

# Nutrient Dynamics and Phytoextraction in Soils Receiving Long-term Manure Application

by

Ikechukwu Vincent Agomoh

A Thesis Submitted to the Faculty of Graduate Studies of

The University of Manitoba

in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Department of Soil Science

University of Manitoba

Winnipeg

Copyright © 2017 by Ikechukwu Vincent Agomoh

## ABSTRACT

Agomoh, Ikechukwu Vincent. Ph.D., The University of Manitoba, February, 2018. Nutrient dynamics and phytoextraction in soils receiving long-term manure application. Major Professor: Francis Zvomuya.

Inclusion of dried distillers grains with solubles (DDGS) in cattle diets may influence the release of plant-available nitrogen (N) from the resulting manure (DGM) relative to manure from cattle fed a regular grain diet (RM). This may be further exacerbated by the use of construction and demolition waste (CDW) components as bedding in beef cattle feedlots.

Laboratory and greenhouse experiments were conducted to determine the effects of RM and DGM containing CDW or peat moss on canola (*Brassica napus* L.) growth, N uptake and the mineralization of N in a Black Chernozem and a Brown Chernozem. The presence of CDW in DGM and RM manure reduced cumulative dry matter yield (DMY) and plant N uptake relative to manure without CDW, while the presence of peat increased DMY and N uptake. Results from the two experiments showed that the addition of CDW decreased organic N mineralization, which may necessitate the application of synthetic N fertilizer to supplement N from CDW-amended DGM and RM manure. Nitrogen mineralization in the DGM- and RM-amended Black Chernozem followed mixed first-order and zero-order kinetics. In general, more organic N was mineralized from RM and DGM at higher temperature, but the mineralization rate constants were not affected by temperature.

We also examined the effectiveness of six 40-day cycles of barley, canola, corn, oat, pea, soybean, and triticale at extracting N and phosphorus (P) from a Dark Brown Chernozem that had received 38 annual applications of manure at  $180 \text{ Mg ha}^{-1} \text{ yr}^{-1}$  (wet wt.). Cereal grains and canola

were more effective in extracting soil nutrients than the legumes. Nutrient phytoextraction was greater at when soil moisture was maintained at 100% than at 50% SFC.

Partial least squares (PLS) regression analysis showed that soil properties measured in 2003 at the end of 30 yr of annual manure application adequately predicted barley grain yield for up to 7 yr following discontinuation of manure application. Our results indicated no evidence of convergence in barley grain yield among the manure treatments 7 yr after discontinuation of manure application, which reflects the high soil nutrient concentrations and the persistence EC effects.

## **ACKNOWLEDGEMENTS**

I will like to express my gratitude to my supervisor, Dr. Francis Zvomuya, for the guidance, advice and encouragement he provided throughout my time as his doctoral student. I see myself extremely blessed to have a supervisor who cared so much about my research, and who responded to my questions promptly. I will also like to thank the members of examination committee; Dr. Xiyang Hao, Dr. Yvonne Lawley, and Dr. Wole Akinremi, for their constant contribution and guidance towards the successful completion of my PhD program. I appreciate Dr. Jeff Schoenau for serving as my external examiner. Special thanks as well to Dr. Monica Benke for compiling part of the data used in chapter 5. I am grateful to the many reviewers who provided helpful comments for improving chapters 2, 3, 4, and 5 when they were submitted for publication in peer-reviewed journals. Many thanks to the Agriculture and Agri-Food Canada (AAFC)'s Research Affiliate Program through the A-base peer review program of AAFC for their financial support. I will like to thank the Faculty of Graduate Studies for providing to me financial awards (International Graduate Students Supplementary Scholarships and Conference Travel Awards).

Thanks to Clarence Gilbertson, David Pearson, Andrew Olson, Brett Hill, Pamela Caffyn, Stephen Tkach, Juliane Buhler, Matt Richards, Timothy Coates, Rob Ellis, Tim Stem, Frauke Fehrmann and Cathy Bay for their technical support. Special thanks to Toby Entz for providing assistance with the experimental design for chapters 3 and 4. Thanks to the staff and graduate students at the Department of Soil Science, University of Manitoba.

