

IMPACT OF CROP ROTATION AND CROP RESIDUE CYCLING ON THE
CADMIUM CONCENTRATION OF THE SUBSEQUENT CROP IN A ROTATION

BY

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

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Department of Soil Science
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**Impact of Crop Rotation and Crop Residue Cycling on the Cadmium
Concentration of the Subsequent Crop in a Rotation**

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ABSTRACT

Eastley, Lisa Marie. M.Sc., The University of Manitoba, August 2008. Impact of Crop Rotation and Crop Residue Cycling on the Cadmium Concentration of the Subsequent Crop In a Rotation. Major Professors: Cynthia Grant and Don Flaten.

Crop rotations can affect both the biological and chemical characteristics of the soil, thus affecting cadmium (Cd) availability. The release of Cd from decomposing crop residue may also influence Cd availability to the following crop. Two growth chamber studies were conducted to study the effect of crop rotation and Cd cycling from crop residue on the Cd concentration of the succeeding crop in a rotation.

The first experiment included treatments of soil where the previous crop was canola (*Brassica napus* L.) or barley (*Hordeum vulgare* L.) and the application of no straw, canola straw or barley straw, equivalent to 5000 kg residue ha⁻¹. The second experiment involved soil application of durum wheat straw equivalent to 5000 kg residue ha⁻¹, containing concentrations of Cd ranging from 50 to 217 µg kg⁻¹. In both experiments the indicator crops of flax (*Linum usitatissimum* L.) and durum wheat (*Triticum turgidum* L.) were grown as indicator crops in clay loam soil in pots, with the chopped straw added to the soil and allowed to decompose for seven weeks prior to seeding.

In both growth chamber experiments, the tissue Cd concentration was higher in flax than in durum wheat. In experiment one, Cd concentration in flax tissue increased with the application of canola and barley straw compared to when no straw was applied,

but the type of straw applied did not affect the Cd concentration in the flax. However, the Cd concentration in durum wheat was not influenced by the application of crop residue. In contrast, the soil from the previous crop influenced the Cd concentration in durum wheat, as the durum wheat Cd concentration increased when grown on the soil where canola was the previous crop. However, the soil from the previous crop of the rotation did not influence Cd concentration in flax. In experiment two, the Cd concentration of flax tissue increased with increasing Cd concentration of the applied durum wheat straw, but durum wheat tissue was not affected. In both experiments the Cd concentration in the test crop tissue was not related to the Zn concentration in crop tissue. The Cd concentration in flax, but not durum wheat may be influenced by Cd released from the decomposition of the crop residue, whereas the Cd concentration in durum wheat and not flax may be influenced by soil effects caused by the crop rotation.

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FOREWORD

This thesis has been prepared in the manuscript format in adherence with the guidelines established by the Department of Soil Science at The University of Manitoba. The Canadian Journal of Soil Science was used as the reference style for this document. Chapter one is a short introduction to explain the reasons for conducting this research project and the focus of the research. Chapter two describes an experiment that tested the effect of crop rotation and straw management on Cd concentration of durum wheat and flax tissue. Chapter three describes an experiment that tested crop straw management, alone, on the Cd concentration of the following crop. The appendices describes auxiliary experiments not directly related to the core of this thesis. Appendix A describes a survey-based field study determining the impact of a variety of soil and crop management factors on the Cd concentration of hard red spring wheat (cv, AC Barrie) across western Manitoba. Appendix B describes a comparison of two analytical methods used for determining Cd concentration in soil samples and crop tissue samples.

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1. GENERAL INTRODUCTION

Cadmium (Cd) occurs naturally in the soil, with total concentrations normally in the range of 0.1-2.0 mg kg⁻¹ (McLaughlin et al. 1999). The Cd concentrations that plants can accumulate without reducing crop growth are higher than concentrations of concern for toxicity in the human diet. Thus, consumption of high-Cd crops can allow the movement of Cd from the soil into the human food chain.

Over the long-term, consumption of excess Cd in the diet can have a number of negative health effects. Chronic Cd toxicity can lead to “Itai itai” disease (McLaughlin et al. 1999), where the reabsorption function of the kidney is decreased and osteomalacia and bone fractures can occur. The risk of Cd directly entering the human food chain via plant material prompted the World Health Organization (WHO) to establish a maximum provisional tolerable intake limit for an adult of 60 to 70 µg Cd per day (World Health Organization 1972). The concern that excess Cd might enter the human food chain in agricultural crops also led the Codex Alimentarius Commission of FAO/WHO to propose the establishment of a limit of 0.1 mg kg⁻¹ for cereal grains and oilseeds traded on international markets (World Health Organization 1972).

Many processes are involved in the movement of Cd into a crop. However, adsorption and desorption are the most important chemical processes that control Cd availability, with soil pH having a large effect on the adsorption and desorption process of Cd (Christensen and Haung 1999; Swift and McLaren 1991; McLaren et al. 1998).

Biological processes in the rhizosphere may also affect the availability of Cd to the subsequent crop in the rotation. The various processes that regulate the availability of trace element can be altered by agricultural practices. Two examples of management practices that may affect Cd concentration in Cd sensitive crops are crop rotation and crop residue management.

The use of a diverse crop rotation can be a beneficial cultural practice for grain production. Diversified crop rotations can break insect and disease cycles, manage weed populations and slow the development of herbicide resistance, reduce economic risk and improve crop yields. However, the changes in the soil environment due to the preceding crop in a rotation may influence the availability of Cd for the subsequent crop in the rotation. For example, in field studies conducted in Manitoba, flax grown after canola had higher Cd concentration compared to flax grown after durum wheat (Grant 2003), indicating that the preceding crops can somehow modify the mineral availability for the subsequent crop in a rotation. However, the role or the relative importance of crop residue or soil factors in increasing Cd concentration of the flax crop grown in this rotation was not determined.

Crop rotations can also affect the biological activity in the soil, since the population of mycorrhizae and other microorganisms depends on the crop species grown. Mycorrhiza refers to a symbiotic association between plant roots and soil fungi. The fungal components extend beyond the plant roots into the soil and increase the movement of non-mobile nutrients such as P and Zn to the plant, in return for photosynthate from the plant. However, evidence is inconsistent as to the role mycorrhizae hyphae have on Cd availability (Galli et al. 1994, Heggo et al. 1990), suggesting that many factors are

involved in the ability of mycorrhizae fungi to absorb non-essential plant nutrient such as Cd.

Crop residue management could also influence the availability of Cd to the subsequent crop in the rotation. In western Canada, straw can be retained on a field, removed from the field, or sometimes burned. Retention of straw on a field has many benefits in improving soil quality and decreasing the nutrient removal. Important nutrients are released from the decomposition of crop residue; however, the decomposition of crop residue may also release non-nutrients such as Cd. Andersson and Siman (1991) reported that Cd concentration was higher in crops where the residue had been returned to the soil as compared to where it was removed. The increased Cd concentration may be due to direct release of Cd from the residues, or changes in other soil factors affected by residue decomposition that could indirectly affect Cd availability.

Decomposition of straw may indirectly increase Cd availability through decreasing soil pH. As residues decompose, they tend to acidify the soil (Power et al. 1998). Since pH is a major factor determining Cd availability, a change of pH due to decomposition may affect Cd availability (Christensen 1984; Naidu et al. 1994). Also, the decomposition of straw will release organic acids. Organic acids can dissociate to form anions, which in turn form soluble complexes with Cd in the soil solution (Jones and Darra 1994). The formation of soluble metal organic complexes can increase the phytoavailability of Cd to plants by preventing adsorption or precipitation of Cd to soil surfaces.

In this project, growth chamber studies were conducted to evaluate the impact of crop rotation and crop residue on the Cd concentration in durum wheat and flax. The

influence of crop residue and crop rotation were measured by determining the Cd concentration of durum wheat and flax: i) following the addition of canola straw, and barley straw, on soil where canola was the previous crop or on soil where barley was the previous crop; ii) following the addition of durum wheat straw containing concentration of Cd ranging from 50 to 217 $\mu\text{g kg}^{-1}$.

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2. IMPACT OF CROP ROTATION AND CROP RESIDUE MANAGEMENT ON THE CADMIUM CONCENTRATION OF DURUM WHEAT AND FLAX

2.1 Abstract

A growth chamber experiment was conducted to determine if crop rotation or residue management could be used to manage Cd concentration of Cd sensitive crops. Flax (*Linum usitatissimum* L.) and durum wheat (*Triticum turgidum* L.) were grown in a clay loam soil, in pots with treatments of soil where the previous crop was either canola (*Brassica napus* L.) or barley (*Hordeum vulgare* L.) and with or without the addition of canola or barley straw residues. The chopped straw was added to the pots at a rate of 5000 kg ha⁻¹ and allowed to decompose for seven weeks prior to seeding. The above-ground biomass of flax and durum wheat was collected at 50 days after emergence or at complete emergence of the inflorescence on the main stem of each plant in a pot. Cadmium concentration was higher in the tissue of flax than durum wheat. The Cd concentration in the durum wheat tissue was higher when grown on the soil where canola rather than barley was the previous crop. In contrast, Zn, an element chemically similar to Cd, showed the opposite effect, with a lower concentration in durum wheat tissue grown where barley rather than canola was the preceding crop. In contrast to the effect of soil from the previous crop of the rotation, crop residue source influenced the Cd concentration of flax tissue but did not affect durum wheat. Cadmium concentration

increased with the application of canola and barley straw as compared to where no straw was added, while Cd concentration of durum wheat tissue was not affected by straw addition. Therefore, the Cd concentration in tissue of flax, but not durum wheat may be influenced by the application of straw, presumably due to the release of Cd from the decomposition of the crop residue. Durum wheat but not flax appears to be influenced by the effect of the previous crop on the soil environment.

2.2 Introduction

Cadmium is not phytotoxic at low concentrations and as a result plants can accumulate high concentrations of Cd in their tissue and grain without decreasing plant growth. Thus, plant accumulation of Cd from the soil can allow Cd to enter the human food chain directly through consumption of high-Cd plant material.

The risk of Cd entering the human food chain via plant material prompted the World Health Organization (WHO) to establish a maximum provisional tolerable intake limit for an adult of 60 to 70 μg Cd per day (World Health Organization 1972). The concern that excess Cd might enter the human food chain in agricultural crops also led the Codex Alimentarius Commission of FAO/WHO to propose the establishment of a limit of 0.1 mg kg^{-1} for cereal grains and oilseeds traded on international markets (World Health Organization 1972). Many of the crops produced in Canada, including durum wheat and flax, may exceed these proposed limits (Li et al. 1997). Therefore, it would be beneficial to develop crop management practices to reduce the concentration of Cd in crops that are a part of the human diet.

Crop rotations are an important agricultural management practice in sustainable crop production. Diverse crop rotations are beneficial in reducing diseases, weeds and insect pests, thus encouraging higher crop yields (Entz et al. 2002). Crop rotations may also be used to improve soil physical and chemical factors, promoting both higher crop yields and long-term soil quality. The changes in the soil environment due to crop rotations may also have a significant effect on the availability of Cd for the subsequent crop in the rotation. Cadmium availability in the soil solution is primarily controlled by adsorption and desorption reactions. Different agricultural crops can cause changes to the soil environment that may modify adsorption or desorption of Cd, resulting in an increase or decrease in Cd availability. For example, Oliver et al. (1993) stated that legumes could acidify the rhizosphere by releasing protons from the roots. The researchers attributed higher Cd concentrations in mature wheat grain grown after lupines as compared to wheat grown after wheat to the acidification of the soil. Oilseed crops can also acidify the soil for the succeeding crop. For example, Santonoceto et al. (2002) stated *Brassica spp.* can acidify the soil because the deep roots remove cations deep in the soil profile for production of biomass and seed. Acidification of soil has been connected to observed increases of Cd in a growing crop (Grant et al. 1997). Therefore, the choice of a crop grown within a rotation may influence the availability of Cd through effects on soil acidification.

Agricultural crops can release root exudates to improve nutrient acquisition. Canola and wheat (*Triticum spp.*) have been identified as crops that release root exudates into the soil solution (Bertin et al. 2003). The release of root exudates by canola and wheat roots could affect phytoavailability of Cd by altering soil chemistry. The

components of root exudates may decrease the pH of the soil, or may be able to form soluble complexes with Cd, increasing Cd availability to plants (Bertin et al. 2003). However, the root exudates of the crop being produced would probably have more of an affect on the Cd availability than would the root exudates of the previous crop in the rotation.

Crop rotations can also affect the biological activity of the soil, since the population of mycorrhizal fungi and other microorganisms is strongly affected by the growing plant species. The majority of plant species will form associations with mycorrhizal fungi, affecting the ability of the crop to access non-mobile nutrients such as P and Zn in the soil. All non-host crops for mycorrhizae exist within the *Brassica* and *Chenopodiaceae* families (Smith et al. 2004). As a result, canola allows minimal mycorrhizal association to develop during the growing season, while crops such as flax or wheat and barley will more readily form associations. The degree of mycorrhizal association may influence the Cd uptake by the plant (Galli et al. 1994).

