (370 kg/ha). The grazed forest had amorphous SiO₂ amounts of 23,600 kg/ha and water soluble levels at 346 kg/ha. This study demonstrates how differences in vegetation and management can very easily effect the terrestrial Si cycle.

Silica cycling in agricultural sites have not been as extensively studied as in the forested areas and no significance has been placed on crop type thus far. A long term wheat residue management field experiment was initiated in 1931 at the Pendleton Agriculture Research Center in northeastern Oregon on a Walla Walla silt loam. In 1984, Douglas et al. conducted a study on the silicic acid and oxidizable carbon movement within these research plots. They found that H_4SiO_4 levels tended to decrease with soil depth. Within the top 15 cm, H_4SiO_4 levels ranged from 22.5-38.6 g Si/m². The 15-30 cm depth range had showed values between 28.1-38.6 g Si/m². Both the 30-45 and 45-60 cm depth ranges had H_4SiO_4 ranging from 22.5-33.7 g Si/m². Penetrometer readings were

also taken. The sites and the depths requiring the greatest amount of force also had the highest amounts of Si. Twenty-two years later, Gollany et al. (2006) conducted another study on these sites looking at the source carbon (C) and nitrogen (N) fertilization effects on carbon storage and soluble silica (Si_{ws}) levels. All plots had approximately the same amount of Si_{ws} ranging between 18.6-26.3 g Si/m² in the Ap horizon and 11.4-13.2 g Si/m² in the BA horizon for 75 years of establishment. The goal of the present research is to determine whether long term cropping with cereals versus cropping systems including canola affect soil Si levels or surface resistance through a laboratory incubation and field survey.

MATERIALS AND METHODS

In order to determine the relationships among cropping systems, residue decomposition, and soil Si from various crops, soils from different cropping systems were incubated for 28 days. Each sample contained 250 g of Ritzville series silt loam acquired from Ralston, WA (See table 4.1). The soil was collected from the top 15 cm of the surface from two different fields. The first field has been cropped in a cereals rotation (winter wheat, summer fallow, barley, and spring wheat) for over 50 years and the second field has been cropped in a canola, summer fallow, winter wheat rotation for approximately 29 years. Both soils were air dried and sieved through a two mm sieve. A total of 84 samples of wheat soil and 84 samples of canola soil were created following this procedure. Four levels of H_4SiO_4 solution were then randomly applied to 21 samples of each soil type: 0 g/pot (control), 0.105 g/pot (low), 1.05 g/pot (medium), and 10.5 g/pot (high). The low value is approximately equivalent to the amount of Si found in two g of canola residue and the medium value represents the amount found in two g of wheat

residue. Samples were placed in polyethylene containers, arranged in a completely randomized design and stored in a room temperature oven (25°C). Every three days samples were brought up to field capacity (~23%) in order to simulate a wetting and drying cycle. Prior to watering, each sample was weighed to determine the amount of water loss between wetting cycles and to ensure samples were all receiving approximately the same amount of water. Samples were destructively sampled every 1, 3, 7, 10, 14, 21, and 28 days. Analyses included: surface resistance, moisture, water soluble silica (Si_{ws}), amorphous silica (Si_{am}), crust thickness, and scanning electron microscope (SEM).

In addition to the laboratory incubation, a field survey was conducted in order to determine the long term influence canola may have on a rotation in a field setting. Six research locations were chosen throughout eastern Washington: Davenport, Reardan, Ralston, LaCrosse, Odessa Irrigation Site #1, and Odessa Irrigation Site #2. Depending on the rotation history and presence of canola at the research site anywhere between two and six fields were sampled at each location. The representative field was sampled 10 times by a field penetrometer (fieldscout SC 900) and three one-foot soil cores were taken. Each soil core was separated into 0-10 cm, 10-20 cm, and 20-30 cm depth increments and sampled for moisture, Si_{ws}, Si_{am}, and pH. A summary of each site's general conditions can be seen in Figure 4.1.

<u>Laboratory analyses</u>

During the laboratory incubation, the surface strength was determined by using a Humboldt MFG. Co. pocket penetrometer. Measurements always occurred at the end of each wetting and drying cycle in order to maintain consistency between measurements.

The field survey utilized a field penetrometer (fieldscout SC 900) that has the ability to sample up to a 46 cm depth. Moisture was determined by weighing a portion of each sample, placing it in a drying oven for 48 hours, and recording the dry weight. Moisture was recorded in addition to water loss in the laboratory incubation in order to be sure the samples were all at the same moisture level at the time of sampling.

For both the laboratory incubation and the field survey, Si_{ws} extraction methods from Albrecht et al. (2005) were followed. Five grams of soil and 25 mL of distilled water were placed in polyethylene tubes, shaken for 30 minutes, allowed to settle overnight, centrifuged for 10 minutes, and then filtered with Whatman No. 42 filter paper. Amorphous soil silica is operationally defined as the Si extracted by a Na_2CO_3 solution (Follett et al., 1965). This method is used due to the fact that the solubility of Si_{am} strongly increases at higher pH levels. For both the laboratory incubation and the field survey, one gram of soil and 25 mL of 0.5 M Na₂CO₃ solution were combined in a polyethylene tube, shaken in an 80°C water bath for 10 minutes at 100 RPM. Samples were then allowed to cool, settle at room temperature and centrifuged at 2,500 RPM for 10 minutes. The extract solution was filtered with Whatman No. 42 filter paper and stored in a cool environment until analysis. Both Si_{ws} and Si_{am} solutions were analyzed using colorimetric procedures outlined by Van der Vorm (1987). One mL of extraction was pipetted into a small polyethylene container and diluted to three mL with DI water. Samples extracted with sodium carbonate had one mL HCl added and a few drops of potassium permanganate to adjust the color from a slightly vellow solution to a clear solution. After adding one mL of 0.5 M H₂SO₄, samples were agitated and incubated at 40.2°C for 20 minutes. One mL of 5% ammonium molybdate was added, agitated, and

left to sit for five minutes. One mL of both 5% oxalic acid and 1.5% ascorbic acid were also added. Solution level was adjusted to 10 mL with DI water and agitated. Samples were left to sit for 20 minutes and then read with a spectrophotometer set at an absorbance of 700 nm. If color was too dark samples were diluted 10 times in order to reach an attainable reading. Upon destructive sampling at different time points, crust thickness was measured in mm using a caliper.

Scanning Electron Microscopy (SEM) analysis was done on samples randomly selected from each treatment. A sub-sample was taken from the selected specimen before and after the final soil silica extraction. Samples were thoroughly dried and carefully removed from their containers. The crust samples were fixed to a SEM stub by firmly pressing the stub covered with a carbon tab against the targeted area of the soil crust. Loose soil and large pieces of organic matter were removed. The samples were then coated with a thin layer of gold ~40 nm in thickness using a sputter coater. This thickness was chosen after trial and error with soil samples to determine how much gold was required to keep the samples from charging while in the SEM. Once this process was complete the samples were placed in the Hitachi SEM and micrographs were taken at 50, 100, 300, 500, and 2k times magnification.

The soil solution pH was determined in a 1:1 soil to water solution. For each sampling period 10 g of soil and 10 g of water were placed into a polyethylene container and shaken vigorously for 30 seconds. Samples were allowed to settle for 15 minutes then agitated before the pH meter was submerged into the soil slurry.

Calculations

Soil Si = Si ppm * (mL of extractant/g of soil) * (1 L/1000 mL) * (1000 g/1 kg) * 10 (if

diluted) = g of Si/kg of soil

Statistical analyses

For the laboratory incubation, the soil parameters were analyzed individually using a two factor completely randomized design. The two factors considered were soil type and SiO₂ treatment. For the field survey, each research location was analyzed individually using a two factor randomized complete block design. The two factors considered were: crop rotation and depth increment. The analyses were done using the PROC GLM procedure in SAS 9.3 at a 95% confidence interval via Tukey's method of comparison.

Soil Parameter	Wheat Soil Initial Value	Canola Soil Initial Value
% Sand	18.6	13.1
% Silt	67.8	70.6
% Clay	13.6	16.3
% Moisture	16.5	12.2
pH	5.5	5.6
Total C %	0.99	1.1
Total N %	0.09	0.11
$NO_3 mg/kg$	8.0	21.0
NH_4^+ mg/kg	1.0	0.7
Water soluble Si (g/kg soil)	0.005	0.008
Amorphous Si (g/kg soil)	1.85	1.42
Bulk Density (g/cm ³)	1.4	1.4

Table 4.1. Initial soil data



Figure 4.1. General description of each site including: annual rainfall, soil type, years since introduction of canola, and the rotations sampled. NTF = no till fallow, SW = spring wheat, WW = winter wheat, C = canola, BAR = barley, SF = summer fallow, PEA = peas, and POT = potatoes

RESULTS

Laboratory incubation

Water loss & soil moisture

Water loss between each wetting cycle was fairly consistent between H_4SiO_4 treatments. For both soil types, the control lost significantly less water (p-value < 0.0001) when compared to the treatments (see Table 4.2). This might be the result of Si_{am} filling pores and increasing the capillarity of water to the surface compared to the controls. Each treatment with soil collected from the field previously cropped in wheat had slightly more water loss than that collected from the canola field. Visual cracking occurred on the soil surface between wetting and drying cycles for all treatments. Soil moisture varied slightly throughout the incubation however no statistical differences were found.