I appreciate Dr. Donald Thompson for all your support, kindness and giving me a ride daily to the AAFC research centre when I lived in Lethbridge, Alberta. To my mum and siblings, I appreciate your continual prayers and supports. To my dad, Mr. Friday Agomoh (deceased), you left during the early days of my Ph.D program and I appreciate the good training you gave my siblings and I. You

taught us that life is full of good times and unpleasant times and having a good attitude in the face of adversity will make an individual to overcome any challenge. You will always be remembered. Finally, special thanks to my wife, Fehintoluwa, for her constant support, patience, prayer, and love and to our precious daughter, Amy you are a great blessing to me and will always be.

## FOREWORD

This thesis was prepared in manuscript format in accordance with the thesis guidelines of the Department of Soil Science, University of Manitoba.

Chapter 2 has been published:

**Agomoh, I., Zvomuya, F., Hao, X. Akinremi, O.O. and McAllister, T. A. 2017.** Nitrogen mineralization in Chernozemic soils amended with manure from cattle fed dried distillers grains with solubles. *Soil Sci. Soc. Am. J.* doi:10.2136/sssaj2017.08.0282.

Chapter 3 has been published:

**Agomoh, I., Zvomuya, F., Hao, X. and McAllister, T. A. 2017.** A bioassay of nitrogen availability in soils amended with manure from cattle fed DDGS: Effects of construction/demolition waste and peat moss. *Can. J. Soil Sci.* doi:10.1139/cjss-2017-0031.

Chapter 4 has been published:

**Agomoh, I., Hao, X. and Zvomuya, F. 2017.** Phytoextraction of nitrogen and phosphorus by crops grown in a heavily manured Dark Brown Chernozem under contrasting soil moisture conditions. *Int. J. Phytorem.* doi:10.1080/15226514.2017.1319333.

Chapter 5 has been accepted for publication:

**Agomoh, I., Zvomuya, F., Hao, X. and Benke, M. 2017.** Modeling barley in a Dark Brown Chernozem following discontinuation of long-term manure application. *Soil Sci. Soc. Am. J.*

# TABLE OF CONTENTS

	Page
ABSTRACT.....	ii
ACKNOWLEDGEMENTS .....	iv
FOREWORD .....	vi
TABLE OF CONTENTS.....	vii
LIST OF TABLES .....	xi
LIST OF FIGURES .....	xiii
1. GENERAL INTRODUCTION.....	1
1.1. Dried Distillers Grains with Solubles in Cattle Diets .....	2
1.2. Forms of Nitrogen in Manure and the Use of Nitrification Inhibitors .....	3
1.3. Construction and Demolition Waste and Peat Moss .....	4
1.4. Nutrient Removal in Heavily Manured Soils .....	6
1.5. Predicting Crop Yield in a Heavily Manured Soil .....	8
1.6. Thesis Outline .....	10
1.7. References .....	10
2. NITROGEN MINERALIZATION IN CHERNOZEMIC SOILS AMENDED WITH MANURE FROM CATTLE FED DRIED DISTILLERS GRAINS WITH SOLUBLES .....	19
2.1. Abstract .....	19
2.2. Introduction .....	20
2.3. Materials and Methods .....	22
2.3.1. Soil .....	22
2.3.2. Manure .....	23
2.3.3. Experimental Design and Setup.....	24
2.3.4. Calculations and Statistical Analysis .....	26
2.4. Results and Discussion.....	29
2.4.1. Nitrogen Mineralization Kinetics for the Black Chernozem .....	29
2.4.2. Mineralized Nitrogen Concentration in the Brown Chernozem .....	37
2.5. Conclusions .....	38
2.6. References .....	39