Mycorrhizal association in soybeans was reported to increase the Cd concentration of the plant tissue on high Cd-contaminated soils but mycorrhizal association also increased Cd uptake on lower Cd- contaminated soils (Heggo et al. 1990). Limited information is available as to the role of mycorrhizae in Cd accumulation with crops, although it does appear there are many factors that will influence the ability of mycorrhizae to absorb Cd.

Mycorrhizae could also change Cd availability through a Cd-Zinc (Zn) interaction. Zinc and Cd are chemically similar and will compete for the same binding sites in different soil and plant systems (Grant and Bailey 1997). Therefore, factors that

increase concentrations of Zn could decrease plant uptake of Cd. Galli et al. (1994) observed that mycorrhizae enhanced wheat uptake of Zn from the soil solution. Thus, mycorrhizae may decrease Cd availability by increasing wheat uptake of Zn. For example, Grant and Bailey (1997) observed an inverse relationship between Zn and Cd concentration in flax regardless of whether the change in Zn concentration was caused by differences in soil type or by application of Zn or P fertilizer. The interaction between Zn and Cd appeared to be based solely on the Zn concentration in the plant. Therefore, the increase in crop Zn concentration as a result of mycorrhizae may decrease the crop Cd concentration.

Management of the straw produced during a crop year may also affect the availability of Cd to the subsequent crop in a rotation. Straw material can be retained or removed from a cropping system. The removal of straw from a cropping system would decrease Cd availability as the Cd contained in the straw is completely removed from the system. The retention of straw in a cropping system would contribute to the soil organic matter content of the soil, which may decrease Cd availability. For example, Jones and Johnston (1989) attributed a decrease in DTPA-extractable Cd in a manured soil to the increased organic matter content of the soil following the manure application. However, the Cd released from the decomposing straw retained in a field may increase Cd availability. Studies have reported Cd concentration was higher in crops where the residue had been returned to the soil as compared to when the residue was removed (Andersson and Siman 1991).

The impact of crop residue on Cd uptake by the following crop may also be related to the type of residue being incorporated into the soil. For example, wheat grown

on flax straw had higher concentrations of grain Cd compared to wheat seeded following fallow, spring wheat, fall rye or grain lentil (Dr. Fernando Selles unpublished). The observed increase in Cd concentration with the addition of flax straw was attributed to the higher Cd concentration of the flax residue as compared to the wheat, rye or lentil residue.

Another aspect of crop residue decomposition that may affect Cd availability is the release of organic acids. Cadmium exists in solution mostly as the divalent cation, Cd^{2+} . However, the formation of soluble complexes between organic acids and divalent Cd^{2+} can increase the phytoavailability of Cd by preventing adsorption or precipitation of Cd to soil surfaces. The availability of Cd could also be dependent on the rate of straw decomposition and the intensity of all the processes by which these residues affect Cd availability. For example, the C:N ratio of the straw and the lignin and the carbohydrate composition of the residue will affect the rate of residue decomposition (Reinertsen et al. 1984). In pot studies conducted by Janzen and Kucey (1988), the decomposition rate of different plant residues was affected by crop species and the crop nutrient content. The water content of the soil will also affect the decomposition of straw material, since soil water is essential for the activity and growth of soil organisms. Soil aeration will also affect straw decomposition, as restricted aeration could inhibit respiration of soil organisms and plant roots (Van Gestel et al. 1993).

Previous studies have shown that the preceding crop in a rotation and straw management could be responsible for increased Cd availability (Andersson and Siman 1991; Grant 2003). However, it has not been determined if it is the soil or the plant factors affected by crop rotation or straw management which is the major cause for the

observed changes in Cd concentration of the subsequent crop. Also, the magnitude of the effects of crop rotation or straw management on Cd availability has not been defined. Therefore, this study was conducted to evaluate the potential effects of preceding crop focusing on canola and barley on Cd accumulation in the subsequent crop of flax or durum, differentiating between the effects of crop residue and direct effects of crop sequence on soil characteristics.

2.3 Methods and Materials

Growth chamber studies were conducted at the Agriculture and Agri-Food Canada (AAFC) Brandon Research Station using a Newdale clay loam soil (Orthic Black Chernozemic). The soil was collected from AAFC Brandon Research Centre's Phillips Research farm (50°01'17.04 N 49°53'2.88 W). The soil collected was a composite sample from four replications of plots where the previous crop was either canola or barley. The soil was collected from the 0-15 cm depth in September, 2005 after crop harvest and tillage. The soil was dried to approximately 15% moisture and passed through a 1-cm mesh screen. Five kg of soil was placed in each individual pot in the study and the bottoms of the pots were sealed to prevent drainage.

Potassium chloride (2 M KCl) extractable NO_3^- -N and NH_4^+ -N, sodium bicarbonate extractable P and DTPA extractable Cd and Zn in the soil are reported in Table 1 (Carter 1993). The analyses were conducted using a Technicon II Autoanalyzer for NO_3^- N and P, and a Varian Spectr AA-400 graphite furnace atomic absorption spectrometer with deuterium background correction for Cd analysis. Soil pH was

measured using a soil deionized water ratio of 1/2 ww⁻¹. The pH of the soil was measured using an Orion SA 520 pH meter with a Ross “Sure Flow” electrode.

Table 2.1. The physical and chemical characteristics of the experimental soil

Characteristics	Barley soil	Canola soil
Soil Association	Newdale	Newdale
Soil Texture	Clay loam	Clay loam
Soil pH	7.00	7.54
Organic Matter (%)	4.4	5.4
CEC (cmol ⁺ kg ⁻¹)	35.0	32.5
Extractable Cd (µg kg ⁻¹)	145	147
Extractable Zn (mg kg ⁻¹)	1.13	1.20
Extractable N (mg kg ⁻¹)	9.0	11
Extractable P (mg kg ⁻¹)	15	12
Total Cd (µg kg ⁻¹)	420	460

Water-holding capacity for the soil used in the study was determined using the pill bottle method. In this method, a measured amount of soil was added to each pill bottle, duplicating the bulk density for the pot study. Water was added to each bottle to produce a range of soil moisture contents bracketing the expected water holding capacity of the soil. The bottles were checked 24 hrs after the water was added and the bottle in which the wetting front had moved to the bottom of the bottle but where excess water had not accumulated at the bottom of the bottle was considered to have a water content that is equivalent to the water-holding capacity of the soil under pot study conditions.

Canola and barley residue (straw) was collected from a research site located on the AAFC Research Centre in Brandon, MB (49°52'04.01"N 99°58'15.42"W). The canola and barley straw was collected at harvest as a composite sample of all four replicates of the same treatment. A sample of each type of straw was ground and analyzed for Cd, Zn and P (Table 2.2) after a nitric-perchloric acid digestion (Westerman 1990). Straw

collected from the field study was chopped into lengths of approximately 2 cm. The chopped straw was added to the 5 L cylindrical plastic pot and mixed thoroughly with the soil at a rate equivalent to 5000 kg straw ha⁻¹.

Table 2.2. Cd, Zn, P concentration of the canola and barley straw added to the pots

	Canola Straw	Barley Straw
Cd ($\mu\text{g kg}^{-1}$)	250	223
N (mg kg^{-1})	600	1000
P (mg kg^{-1})	642	1032
Zn (mg kg^{-1})	8.0	14.8

The chopped canola or barley straw (straw source) was mixed with soil that had grown either canola or barley as the preceding crop (soil source). An additional straw treatment was included in which straw was not added. The resulting straw and soil treatments were:

- (1) No straw into soil with barley as the previous crop
- (2) Canola straw into soil with barley as the previous crop
- (3) Barley straw into soil with barley as the previous crop
- (4) No straw into soil with canola as the previous crop
- (5) Canola straw into soil with canola as the previous crop
- (6) Barley straw into soil with canola as the previous crop

Straw decomposes between growing seasons, releasing metals and nutrients into the soil solution. The decomposition occurs for about four weeks in the fall in the period after harvest and before the ground is frozen and begins again in spring once the soil warms up. Therefore, the pots were placed in the growth chambers for a seven-week incubation period at 22°C without light, to allow straw decomposition to occur.

Flax (*Linum usitatissimum* L. cv. AC Lighting) and durum wheat (*Triticum turgidum* L. cv AC Melita) were the test crops used in this study to measure Cd bioavailability from each straw and soil treatment. Fifteen seeds of flax or durum wheat were seeded into each pot and the soil was watered to field capacity. After emergence seedlings were thinned to six per pot for both flax and durum wheat.

In all treatments, reagent grade ammonium nitrate was added at a rate of 314 mg N pot⁻¹ for durum wheat and 157 mg N pot⁻¹ for flax and reagent grade monoammonium phosphate was added at a rate of 31 mg P pot⁻¹ for both test crops. Both fertilizers were dissolved in distilled deionized water and applied in solution. Phosphate was banded beside the seed with both durum wheat and flax. Ammonium nitrate was banded below the seed with durum wheat and beside the seed with flax. An additional 157 mg N pot⁻¹ as reagent grade ammonium nitrate was added as a solution to each pot after four weeks of growth.

Two trials of the experiment were conducted to represent two growing seasons in the field. Each trial had four replications of each treatment, arranged in a randomized complete block design. The growth chamber was maintained at a 16 hr light period and a 8 hr dark period with temperature at 22°C day/15°C night. The humidity was maintained at 50%. During the growth period the treatments were monitored daily and watered to field capacity by weight once the soil moisture level decreased to 60% of the estimated water-holding capacity.

The above-ground biomass for durum wheat was collected from each pot at complete emergence of the inflorescence on the main stem of each plant. For pots where all plants did not reach the complete emergence of the inflorescence growth stage, the

above-ground plant material was collected 50 days after emergence. The above-ground plant material for flax was collected 50 days after emergence. The plant material was cut using stainless steel scissors 1 cm above the soil level. The fresh weight of the plants was measured and recorded. The plants were then dried at 50°C until the weight was constant. Then the dry weight was recorded and samples ground. Samples were digested in nitric-perchloric acid and the Zn and P concentration in digests were determined on an ARL 3520 inductively coupled plasma spectrometer (ICP). Cadmium concentration of samples was determined using a Varian AA400 graphite furnace atomic absorption spectrophotometer with deuterium background correction (Jiao et al. 2004). Also at the end of the experiment, the pH of the soil was measured for each treatment using an Orion SA 520 pH meter with a Ross “Sure Flow” electrode.

Statistical analysis was performed using the SAS General Linear Models procedure (SAS Institute, 1990). The experiment was analyzed as a factorial randomized block design. The data from the two runs were combined as they were treated as random effects. The experiment data had approximately normal distributions and skewness less than 0.5.

2.4 Results and Discussion

Flax and durum wheat both tend to accumulate relatively high concentrations of Cd in their tissue. However, in this study the overall mean Cd concentration of the above ground biomass was higher in flax (708 $\mu\text{g Cd kg}^{-1}$) than in durum wheat (492 $\mu\text{g Cd kg}^{-1}$). The difference in Cd concentration recorded between flax and durum wheat was not a result of the biomass difference because there was not a significant difference between the biomass of the two test crops ($P > 0.05$). The difference observed in the Cd

concentration for flax and durum wheat corresponds to genetic differences reported in the literature and was expected based upon previous research (Jiao et al. 2004). In this study, the focus was to determine the magnitude of crop rotation and straw management effects on Cd availability for the subsequent crop of the rotation.

In this study the preceding crop affected Cd concentration of the test crop tissue, with a significant interaction occurring between the test crop and the preceding crop that had been grown in the soil (Table 2.3). Therefore, the effects of soil source were evaluated separately for flax and durum wheat. However, there was no significant interaction between straw treatment and test crop, so the response of the two test crops to straw treatments will be discussed together.

Table 2.3. Analysis of variance showing the effects of soil and straw source treatments on Cd concentration of the test crop tissue

ANOVA			
Treatment	<i>df</i>	<i>F value</i>	<i>P > F</i>
Soil Source	1	4.85	0.0309
Test Crop	1	74.78	<0.001
Straw Source	2	0.53	NS ²
Soil Source x Test Crop	1	6.61	0.0122
Soil Source x Straw Source	2	0.84	NS
Test Crop x Straw Source	2	1.73	NS
MSE			14516
CV			20.11

²NS, not significant ($P > 0.05$)

2.4.1 Impact of soil source on the Cd concentration of durum wheat tissue. Durum wheat had a higher concentration of Cd in the above ground plant material when grown on the soil where canola was the previous crop compared to the soil where barley was the previous crop (Table 2.4). The increased Cd concentration of durum wheat grown in soil where canola rather than barley was the previous crop may be a result of biological changes in the soil environment with the different crop rotations. As research has shown, mycorrhizae can decrease or increase Cd concentration in plant tissue (Galli et al. 1994;

Heggo et al. 1990). The varying results of mycorrhizae and Cd availability demonstrate that the role of mycorrhizae in Cd availability is influenced by many factors, one of which could be the species of the previous crop. Thus, the higher Cd concentration in durum wheat following canola rather than barley may be due to the different relationship between the previous crops and the mycorrhizae population. However, the mycorrhizal populations were not measured in this study and so the role mycorrhizae had in the Cd availability in this experiment is unknown.