Surface Resistance

When comparing the two soil types, the soil previously cropped in wheat showed significantly higher surface resistance for all H_4SiO_4 treatments compared to canola (p-value <0.0001). A significant effect was also seen among treatments (p-value <0.01) (Table 4.2) with wheat high having the highest surface strength at 0.74 kg/cm². Such resistance differences supported the hypothesis that higher Si would increase the surface resistance and the wheat soil also increased resistance.

Crust Thickness

There was no significant difference seen between soil types. A significant difference (p-value < 0.0001) was found between the treatments and the control (Table 4.2) with the control being at least two mm thinner supporting the hypothesis that Si additions would lead to deeper crusting.

Soil Si

Overall, the soil previously cropped in wheat had significantly higher amounts of Si_{am} for all treatments (p-value 0.0001). The Si_{am} showed the expected pattern with the highest significance found between the two highest treatment applications (p-value of < 0.0001) (Table 4.3). These results support the hypothesis that soils predominantly cropped in wheat over a long period of time will have higher levels of Si_{am} and the addition of H_4SiO_4 also increases Si_{am} . Water soluble Si was variable and did not have high enough levels to show significance.

SEM

The pictures taken with the Hitachi SEM can be found in Appendix II. Although visual differences could be seen between treatments, a way to quantify the amount of Si_{am} within the images has yet to be determined.

Treatment	Water Loss (g/pot/watering cycle)	Surface Resistance (kg/cm ²)	Crust Thickness (mm)		
		Wheat	, , , , , , , , , , , , , , , , , , ,		
High	55.4 a	0.74 a	25.3 a		
Medium	55.5 a	0.71 ba	24.6 a		
Low	55.5 a	0.67 bac	23.8 a		
Control	52.4 b	0.56 dc	20.3 b		
	Canola				
High	54.4 a	0.68 ba	24.5 a		
Medium	54.6 a	0.65 bac	24.8 a		
Low	54.6 a	0.60 bdc	23.5 a		
Control	51.4 b	0.51d	21.2 b		

Table 4.2. Average water loss, surface resistance, and crust thickness by treatment

Treatment	Siws (g/kg soil)	Si _{am} (g/kg soil)			
	Wheat				
High	0.007 a	6.51 a			
Medium	0.005 a	2.54 c			
Low	0.004 a	2.03 c			
Control	0.007 a	2.12 c			
	Canola				
High	0.006 a	5.01 b			
Medium	0.004 a	2.22 c			
Low	0.006 a	1.82 c			
Control	0.005 a	1.83 c			

Table 4.3. Mean Si_{ws} , and Si_{am} per treatment

Field survey

Surface resistance

Davenport and Reardan consistently showed the highest penetration resistance from approximately 137.9 kPa to a resistance impenetrable by the field penetrometer at the depths of 10-20 cm (see Tables 4.4&4.5). The highest resistance at this depth was found in the fields most recently producing winter wheat. The resistance found at the surface depths (0-10 cm), was less in the fields most recently producing canola than those producing cereal crops, as seen in figures 4.2 & 4.3. However the resistance values at these sites were highly variable among rotations and only seemed to show much significance by depth suggesting these differences are primarily due to the specific climate, soil type, and other management practices. Ralston and LaCrosse also had the highest penetration resistance occur at the 10-20 cm depth ranging from 110.3 to 241.3 kPa (see Tables 4.6&4.7); however, the fields most recently cropped in canola showed the highest resistance at both the 0-10 cm and 10-20 cm depth ranges (see figures 4.4&4.5). The irrigated sites had significantly lower penetrometer readings, which may be due primarily to the more frequent watering and more intense tillage practices (Tables

4.8&4.9). The highest resistance for these sites was found at the lower depth range of 20-30 cm rather than the 10-20 cm range as seen at the other sites possibly due to the use of disc rippers. Despite these lower numbers, irrigated site #2 showed higher penetration numbers in the field in which canola has never been a part of the rotation (see figure 4.6).

Moisture

Both Davenport and Reardan showed an increase in moisture with increasing depth in the soil profile. The fields in Davenport, which are currently in fallow showed significantly more moisture than those that were not in fallow (Table 4.4). Reardan moisture levels also varied with crop rotation. The field most recently cropped in canola and previously winter wheat (C/WW/BAR) had the highest overall average moisture content (23%) followed by the field that has never had canola in the rotation (16%) and the field most recently cropped in winter wheat and previously canola (WW/C/BAR) had the least amount of moisture (8%) (Table 4.5). The research plots in Ralston contained the most moisture in the 10-20 cm and 20-30 cm depths (Table 4.6). Crop rotation also proved to be significant for moisture levels (p-value <0.0001). The field that has never had canola in the rotation (WW/BAR/SW) had the most moisture with an overall average value of 13% followed by a field most recently cropped in winter wheat and previously cropped in canola (WW/SF/C/SF) (11%) and the field most recently cropped in canola (C/SF/WW/SF) has the lowest average moisture value (8%). The moisture values in LaCrosse actually showed the opposite trend with depth, the highest values were seen in the top 0-10 cm in all crop rotations (Table 4.7). The WW/C/SF field showed the highest overall average moisture value followed by the field that has never had canola and the field with the lowest moisture levels was the C/SF/WW field. The moisture values at the

irrigated sites were highly variable most likely due to the amount of irrigation and the irrigation schedule (Tables 4.8&4.9)

pН

The most significant factor determining pH levels at all the research sites is depth. In Davenport the top 0-10 cm had an average value of 5.1 while the 10-20 cm depth had an average of 5.0 followed by a slightly more calcareous 20-30 cm with an average pH of 6.2 (Table 4.4). The research plots in Reardan showed a similar trend, as soil depth increased so did pH (Table 4.5). The C/WW/BAR field had a slightly calcareous overall pH with a value around 8.2 followed by the field that has not had canola (6.4) and finally the WW/C/BAR field with an average pH of 5.5. Both Ralston and LaCrosse only showed significant differences with depth. Both sites had an average pH value of 5.4 at the 0-10 cm depth, 5.2 at the 10-20 cm depth, and 6.1 at the 20-30 cm depth across all rotations (Tables 4.6&4.7). Irrigated site #1 for both crop rotations showed an increase in pH with an increase in depth (Table 4.8). The average pH for the field most recently grown in canola was significantly higher (6.2) than the field that has not had canola in the rotation (5.5). Irrigated site #2 showed a decrease in pH with an increase in depth (Table 4.9).

Soil Si

The amorphous Si levels in Davenport did not change significantly with crop rotation, but depth did prove to be significant (p-value = 0.0022). The highest amount of Si_{am} was found in the 0-10 cm depth range followed by the 20-30 cm range. The 10-20 cm depth had the least amount of Si_{am} (Table 4.4). In Reardan the Si_{am} levels decreased with increasing depth. The 0-10 cm depth has significantly more Si_{am} (p-value = 0.0173)

compared to the lower depths (Table 4.5). The research plots in Ralston had the highest accumulation of Si_{am} out of all sites sampled. Depth was the primary factor determining Si_{am} levels with levels decreasing with increasing depth (Table 4.6). The Si_{am} levels in LaCrosse did not show significance with depth. Crop rotation, however, did seem to have a slight effect. The C/SF/WW field had the highest amount of Si_{am} followed by the WW/C/SF field and lastly the field with no canola in the rotation had the least amount of Si_{am} (Table 4.7). Irrigation site #1 had the most Si_{am} levels in the no canola present rotation and did not change much with depth (Table 4.8). Irrigation site #2 had the most Si_{am} in the top 0-10 cm depth and the levels decrease with increasing soil depth (Table 4.9).