3. A BIOASSAY OF NITROGEN AVAILABILITY IN SOILS AMENDED WITH MANURE FROM CATTLE FED DDGS: EFFECTS OF CONSTRUCTION/DEMOLITION WASTE AND PEAT MOSS.....	44
3.1. Abstract .....	44
3.2. Introduction .....	44
3.3. Materials and Methods .....	46
3.3.1. Organic Amendment .....	46
3.3.2. Soils.....	47
3.3.3. Experimental Design and Setup.....	47
3.3.4. Leaching Events .....	48
3.3.5. Harvesting .....	48
3.3.6. Laboratory Analysis.....	49
3.3.7. Calculations.....	50
3.3.8. Statistical Analysis.....	51
3.4. Results .....	52
3.4.1. Manure and Soil Properties.....	52
3.4.2. Cumulative Dry Matter Yield .....	52
3.4.3. Cumulative Nitrogen Uptake by Canola.....	61
3.4.4. Amendment-derived Nitrogen Uptake.....	63
3.4.5. Apparent Nitrogen Recovery .....	64
3.4.6. Soil Available Nitrogen .....	66
3.4.7. Apparent Organic Nitrogen Mineralization .....	69
3.4.8. Potentially Leachable Nitrate Nitrogen .....	71
3.5. Discussion .....	74
3.6. Conclusions .....	78
3.7. References .....	78
4. PHYTOEXTRACTION OF NITROGEN AND PHOSPHORUS BY CROPS GROWN IN A HEAVILY MANURED DARK BROWN CHERNOZEM UNDER CONTRASTING SOIL MOISTURE CONDITIONS.....	82
4.1. Abstract .....	82
4.2. Introduction .....	82
4.3. Materials and Methods .....	84



4.3.1.	Soil Collection and Analysis.....	84
4.3.2.	Experimental Setup.....	84
4.3.3.	Laboratory Analysis.....	85
4.3.4.	Calculations.....	86
4.3.5.	Statistical Analysis.....	87
4.4.	Results .....	87
4.4.1.	Initial Soil Properties .....	87
4.4.2.	Change in Dry Matter Yield .....	88
4.4.3.	Change in Nitrogen Uptake .....	92
4.4.4.	Soil Available Nitrogen .....	93
4.4.5.	Change in Phosphorus Uptake .....	94
4.4.6.	Soil Available Phosphorus.....	95
4.4.7.	Cumulative Nitrogen Uptake .....	95
4.4.8.	Cumulative Phosphorus Uptake.....	96
4.5.	Discussion .....	97
4.6.	Conclusions .....	101
4.7.	References .....	101
5.	MODELING BARLEY YIELD IN A DARK BROWN CHERNOZEM FOLLOWING DISCONTINUATION OF LONG-TERM MANURE APPLICATION .....	105
5.1.	Abstract .....	105
5.2.	Introduction .....	106
5.3.	Materials and Methods .....	108
5.3.1.	Site Description.....	108
5.3.2.	Soil Collection .....	109
5.3.3.	Laboratory Analysis.....	110
5.3.4.	Weather Data .....	110
5.3.5.	Statistical Analysis.....	111
5.4.	Results and Discussion.....	113
5.4.1.	Precipitation and Irrigation .....	113
5.4.2.	Soil Properties.....	113

5.4.3. Grain Yield Trends and Changes in Soil Properties after Discontinuation of Manure Application.....	116
5.4.4. Predicting Grain Yield following Discontinuation of Long-term Manure Application... 122	
5.4.5. Predicting Grain Yield for the Control Plot.....	126
5.5. Conclusions .....	132
5.6. References .....	133
6. OVERALL SYNTHESIS .....	138
6.1. Relevant Findings and Implications of the Research .....	138
6.2. Recommendations .....	143
6.3. References .....	145
APPENDICES .....	148
Supplementary Information: Chapter 5 .....	148