Another possible explanation for the previous crop's soil effect is differences in root systems of the preceding crops. The root structure of the different crops can alter the effect that the root systems will have on the chemical environment of the soil solution and so can influence the availability of Cd to the subsequent crop of the rotation. Canola plants have a tap root system with root hairs branching from the taproot while cereal crops have a fibrous root system. The taproot structure allows canola plants to access greater depths of soil compared to cereal crop root systems (McConkey 2002). The growth of canola roots deeper into the soil profile can contribute to acidification of the soil due to the cycling of cations from the lower soil profile into the top soil (Santonoceto et al. 2002). In this study the soil pH for both of the soil sources was in the neutral range, with soil pH being slightly higher where canola rather than barley was the preceding crop. However, Cd availability would be expected to decrease with a rise in soil pH; therefore, the higher Cd concentration is durum wheat grown after canola as compared to after barley was not a function of effects on soil pH (Table 2.5).

Table 2.4. Cd, Zn and P concentrations in durum wheat tissue as affected by soil source and straw source treatments.

Soil Source Treatment	Straw Source Treatment											
	Cd concentration				Zn concentration				P concentration			
	No Straw	Canola Straw	Barley Straw	Mean	No Straw	Canola Straw	Barley Straw	Mean	No Straw	Canola Straw	Barley Straw	Mean
-----($\mu\text{g Cd kg}^{-1}$)-----				-----(mg Zn kg^{-1})-----				-----(mg P kg^{-1})-----				
Barley	465	404	427	432	19.9	17.8	16.7	18.1	1957	1815	1739	1837
Canola	542	550	566	553	16.2	15.8	15.6	15.9	1522	1380	1435	1446
Mean	504	477	496		18.1	16.8	16.1		1740	1597	1587	
ANOVA	<i>df</i>		<i>P > F</i>		<i>df</i>		<i>P > F</i>		<i>df</i>		<i>P > F</i>	
Straw Treatment	2		NS ^z		2		0.0204		2		0.0314	
Soil Treatment	1		<0.001		1		0.0002		1		<0.0001	
Soil Treatment x Straw Treatment	2		NS		2		NS		2		NS	
MSE			146				3.55				27732	
CV			20.45				11.09				13.5	

^zNS, not significant ($P < 0.05$)

Table 2.5. Effect of straw source treatment and soil source treatment on soil pH of pot soil at the end of the growing period of durum wheat

Soil Source Treatment	Straw Source Treatment			
	No Straw	Canola Straw	Barley Straw	Mean
	-----(pH)-----			
Barley	6.9	7.1	7.0	7.0
Canola	7.0	7.1	7.7	7.3
Mean	6.9	7.1	7.4	
ANOVA	<i>df</i>			<i>P > F</i>
Straw treatment	2			NS ²
Soil treatment	1			<0.001
Straw treatment x Soil treatment	2			NS
MSE				3.62
CV				0.067

²NS, not significant ($P > 0.05$)

The decomposing root hairs remaining in the soil from the previous crop could have affected Cd availability to the growing test crop. Although the soil was sieved and the bulk of the root material was removed from the soil source, small pieces of root material could still remain in the soil sources used in this experiment. Decomposition of the previous crops root material could have released Cd tied up in the root material. A release of Cd into the soil solution from the previous crop root material would increase the availability of Cd to the growing crop. As a result, the amount of root material returned to the soil of the previous crop could become a factor in the Cd availability of the growing crop. Many factors will determine the root mass of a growing crop. As explained earlier, different crop species have different root structures, which can affect the amount of root material accumulated. Also environmental conditions during the growing season of the crop will influence the developing root mass. However, in this study we did not measure the amount of root material remaining from the previous crop following the process of sieving. Therefore, the contribution of decomposition of the previous crop root material to the Cd concentration of the soil solution for the growing crop is not known.

2.4.2 Impact of soil source on the Zn and Cd interaction in durum wheat. The Zn and Cd interaction was also investigated to determine the effect of changes in Zn concentration on the Cd concentration in durum wheat in this study. Zinc is an essential nutrient for crop production that can be affected by crop history (Havlin et al. 1999). Zinc concentration in durum wheat was lower when durum wheat was grown in the soil where the previous crop was canola rather than barley (Table 2.4). Therefore, the effect of the previous crop on Zn concentration was opposite to the effect on Cd concentration. In terms of the Zn and Cd interaction, the lower Zn concentration in durum wheat may have allowed greater uptake and translocation of Cd, contributing to the higher Cd concentration in durum wheat following canola.

Canola is a non-mycorrhizal crop and the lack of mycorrhizal colonization in the canola may have decreased the formation of mycorrhizae in the following durum wheat crop. Mycorrhizal fungi can increase the ability of plants to absorb immobile nutrients such as Zn. Galli et al. (1994) stated that mycorrhizal populations enhance the uptake of Zn by plants. Thus, it is possible that a decrease in the mycorrhizal population in the soil where canola was grown previously may have decreased Zn concentration in durum wheat, allowing higher accumulation of Cd. However, as mentioned previously, the mycorrhizal populations were not measured in this study, so this hypothesis cannot be confirmed or refuted.

2.4.3 Impact of soil source on the P, Zn and Cd interaction for durum wheat. The P, Zn and Cd interaction is an important interaction to consider in explaining differences observed in Cd concentration in plant tissue. As previously stated, the Cd concentration

in plants can be affected by the P and Zn interaction (Jiao et al. 2004; Grant and Bailey 1993; Grant and Bailey 1998). However, in this study both P and Zn concentrations in durum wheat were higher when grown on soil where the previous crop was barley rather than canola (Table 2.4). As a result, the higher concentrations of P after barley did not restrict the uptake of Zn and the higher Cd concentration in durum wheat following canola does not appear to be a result of changes in P availability influencing the Zn and Cd interaction.

2.4.4 Impact of soil source on the Cd concentration in flax. In contrast to the results for durum wheat, the Cd concentration in flax was not affected by the soil of the preceding crop (Table 2.6). To understand why the soil of the previous crop did not affect the Cd concentration in flax, it is important to consider other factors within the soil system that may affect the Cd availability to flax.

2.4.5 Impact of soil source on the Zn and Cd interaction in flax. The Zn and Cd interaction can influence Cd availability, with increases in tissue Zn concentration decreasing tissue Cd concentration (Choudhary et al. 1994). In this study, the Zn concentration in flax was higher when grown in soil where the previous crop was barley as compared to when it was canola (Table 2.6). However, the Cd concentration in flax was similar among all soil source treatments. Therefore, the increase in Zn concentration in flax where barley was the preceding crop was not associated with a corresponding significant decrease in Cd concentration.

2.4.6 Impact of soil source on the P, Zn and Cd interaction for flax. Another important interaction that can influence Cd concentration is the P, Zn and Cd interaction.

As stated previously, P and Zn interactions can affect Cd concentration in plant tissues, with increasing P concentration reducing Zn concentration and possibly increasing Cd concentration (Jiao et al. 2004). However, in this study the P concentration in flax was similar among all the treatments (Table 2.6). Thus, no interaction occurred between P and Zn that affected the Cd concentration in flax, similar to the observation for P with Zn and Cd in durum wheat. Therefore, flax and durum wheat reacted differently to the soil effects of the preceding crop, although a P, Zn and Cd interaction could not explain the different responses of the two crops to the soil effects.

2.4.7 Impact of straw management on Cd concentration in durum wheat and flax.

Cadmium concentration in durum wheat was unaffected by the application of straw, being similar where canola, barley or no straw was applied (Table 2.6). However, the Cd concentration of flax tissue was higher when either canola or barley straw was added to the pot as compared to the control treatment where residue was not applied (Table 2.6). Andersson and Siman (1991) reported Cd concentration was higher in barley, rye and winter wheat when the residue had been returned to the soil as compared to when it was not. The higher Cd concentration in flax with the addition of crop residue may be a result of the release of Cd from the decomposing residue increasing the soil solution Cd available for flax uptake. Addition of canola or barley straw increased the Cd concentration in flax to a similar extent ($P > 0.05$). Therefore, the crop residue effect on Cd accumulation by flax was insensitive to the type of straw added.

Table 2.6. Cd, Zn and P concentration in flax tissue, as affected by crop rotation soil treatment

Soil Source Treatment	Straw Source Treatment											
	Cd concentration				Zn concentration				P concentration			
	No Straw	Canola Straw	Barley Straw	Mean	No Straw	Canola Straw	Barley Straw	Mean	No Straw	Canola Straw	Barley Straw	Mean
	-----($\mu\text{g Cd kg}^{-1}$)-----				-----(mg Zn kg^{-1})-----				-----(mg P kg^{-1})-----			
Barley	664	717	748	710	12.9	13.6	13.0	13.2	2849	3028	2999	2959
Canola	657	775	792	741	11.4	12.4	9.8	11.2	2554	2938	2661	2718
Mean	660	746	770		12.1	13.0	11.4		2702	2983	2830	
ANOVA	<i>df</i>			<i>P > F</i>	<i>df</i>			<i>P > F</i>	<i>df</i>			<i>P > F</i>
Straw treatment	2			0.009	2			NS ^z	2			NS
Soil treatment	1			NS	1			0.0148	1			NS
Soil treatment x Straw treatment	2			NS	2				2			NS
MSE				103.63				6.24				54323
CV				21.79				20.44				15.6

^zNS, not significant ($P > 0.05$)

For example, the decomposition of the added straw might have indirectly increased Cd availability through reduction in soil pH. However, at the end of the growing period the soil pH was not significantly different when straw was added as compared to when straw was not added for both crops (Table 2.5 and 2.7).

Table 2.7. Effect of the straw source and soil source on soil pH of the pot soil at the end of the growing period for flax.

Soil Source Treatment	Straw Source Treatment			Mean
	No Straw	Canola Straw	Barley Straw	
	----- (pH) -----			
Barley	7.4	7.3	7.0	7.2
Canola	7.0	7.6	7.2	7.3
Mean	7.2	7.4	7.1	
ANOVA	<i>df</i>	<i>P > F</i>		
Straw treatment	2	NS ²		
Soil treatment	1	<0.001		
Straw treatment x Soil treatment	2	NS		
MSE		0.059		
CV		0.028		

²NS, not significant (P>0.05)

Organic acids released from the decomposing straw may also contribute to increased availability of Cd. Reber and Schara (1971) reported that the water-soluble fraction of wheat straw, which includes sugars, starch, organic acids and proteins, is readily decomposed followed by fats, waxes, resins and oils. Organic acids can dissociate to form anions, which form soluble complexes with Cd in the soil solution increasing the phytoavailability of Cd (Jones and Darrah 1994). In this study, the concentration of Cd increased in flax when straw was added as compared to when straw was not added (Table 2.6). Thus, the release of organic acids from the added crop residue may have contributed to the increased Cd concentration in flax.

Zinc is an essential nutrient and crop residues contain Zn, usually at higher concentrations than Cd (Jiao et al. 2004). Thus, the release of Zn from crop residues may

influence plant available Zn, just as Cd release from crop residues can influence Cd availability. The initial Zn concentration in canola straw was higher than in the barley straw. However, in this study, increases in the Zn concentration of the test crop were not related to the Zn concentration of the applied straw (Table 2.6.). Therefore, the initial Zn concentrations of straw did not affect Zn concentration in durum wheat tissue in this experiment.

The Zn concentration in flax was not affected by straw treatments (Table 2.6). Therefore, the increase observed in the Cd concentration of flax tissue due to the application of canola or barley straw was not due to Zn competition influencing Cd uptake or translocation. Given the lack of evidence for indirect effects of straw decomposition on the release of soil Cd, it appears that the Cd concentration in flax increased primarily due to the release of Cd from the decomposition of the straw. However, the reasons for flax responding to this release of Cd from straw and durum wheat not responding are not known.

2.5 Conclusions

The effect of preceding crop on the soil and crop residues influenced the Cd concentration in the subsequent crop. However, the Cd concentration in the two test crops of durum wheat and flax responded differently to the previous crop's soil and crop residues. Durum wheat accumulated higher concentrations of Cd when grown in soil where canola rather than barley was the previous crop. The lower level of mycorrhizae following a non-mycorrhizal crop such as canola could be the reason for the observed higher Cd concentration of durum wheat following canola as compared to barley.

Alternately, the decomposition of root material from the previous canola crop that remained in the soil source may have released Cd, thereby increasing Cd concentration in the soil solution. However, in this study the specific mechanism causing the preceding crop effects could not be determined.

The addition of crop residues from either canola or barley significantly increased Cd concentrations in the flax tissue. The increased concentration of Cd observed in flax appears to be a response to the Cd released during the decomposition of the added straw.

This study determined that previous crop's growth can alter the soil environment increasing Cd availability to the subsequent crop. Also, straw management affected Cd availability to the subsequent crop, with concentration of Cd in flax tissue increasing when crop residues were returned to the soil. However, the effect of each individual agronomic practice differed between the test crops, with durum wheat responding to changes in the soil and flax responding to changes in residue management. Thus, crop rotation and residue management practices for minimizing Cd concentrations in crops will have to be developed for each individual plant species. The increased availability of Cd is not determined singly by the previous crop's soil or crop residue but is a result of an interaction between the growing crop species, the previous crop and straw management practice.