The water soluble Si levels significantly changed with soil depth across all research sites. In Davenport, the NTF plots had the highest amount of Si_{ws} in the top 0-10 cm and slightly decreased with soil depth. The fields that were not in fallow however showed the highest Si_{ws} in the 20-30 cm depth (Table 4.4). Although the distribution of Si_{ws} was different among such cropping rotations the overall total Si_{ws} was similar. In Reardan, crop rotation also showed significance (p-value = 0.0002). The field most recently cropped in canola had the highest total Si_{ws} with the highest accumulation in the 0-10 cm depth. The WW/C/BAR and no canola rotation accumulated Si_{ws} in the 20-30 cm (Table 4.5). The Ralston and LaCrosse research plots did not differ between crop rotations, but depth did prove to be significant (p-value <0.0001) with the most Si_{ws} accumulating in the 20-30 cm depth followed by the 10-20 cm depth and the least amount in the 0-10 cm depth (Table 4.6). Irrigation site #1 showed differences between both crop rotations (p-value = 0.0065) and depth (p-value = 0.0109). The field most recently

cropped in canola had a higher amount of Si_{ws} with a value of 1.47 g Si/kg soil compared to the field that has no canola in the rotation with a value of 1.28 g Si/kg soil. In both fields the Si_{ws} accumulated mostly in the 20-30 cm depth followed by 10-20 cm and finally 0-10 cm (Table 4.8). Irrigated site #2 did not show a difference in Si_{ws} levels between fields however a decrease with an increase in depth was seen (Table 4.9).

Table 4.4. Penetrometer (kPa), moisture (%), pH, Si_{am} (g Si/kg soil), and Si_{ws} (g Si/kg soil) values for each crop rotation by depth located in Davenport, WA

Depth	NTF/SW/SW	SW/WW/SW	NTF/SW/C	SW/WW/C	WW/C/SW	C/SW/WW
(cm)						
		I	Penetrometer (l	kPa)		
0-10	91.9 bc	110.2 b	59.7 с	114.8 b	110.2 b	78.1 b
10-20	133.2 ba	154.1 a	152.0 a	151.6 a	N/A	161.5 a
20-30	140.3 ba	127.3 ba	126.9 b	147.4 ba	N/A	N/A
			Moisture (%)		
0-10	10.00 dc	5.33 d	13.00 bc	5.00 d	5.33 d	4.66 d
10-20	18.33 ba	8.33 dc	18.00 ba	7.66 dc	8.66 dc	8.00 dc
20-30	19.00 a	8.66 dc	23.00 a	9.00 dc	9.66 dc	9.00 dc
			pН			
0-10	5.11 dc	5.12 dc	5.06 d	5.11 dc	5.18 bdac	5.07 d
10-20	4.87 d	5.13 bdc	5.36 bdac	5.09 d	4.95 d	4.84 d
20-30	6.25 a	6.20 bac	6.40 a	6.24 ba	6.20 bac	5.88 bdac
			Si _{am} (g Si/kg so	oil)		
0-10	57.66 a	36.19 a	32.62 a	37.93 a	30.47 a	46.45 a
10-20	16.68 a	18.04 a	15.20 a	26.30 a	18.46 a	20.81 a
20-30	17.89 a	32.06 a	18.21 a	26.72 a	26.07 a	42.55 a
Si _{ws} (g Si/kg soil)						
0-10	1.55 bac	1.19 bc	1.82 a	1.25 bc	1.17 c	1.33 bac
10-20	1.20 bc	1.38 bac	1.27 bc	1.30 bc	1.44 bac	1.47 bac
20-30	1.25 bc	1.64 ba	1.33 bac	1.55 bac	1.78 a	1.82 a

Depth (cm)	WW/BAR/SW	WW/C/BAR	C/WW/BAR		
	Penetrometer (kPa)				
0-10	114.8 c	119.4 c	105.6 c		
10-20	199.8 a	166.3 ab	161.2 ab		
20-30	150.2 bc	N/A	154.0 b		
	Moist	ure (%)			
0-10	14.33 a	5.66 a	22.33 a		
10-20	15.66 a	9.00 a	24.00 a		
20-30	18.50 a	9.33 a	23.66 a		
		рН			
0-10	6.26 bc	5.59 cd	7.99 ba		
10-20	6.47 bc	5.12 cd	8.59 a		
20-30	6.86 bc	6.18 bcd	8.48 a		
	Si _{am} (g	Si/kg soil)			
0-10	43.54 a	26.75 ba	31.60 ba		
10-20	17.77 ba	13.19 ba	28.35 ba		
20-30	19.24 ba	8.61 b	23.65 ba		
Si _{ws} (g Si/kg soil)					
0-10	0.88 b	0.87 b	1.47 a		
10-20	0.91 b	0.97 b	1.23 ba		
20-30	0.91 b	1.18 ba	1.13 ba		

Table 4.5. Penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil), and Si _{ws} (g Si/kg
soil) values for each crop rotation by depth located in Reardan, WA

Table 4.6. Penetrometer (kPa), moisture (%), pH, Si_{am} (g Si/kg soil), and Si_{ws} (g Si/kg soil) values for each crop rotation by depth located in Ralston, WA

Depth (cm)	WW/BAR/SW	WW/SF/C/SF	C/SF/WW/SF		
	Penetrometer (kPa)				
0-10	41.3 e	52.8 ed	80.4 bced		
10-20	114.8 bcad	146.4 ba	155.0 a		
20-30	100.5 bcaed	114.8 bcad	157.9 a		
	Mois	ture (%)			
0-10	12.53 cbad	8.38 cbd	7.95 d		
10-20	14.24 a	12.84 cba	9.81 cbad		
20-30	14.46 a	13.62 ba	8.63 cbd		
		pH			
0-10	5.23 cb	5.99 ab	5.25 cb		
10-20	5.51 acb	5.57 acb	5.17 cb		
20-30	6.52 a	6.19 ab	5.99 ba		
	Si _{am} (g	Si/kg soil)			
0-10	54.52 ba	50.54 bac	45.73 bac		
10-20	59.10 a	48.76 bac	39.33 bac		
20-30	21.74 ba	47.93 bac	23.12 bac		
	Si _{ws} (g	Si/kg soil)	•		
0-10	0.51 c	0.57 c	0.48 c		
10-20	0.65 bac	0.73 bac	0.76 cab		
20-30	0.90 a	0.78 cab	0.86 ab		

Depth	PEA/SW/WV	WW/C/SF	C/SF/WW
	Pene	trometer (kPa)	·
0-10	50.5 d	75.8 c	57.4 d
10-20	89.0 bc	169.4 a	120.6 ab
20-30	N/A	134.9 ab	N/A
	N	loisture (%)	
0-10	16.52 a	17.21 a	13.52 bdac
10-20	14.94 ba	13.68 bac	10.75 ebdcf
20-30	8.21 ef	8.65 edf	9.76 edcf
		pH	
0-10	5.41 bdc	5.33 bdc	5.67 bdac
10-20	4.77 d	4.91 d	5.34 bdc
20-30	6.54 a	5.47 bdc	6.14 bac
	Sian	n (g Si/kg soil)	
0-10	28.34 a	24.63 a	34.63 a
10-20	13.61 bc	18.95 a	35.62 a
20-30	9.86 c	22.06 a	38.00 a
	Siw	_s (g Si/kg soil)	
0-10	0.50 e	0.58 ed	0.62 edc
10-20	0.67 ebdc	0.62 edc	0.77 ebdac
20-30	1.00 a	0.75 ebdac	0.93 ba

Table 4.7. Penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil), and Si _{ws} (g Si/kg
soil) values for each crop rotation by depth located in LaCrosse, WA

Table 4.8. Irrigated site #1 penetrometer (kPa), moisture (%), pH, Si _{am} (g Si/kg soil), and
Si _{ws} (g Si/kg soil) values for each crop rotation by depth located in Odessa, WA

Depth (cm)	WW/WW/POT	C/WW/WW			
	Penetrometer (kPa)				
0-10	29.9 b	39.0 b			
10-20	81.8 ba	104.3 a			
20-30	124.3 a	135.7 a			
	Moisture (%)				
0-10	17.66 a	7.00 b			
10-20	16.33 a	15.33 a			
20-30	11.00 ba	14.00 ba			
	рН				
0-10	5.33 b	5.88 ba			
10-20	5.36 b	6.24 a			
20-30	5.87 ba	6.40 a			
	Si _{am} (g Si/kg soil)				
0-10	35.47 a	36.91 a			
10-20	35.77 a	29.30 b			
20-30	35.73 a	31.11 ba			
Si _{ws} (g Si/kg soil)					
0-10	1.12 b	1.36 ba			
10-20	1.26 ba	1.50 a			
20-30	1.45 a	1.55 a			