## LIST OF TABLES

Table	Page
Table 2.1 Initial properties of soils and organic amendments used for the incubation study.....	23
Table 2.2 Kinetic models used in describing N mineralization in a Black Chernozem amended with beef cattle manure. ....	27
Table 2.3 Amendment and temperature effects on mixed first-order and zero-order kinetic model parameters for net N mineralization ( $N_{min}$ ) in a manure-amended Black Chernozem. ....	31
Table 2.4 Amendment and temperature effects on first-order kinetic model parameters for net N mineralization as a percentage of initial organic N concentration ( $\%N_{min}$ ) in a manure-amended Black Chernozem and the $Q_{10}$ function. ....	34
Table 2.5 Amendment, temperature, and incubation time effects on cumulative net N mineralized in Black and Brown Chernozems.....	35
Table 3.1 Initial properties of soils and organic amendments used in the experiment. ....	54
Table 3.2 Chemical properties of peat moss and construction and demolition waste mixed with manure before applying to soil. ....	55
Table 3.3 Treatment effects on canola cumulative dry matter yield (CDMY), cumulative crop N uptake (CNU), amendment-derived N uptake (AmNU), apparent nitrogen recovery (ANR), soil available N (SAN), organic N mineralized ( $N_{min}$ ), potentially-leachable $NO_3-N$ (PLN), and cumulative PLN in Chernozems amended with REG manure.....	56
Table 3.4 Treatment effects on canola cumulative dry matter yield (CDMY), cumulative crop N uptake (CNU), amendment-derived N uptake (AmNU), apparent nitrogen recovery (ANR), soil available N (SAN), organic N mineralized ( $N_{min}$ ), potentially-leachable $NO_3-N$ (PLN), and cumulative PLN in soils amended with DDGS manure. ....	58
Table 4.1 Selected initial physicochemical properties of the soil used in the greenhouse study. ....	88
Table 4.2 Effects of moisture on soil available nitrogen (SAN), soil available phosphorus (P), percent change in dry matter yield ( $\Delta DMY_1$ ), nitrogen ( $\Delta NU_1$ ), and phosphorus ( $\Delta PU_1$ ), and cumulative dry matter yield (CDMY), cumulative nitrogen (CNU) and phosphorus uptake (CPU) of crops grown in a previously heavily manured soil. ....	89

Table 5.1 Descriptive statistics of soil properties measured at the start of the experiment in 1973 and in 2003 after discontinuation of manure application. ....	115
Table 5.2 Soil properties as affected by historical (30 yr) manure application and the number of years elapsed since discontinuation of manure application.....	116
Table 5.3 Variable importance in the projection (VIP) and regression coefficients (b) for soil variables measured in 2003 and used in models for predicting barley grain yield during the 8 yr (2003-2010) following discontinuation of 30 yr of manure application.....	124
Table 5.4 Variable importance in projection (VIP) and regression coefficients (b) values for soil parameters measured in 1973 and used to predict barley grain yield in the control plots.....	128

## LIST OF FIGURES

Figure	Page
Figure 2.1 Mixed first-order plus zero-order kinetic model fits for cumulative N mineralization in a Black Chernozem amended with regular manure (RM) from cattle fed a typical beef cattle finishing diet and manure from cattle fed a DDGS diet (DGM).....	30
Figure 2.2 Temperature effects on first-order kinetic model fits for cumulative N mineralized as percent of organic N applied (%N <sub>min</sub> ) in a Black Chernozem amended with regular manure (RM) from cattle fed a typical beef cattle finishing diet, manure from cattle fed a DDGS diet (DGM), and DGM mixed with CDW (DGM <sub>CDW</sub> ).....	32
Figure 2.3 Effects of temperature on the first-order N mineralization rate constant in a Black Chernozem amended with manure from cattle fed a typical beef cattle finishing diet (RM), manure from cattle fed a DDGS diet (DGM), and DGM mixed with construction and demolition waste (DGM <sub>CDW</sub> ). Error bars represent standard errors of the mean, and bars followed by different letters differ at P < 0.05. ....	33
Figure 2.4 Cumulative N mineralized as a percentage of initial organic N concentration (%N <sub>min</sub> ) of beef cattle manure applied to a Brown Chernozem during the 240-d incubation period. ....	37
Figure 3.1 Cumulative dry matter yield, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across soils for each amendment and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM <sub>p</sub> , RM <sub>CDW</sub> , DGM, DGM <sub>p</sub> , DGM <sub>CDW</sub> , and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at P < 0.05.....	60
Figure 3.2 Cumulative N uptake, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across soils for each amendment and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM <sub>p</sub> , RM <sub>CDW</sub> , DGM, DGM <sub>p</sub> , DGM <sub>CDW</sub> , and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and	

CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at  $P < 0.05$ .....62

Figure 3.3 Amendment-derived N uptake, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across amendment for each soil and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM<sub>p</sub>, RM<sub>CDW</sub>, DGM, DGM<sub>p</sub>, DGM<sub>CDW</sub>, and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at  $P < 0.05$ .....64