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3. THE IMPACT OF CADMIUM RELEASE FROM DECOMPOSITION OF DURUM WHEAT RESIDUE VARYING IN CADMIUM CONCENTRATION ON THE CADMIUM CONCENTRATION OF THE SUBSEQUENT CROP

3.1 Abstract

A growth chamber experiment was conducted to determine the effect of the release of cadmium (Cd) from the decomposition of crop residue on the Cd concentration of the succeeding crop in a rotation. Flax (*Linum usitatissimum* L.) and durum wheat (*Triticum turgidum* L.) were grown in a clay loam soil in pots with application of durum wheat straw containing Cd concentrations ranging from 50 to 217 $\mu\text{g kg}^{-1}$. The plant residue was collected from two sites after the third year of a field study evaluating the impact of long-term phosphate application and concentration of fertilizer Cd on crops. The chopped straw was added to the pots at a rate of 5000 kg ha^{-1} and allowed to decompose for seven weeks prior to seeding. The above-ground biomass of the two test crops, flax and durum wheat, was harvested 50 days after emergence or at complete emergence of the inflorescence on the main stem of each plant. Cadmium concentration was higher in flax than in durum wheat. The site from which the straw originated did not influence the Cd concentration of either test crop biomass. Also, the release of zinc (Zn) and phosphorus (P) from the straw decomposition did not affect the Cd concentration in this study. Cadmium concentration of flax biomass increased with increasing Cd concentration of the durum wheat straw; however, Cd concentration of durum wheat biomass was not

influenced by the Cd concentration of the applied straw. Therefore, Cd concentration of flax, but not durum wheat may be influenced by the Cd concentration of residues from the preceding crop, with the effect presumably being a result of cycling of the Cd released from decomposition of the crop residue.

3.2 Introduction

Cadmium (Cd) is present in all agricultural soils and will accumulate in crops to concentrations proportional to the availability in the soil. The cadmium accumulated in crops can move into the human food chain when the crops are consumed. The movement of Cd from the soil into agricultural crops is regulated by soil physical and chemical properties, weather and plant species that can all be influenced by crop management practices (Grant et al. 1997)

Maintenance of straw in the field can improve soil health, by minimizing soil erosion and increasing organic matter. Crop residue is also an important source of nutrients (Black 1973; Larson et al. 1972). As well as providing essential nutrients, crop residue could also be a source for trace element contaminants such as Cd. Therefore; crop residue management is one agricultural practice that may influence Cd concentration of crops. Crop residues will return varying amounts of Cd to the soil, with the level returned being a function of the amount of residue added and the concentration of Cd that the residue contained. Producers can return or remove residue, which will influence the amount of Cd retained in the system.

In some management systems, crop residues may be removed from a field by either baling or burning the straw. If straw is baled and removed from the field, the Cd

that it contains will also be removed. Burning the straw will eliminate the bulk of the residue; however, the ashes that remain after burning will have a high concentration of Cd. Therefore, the Cd from burnt residues will be available immediately whereas the Cd in unburnt residues will be released as the residue slowly decomposes in the field. Thus, straw management is a method of crop production that might provide an opportunity to reduce Cd concentration.

The amount of Cd supplied by straw during decomposition depends on the initial concentration of Cd in the residue. Fertilizer management could influence the initial Cd concentration in crop residue. Contaminants present in some fertilizer sources can increase concentrations of unwanted metals in the soil such as Cd, resulting in enhanced uptake by crops and higher concentration in straw biomass (Jiao et al. 2004; Grant et al. 1997). Cadmium released from crop residue could increase the uptake of Cd by the following crop. Therefore, Cd uptake by a crop could increase with increased concentration of Cd in the residue of the preceding crop.

As well as having possible direct effects on Cd availability through the release of Cd from the decomposing straw, straw decomposition can lower soil pH. Soil pH is a major factor influencing the phytoavailability of Cd, as it affects the distribution of Cd between the soil and the solute (Christensen 1984; Naidu et al. 1994). Straw management may also indirectly increase Cd availability through changes to the soil solution during the decomposition process. The decomposition process will increase the concentration of organic acids present in the soil solution. Higher levels of organic acids will result in the formation of soluble complexes between Cd and organic acids, which enhances Cd availability to plants (Jones and Darrah, 1994; Nigam et al. 2002).

The overall availability of Cd in the soil solution from crop residue is influenced by the rate of residue decomposition. A wide range of factors that includes temperature, moisture, aeration and incorporation will determine the rate of decomposition (Angers and Recous 1997). The decomposition of crop residue may also indirectly influence Cd availability by influencing the nutrient balance within the soil or the plant system. Decomposing crop residue will release essential plant nutrients such as zinc (Zn) and phosphorus (P) and previous studies have shown that an interaction exists between P, Zn and Cd (Grant and Bailey 1997; Grant et al. 2000).

In summary, crop management practices in Western Canada will influence the soil and plant systems thereby influencing Cd availability. Research conducted has shown that the addition of crop residue increases Cd concentration for the subsequent crop in the rotation (Andersson and Siman 1991). Straw management may directly increase Cd availability with decomposing straw releasing the Cd tied up in the straw material, although the release of Zn and P, from the decomposing straw material may alter the balance in the soil or plant system between Zn, P and Cd. Indirectly, the decomposing plant residue may alter soil's biological or chemical environment, potentially affecting Cd availability to a growing crop. Thus, the objective of this study was to evaluate the influence of Cd released from crop residue on the Cd content of the subsequent crop focusing on two crops of economic significance in western Canada, durum wheat and flax, both of which accumulate relatively high concentrations of Cd in the seed.

3.3 Methods and Materials

Growth chamber studies were conducted at the Agriculture and Agri-Food Canada (AAFC) Brandon Research Station. The soil used in the study was collected from the 0-15 cm depth of a soil mapped as newdale clay loam, Orthic Black Chernozem in September 2005, after harvest of durum wheat and subsequent tillage at the AAFC Brandon Research Centre's Phillips Research farm (50°01'17.04 N 49°53'2.88W). The soil was air-dried and passed through a 1-cm mesh screen. The soil was analyzed for 2 M KCl extractable NO_3^- -N and NH_4^+ -N, 0.5-M sodium bicarbonate extractable P, and DTPA extractable Cd and Zn (Carter 1993). The analyses were conducted using a Technicon II Autoanalyzer for NO_3^- -N and P, and a Varian Spectra AA400 graphic furnace atomic absorption spectrophotometer with deuterium background correction for Cd analysis (Table 3.1). Soil pH was measured with a 1:2 soil:deionized water using an Orion SA 520 pH meter with a Ross "Sure Flow" electrode (Table 3.1).

Table 3.1. The physical and chemical characteristics of the experimental soil

Parameter	
Soil Series	Newdale
Soil Texture	Clay loam
Soil pH	7.4
Organic Matter (%)	4.2
CEC ($\text{cmol}^+ \text{kg}^{-1}$)	32
Extractable Cd ($\mu\text{g kg}^{-1}$)	149
Extractable Zn (mg kg^{-1})	11
Extractable N (mg kg^{-1})	7
Extractable P (mg kg^{-1})	18
Total Cd ($\mu\text{g kg}^{-1}$)	460

Water-holding capacity for the soil used in the study was determined using the pill bottle method. In this method, a measured amount of soil was added to each pill bottle, duplicating the bulk density of the pot study. Water was added at different amounts in each bottle to produce a range of soil moisture contents bracketing the expected water holding capacity of the soil. The bottles were checked 24 hrs after the water was added and the bottle in which the wetting front had moved to the bottom of the bottle but where excess water had not accumulated at the bottom of the bottle was considered to have a water content that is equivalent to the water-holding capacity of the soil under pot study conditions.

Durum wheat straw varying in Cd concentration was collected in August, 2005 from two sites after the third year of a field study examining the impact of long-term phosphate application and concentration of fertilizer Cd on crops. The first site was located at the AAFC Research Center in Brandon, MB (49°52'04.01"N 99°58'15.42"W) and is classified as a Newdale clay loam. The second site was located at AAFC Brandon Research Center's Phillips Research farm (50°01'17.04"N 49°53'12.50"W) and is classified as a Ramada clay. At each site, a composite straw sample was collected at harvest from the four replications of treatments receiving no P fertilizer or 80 kg P ha⁻¹ yr⁻¹ of monoammonium phosphate containing 211 mg Cd kg⁻¹. This provided four types of straw with a range of Cd concentrations from 50 to 217 µg Cd kg⁻¹. The straw was chopped into lengths of approximately 2 cm. A sub-sample of each type of straw was ground and analyzed for Cd, Zn and P after a nitric-perchloric acid digestion (Westerman 1990) (Table 3.2).

Origin of Straw	Cd Treatment for Added Straw ^z	Concentrations in Added Straw			
		Cd (µg kg ⁻¹)	N (mg kg ⁻¹)	P (mg kg ⁻¹)	Zn (mg kg ⁻¹)
Newdale clay loam		50	800	1043	4.9
		217	800	1131	10
Ramada clay		122	600	921	5.7
		173	700	1230	5.9

^zDurum wheat straw was collected from two sites after the third year of a field study examining the impact of long-term application of monoammonium phosphate fertilizer varying in Cd contamination on the grain Cd concentration of the growing crop. Straw from the low Cd treatment was from control plots where no fertilizer P or Cd was added; straw from the high Cd treatment was from plots that had received annual applications of 80 kg P ha⁻¹ using P fertilizer that contained a high concentration of Cd (211 mg kg⁻¹).

Five kg of soil was added to each 5 L cylindrical plastic pot. The pots were sealed to prevent drainage and leaching of nutrients. Each of the four types of chopped straw was added to the pots at a rate equivalent to 5000 kg straw ha⁻¹ and mixed thoroughly into the soil. Straw decomposition is required before the Cd present in the straw becomes available for crop uptake. Straw decomposes between growing seasons, releasing metals and nutrients to the soil solution. The decomposition occurs for approximately four weeks in the fall in the period after harvest and before the ground is frozen. In the spring, decomposition will start once the soil warms. Therefore, the pots were incubated in growth chambers for seven weeks at 22°C without light to simulate straw decomposition between growing seasons, in the fall and prior to crop seeding in the spring.

Flax (*Linum usitatissimum* L. cv Lighting) and durum wheat (*Triticum turgidum* L. cv AC Melita) were used in this study. Fifteen seeds of flax were seeded at a 2-cm depth or fifteen seeds of durum wheat were seeded at a 2.5-cm depth into each pot and

the soil was watered to field capacity. Seedlings were thinned to six per pot for both flax and durum wheat after emergence.

Reagent grade ammonium nitrate was dissolved in deionized water and banded 2.5 cm below the seed row at a rate of 314 mg N pot⁻¹ for durum wheat and 2.5 cm beside the seed-row at 157 mg N pot⁻¹ for flax in both soils. Reagent grade monoammonium phosphate was dissolved in deionized water and banded 2.5 cm beside the seed-row at a rate of 31 mg P pot⁻¹ for both crops in both soils. An additional 157 mg N pot⁻¹ as reagent grade ammonium nitrate was added as a solution applied on the surface then watered into the soil for each pot following four weeks of growth.

The growth chamber was maintained at a 16 hr light period and an 8 hr dark period with temperature at 22°C day/15°C night. The humidity was maintained at 50%. During the growth period the treatments were monitored daily and deionized water was added to each pot to restore the moisture content to field capacity once the soil moisture level had decreased to 60% of the estimate water-holding capacity of the soil field capacity, by weight.

The above ground biomass for durum wheat was collected from each pot at complete emergence of the inflorescence on the main stem of each plant. For pots where all the plants did not reach the complete emergence of the inflorescence growth stage, the above ground plant material was collected 50 days after emergence. The above ground plant material for flax was collected 50 days after emergence. The plant material was cut at 1-cm above the soil level using stainless steel scissors. The fresh weight of the plants was measured; plants were dried at 50°C until weight was constant and the dry weight was recorded. Dried plant material was ground and analyzed. Samples were digested in

nitric perchloric acid and the Zn concentration in digests was determined on an ARL 3520 inductively coupled plasma spectrometer (ICP). Cadmium concentration of samples was determined using a Varian Spectra AA400 graphite furnace atomic absorption spectrophotometer with deuterium background correction (Jiao et al. 2004). Also at the end of the experiment, the pH of the soil was measured in each treatment using an Orion SA 520 pH meter with a Ross “Sure Flow” electrode.

Two trials of the experiment were conducted independently to represent two separate growing seasons in the field. Four replicates of each treatment were included in each of the two trials. The data from the two trials were not significantly different and were treated as random effects within one combined dataset. Statistical analysis was performed using the SAS General Linear Models procedure (SAS Institute, 1996). The statistical analysis was conducted using regression analysis ProcReg and the statistical analysis of transformed data did not produce results that were different from the non-transformed data; therefore, the untransformed data was used in all analyses.

3.4 Results and Discussion

Flax and durum wheat are known to accumulate relatively high concentrations of Cd in the seed, with flax normally accumulating higher concentrations than durum wheat in both the tissue and the seed (Jiao et al. 2004; Li et al. 1997). In this study the overall mean concentration of above-ground biomass concentration of Cd in flax ($705 \mu\text{g Cd kg}^{-1}$) was higher than the Cd concentration in durum wheat biomass ($581 \mu\text{g Cd kg}^{-1}$). Since the dry biomass was not significantly different between the two test crops ($P > 0.05$) the recorded difference of Cd concentration between the test crops is not a result of

accumulated biomass. Therefore, the difference between flax and durum observed in this experiment corresponds to what is reported in the literature.