Depth (cm)	WW/WW/BAR	WW/C/WW	C/C/WW
	Penetro	meter (kPa)	
0-10	39.0 bc	6.9 c	18.4 c
10-20	97.4 ba	33.5 bc	31.2 bc
20-30	134.3 a	91.7 ba	40.7 bc
	Mois	sture (%)	÷
0-10	27.33 a	5.33 d	14.66 c
10-20	19.66 b	7.00 d	20.33 b
20-30	16.00 cb	N/A	17.5 cb
		рН	
0-10	6.27 a	6.70 a	6.65 a
10-20	5.78 a	6.96 a	6.11 a
20-30	5.98 a	N/A	6.13 a
	Si _{am} (g	g Si/kg soil)	
0-10	26.72 ba	36.34 a	26.03 ba
10-20	20.92 ba	24.26 ba	27.02 ba
20-30	21.74 ba	N/A	16.00 b
	Si _{ws} (g	Si/kg soil)	
0-10	0.13	0.18	0.13
10-20	0.10	0.12	0.11
20-30	0.11	N/A	0.08

Table 4.9. Irrigated site #2 penetrometer (kPa), moisture (%), pH, Si_{am} (g Si/kg soil), and Si_{ws} (g Si/kg soil) values for each crop rotation by depth located in Odessa, WA

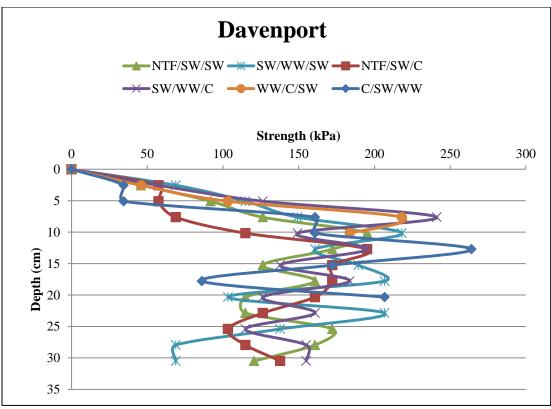


Figure 4.2. Penetrometer measurements in kPa per cm for each crop rotation at the Davenport research site

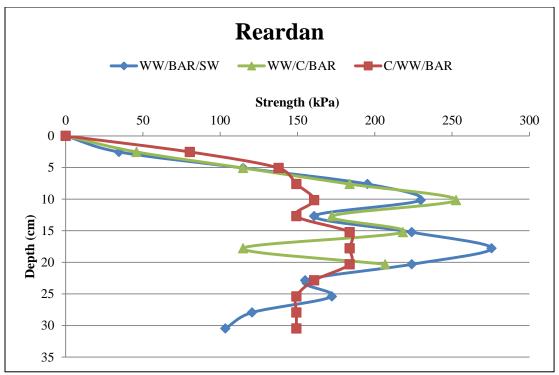


Figure 4.3. Penetrometer measurements (kPa) per cm for each crop rotation at the Reardan research site

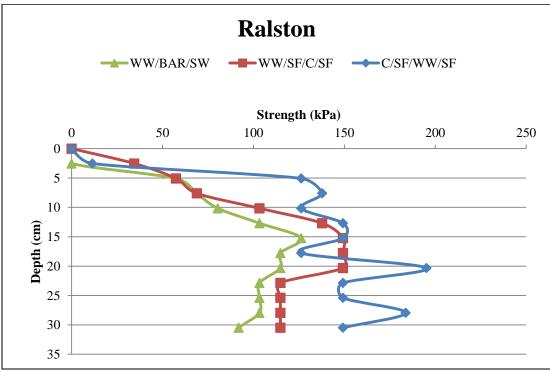
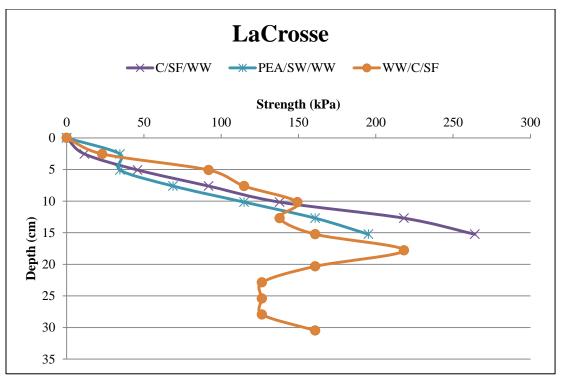
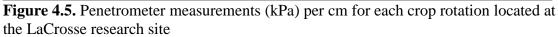


Figure 4.4. Penetrometer measurements (kPa) per cm for each crop rotation located at the Ralston research site





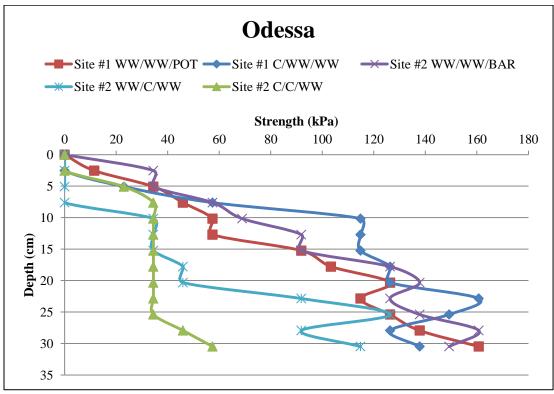


Figure 4.6. Penetrometer measurements (kPa) per cm for each crop rotation located at the Odessa research sites

DISCUSSION

The laboratory incubation with added silicic acid resulted in significant increases in water loss, surface resistance, crust thickness, and Si_{am}. The water loss levels between wetting and drying cycles were consistent among treatments and slightly lower than the control. Surface resistance slightly increased with increasing H₄SiO₄ levels as seen in other field studies (Singleton et al., 1989). However, the biggest difference found was between soil types; the wheat soil had higher surface resistance compared to canola. The soil Si_{am} levels showed a similar relationship, significantly more Si_{am} was present in the wheat soil compared to the canola soil. The H₄SiO₄ treatments did not vary much in Si_{am} except for the high treatment. Although a significant interaction effect was not seen within these results, a relationship has been established with increasing Si levels, increased surface resistance, and crust thickness suggesting that increased H₄SiO₄ does contribute to soil crusting (Belnap, 2001; Ben-Hur and Wakindiki 2002).

The field survey unfortunately did not show any significant differences among crop rotations within or across sites. Perhaps the research sites were not similar enough to obtain significant interactions between such parameters (Wakindiki and Ben-Hur, 2002). This field survey was limited by grower participation, however, care was taken to include two research sites within 40 miles of each other for the Davenport/Reardan, Ralston/LaCrosse, and Odessa area. It may be beneficial for future studies to include sites that are closer together and to take more soil cores within the fields in order to try and establish relationships between the previously mentioned soil parameters.

CONCLUSIONS

The laboratory incubation results confirmed that application of H₄SiO₄ increases water loss, soil Si, surface resistance, and crust thickness. Significant differences were not seen with increasing H₄SiO₄ levels just between the control and the treatments. The soil that was previously cropped in wheat had higher soil Si_{am}, surface resistance, and crust thickness compared to the canola soil demonstrating the influence crop rotation can have on such soil parameters. The field survey, however, did not show much significance with crop rotations regarding soil Si levels or penetration resistance. All parameters measured varied with location and soil depth suggesting that these sites did not have similar enough environmental factors or crop rotations to produce consistent results. Although the results from the field study were inconclusive, effects due to Si treatments and previous crops under controlled conditions warrants further study of field sites.

LITERATURE CITED

- Albrecht, S.L., H.T. Gollany, R.R. Allmaras, C.L. Douglas, and S.M. Copeland. 2005.
 Tillage and nitrogen fertilizer influence on carbon and soluble silica relations in a
 Pacific Northwest mollisol. Soil Sci. Soc. Am. J. 69(4):1102-1109.
- Alexandre, A., J.D. Meunier, F. Colin, and J.M. Koud. 1997. Plant impacts on the biogeochemical cycle of silicon and related weathering processes. Geochim. Cosmochim. Acta. 61(3):677-682.
- Bartoli, F. 1983. The biogeochemical cycle of silicon in two temperate forest ecosystems. Ecol. Bull. 35:469-476.
- Belnap, J. 2001. Comparative structure of physical and biological soil crusts. Biological soil crusts: Structure, function, and management. Springer-Verlag Berlin Heidelberg. Germany.
- Blecker, S.W., R.L. McCulley, O.A. Chadwick, and E.F. Kelly. 2006. Biogenic cycling of silica across a grassland bioclimosequence: grassland silica cycling. Global Biogeochem. Cycles 20(3):1-11.
- Brown, T.H. and R.L. Mahler. 1987. Effects of phosphorus and acidity on levels of silica extracted from a Palouse silt loam. Soil Sci. Soc. Am. J. 51(3): 674-677.
- Clymans, W., E. Struyf, G. Govers, F. Vandevenne, and D.J. Conley. 2011. Anthropogenic impact on amorphous silica pools in temperate soils. Biogeosciences 8:2281-2293.
- Cornelis, J.T., J. Ranger, A. Iserentant, and B. Delvaux. 2010. Tree species impact the terrestrial cycle of silicon through various uptakes. Biogeochemistry 97:231-245.