Figure 3.4 Apparent N recovery, averaged across nitrapyrin rates for each amendment and soil [(a) and (b)] and averaged across amendment for each soil and nitrapyrin application [(c) and (d)] for five growth cycles of canola grown in Chernozemic soils amended with RM, RM<sub>p</sub>, RM<sub>CDW</sub>, DGM, DGM<sub>p</sub>, and DGM<sub>CDW</sub>, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet; and subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively. Error bars represent standard errors of the mean, and bars followed by different letters differ at  $P < 0.05$ .....65

Figure 3.5 Soil available N (i) averaged across soils with (a) and without (b) nitrapyrin addition; (ii) averaged across nitrapyrin in the Black Chernozem (c) and in the Brown Chernozem (d) amended with RM, RM<sub>p</sub>, RM<sub>CDW</sub>, and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, the subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is a mineral fertilizer. Vertical bars represent standard errors of the mean.....67

Figure 3.6 Soil available N in a Black Chernozem [(a) and (b)] and a Brown Chernozem [(c) and (d)] amended with DGM, DGM<sub>p</sub>, DGM<sub>CDW</sub>, and FERT, with [(a) and (c)] or without [(b) and (d)] nitrapyrin application, where DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively, and FERT is a mineral fertilizer. Vertical bars represent standard errors of the mean. ....68

Figure 3.7 Cumulative organic N mineralization ( $N_{min}$ ) averaged across nitrapyrin in a Black Chernozem (a) and a Brown Chernozem (b) and averaged across nitrapyrin application and growth cycles for each amendment and soil (c) following amendment with RM, RM<sub>p</sub>, RM<sub>CDW</sub>, DGM,

DGM <sub>p</sub> , and DGM <sub>CDW</sub> , where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, and subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively. Error bars represent standard errors of the mean, and bars followed by different letters differ at $P < 0.05$ .	70
Figure 3.8 Potentially leachable NO <sub>3</sub> -N (PLN), averaged across nitrapyrin rate in a Black Chernozem [(a) and (c)] and a Brown Chernozem [(b) and (d)] amended with RM, RM <sub>p</sub> , RM <sub>CDW</sub> , DGM, DGM <sub>p</sub> , and DGM <sub>CDW</sub> , where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet; and subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively. Vertical bars represent standard errors of the mean.	72
Figure 3.9 Cumulative PLN, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across soils for each amendment and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM <sub>p</sub> , RM <sub>CDW</sub> , DGM, DGM <sub>p</sub> , DGM <sub>CDW</sub> , and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at $P < 0.05$ .	73
Figure 4.1 Changes in dry matter yield relative to Cycle 1 ( $\Delta\text{DMY}_1$ ) for crops grown in a previously heavily manured soil. Averaged across moisture regimes, the mean dry matter yields in Cycle 1 were 9.32, 9.21, 26.2, 9.13, 4.32, 1.49, and 7.15 g kg <sup>-1</sup> soil for barley, canola, corn, oat, pea, soybean, and triticale, respectively. Vertical bars represent standard errors of the mean.	91
Figure 4.2 Changes in N uptake relative to Cycle 1 ( $\Delta\text{NU}_1$ ) for crops grown in a previously heavily manured soil. Averaged across moisture regimes, the mean N uptake in Cycle 1 were 220, 242, 503, 230, 94, 38, and 230 mg kg <sup>-1</sup> soil for barley, canola, corn, oat, pea, soybean, and triticale, respectively. Vertical bars represent standard errors of the mean.	92
Figure 4.3 Soil residual N by various crops grown repeatedly in a previously heavily manured soil under (a) 100% and (b) 50% soil field capacity. Vertical bars represent standard errors of the mean.	93
Figure 4.4 Change in P uptake (a) relative to Cycle 1 ( $\Delta\text{PU}_1$ ) and soil test (Olsen) P concentration (b) for various crops grown in a previously heavily manured soil. Averaged across moisture regimes,	