Cadmium concentration of the plant tissue was affected by the test crop, with a significant interaction occurring between the test crop and the initial Cd concentration of the added durum wheat straw (Table 3.3). Therefore, the effect of the added durum wheat straw was evaluated separately for flax and durum wheat.

Table 3.3. Analysis of variance for the influence of crop species, origin of straw and straw Cd concentration on Cd concentration of durum wheat and flax

<i>Source</i>	<i>df</i>	<i>F value</i>	<i>P > F</i>
Test crop	1	13.25	0.0007
Origin of straw	1	1.00	NS ^z
Straw Cd treatment	4	1.37	NS
Test crop x origin of straw	1	0.35	NS
Test crop x straw Cd treatment	1	7.19	0.0101
Origin of straw x straw Cd treatment	1	0.03	NS
Test crop x origin of straw x straw Cd treatment	1	2.39	0.0518
MSE		18244	
CV		20.44	

^znot significant (P > 0.05)

3.4.1 Origin of Durum Wheat Straw. In this study, straw was collected from two locations. Location of crop growth can influence the Cd concentrations of plant material (Grant et al. 1997). Differences in Cd content of the crop at different sites may relate to variation in soil characteristics such as parent material, Cd concentration, pH, salinity, organic matter content and soil texture (Grant et al. 1997). The straw collected from the Ramada clay soil site had an average Cd concentration of 148 $\mu\text{g kg}^{-1}$ whereas straw collected from the Newdale clay loam soil site had an average Cd concentration of 134

$\mu\text{g kg}^{-1}$. However, the origin of the straw did not have a significant effect on the Cd content of either flax or durum wheat (Table 3.3).

3.4.2 Impact of durum wheat residue on flax tissue Cd concentration. Application of durum wheat straw with high concentration of Cd increased the Cd concentration of the growing flax plant (Table 3.4). A regression analysis and coefficients of determination provide evidence that the Cd concentration in flax tissue was linearly related to the Cd concentration of the incorporated straw (Figure 3.1). Thus, as the Cd concentration in the durum wheat straw increased, so did the availability of Cd for uptake by the flax plant. Andersson and Siman (1991) also reported Cd concentration was higher in crops when the residue had been returned to the soil as compared to when it was not. In this experiment the relationship between the Cd concentration in the added durum wheat straw and the Cd concentration in the flax supports the conclusion that the increase in Cd concentration for the growing flax crop was due to the release of Cd from the decomposing straw (Table 3.4).

3.4.3 Impact of straw decomposition process on pH of soil. The decomposition of the added durum wheat straw may alter the soil pH, affecting the availability of Cd to the growing flax plant. During the decomposition process, the microbial community within the soil can decrease soil pH by releasing nutrients and organic acids from the plant material (Power et al. 1998). As explained previously, the soil pH has a major influence on the availability of Cd to the growing plant (Christensen 1984). However, the ANOVA for the pH at the end of this experiment showed that soil pH was not affected by the decomposition of the different types of added durum wheat straw ($P > 0.05$).

Furthermore, the major differences in soil pH among treatments were not related to the concentration of Cd in flax tissue ($P > 0.05$).

Origin of Straw	Cd treatment for added straw ²	Concentrations in flax tissue					
		Cd ($\mu\text{g kg}^{-1}$)		Zn (mg kg^{-1})		P (mg kg^{-1})	
Newdale clay loam	0 g Cd ha ⁻¹ y ⁻¹	622		4.71		2431	
	74 g Cd ha ⁻¹ y ⁻¹	772		4.78		2437	
Ramada clay	0 g Cd ha ⁻¹ y ⁻¹	663		4.41		2571	
	74 g Cd ha ⁻¹ y ⁻¹	765		4.63		2378	
Group Means							
Newdale clay loam		697		4.78		2434	
Ramada clay		714		4.52		2474	
0 g Cd ha ⁻¹ y ⁻¹		642		4.59		2501	
74 g Cd ha ⁻¹ y ⁻¹		768		4.71		2408	
<i>ANOVA</i>		<i>df</i>	<i>P > F</i>	<i>df</i>	<i>P > F</i>	<i>df</i>	<i>P > F</i>
Origin of Straw		1	NS ^y	1	NS	1	NS
Straw Cd		1	0.024	1	NS	1	NS
Origin x Straw Cd		1	NS	1	NS	1	NS
MSE		2166		3.13		2.28	
CV		20.84		38.0		25.8	

²Durum wheat straw was collected from two sites after the third year of a field study examining the impact of long-term application of monoammonium phosphate fertilizer varying in Cd contamination on the grain Cd concentration of the growing crop. Straw from the low Cd treatment was from control plots where no fertilizer P or Cd was added; straw from the high Cd treatment was from plots that had received annual applications of 80 kg P ha⁻¹ using P fertilizer that contained a high concentration of Cd (211 mg kg⁻¹).

^yNS, not significant ($P < 0.05$)

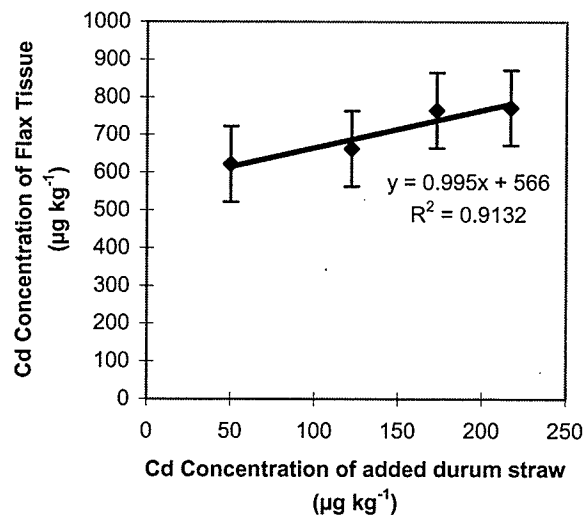


Figure 3.1. Cd concentration of flax tissue as a function of Cd concentration of applied durum wheat residue. ($P = 0.044$)

3.4.4 Impact of crop residue on the Zn, P and Cd interaction in flax. Zinc (Zn) is an essential nutrient for crop production and crop residues contain Zn, usually at a substantially higher concentration than Cd (Jiao et al. 2004). Therefore, crop residues may influence plant-available Zn, just as crop residue influences plant-available Cd. As explained previously, Zn and Cd are chemically similar and may compete for the same binding sites in different soil and plant systems (Grant and Bailey 1997). Therefore, Zn released from decomposing residue may influence the Cd accumulation in crops. However, the Zn effect on the Cd concentration of different crops can be variable. For example, Williams and David (1976) reported an increase in plant Cd concentration due to Zn application, whereas White et al. (1981) reported no effect on Cd concentration with Zn application. The differences among the results may relate to the relative Cd and Zn concentrations, soil properties and plant characteristics involved in each individual study.

In this study, the Zn concentration of the straw applied ranged from 4.9 to 10.4 mg kg⁻¹ and the Zn concentration in the growing flax tissue ranged from 4.4 to 4.8 mg kg⁻¹ (Table 3.4). The Zn concentration in the tissue of the growing flax plant was not significantly affected by the Zn concentration in the added durum wheat straw. Zinc response in the flax tissue to the Zn concentration of the added straw differed from the response of Cd in flax tissue to the Cd concentration in the straw applied. The Cd concentration in the flax tissue increased as the Cd concentration in the applied straw increased. As Zn is an essential nutrient, the plants may actively regulate Zn concentration to maintain Zn concentration within the acceptable levels, while the concentration of the non-essential Cd may not be actively regulated by the plant.

However, since the Zn concentration was not affected by the straw treatments, the observed differences in Cd concentration in flax did not appear to be due to Zn competition reducing Cd uptake or translocation.

Phosphorus (P) fertilization can reduce Zn concentration in crop biomass, resulting in Zn deficiencies for many crops, such as flax, cotton, sunflower, and wheat (Moraghan 1984, Grant and Bailey 1993, Grant and Bailey 1989). Grant et al. (2000) explain that reduced Zn concentrations could be due to the interactions between Zn and P in the soil, interference of P with the Zn uptake process, P toxicity with restricted Zn supply, or interaction within the plant leading to reduced phytoavailability. The P and Zn interaction can affect the Cd concentration in plant tissues. However, as was seen for Zn concentration in flax, the P concentration in flax was not affected by the straw treatments (Table 3.4). However, since the P and Zn concentration were similar in both treatments, a significant relationship did not exist between Zn and P; so clearly the P concentration was not affecting the Zn concentration in flax. Therefore, the observed effects of crop residue on Cd concentration in flax were not a result of changes in Zn or P concentration in the plant.

3.4.5 Impact of durum wheat residue on durum wheat tissue Cd concentration. The durum wheat straw applied to both flax and durum wheat contained Cd concentrations ranging from $50 \mu\text{g kg}^{-1}$ to $217 \mu\text{g kg}^{-1}$. The release of Cd from this straw during decomposition was expected to increase Cd availability in proportion to the straw's Cd concentration, as had occurred in the flax treatments (Table 3.4). The lack of relationship between the Cd concentration of the growing durum wheat and the Cd concentration of the added durum wheat straw may indicate that the availability of Cd in the soil solution

was not the limiting factor for uptake of Cd by the durum wheat. In contrast to the results for flax, regression analysis showed that the concentration of Cd in durum wheat was not affected by the concentration of Cd in the added straw. Therefore, the release of Cd from decomposing straw does not appear to be a major factor affecting Cd concentration in durum wheat (Table 3.5).

Origin of Straw	Cd treatment for added straw ^z	Concentrations in durum wheat tissue					
		Cd ($\mu\text{g kg}^{-1}$)		Zn (mg kg^{-1})		P (mg kg^{-1})	
Newdale clay loam	0 g Cd ha ⁻¹ y ⁻¹	592	8.91	2181			
	74 g Cd ha ⁻¹ y ⁻¹	570	10.30	2247			
Ramada clay	0 g Cd ha ⁻¹ y ⁻¹	618	10.41	2441			
	74 g Cd ha ⁻¹ y ⁻¹	595	11.32	2003			
Group Means							
Newdale clay loam		581	9.61	2214			
Ramada clay		606	10.87	2222			
	0 g Cd ha ⁻¹ y ⁻¹	605	9.66	2311			
	74 g Cd ha ⁻¹ y ⁻¹	583	10.81	2125			
<i>ANOVA</i>		<i>df</i>	<i>P > F</i>	<i>df</i>	<i>P > F</i>	<i>df</i>	<i>P > F</i>
Origin of Straw		1	NS ^y	1	0.0273	1	NS
Straw Cd		1	NS	1	NS	1	0.0428
Origin x Straw Cd		1	NS	1	NS	1	NS
MSE		146.80		2.279		6042	
CV		20.82		14.75		11.08	

^zDurum wheat straw was collected from two sites after the third year of a field study examining the impact of long-term application of monoammonium phosphate fertilizer varying in Cd contamination on the grain Cd concentration of the growing crop. Straw from the low Cd treatment was from control plots where no fertilizer P or Cd was added; straw from the high Cd treatment was from plots that had received annual applications of 80 kg P ha⁻¹ using P fertilizer that contained a high concentration of Cd (211 mg kg⁻¹).

^yNS, not significant ($P > 0.05$)

3.4.6 Impact of crop decomposition process on pH of the soil. As in the flax portion of the study, none of the soil or residue treatments affected the bulk soil pH at the end of the durum wheat pot experiment ($P > 0.05$). Therefore, for both crops, pH of the soil was not a significant factor affecting crop response in this study. The concentration of organic acids was probably similar in each straw treatment; therefore, Cd availability in each straw treatment would be affected equally by the concentration of organic acids.

3.4.7 Impact of crop residue on the Zn, P and Cd interaction in durum wheat. As with flax, the Zn concentration in the durum wheat tissue was not related to the concentration of Zn within the durum wheat straw added in this experiment ($P > 0.05$). Therefore, there was no evidence for a Zn and Cd interaction. Since there is no Zn interaction with Cd then the indirect interaction between P and Cd through P influence on Zn is also not observed in this study ($P > 0.05$).

3.5 Conclusions

Durum straw with varying concentrations of Cd was added to soil to determine if the Cd present in the straw influenced Cd accumulation by durum wheat and flax. The Cd concentration in flax tissue increased with increasing Cd concentration in the added straw. The close relationship between the Cd concentration in the flax and the Cd concentration in the applied straw supports the conclusion that the release of Cd from the decomposing straw residue directly increased the Cd concentration of the flax. Other factors that could be affected by decomposition of straw material, such as soil pH or organic acid concentration, did not significantly affect the Cd concentration in the flax tissue. Therefore, the release of Cd from the added straw apparently increased the availability of Cd to flax. However, durum wheat Cd concentration did not increase with the increased concentration of Cd in the applied straw. The difference in the Cd concentration response of the two crops was a result of genetic differences that were not specifically evaluated in this trial.