- Cornelis, J.T., H. Titeux, J. Ranger, and B. Delvaux. 2011. Identification and distribution of the readily soluble silicon pool in a temperate forest soil below three distinct tree species. Plant and Soil 342:369-378.
- Douglas, C.L., R.R. Allmaras, and N.C. Roager. 1984. Silicic acid and oxidizable carbon movement in a Walla Walla silt loam as related to long term management. Soil Sci. Soc. Am. J. 48:156-162.
- Epstein, E. 1999. Silicon. Annu. Rev. Plant Biol. 50(1):641-664.
- Follett, E.A.C., W.J. McHardy, B.D. Mitchell, and B.F.L. Smith. 1965. Hemical dissolution techniques in the study of soil clays: Part1. Clay Minerals 6:23-34.
- Gollany, H.T., R.R. Allmaras, S.M. Copeland, S.L. Albrecht, and C.L. Douglas. 2006. Incorporated source carbon and nitrogen fertilizer effects on carbon storage and soluble silica in a Haploxeroll. Soil Sci. 171(8):585-597.
- Ma, J.F., and N. Yamaji. 2006. Silicon uptake and accumulation in higher plants. Trends Plant Sci. 11(8):392-397.
- Richmond, K.E., and M. Sussman. 2003. Got silicon? The non-essential beneficial plant nutrient. Curr. Opin. Plant Biol. 6(3):268-272.
- Singleton, P.L., M. Mcleod, and H.J. Percival. 1989. Allophane and halloysite content and soil solution silicon in soils from rhyolitic volcanic material, New Zealand. Australian Journal of Soil Research 27:67-77.
- Van der Vorm, P.D.J., 1987. Dry ashing of plant-material and dissolution of the ash in HF for the colorimetric determination of silicon. Commun. Soil Sci. Plant Anal. 18(11):1181-1189.

Wakindiki, I.I.C. and M. Ben-Hur. 2002. Soil mineralogy and texture effects on crust micromorphology, infiltration, and erosion. Soil Sci. Soc. Am. J. 66:897-905.

CHAPTER 5

CONCLUSIONS

A few conclusions can be drawn from the experimental results throughout this thesis. The greenhouse results expressed the importance of crop type on both fiber and Si allocation. Wheat accumulated significantly more Si and hemicellulose while canola accumulated higher amounts of lignin, cellulose, and other soluble components. The increased fertilizer rates increased yields, but did not affect the fiber synthesis or Si uptake. Although the fertilizer treatments did not affect crop uptake of Si in the greenhouse, the laboratory incubation in which fertilizer was applied to the soil surface did show an effect on soil parameters. The treatments without fertilizer had significantly higher values for pH, Si_{am}, Si_{ws}, and surface strength. This suggests that at higher pH levels more Si is adsorbed to soil surfaces allowing Si to stay within the top few mm of the soil rather than being translocated lower in the soil profile. Higher amounts of Si may then be able to act as cementing agents by coating the insides of the soil pores resulting in increased surface resistance. The decomposition study showed that over a period of 12 weeks, the concentration of Si in both residue types increases and the more soluble components are being decomposed first. However when taking the residue weights into consideration, the grams of Si slightly decreases with time. This suggests that there is a soluble or readily available pool of Si within the residue that can be rapidly decomposed and released back into the soil system. The rotation comparison incubation showed that additions of silicic acid, regardless of the amount, increased water loss, Si_{ws}, Si_{am}, surface resistance, and crust thickness. These results suggest that any addition of Si to the soil may result in a higher potential and severity of soil crusting. The soil which has been

traditionally cropped in a cereal rotation for over 50 years did show significantly higher surface resistance and Si_{am} values when the highest amount of silicic acid was applied compared to the soil in which canola has been a part of the rotation for 29 years. This relationship suggest that long term crop rotations do influence surface strength and Si_{am} levels therefore affecting soil crusting potential. The field survey did not show any significant trends with crop rotation however the environmental factors were variable among sites which may have caused such variable results.

The main conclusion to be drawn from this research is that in arid and semi-arid areas, where soils are more susceptible to crusting, it may be beneficial to consider both the structural composition (specifically Si levels) of crops and fertilizer use within the cropping system. Long-term cereal rotations and fertilizer additions may increase soil Si levels and therefore potential surface crusting. Therefore the introduction of crops that accumulate less Si, such as canola or other dicots, may aid in decreasing the severity or occurrence of soil crusting aiding in seedling emergence and subsequent crop productivity.

APPENDIX I

PILOT INCUBATION: EFFECTS OF WHEAT AND CANOLA RESIDUE ON SOIL CRUSTING AND SILICON LEVELS

RESEARCH GOAL

The overall goal of this preliminary research was to establish some connections between differing silica levels in wheat and canola grown under different fertilizer conditions and its effect on soil crusting.

Hypotheses:

- Treatments with wheat residue will have more silica in the soil from decomposition of residue and therefore have a higher surface strength compared to treatments with canola residue.
- Residue grown under the high N rates would lower the soil pH and therefore increase the amount of Si in soil solution.

MATERIALS AND METHODS

In order to apprehend the relationship between the decomposition of Si from various crops an incubation was initiated for a period of 63 days. Each sample contained 250 g of Warden series sandy loam acquired from the Prosser Research Station (See table AI-1). Soil was air dried and sieved through a two mm sieve prior to incubation. The residue used in this study was grown at the Palouse Conservation Farm Station under two different nitrogen rates: 0 lbs/acre for wheat and canola, 150 lbs/acre for wheat, and 160 lbs/acre for canola (See table AI-2). The residue was cut into small pieces ranging from two to five cm. Two g of residue was added to each 250 g soil sample and thoroughly mixed. A control treatment was also employed consisting of only air dried soil. Samples were placed in polyethylene containers, arranged in a completely randomized design and stored in a room temperature oven (25°C). Every three days samples were brought up to field capacity (~23%) in order to simulate a wetting and drying cycle. Prior to watering, each sample was weighed to determine the amount of water loss between wetting cycles and to ensure samples were all receiving approximately the same amount of water. Subsamples were taken every 0, 1, 3, 7, 10, 14, 21, 28, 35, 42, 49, 56, and 63 days and analyzed for pH, total C & N, NO₃⁻ & NH₄⁺, water soluble Si (Si_{ws}), amorphous Si (Si_{am}), and surface strength. Sub-samples were taken at random after the completion of the incubation for analysis using a scanning electron microscope (SEM).

Soil solution pH was determined by using a 1:1 soil to water solution. For each sampling period 10 g of soil and 10 g of water were placed into a polyethylene container and shaken vigorously for 30 seconds. Samples were allowed to settle for 15 minutes then agitated before the pH meter was submerged into the soil slurry. Total soil C and N was analyzed using a LECO CN2000. Soil nitrate and ammonium amounts were deduced by conducting KCl extracts with a soil to KCl ration of 1:4 and analyzed via the lachet XYZ autosampler ASX 500-series.

Water soluble silicon extraction methods from Albrecht et al. (2005) were followed. Five g of soil and 25 mL of DI water were placed in polyethylene tubes, shaken for 30 minutes, allowed to settle overnight, centrifuged for 10 minutes, and then filtered with Whatman No. 42 filter paper. Amorphous soil silicon was extracted by following the method of Follet et al. (1965). This method is used due to the fact that the solubility of Si_{am} strongly increases at higher pH levels. One g of soil and 25 mL of sodium carbonate were combined in a polyethylene tube, shaken in an 80°C water bath for 10 minutes at

100 RPM's. Samples were then allowed to cool and settle at room temperature and centrifuged at 2,500 RPM's for 10 minutes. Extracted solution was filtered with Whatman No. 42 filter paper and stored in a cool environment until analysis. Both Si_{ws} and Si_{am} solutions were analyzed using colorimetric procedures outlined by Van der Vorm (1987). One mL of extraction was pipetted into small polyethylene container and diluted to three mL with DI water. Samples extracted with sodium carbonate had one mL HCl added and a few drops of potassium permanganate to adjust the color from a slightly yellow solution to a clear solution. One mL of 0.5 M H2SO4 was added then samples were agitated and incubated at 40.2°C for 20 minutes. One mL of 5% ammonium molybdate was added, agitated, and left to sit for five minutes. One mL of both 5% oxalic acid and 1.5% ascorbic acid were also added. Solution level was adjusted to 10 mL with DI water and agitated. Samples were left to sit for 20 minutes and then read with a spectrophotometer set at an absorbance of 700 nm. If color was too dark samples were diluted 10 times in order to reach an attainable reading.