mean P uptake values in Cycle 1 were 31.3, 35.6, 69.4, 44.3, 8.02, 7.54, and 30.5 mg kg <sup>-1</sup> soil for barley, canola, corn, oat, pea, soybean, and triticale, respectively. Vertical bars represent standard errors of the mean. ....	95
Figure 4.5 Cumulative N (CNU), and P uptake (CPU) by six cycles of barley, canola, corn, oat, pea, soybean, and triticale grown in a heavily manured soil at 50% and 100% soil field capacity (SFC). Error bars represent standard errors of the mean. Bars with the same letter are not significantly different ( $\alpha = 0.05$ , Turkey's test). ....	96
Figure 5.1 Annual growing season precipitation, amount of irrigation water applied, and average growing season temperature during the eight years (2003-2010) following discontinuation of 30 yr of manure application. The 30-yr (1981-2010) average growing season precipitation is 220 mm and the average growing season temperature is 16°C (Environment Canada 2016). ....	114
Figure 5.2 Barley grain yield during the 7 yr following discontinuation of 30 yr of annual beef cattle manure applications at different rates. ....	117
Figure 5.3 Trends in coefficient of variation (CV) of barley grain yield during the 7 yr following discontinuation of 30 yr of annual beef cattle manure application. ....	119
Figure 5.4 Soil nitrate N (NO <sub>3</sub> -N) concentration in the 0-60 cm soil layer during the 5 yr following discontinuation of 30 yr of annual beef cattle manure application at different rates. ....	120
Figure 5.5 Soil test P concentration in the 0-15 cm soil layer during the 5 yr following discontinuation of 30 yr of annual beef cattle manure applications at different rates. ....	122



## **1. GENERAL INTRODUCTION**

The ability of agricultural soils to supply essential nutrients is vital for crop production. However, the soil's capacity to supply these essential nutrients may be limited due to a decline in the soil organic matter content (Dick, 1992; Reeves, 1997). In order to replenish the soil nutrient reserve following crop harvest, synthetic fertilizers or organic amendments such as livestock manure, compost or crop residue are usually applied on agricultural fields (Schoenau and Davis, 2006). Apart from supplying nutrients, livestock manure has been shown to improve soil physical properties as well (Miller et al., 2002, 2015). Miller et al. (2002) studied the effects of beef cattle manure application on soil physical properties such as soil temperature, bulk density and penetration resistance. The authors reported that soil bulk density and penetration resistance decreased with increasing soil organic carbon from manure application. Organic amendment application has also been found to increase soil microbial populations, thereby improving nutrient cycling processes (Dick, 1992; Marschner et al., 2003; Saviozzi and Cardelli, 2014).

Large volumes of manure are generated annually, especially from concentrated animal feeding operations (CAFO) (Simard et al., 1995). In Canada, there are currently 13.2 million head of cattle (Statistics Canada, 2016), which is a 2.3 percent increase from the 12.9 million head of cattle in 2011 (Statistics Canada, 2011). The majority of Canada's cattle feedlot operations are in the western provinces, mostly in Alberta (Vergé et al., 2008; Olson et al., 2009; Beauchemin et al., 2010). The large amounts of manure generated annually from these feedlots pose a serious challenge with respect to disposal (Volf et al., 2007). Traditionally, manure is spread on agricultural fields in the vicinity of the feeding operations due to the high cost involved in hauling manure over long distances (Larney et al., 2006; Volf et al., 2007).