The release of plant-available Cd through decomposition of straw appears to be a major factor increasing Cd concentration in flax. Therefore, agronomic practices that can

decrease the amount of straw remaining on a field might reduce plant-available Cd and reduce the Cd concentration of flax or possibly in other high-Cd accumulating dicotyledenous plant species, in crops where high Cd concentration is of concern. A similar strategy for reducing Cd uptake in durum wheat does not appear to be warranted.

3.6 References

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4. OVERALL SYNTHESIS

The role of crop rotation and crop residue on Cd concentration of durum wheat and flax was evaluated in two growth chamber studies. The concentration of Cd in flax tissue increased with the addition of either barley or canola straw, as compared to where residue was not added, but showed no difference in Cd concentration if grown on soil where canola was the previous crop as compared to soil where barley was the previous crop. In contrast, the Cd concentration in durum wheat was not affected by the addition of crop residue, but the Cd concentration significantly increased by soil-mediated effects of crop rotation. The Cd concentration of the durum wheat had a higher Cd concentration when grown on soil where canola was the previous crop compared to soil where barley was the previous crop. A subsequent study confirmed that Cd concentration of durum wheat was unaffected by addition of durum wheat straw with widely varying Cd concentrations, while Cd concentration of flax increased with increasing Cd concentration in the applied straw. The response of flax tissue Cd to the Cd concentration of the residue supports the conclusion that crops residues can increase the Cd concentration of flax by release of Cd into the soil solution from the decomposing residues.

In both growth chamber experiments, crop residue increased the Cd concentration of flax but not of durum wheat. The difference between the two species may be related to a range of factors including the production of root exudates, the concentration of Cd accumulated, the different strategies of obtaining trace elements from the soil, or possible

translocation of Cd from the roots to the shoots by a dicotyledonous plant as compared to a graminaceous plant. Determination of the specific reasons causing the difference between the two species was beyond the scope of this study. However, in this study we determined that the significance of crop management on the Cd concentration of the subsequent crop depends on the type of crop that is to be grown in the following year.

It is important to have a better understanding of the impact that crop rotation and straw management will have on the Cd concentration of the subsequent crop in the field. As society becomes more health conscious and quality of grain entering domestic and foreign markets is becomes more closely scrutinized, the potential to minimize Cd concentration in high Cd accumulating crops at low costs (i.e., crop rotation planning or straw removal) would become more appealing to producers.

The increased production of high accumulating crops such as soybeans in Western Canada, and the movement of flax seed into the health food market also increase the need to be able to control Cd concentrations of the crops grown in Western Canada. Thus, the opportunity of decreasing Cd availability through the proper planning of crop rotation or the removal of straw is very appealing as growers in Western Canada are able to apply these management practices relatively easily if there is an economic benefit. For example, crop residue removal can be done simply with baling the straw remaining on the field after harvest. Crop rotations are currently designed to minimize disease, insect and herbicide resistant pressure and to manage economic risk; thus, the potential to minimize Cd concentration through proper crop planning could be an additional factor for farmers to consider when deciding on the cropping plans for a certain field in their farming operation.

The growth chamber study proved that crop rotation and straw management can influence Cd concentration in certain crops; however, the growth chamber is a very controlled environment. The restricted rooting volume and controlled environmental conditions may accentuate differences in Cd concentration. The relationship between crop rotation and straw management should be studied in field experiments to determine if the effects observed in pot studies also occur under field conditions.

Further studies are needed to investigate Cd phytoavailability following canola and to determine if the depressed mycorrhizal population is a major factor involved with the observed higher concentration in the subsequent crop in a rotation following canola. Also, the specific factors leading to the difference between durum wheat and flax response to crop residue Cd should be determined, since the two crops consistently reacted differently to the addition of crop residues in this study.

This thesis provides another important step to understanding the availability of Cd in soil. Previous researchers have speculated that the crop history and crop residue were responsible for the increased Cd concentration observed in some situations. This thesis confirms that the crop rotation and crop residue cycling influence the Cd concentration in the subsequent crop and that this influence with the crop species being grown.

5. APPENDICES

5.1 Appendix A

A review of a survey based field study determining the impact of crop rotation and straw management on the Cd concentration of the Cd concentration in hard red spring wheat (cv, AC Barrie)

A.1. Abstract

A survey was completed for each individual field of AC Barrie grown in southwestern Manitoba for Warburtons Family Bakery Ltd. in the 2005 growing season. In the fall of 2005, soil and grain samples were collected from each individual field involved in the study and analyzed for Cd concentration.

A multivariate analysis of the data set did not identify any strong correlations between soil characteristics or cropping practices and grain Cd or soil Cd concentration. The characteristics used in this analysis were grain Cd concentration, soil Cd concentration, location of field, tillage type, previous crop, soil series, soil texture, total N, P, total S, K, Ca, Mg, Zn, Cu, Fe and Mn.

A.2 Introduction

It is important to determine agronomic management practices that can reduce Cd availability. In the previous studies it was found the Cd concentration of durum wheat was significantly increased when durum wheat was grown on soil when canola was the

previous crop compared to when barley was the previous crop. Also the data collected from previous studies showed that the Cd concentration in flax increased as the Cd concentration of crop residue increased. However, these observations occurred in growth chamber studies where many factors were controlled and the variability of the study was minimized. Results of growth chamber studies are therefore not necessarily representative of the results that may occur in the field. Although crop rotation and straw management had a significant influence on the Cd availability in a growth chamber study, in a field scale study many factors may minimize or overcome the effect of crop rotation or straw management on the availability of Cd.

Fertilizer management in different crop systems can also significantly influence Cd availability and can differ from growth chamber to field situations. Fertilizers are a primary input for most cropping systems. The application of commercial synthetic fertilizers will alter the natural chemical processes and contaminants present in some fertilizer can increase concentrations of unwanted metals in the soil (Jiao et al. 2004; Grant et al. 1997). However, in growth chamber studies reagent grade fertilizer are commonly used. This grade of fertilizer minimizes the concentration of contaminants compared to commercial fertilizer and presents another factor that is controlled in the growth chamber that is not at the field scale level.

The objective for this study was to evaluate the potential influence of management practices on Cd accumulation at a field scale. It would be beneficial for producers to be able to select management practices that would reduce the Cd concentration of sensitive crops. An integrated management program that would decrease

Cd availability is a very important step to improve grain quality, thereby possibly opening new marketing opportunities.

A.3 Methods and Materials

A survey was completed on the producer's management practices for 36 fields of AC Barrie A hard red spring wheat variety grown under contract to Warburtons Family bakery Ltd. in the 2005 growing season. The survey included questions on fertilizer application, straw management from the previous year and the previous crop of the particular field.

In the fall of 2005, soil samples were collected from each individual field in the study. A random sampling pattern was followed on each field. Twenty to twenty-five subsamples were combined together to generate one composite sample from each field for the 0-15 cm depth and one for 15-30 cm. The samples were dried at room temperature for a week, then ground. The samples were analyzed for sodium bicarbonate extractable N, P and DTPA extractable Cd and Zn (Carter 1993). The analyses were conducted using a Technicon II Autoanalyzer (NO₃-N, P) and a Varian SpectrAA-400GFAAS with deuterium background correction (Cd).

Grain samples were collected by Warburtons. Each producer is required to send samples to the company to determine baking quality. Therefore, Warburtons sub-sampled 200 g from the growers' grain samples. The grain was then ground and analyzed. Samples were digested using nitric-perchloric acid and analyzed for nutrient content with an ICP and for Cd using a Varian AA with a graphite furnace (Jiao et al. 2004).

A multivariate analysis of the data set did not identify any strong correlations between soil characteristics or cropping practices with grain Cd or soil Cd. The characteristics used in this analysis were grain Cd, soil Cd, location of field, tillage type, previous crop, soil series, soil texture, pH, conductivity, total N, P, total S, K, Ca, Mg, Zn, Cu, Fe and Mn.

The initial analysis conducted on the collected data included 36 fields, representing eleven different soil series and eleven different soil textures. Therefore, high variability due to the large number of soil series and soil textures in the data set could conceal relationships between Cd concentrations in the crop and either soil or management factors of interest. Therefore, to minimize the variation occurring in the data set, a second multivariate analysis was conducted which included only fields in which the region was identified to have a soil series of Newdale or Oxbow with a soil texture of loam to clay loam. The Newdale soil series was isolated from the original data set because this was the most common soil series in the original data set as six fields were classified under the Newdale soil series. An additional six fields from the Oxbow series were selected from the original data set because this association is classified as a loam to clay loam glacial till similar to the clay loam soil for the Newdale soil series. A second multivariate analysis was then conducted on these twelve fields; however, again no distinct relationship was observed between any soil or agronomic characteristic and grain Cd or soil Cd concentration.

A.4 Results and Discussion

A.4.1 The Effect of Crop Rotation on Cadmium Concentration of AC Barrie Wheat. No distinct relationships between the previous crop or the tillage practice and the grain Cd or soil Cd concentration were observed under field conditions (Figures C.1 and C.2). The absence of a distinct pattern may be related to variability within the field. The fields chosen for sampling belonged to farmers that grew AC Barrie spring wheat for Warburtons Family Bakery Ltd. and were willing to participate in the study. The samples were collected from fields located across western Manitoba, from many different soil series and soil textures. The different fields would also have had different long-term management practices and weather. As a result, the variability within the data was high, thus the actual relationship between Cd concentration and management practices may be obscured by differences in these other environmental factors.

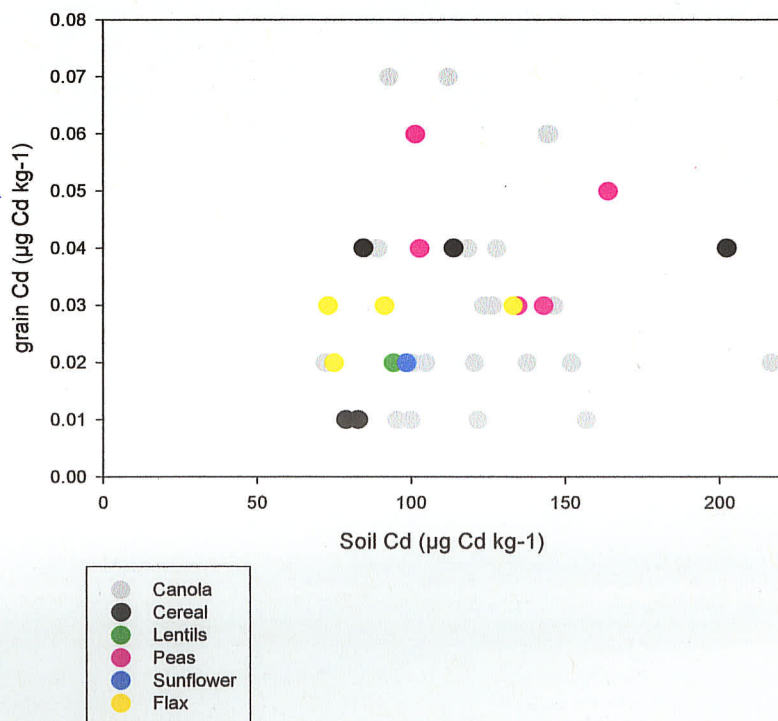


Figure A.1. Grain Cd concentration as a function of soil Cd concentration for various crops. The y variable is grain Cd ($\mu\text{g kg}^{-1}$), the x variable is the soil Cd ($\mu\text{g kg}^{-1}$)

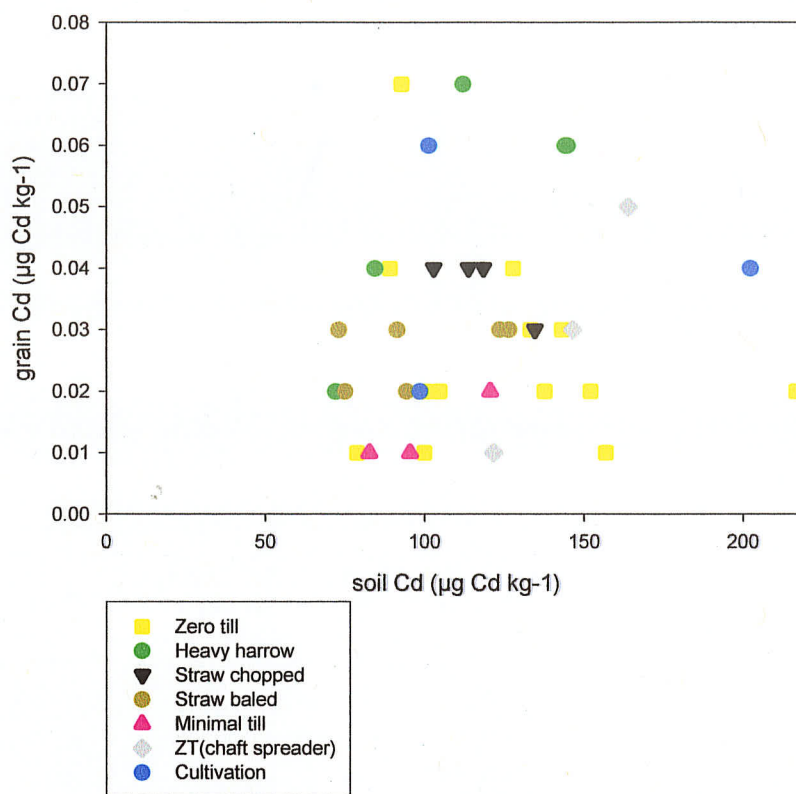


Figure A.2. Grain Cd concentration as a function of soil Cd concentration for various tillage operations.