In order to determine surface strength a Humboldt MFG. CO. pocket penetrometer was used. Measurements with the penetrometer always occurred at the end of each wetting and drying cycle in order to maintain consistency between measurements.

SEM analysis was done on samples acquired after the incubation was complete. A randomly selected sample from each treatment within all three incubations was used for SEM analysis. A sub-sample was taken from the selected specimen's before and after the final soil silica extraction. Samples were thoroughly dried and carefully removed from their containers. The crust samples were fixed to a SEM stub by firmly pressing the stub covered with a carbon tab against the targeted area of the soil crust. Loose soil and large

pieces of organic matter were removed. The samples were then coated with a thin layer of gold ~40 nm in thickness using a sputter coater. This thickness was chosen after trial and error with soil samples to determine how much gold was required to keep the samples from charging while in the SEM. Once this process was complete the samples were placed in the Hitachi SEM and micrographs were taken at 50, 100, 300, 500, and 2k times magnification. Images can be seen in Appendix II.

Calculations

Total nitrate/ammonium = ppm * (0.025 L KCl/(5 g soil/MF)) = mg/g

MF = moisture factor (wet weight/dry weight)

Soil Si = Si ppm * (mL of extractant/g of soil) * (1 L/1000 mL) * (1000 g/1 kg) * 10

(only if diluted) = mg of Si/kg of soil

Statistical analyses

Data was analyzed by the PROC GLM procedure in SAS 9.3 at a 95% confidence interval using Tukey's method of comparison.

Soil Parameter	Initial Value
% Sand	46.1
% Silt	46.1
% Clay	7.8
% Moisture	3.2
pH	8.21
Total C %	0.52
Total N %	0.02
NO ₃ mg/g	2.6×10^{-2}
$NH_4^+ mg/g$	2.7 x 10 ⁻⁴
Water soluble Si (mg/kg soil)	66.3
Amorphous Si (mg/kg soil)	214.3
Bulk density (g/cm ³)	1.5

Table AI-1. Initial soil data

Table AI-2. Residue analysis

Residue Type	C:N	%NDF	%ADF	%ADL	%Ash	%Si
Wheat 0 lbs N	138.0	73.9	45.3	11.3	0.11	0.81
Wheat 150 lbs N	138.0	74.0	50.7	9.8	0.08	0.72
Canola 0 lbs N	115.8	65.5	51.1	10.4	0.12	0.01
Canola 160 lbs N	42.9	67.3	52.5	12.8	0.16	0.01

RESULTS

Water loss

No significant difference was seen over time or between treatments for water loss (see Table AI-3). This suggests that the soil moisture was kept at a consistent level throughout the experiment and treatment did not have an effect on rate of water loss.

pH

pH varied slightly at each sampling period (between 8.0-8.8) and the only

significant difference found was between the treatments and the control (p-value < 0.01). It was expected that the treatments with biomass grown under higher amounts of fertilizer would have slightly lower pH compared to the other treatments. This trend was shown in the overall averages, however it was only slight (see Table AI-3 and Figure AI-1).

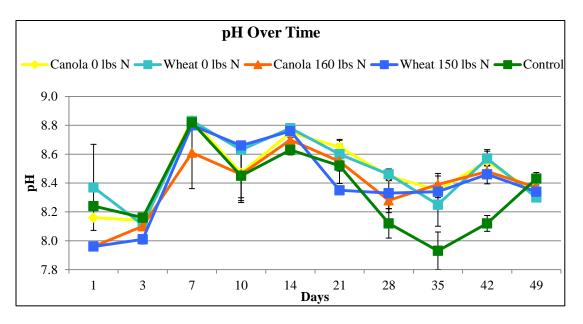


Figure AI-1. Graph of pH over time

Total C&N

The total soil C data showed a significant effect due to treatment (p-value < 0.001). Canola 0 lbs N ranked the highest with a value of 59% C followed by canola grown at 160 lbs N (58% C), wheat 150 lbs N (56% C), wheat 0 lbs N (54% C), and finally the control (50% C). Total soil N also showed a significant treatment effect (p-value < 0.001). Canola 160 lbs N ranked the highest with a value of 4% N followed by canola 0 lbs N (~3% N), control (~3% N), wheat 150 lbs N (~3% N), and wheat 0 lbs N (~3% N). When the C:N values were calculated, they followed the same trend as seen in the pH results (see Table AI-3). No pattern was seen over time and the biggest contrast was between the residues grown without fertilizer and residues grown with a higher rate of fertilizer (p-value < 0.01) due to more available N.

Surface resistance

The purpose for this measurement was to establish a connection between crop type and soil crusting. It was hypothesized that the wheat residue would contribute to soil crusting more than the canola due to the higher content of silica within the straw. Over time however, this relationship was not clearly defined (Figure AI-2) and the only significant difference found (p-value <0.0001) was that the control had a much lower average surface resistance compared to the treatments (see Table AI-3). This conclusion in itself is somewhat of an anomaly because it has been found that generally surface residue helps protect the soil surface from raindrop impact and therefore reduces soil crusting.

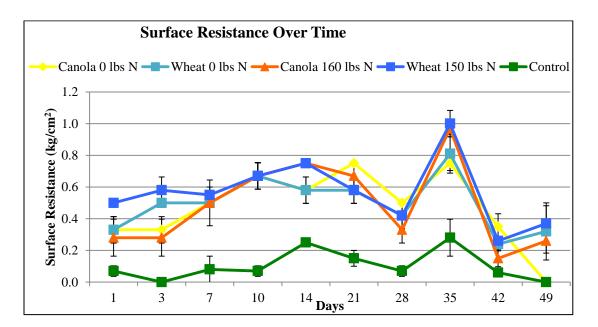


Figure AI-2. Graph of surface resistance for each treatment over time

Treatment	Water Loss (g)	рН	C:N	Surface Resistance (kg/cm ²)
Wheat 0 lbs N	47.8 a	8.49 a	20.1 a	0.55 a
Wheat 150 lbs N	48.2 a	8.40 ab	20.0 ab	0.47 a
Canola 0 lbs N	48.6 a	8.47 a	22.0 a	0.47 a
Canola 160 lbs N	48.1 a	8.39 ab	16.3 b	0.45 a
Control	48.1 a	8.34 b	19.9 ab	0.11 b

Table AI-3. Average water loss (g), pH, C:N, and surface resistance (kg/cm²) per treatment

Soil Si

Crop type seemed to have the biggest effect (p-value < 0.05) on the total Si_{am} (including Si_{ws}) as wheat 0 lbs N had the highest (393.2 mg Si/kg soil) followed by wheat 150 lbs N (349.0 mg Si/kg soil), canola 0 lbs N (341.5 mg Si/kg soil), canola 160 lbs N (336.6 mg Si/kg soil), and the control (281.4 mg Si/kg soil). When looking just at the Si_{ws} there was a significant treatment effect (p-value <0.0001). Wheat 150 lbs N showed the highest average (84.0 mg Si/kg soil) followed by wheat 0 lbs N (74.4 mg Si/kg soil), control (65.6 mg Si/kg soil), canola 0 lbs N (63.4 mg Si/kg soil), and canola 160 lbs N (62.9 mg Si/kg soil). When the Si_{ws} fraction is subtracted from the total Si_{am} wheat 0 lbs N had the highest with 317.3 mg Si/kg soil followed by canola 0 lbs N (279.4 mg Si/kg soil), canola 160 lbs N (272.4 mg Si/kg soil), wheat 150 lbs N (266.0 mg Si/kg soil), and the control (214.3 mg Si/kg soil) (see Table AI-4 and Figure AI-3). These results suggest that as wheat breaks down in the soil it releases more Si into the soil compared to canola.

Table AI-4. Mean amorphous + water soluble Si, water soluble Si, and amorphous Si per	
treatment.	