### **1.1. Dried Distillers Grains with Solubles in Cattle Diets**

Bioethanol production in Canada was 1.75 billion liters in 2016, which represents a 1.4 percent increase from 2015 production levels of 1.725 billion liters (EPM, 2016; USDA Foreign Agricultural Service, 2016). In the United States, approximately 55.6 billion liters of ethanol were produced in 2015 (RFA, 2016). Corn is the main feedstock used in the production of ethanol in the United States and eastern Canada, while wheat is commonly used in western Canada (Li et al., 2011; Hünerberg et al., 2013b). This steadily growing bioethanol industry generates large amounts of dried distillers grains with solubles (DDGS) as a by-product. Approximately 0.26 million tonnes of wheat DDGS per year were produced by Canadian ethanol plants in 2009/2010, with an estimated value of \$51 million (International Grains Council, 2010). The DDGS is typically fed to feedlot animals (Li et al., 2011; FOBI Network, 2013), thus modifying the diet typically fed to these animals. Most cattle feedlots in Canada include either wheat- or corn-based DDGS in the cattle diets (Li et al., 2011; Hünerberg et al., 2013b). Diet modification, along with feeding practices, affect manure properties such as pH and ion composition (Hao et al., 2009; Hünerberg et al., 2013a). The starch content of DDGS is low because most of the starch in the grain is fermented to ethanol during the dry milling process, while concentrations of protein, fibre and minerals, which are unchanged chemically during the process, are almost three times those in the original grain (Klopfenstein et al., 2008; Meyer et al., 2010; Liu, 2011; Hünerberg et al., 2013a). Typically, DDGS is included in the finishing cattle ration at 40% of diet dry matter with no negative impact on the growth performance of the animal (Gibb et al., 2008; Klopfenstein et al., 2008; Walter et al., 2010). A typical western Canadian beef finishing diet consists of 85% barley grain, 10% barley silage, and 5% mineral supplement (Hao et al., 2009).

Although DDGS is an excellent source of protein, energy and fibre for ruminants, studies have shown that its inclusion in cattle diets results in increased N and P concentrations in manure,

which may pose a risk to the environment when land applied (Klopfenstein et al., 2008; Hao et al., 2009; Hünérberg et al., 2013a). Hao et al. (2009) reported a positive correlation between manure total P (TP) and  $\text{NH}_4\text{-N}$  and the feed TP and crude protein fed to cattle in a feedlot. Research has shown that dietary N and P intake influences the forms and concentrations of N and P excreted by dairy and beef cattle (Ebeling et al., 2002; Hao et al., 2009; Hristov, 2013; Kebreab et al., 2013). Often, the partial replacement of barley grain with DDGS in beef rations results in the protein requirements of the animal being exceeded, resulting in increased excretion of both fecal and urinary N relative to cattle that are fed barley grain diets (Hao et al., 2009; McGinn et al., 2009; Hünérberg et al., 2013a).

## **1.2. Forms of Nitrogen in Manure and the Use of Nitrification Inhibitors**

Ammonium ( $\text{NH}_4$ ) and urea N are the two main forms of N in livestock manure that are immediately available for plant uptake and also susceptible to loss to the environment following land application of manure (Egball, 2000). Through the nitrification process,  $\text{NH}_4$  in manure is converted to nitrate ( $\text{NO}_3$ ), which may be susceptible to leaching, especially in a medium or coarse textured soil when manure application is followed by significant precipitation (Paul and Beauchamp, 1994; Calderón et al., 2004; Olson et al., 2009). Ammonium N in the manure is also subject to loss through  $\text{NH}_3$  volatilization or N immobilization, making the inorganic N unavailable for plant uptake (N'Dayegamiye and Isfan, 1991; Paul and Beauchamp, 1994; Hao et al., 2009).

Minimizing or blocking nitrification is one approach to enhancing N availability when manure is land applied. Studies have shown that nitrification inhibitors (NI), such as nitrapyrin, increase the efficiency of fertilizer N use and the yield of canola (Bailey, 1990). Nitrapyrin has also been found to increase fertilizer N recovery by spring wheat (Crawford and Chalk, 1993). Nitrification inhibitors work by delaying the conversion of  $\text{NH}_4\text{-N}$  by *Nitrosomas* spp. to  $\text{NO}_3$ ,

prolonging the availability of N in the root zone (Wolt, 2000, 2004; Subbarao et al., 2006). Nitrification inhibitors have been effective with inorganic N fertilizer (Ronaghi et al., 1993; Peng et al., 2015) and manure (Calderón et al., 2005; Ruser and Schulz, 2015) in reducing denitrification, minimizing nitrate leaching and improving N utilization by crops. They have also been reported to reduce soil nitrate leaching from animal urine patches by 63% (Di and Cameron, 2007). Others have reported no effects of nitrapyrin on nitrification when applied with either inorganic N fertilizer or manure (Bailey, 1990; Wolt, 2000; Randall and Vetsch, 2005).