The large area which the study included had various environmental conditions during the 2005 growing season. Grant et al. (2000) reported environment influenced the concentration of Cd and Zn of flax seed. Rainfall and temperature average were not consistent between the regions in which the fields were located. Therefore, the weather of the different regions where the fields were located could also have affected Cd concentration.

Management practices altering the soil environment of individual fields may also have contributed to variation of Cd availability. Fertilizer application will affect soil chemical processes and the contaminants present in some fertilizer sources can increase

concentrations of unwanted metals in the soil (Jiao et al. 2004; Grant et al. 1996).

Phosphate fertilizers are a major source of Cd input into agricultural systems. In the current study, P fertilizer was applied to every field in this study; however, the rates varied substantially from field to field. Therefore, the different rate of fertilizer applied may have contributed to the variability of Cd within the experiment.

Monammonium phosphate fertilizer was the most common type of P fertilizer used by the growers involved in this study. However, the concentration of Cd in monoammonium phosphate fertilizer varies substantially, depending on the source of phosphate rock used for manufacturing the fertilizer. Therefore, the origin of the fertilizer may be an important factor to consider as the Cd concentration. In this study, six different sources of phosphate fertilizer may have contributed to the variability in the study, resulting in a minimization of the relationship between agronomic management strategies and Cd concentration.

Nitrogen is also a major fertilizer nutrient applied to agricultural land in Western Canada. Nitrogen fertilizer can increase the availability of Cd, although the fertilizer does not contain significant concentration of Cd (Grant et al. 1997). Increased Cd availability may be related to changes within the soil environment with the application of N fertilizer. In the current study, N fertilizer was applied using different types and amounts; therefore, Cd phytoavailability may have been affected depending upon the N fertilizer practice of the individual farmer. These are examples of the types of variability that probably obscured our ability to isolate the effect of individual factors affecting wheat grain Cd concentrations within the large survey based field-scale survey.

A.5 Conclusions

This field scale study did not show any distinct relationship between previous crops or straw management and soil or grain Cd concentration. The design of the study resulted in large variation between many factors within the 39 fields used in this study which may have contributed to the inability to find a distinct relationship between any individual factors and grain Cd concentrations. The soil physical and soil fertility, microclimate, past cropping history, fertilizer application, seed source, seeding date, seeding rate of the individual fields differed among the fields. Thus, we assumed that these cropping characteristics contributed to the variation of the experiment. The range of confounding factors presumably concealed any potential differences in Cd concentration due to the two single factors that were evaluated.

Notwithstanding these results, it may be possible to influence Cd by management practices within a particular field even though we were unable to identify the impact of these factors across a large number of fields. An individual farmer may be able to determine the best cropping or straw management practices that can minimize Cd concentration in grain produced for separate fields in his farm operation. As the individual farmer would have the knowledge of the variation in terms of soil productivity, physical properties, soil fertility practices. The variation that was presumed to have concealed the relationship in this study could be minimized or factored into the analysis on a smaller scale when the field history is known.

A.6 References

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5.2. Appendix B

Comparing the accuracy of plant and soil extraction methods for determining Cd concentration

B.1. Abstract

Different soil and plant extraction methods for Cd analysis were compared using grain and soil samples collected from western Manitoba, Canada. The total soil Cd concentration was determined by ICP-MS after utilizing two different types of digestion procedures, four-acid procedure and the aqua regia digestion procedure. The four-acid digestion procedures resulted in a higher Cd mean concentration at $0.039 \mu\text{g Cd kg}^{-1}$ compared to the Cd concentration mean of the aqua regia digestion procedure at $0.029 \mu\text{g Cd kg}^{-1}$.

The total grain Cd concentration in samples of AC Barrie a spring wheat variety collected in the fall of 2005 was determined by ICP-MS utilizing two different types of extraction procedures, ashed vegetation and microwave digestion. The microwave preparation procedure achieved a significantly higher Cd concentration mean at $0.0423 \mu\text{g Cd kg}^{-1}$ as compared to the ashed vegetation preparation method which achieved a Cd concentration mean of $0.0308 \mu\text{g Cd kg}^{-1}$.

B.2 Introduction

There are many methods accepted for determining Cd concentration in plant and soil material. The Codex Alimentarius Commission proposed the establishment of a limit

of 0.1 mg kg^{-1} for cereal grains and oilseeds traded on international markets, thus the relevance of the difference between extraction methods could become very important in the future when grain shipments can be accepted or rejected based on Cd concentrations.

The reliability of a soil extraction method depends on the ability of an extractant to extract a plant nutrients in quantities related to plant requirements and this is dependent on the reactions that control the desired trace element supply and availability.

For plant analysis, after sampling the plant material is ground and the trace metal concentration is determined following digesting or ashing of the plant material. Almas and Singh (2001) describe using dry-ashing of the plant samples followed by dissolution in a 1:2 concentrated HCl-HNO₃ mixture. However, loss of volatile elements can occur during the ashing process; therefore, some laboratories choose to use a microwave digestion, in which the digestion is conducted in an enclosed chamber, eliminating the loss of volatile elements.

The objective of this study was to evaluate the relative performance of two different extraction and sample preparation techniques for determining Cd concentrations in soil and plant material.

B.3. Methods and Materials

This study was conducted in western Manitoba Canada, in collaboration with Agricore United and Warburtons Family Bakery Ltd. In the fall of 2005, soil samples were collected from field across western Manitoba, Canada. A random sampling pattern was followed on each field. Twenty to twenty-five subsamples were combined together to generate one composite sample from each field for the 0-15cm depth and one for 15-30

cm. The samples were dried at room temperature for a week, then ground. The total soil Cd concentration was determined by the ICP-MS after utilizing two different types of digestion procedures. In the four acid procedure the samples were dissolved beginning with hydrofluoric acids (HF) , followed by a mixture of nitric (HNO₃-) and perchloric (HClO₄) , then heated using precise programmer- controlled heating in several ramping and holding cycles which take the sample to dryness. After dryness is achieved, samples are brought back into solution using hydrochloric acid (HCl) (Sheppard et al. 1996). In the aqua digest the samples were dissolved with a combination of concentrated HCl and HNO₃ acids (Activation Laboratories Ltd. Ancaster ON).

Grain samples were collected by Warburtons in the fall of 2005. Each producer is required to send samples to the company to determine baking quality. Therefore, Warburtons sub-sampled 200g from the growers' grain samples. The grain was then ground and analyzed. Total grain Cd concentration was determined by the ICP-MS after utilizing two different types of digestion procedures. Ashed digestion, samples were dry ashed at low temperature (480°C) and a proprietary acid digestion was performed on the ash. In the second procedure, the samples were prepared using an acid/microwave digestion. Statistical analysis was conducted by single-degree of freedom orthogonal contrasts, using the GLM procedure of the SAS.

B.4. Results and Discussion

B.4.1 Soil Extraction Methods. Two different soil extraction methods were used to determine the total extractable soil Cd. The ultratrace 4 digestion procedures resulted in a higher mean concentration at 0.039 µg Cd kg⁻¹ compared to ultratrace 1 at 0.029 µg Cd

kg⁻¹ (Table D.1). A t-test and a contrast analysis on the data of the two types of digestions demonstrated the ultratrace 4 digestion achieved soil Cd concentrations significantly greater than those from the ultratrace 1 digestions procedure (Table D.1). The lower concentration of Cd extracted by the ultratrace 1 digestion procedure may be a result of the extraction not being able to extract all the elements from the mineral matrices.

Table B.1. Cd concentration in soil samples ($\mu\text{g Cd kg}^{-1}$) obtained through four-acid and aqua digestion for soil samples

Soil Digestion Treatment	N	Cd ($\mu\text{g Cd kg}^{-1}$)			
		Mean	Standard deviation	Minimum value	Maximum value
Four-acid	44	0.039	0.08685	0.02	0.06
Aqua	44	0.029	0.07053	0.015	0.046
Contrast	<i>df</i>	<i>P</i> > <i>F</i>			
Four-acid vs. Aqua	1	<0.0001			
T-test		t value	<i>P</i> > t		
Four-acid vs. Aqua	44	8.95	<0.0001		

B.4.2 Plant extraction methods. Two methods were used to analyze the grain samples collected from the fields involved in the study. The analytical results were obtained by ICP-MS analysis after utilizing two different types of preparation procedures. In the ashing procedure, samples were dry ashed at low temperature (480°C) and a proprietary acid digestion was performed on the ash. In the second procedure, the samples were prepared using an acid/microwave digestion. This method is conducted in an enclosed container therefore decreasing the potential of loss of elements which exists in the ashing digestion procedure. The microwave preparation procedure resulted in a significantly higher mean concentration of 0.0423 $\mu\text{g Cd kg}^{-1}$ as compared to the ashed vegetation preparation method which achieved a mean concentration of 0.0308 $\mu\text{g Cd kg}^{-1}$.

¹ (Table D.2). The ashed sample method can be assumed to have lost some of the element during the digestion process because the procedure is not enclosed which occurs with the microwave digestion.

Plant Digestion Treatment	N	Cd concentration ($\mu\text{g Cd kg}^{-1}$)			
		Mean	Standard deviation	Minimum value	Maximum value
Ashing	37	0.031	0.017	0.01	0.07
Microwave digestion	37	0.042	0.016	0.010	0.100
Contrast	<i>df</i>	P > F			
Ashing vs. Microwave digestion	1	0.0026			
Difference	<i>df</i>	t value	Pr > t		
Ashing vs. Microwave digestion	36	4.47	<0.0001		

The difference observed between the two plant extraction methods is relevant especially in the future if the Codex Alimentarius Commission is successful in establishing the limit of 0.1mg Cd kg^{-1} for cereal and oilseed grains traded on the international market. Grain exceeding the established limit may be refused by international ports; therefore, the plant extraction method used by the country that is exporting the grain and the country importing the grain will have to be compatible for the countries to avoid trade problems on the international market, resulting in costs for the exporting company and possible with the primary producer.

B.5 Conclusions

The two soil analysis method to determine total Cd concentration used in this study differed in their recovery of soil Cd. Thus, it is important to understand that soil analyses are only a gauge of plant-available Cd within the soil. The results reported by the soil analysis are not absolute measures of the availability of Cd to the crop and may not be appropriate for comparing experimental results when different extraction methods are used. It is important to consider the type of extraction process used and if the extracts are suitable for the element of interest and the purpose of the measurement.

The two plant analysis method to determine Cd concentration used in this study differed significantly. Again it is important to understand the process and the extracts used and make sure it is appropriate for the elements of interests. The reliability of extraction methods for determining Cd concentration will become an issue in the future as the Codex Alimentarius Commission sets acceptable Cd concentration for grain traded internationally and exporting/importing countries determine the methods that will be used to determine the Cd concentration of grain that is to be exported or imported.

B.6 References

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5.3. Appendix C

Previous crop, tillage, soil series, macro and micro nutrient from 36 fields across western Manitoba.

Table C.1. The main soil series and texture of each field sampled across western Manitoba in fall 2005

Field	Soil Classification	
	Soil Series	Soil Texture
1	Beresford	Clay loam
2	Beresford	Clay loam
3	Newdale	Clay loam
	(Undulating phase)	
4	Carroll	Clay loam to fine loam
5	Oxbow	Loam to clay loam
	(smooth phase)	
6	Oxbow	Loam to clay loam
	(smooth phase)	
7	Harding	Clay to silty clay
8	Arrow Hills-Lenore Combination	
9	Carroll	Clay loam
	(Till substrate phase)	
10	Harding	Clay to silty clay
11	Souris	Light fine sandy loam
12	Heaslip Complex	texture complex
13	Glenbro	Fine sandy loam-light clay loam
14	Carroll (Heavy sub phase)	Clay loam to fine loam
15	Waskada	Heavy loam to clay loam
	(undulating phase)	
16	Waskada	Clay loam
	(smooth phase)	
17	Arrow Hills-Lenore Combination	
18	Waskada	Clay loam
	(smooth undulating)	
19	Newdale	Clay loam
20	Newdale	Clay loam
21	Newdale	Clay loam
22	Heaslip Complex	
23	Oxbow	Loam to clay loam
24	Oxbow	Loam to clay loam
25	Coulter	Silty clay to clay
26	Waskada	Clay loam
	(smooth phase)	
27	Arrow Hills-Lenore Combination	
28	Newdale	
	(Undulating phase)	Clay loam
29	Newdale	
	(Undulating phase)	Clay loam
30	Carroll	
	(Till substrate phase)	Clay loam
31	Souris	Fine loams
32	Newdale	Clay loam
33	Souris	Loamy fine sand
34	Carroll	Clay loam
35	Oxbow	
	(smooth phase)	Loam to clay loam
36	Waskada	
	(smooth phase)	Clay loam

Table C.2. The nutrient analysis of the fields sampled across western Manitoba in fall 2005