Treatment	Mean Si _{am} + Si _{ws} (mg/kg soil)	Mean Si _{ws} (mg/kg soil)	Mean Si _{am} (mg/kg soil)
Wheat 0 lbs N	393.2 a	74.4 ba	317.3 a
Wheat 150 lbs N	349.0 a	84.0 a	266.0 ba
Canola 0 lbs N	341.5 a	63.4 c	279.4 a
Canola 160 lbs N	336.6 ba	62.9 c	272.4 ba
Control	281.4 b	65.6 bc	214.3 b

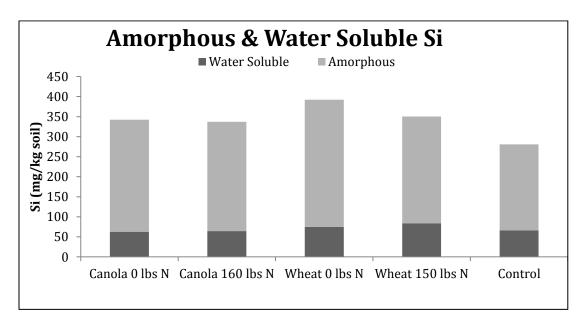


Figure AI-3. Amorphous & water soluble Si per treatment

LITERATURE CITED

- Albrecht, S.L., H.T. Gollany, R.R. Allmaras, C.L. Douglas, and S.M. Copeland. 2005.
 Tillage and nitrogen fertilizer influence on carbon and soluble silica relations in a
 Pacific Northwest mollisol. Soil Sci. Soc. Am. J. 69(4):1102-1109.
- Follett, E.A.C., W.J. McHardy, B.D. Mitchell, and B.F.L. Smith. 1965. Hemical dissolution techniques in the study of soil clays: Part1. Clay Minerals 6:23-34.
- Van der Vorm, P.D.J., 1987. Dry ashing of plant-material and dissolution of the ash in HF for the colorimetric determination of silicon. Commun. Soil Sci. Plant Anal. 18(11):1181-1189.

APPENDIX II

SEM ANALYSES AND IMAGES

INTRODUCTION

Analysis of soil samples through a scanning electron microscope (SEM) is a common practice in order to examine the structure and morphology of soil crusts (Ben-Hur and Wakindiki 2002; Dietrich et al. 2003; Farmer, Delbos, and Miller 2005; Taylor, Raupach, and Chartres 1990; Remley and Bradford 1989; Wickramasinghe and Rowell 2005). One of the objectives of this study is to utilize SEM analysis in order to get a closer look at the soil surface under various treatments. To determine the relationship between amounts of silica available in soil and soil crusting two incubations were employed during the summer of 2012.

MATERIALS & METHODS

Samples were placed in polyethylene containers, arranged in a randomized block design and stored in a room temperature oven (25°C). Each sample contained 250 g of soil. Soil was air dried and sieved through a two mm sieve prior to incubation. Every three days samples were brought up to field capacity in order to simulate a wetting and drying cycle. Samples were watered with a squirt bottle to simulate the force of rain. Prior to watering, each sample was weighed to determine the amount of water loss between wetting cycles and to ensure samples were all receiving approximately the same amount of water. Analyses that took place included: pH, total C and N, Si content, surface resistance, and crust thickness. In addition to the specific treatments for the incubations a control treatment was employed consisting of only air-dried soil. • Residue comparison (APPENDIX I)

Soil used in this incubation was a Warden series silt loam acquired from the Prosser Research Station. Two treatments were applied: wheat and canola residue. Both residues were grown at the Palouse Conservation Farm Station. The wheat was fertilized at a nitrogen rate of 150 lbs/acre and the canola was fertilized at a nitrogen rate of 160 lbs/acre. The residue was cut into small pieces ranging from two to five cm. Two g of residue was added to each soil sample and thoroughly mixed into the soil.

• Cropping history comparison (CHAPTER 4)

The soil used in this incubation was a Ritzville silt loam acquired from research plots located in Ralston, WA. The soil was collected from the top 15 cm after harvest from a field previously cropped in wheat and one previously cropped in canola. Three levels of silicic acid were applied to the corresponding soil samples: low 0.105 g/pot, medium 1.06 g/pot, and high 10.5 g/pot.

<u>SEM sample prep</u>

SEM analysis was done on samples acquired after the incubation was complete. A randomly selected sample from each treatment within all three incubations was used for SEM analysis. A sub-sample was taken from the selected specimen's before and after the final soil silica extraction. Samples were thoroughly dried and carefully removed from their containers. The crust samples were fixed to a SEM stub by firmly pressing the stub covered with a carbon tab against the targeted area of the soil crust. Loose soil and large pieces of organic matter were removed. The samples were then coated with a thin layer of gold ~40 nm in thickness using a sputter coater. This thickness was chosen after trial and

error with soil samples to determine how much gold was required to keep the samples from charging while in the SEM. Once this process was complete the samples were placed in the Hitachi SEM and micrographs were taken at 50, 100, 300, 500, and 2k times magnification.

RESULTS & DISCUSSION

Residue comparison

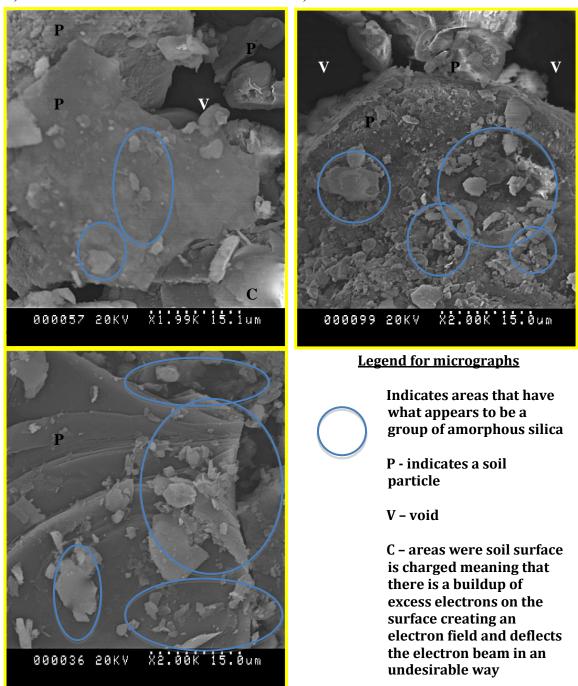
The micrographs for this incubation showed a visual difference in amorphous silica levels between treatments. The amorphous silica (Si_{am}) is considered the small particles located on the surface of the soil. Areas with higher concentrations of Si_{am} are circled and indicated on the micrographs. The control appears to have the least amount of amorphous Si and the wheat showed the most (see Figure A2-1). These images coincide with our hypothesis that as wheat decomposes it does in fact result in more amorphous silica on the soil surface. Other parameters measured in this incubation such as average soluble silica and surface resistance (the force required to penetrate the crust) depict the same pattern between treatments as seen on the micrographs. The control had the lowest values for Si and surface resistance and the wheat treatment had the highest. The pre-extraction samples (see Figure A2-1) in comparison to the post-extraction samples (see Figure A2-2) show that most of the particles seen on the soil surface were removed after the extraction therefore suggesting that what the images are showing in the pre-extraction micrographs is in fact amorphous Si.

Cropping history comparison

The micrographs from both soils showed that the amount of amorphous silica on the soil particles increased with increasing silica levels added to the two soils. When

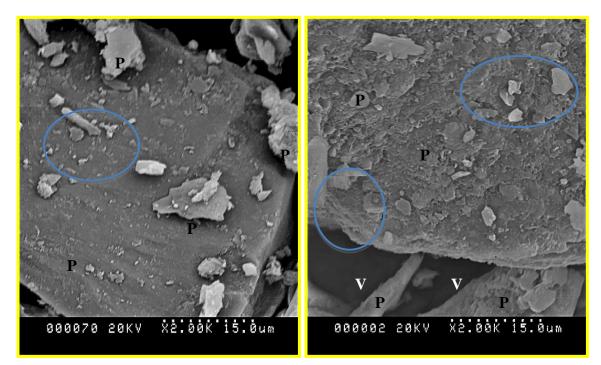
103

comparing micrographs from both soils (see figures A2-3&A2-5) it appears there is less amorphous Si present on the soil particles from the canola soil. These results suggest that the soil previously cropped with wheat in general has more silica present compared to the samples that were previously cropped in canola. This again strengthens the hypothesis that wheat residue may contribute more silica to the soil than canola. Other measurements of soil parameters during the incubation supports what is being seen on the micrographs. Surface resistance, crust thickness, and soil Si all increased from the control to the high treatment and on average the soil previously cropped in wheat had higher values for these parameters (see Chapter 4). The pre versus post-extraction micrographs for both soils showed similar results as the residue comparison. The particles seen on the soil surface in the post-extraction samples were much less than in the pre-extraction samples suggesting that the particles were amorphous silica and that the extraction process was successful. **Figure A2-1.** Pre-extraction SEM micrographs of residue comparison incubation at ~2Kx magnification.



b) Canola

Figure A2-2. Post-extraction SEM micrographs of residue comparison incubation at 2Kx magnification.