Field	Nutrients					
	Ca	Mg	Zn	Cu	Fe	Mn
	(mg kg ⁻¹)					
1	5989	1295	0.73	1.51	17.3	12.8
2	5989	829	1.22	1.1	42.1	25.5
3	4989	657	1.63	0.99	53.9	23.3
4	5230	722	0.69	0.6	33.9	11.4
5	5391	681	0.74	0.9	34.8	19.2
6	5042	994	0.89	0.8	36.3	22.6
7	6768	1143	0.56	1.9	16.7	10.1
8	5165	923	1.3	1.3	48.6	24.6
9	4688	888	1.32	1.1	43.4	20.7
10	5183	1218	0.89	1.3	39.3	17.2
11	4834	1016	0.76	0.5	27.4	15.5
12	5895	662	1.62	1.4	51	25.2
13	2408	373	0.81	0.7	75.6	28.4
14	2755	709	2.9	1.3	137.6	57.7
15	4746	500	0.95	0.7	12.3	7
16	4856	534	0.89	0.8	31.2	20.2
17	3876	548	1.17	0.5	65.6	43.1
18	5194	462	1.26	0.7	50.7	26.6
19	5688	773	0.98	0.9	43.9	28.5
20	5400	975	1.62	1.1	82.2	40.2
21	5955	1089	0.98	1.1	37.8	17.2
22	5228	718	1.9	1.0	81.6	33.3
23	4140	761	4.5	1.3	82.5	40.8
24	4745	429	1.72	1.0	39.7	19.2
25	4762	1009	1.2	1.5	49.1	20.3
26	5398	691	1	0.9	57.7	35.0
27	2890	568	1.79	0.7	178.6	133.2
28	5240	638	1.45	0.8	23.4	11.6
29	4034	650	1.43	0.9	67.5	30.3
30	4254	758	2.78	0.9	72.8	41.3
31	5073	783	0.5	0.9	18.8	12.7
32	5323	796	1.11	0.9	65.2	36.7
33	4533	565.0	0.52	0.7	17.1	8.9
34	4484	458.2	0.57	0.5	45.9	25.3
35	4588	707.7	1.12	0.8	87.2	52.5
36	5024	509.1	0.73	0.7	21.9	15.4

Table C.3. The macro nutrient analysis of the soil samples collected across western Manitoba

Field	Macro Nutrient analysis of soil collected			
	NH ₄ and NO ₃	P ₂ O ₅	SO ₄	K
	----- (mg kg ⁻¹) -----			
1	10.1	21.8	111.6	450.4
2	15.3	21.8	227.9	443.5
3	11.8	16.5	187.4	331.3
4	11.2	12.0	8.8	196.7
5	11.0	20.3	1231.7	254.9
6	9.1	15.5	1697.2	287.8
7	8.6	10.0	20.2	508.6
8	12.2	14.8	241.2	333.0
9	12.8	10.7	203.9	275.5
10	13.6	9.8	731.4	346.2
11	25.7	9.0	412.5	373.5
12	116.0	18.8	1312.6	391.5
13	8.1	11.0	18.7	170.9
14	22.9	34.3	728.4	475.7
15	13	12.5	160.9	277.9
16	18.2	15.3	97.8	460.5
17	10.6	17.3	735.5	320.4
18	47.4	12.3	50.2	365.0
19	10.2	13.5	15.6	244.4
20	13	31.5	392.9	337.5
21	34.5	16.5	524.1	281.2
22	31.3	20.0	692.5	415.5
23	37.1	16.5	146.3	512.0
24	7.7	13.8	37.9	255.9
25	18.1	18.8	16.6	368.9
26	12.7	10.5	443.4	403.7
27	10.4	24.8	8.6	395.6
28	17.1	10.5	1129.4	316.2
29	8.6	10.8	5.6	277.2
30	17.7	30.3	308.7	493.7
31	16.4	8.3	1658.4	252.8
32	13.0	14.3	13.3	341.5
33	12.1	12.0	363.3	163.6
34	5.8	18.3	1027.8	135.3
35	8.0	13.3	180.1	184.4
36	17.4	19.5	79.9	343.6

5.4. Appendix D

Soil and grain Cd concentrations of 36 fields across western Manitoba

Table D.1. The soil Cd concentration and the grain Cd concentration for the soil samples collected across western Manitoba

Field	Soil	Grain
	-----($\mu\text{g Cd kg}^{-1}$)-----	-----($\mu\text{g Cd kg}^{-1}$)-----
1	152	0.02
2	145	0.06
3	118	0.04
4	100	0.02
5	126	0.03
6	124	0.03
7	93	0.07
8	122	0.01
9	146	0.03
10	95	0.01
11	72	0.02
12	105	0.02
13	112	0.07
14	217	0.02
15	89	0.04
16	138	0.02
17	100	0.01
18	121	0.02
19	128	0.04
20	157	0.01
21	144	0.06
22	99	0.02
23	202	0.04
24	84	0.04
25	83	0.01
26	114	0.04
27	79	0.01
28	135	0.03
29	103	0.04
30	164	0.05
31	101	0.06
32	143	0.03
33	75	0.02
34	73	0.03
35	91	0.03
36	133	0.03

5.5. Appendix E
Soil Characteristics of 36 fields across western Manitoba

Table E.1. Soil characteristics of the soil samples collected from across western Manitoba

Field	Soil Characteristics	
	PH	Conductivity ----- $(ds\ m^{-1})$ -----
1	7.4	0.57
2	7.6	0.82
3	7.5	0.67
4	7.4	0.48
5	7.7	1.33
6	7.7	4.09
7	7.5	0.5
8	7.4	0.52
9	7	0.84
10	7.7	0.65
11	7.6	1.11
12	7.3	2.94
13	6.3	0.28
14	6.5	1.27
15	7.8	0.73
16	7.7	0.63
17	7.7	1.14
18	7.5	1.27
19	7.9	0.48
20	7.5	0.93
21	7.7	0.96
22	7.4	1.04
23	7.3	0.95
24	7.7	0.86
25	7.4	0.53
26	7.8	1.06
27	6.9	0.38
28	7.5	0.76
29	7.3	0.4
30	7.2	0.46
31	7.9	1.65
32	7.8	0.51
33	7.8	1.04
34	7.5	3.17
35	7.7	0.86
36	7.7	0.58

5.6. Appendix F

The average and standard deviation of Cd concentration in relation to the previous crop grown in 2004 for 36 fields across western Manitoba

Table F.1. Soil Cd concentration in relation to the previous crop grown					
Crop	N	Soil			
		Cd Concentration	Standard Deviation	Minimum value	Maximum value
------(mg L ⁻¹)-----					
Canola	22	122	30	72	216
Cereal	5	112	52	78	202
Lentils	1	94	0	94	94
Peas	5	129	26	101	163
Sunflowers	1	98	0	98	98
Flax	4	93	27	73	133

5.7. Appendix G

Contrast analysis of Soil two different digestion procedure for determining the Se concentration of Hard Red Spring Wheat (cv. AC Barrie) and the Average Se concentration of Hard Red Spring wheat in relation to the crop grown in 2004

Soil Digestion Treatment	N	Se (mg kg ⁻¹)			
		Mean	Standard deviation	Min. value	Max value
Four-acid	44	0.8	0.2	0.3	1.2
Aqua	44	0.7273	0.159	0.4	1.0
Contrast	<i>df</i>	<i>P > F</i>			
Four acid vs. Aqua	1	0.403			
T-test	N	t value	<i>P > t </i>		
Four-acid vs. Aqua	44	0.92	0.3628		

Crop	N	Soil
		Se Concentration ----- (mg L ⁻¹) -----
Canola	22	0.004
Peas	5	0.003
Flax	4	0.009
Cereal	5	0.003
Lentils	1	0.003
Sunflowers	1	0.003

Table G.3. The average Grain Se concentration in relation to the previous crop grown

Crop	N	Grain
		Se Concentration -----(mg kg^{-1})-----
Canola	22	0.29
Peas	5	0.25
Flax	4	0.20
Cereal	5	0.29
Lentils	1	0.51
Sunflowers	1	0.20

5.8. Appendix H

The eight highest grain and soil Se samples in relation to the soil type

Table H.1. The eight highest grain Se concentrated sample related to the major soil type of the field from which the sample was collected

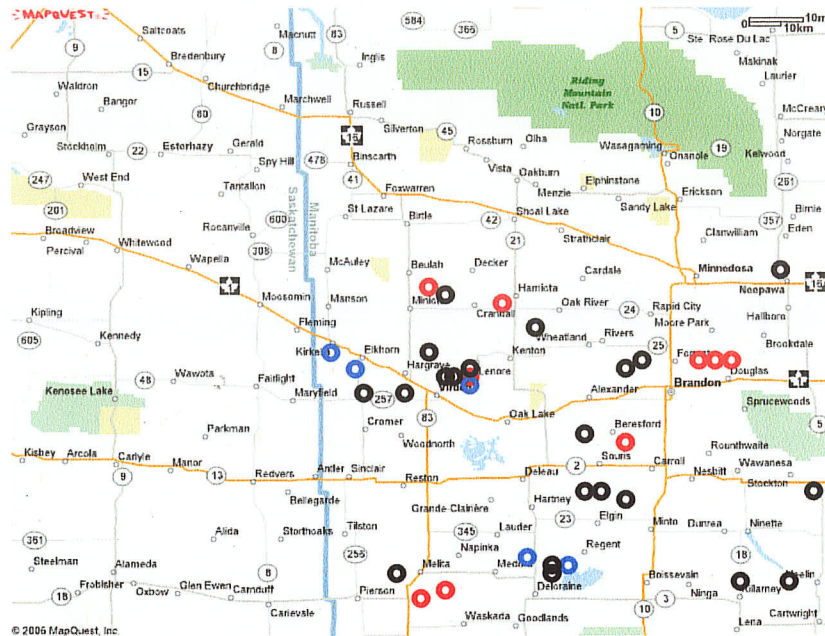
Field	Grain Se	Soil Type
	-----(mg kg^{-1})-----	
9	0.48	Carrol Clay Loam
19	0.54	Newdale Clay Loam
20	0.47	Newdale Clay Loam
25	0.38	Coulter Silty Clay
	0.51	Newdale Clay Loam
32	0.43	Newdale Clay Loam
10	0.53	Harding Clay
11	0.38	Souris light fine Sandy Loam

Table H.2. The eight highest soil Se samples reported with the major soil type of the field

Field	Soil Se	Soil Type
	----- (mg L^{-1})-----	
10	0.01	Harding Clay
34	0.03	Carroll Clay Loam
15	0.01	Waskada Heavy Loam to Clay Loam
11	0.01	Newdale Clay Loam
3	0.01	Newdale Clay Loam
28	0.01	Newdale Clay Loam

5.9. Appendix I

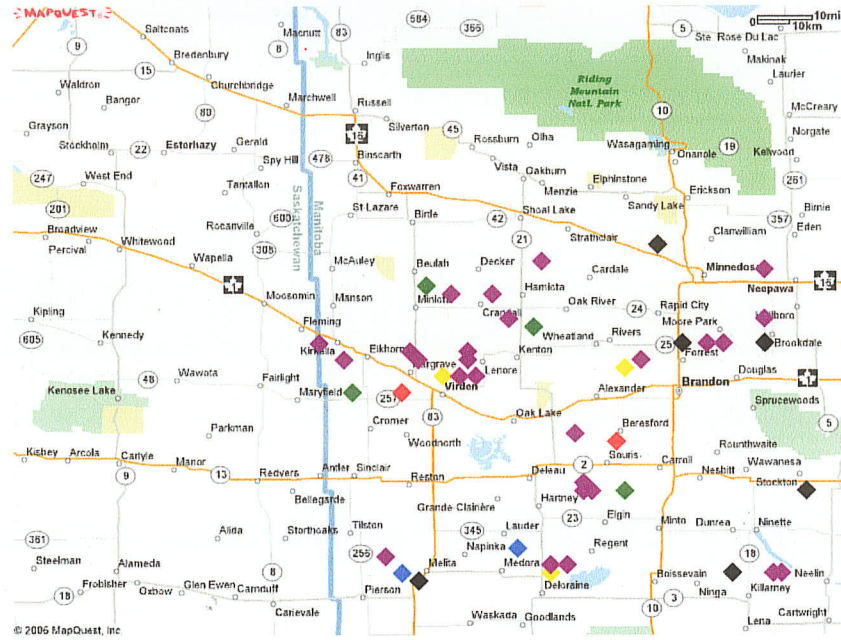
A vision representation of the Se grain and soil concentration across western Manitoba



Grain Se Levels

- 0.5-0.4 mg kg⁻¹
- 0.3-0.2 mg kg⁻¹
- <0.2 mg kg⁻¹

Figure I.1. A map representing the concentration of grain Se in accordance to the area in western Manitoba



Soil Se Levels

- ◆ 0.01-0.02 mg Se L⁻¹
- ◆ 0.008-0.009 mg Se L⁻¹
- ◆ 0.006-0.007 mg Se L⁻¹
- ◆ 0.004-0.005 mg Se L⁻¹
- ◆ 0.002-0.003 mg Se L⁻¹
- ◆ 0.001 mg Se L⁻¹

Figure I.2. A map demonstrating the concentration of Se in the soil samples collected in the fall of 2005.