a) Control

b) Canola

c) Wheat

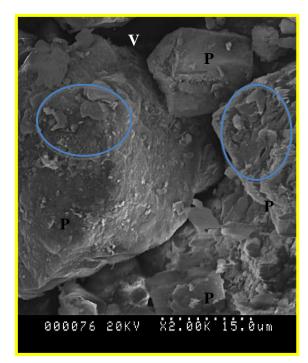


Figure A2-3. Pre-extraction SEM micrographs of the cropping history comparison incubation (previously cropped in wheat) at 2Kx magnification.

a) Control



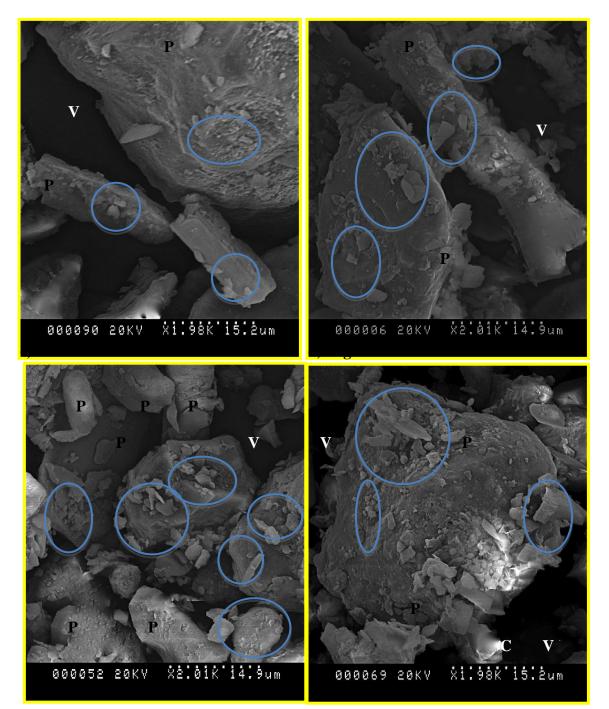


Figure A2-4. Post-extraction SEM micrographs of cropping history comparison incubation (previously cropped in wheat) at 2Kx magnification.

a) Control

b) Low

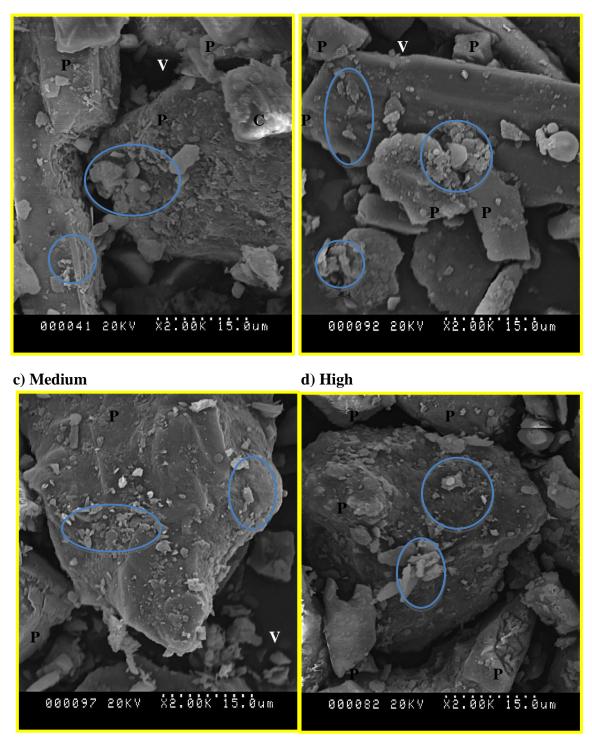
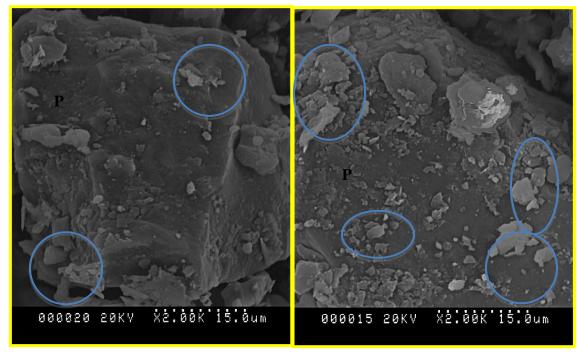


Figure A2-5. Pre-extraction SEM micrographs of cropping history comparison incubation (previously cropped in canola) at 2Kx magnification.



b) Low



c) Medium

d) High

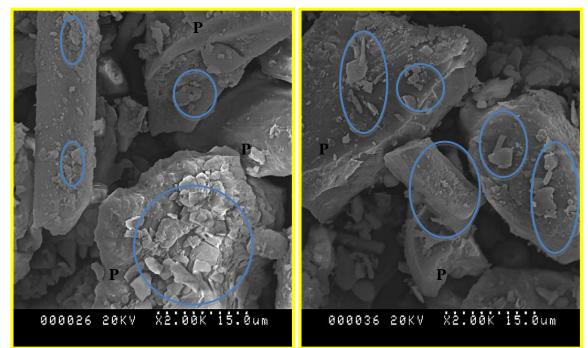
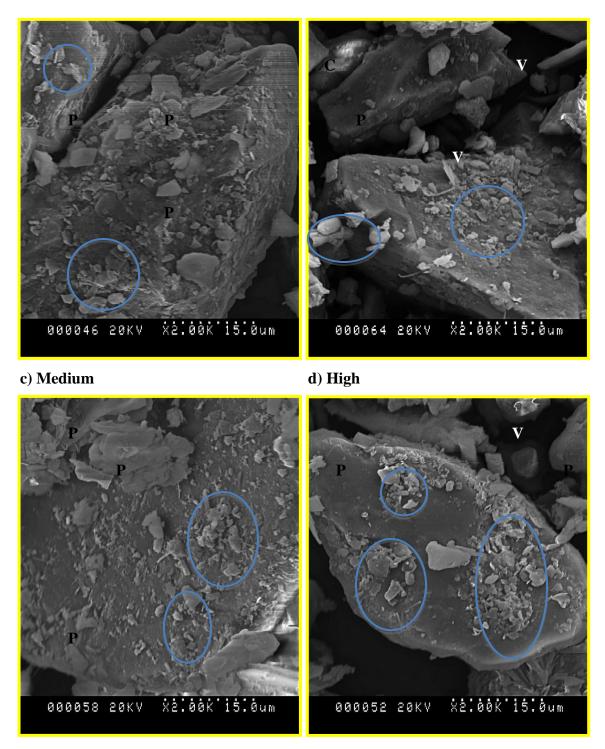


Figure A2-6. Post-extraction SEM micrographs of cropping history comparison incubation (previously cropped in canola) at 2Kx magnification.

a) Control

b) Low



CONCLUSION

The coinciding micrograph images and measurements of other soil parameters suggests that crops with higher silica levels such as wheat contribute more amorphous silica to the soil and therefore may contribute to soil crusting. When comparing the pre-extraction and post-extraction micrographs there is visual evidence suggesting that the extraction process of soil silica was successful. The micrographs also confirm that the amorphous pool of Si is the dominant form of silica affecting crust parameters. Future work with the micrographs obtained will include attempting to quantify the amounts of amorphous Si seen on the soil surface through image software.

LITERATURE CITED

- Ben-Hur, M., and I. I. C. Wakindiki. 2002. Soil mineralogy and texture effects on crust micromorphology, infiltration, and erosion. Soil Sci. Soc. Am. J. 66(3):897–905.
- Dietrich, D., S. Hinke, W. Baumann, R. Fehlhaber, E. Bäucker, G. Rühle, O. Wienhaus, and G. Marx. 2003. Silica accumulation in *Triticum Aestivum L.* and *Dactylis Glomerata L.* Analytical and Bioanalytical Chemistry 376(3):399–404.
- Farmer, V.C., E. Delbos, and J.D. Miller. 2005. The role of phytolith formation and dissolution in controlling concentrations of silica in soil solutions and streams. Geoderma 127(1-2):71–79.
- Remley, P. A., and J. M. Bradford. 1989. Relationship of soil crust morphology to interrill erosion parameters. Soil Sci. Soc. Am. J. 53(4): 1215–1221.
- Sauer, D., L. Saccone, D.J. Conley, L. Herrmann, and M. Sommer. 2006. Review of methodologies for extracting plant-available and amorphous Si from soils and aquatic sediments. Biogeochemistry 80(1):89–108.
- Taylor, R. M., M. Raupach, and C. J. Chartres. 1990. Simula-tion of soil reactions: aluminium-iron (III) hydroxy species react with silica to give deposits on particle surfaces. Clay Minerals 25(3):375–389.
- Wickramasinghe, D. B., and D. L. Rowell. 2005. The release of silicon from amorphous silica and rice straw in Sri Lankan soils. Biology and Fertility of Soils 42(3):231– 240.