Manure N also exists in organic forms, which must be mineralized to inorganic N by soil microorganisms before it becomes available for plant uptake (Qian and Schoenau, 2002; Powell et al., 2006; Helgason et al., 2007). When manure is land applied, its C/N ratio will influence the organic N mineralization process (Paul and Beauchamp, 1994; Gagnon and Simard, 1999; Qian and Schoenau, 2002).

### **1.3. Construction and Demolition Waste and Peat Moss**

Studies have shown that the nutrient content of manure is greatly influenced by the type and quantity of bedding material used in CAFOs (Choudhary et al., 1996; Miller et al., 2003; Helgason et al., 2005). In cattle feedlots in western Canada, animals are kept warm during the winter months via use of high volumes of bedding material to provide insulation from snow or the frozen ground (Miller et al., 2006; Larney et al., 2008). Recently, in southern Alberta, there has been a gradual shift from the conventional use of cereal (barley) straw as bedding material to wood shavings and sometimes woodchips mixed with drywall from construction and demolition waste (CDW) (Miller et al., 2006; Larney et al., 2008; Hao et al., 2014).

In Canada and the United States, construction and demolition waste is largely buried in landfills, with little or no effort made to recycle it (Sandler, 2003; Yeheyis et al., 2013). The use of

CDW as bedding for cattle in feedlots or as a co-amendment with manure destined for land application may be a viable alternative to landfilling (Korcak et al., 2000). The drywall, also known as wall-board, gypsum board, or sheetrock, is composed primarily (85-90%) of gypsum (calcium sulfate), while woodchips, which are characterized by a high C/N ratio ( $> 400:1$ ), have been widely used as a bulking agent during manure composting (Larney et al., 2008; Miller et al., 2010; Naeth and Wilkinson, 2013). Gypsum is typically applied to agricultural land as a soil amendment (Korcak et al., 2000; Naeth and Wilkinson, 2013). Recently, drywall or a mixture of drywall and woodchips has been used as a bulking agent during composting of dairy and beef cattle manure and biosolids (Saludes et al., 2008; Naeth and Wilkinson, 2013; Hao et al., 2014). The implication is that mixing cattle manure with CDW could affect N availability in soil when the manure is land applied. The high C/N ratio of the wood chips in CDW could increase the C/N ratio of the manure, resulting in N immobilization following soil application. Several studies have shown a strong correlation between the C/N ratio of manure and the amount of N mineralized (Paul and Beauchamp, 1993; Gagnon and Simard, 1999; Qian and Schoenau, 2002). In their study, Gagnon and Simard (1999) reported a negative linear relationship between the C/N ratio of dairy manure and the amount of N mineralized after 13 wk of incubation. In a field study, Eghball (2000) reported that approximately 20% of the organic N in cattle manure applied the previous fall was mineralized during the first growing season.

Peat moss has been used as a growth medium in horticulture, agriculture, and forestry and has also been used as a bulking agent for cattle manure composting (N'Dayegamiye and Isfan, 1991; Gagnon et al., 1997; Chong, 2005; Jayasinghe et al., 2010). Peat moss has been shown to exhibit porosity and aeration properties that are comparable to composts and other organic by-products (Chong, 2005). Saviozzi and Cardelli (2014) found that the addition of peat moss to soil lowered soil pH, reflecting its acidifying properties, with most of the organic C in the peat moss having

undergone intense mineralization after 60 d of incubation, similar to pelleted cow manure and green waste compost. In another study, Gagnon et al. (1997) reported an increase in wheat yield and N uptake in plots amended with peat moss-based commercial compost relative to farm composts. The author attributed this increase to the high stable C and CEC of the peat moss, which retains  $\text{NH}_4\text{-N}$  on its exchange sites, thereby making N more readily available for plant uptake.

When mixed with manure or used as bedding, peat moss and CDW may alter the N mineralization process in manure, which could influence nutrient release following application of the manure to soil. Many of the published studies have investigated the effects of peat moss and CDW as compost bulking agents (N'Dayegamiye and Isfan, 1991; Gagnon et al., 1997; Naeth and Wilkinson, 2013; Hao et al., 2014). To our knowledge, there is a dearth of published research on the agronomic and environmental risks associated with land application of manure containing CDW. Therefore, the research reported in Chapter 2 of this thesis examined the effect of CDW on the rate and extent of N mineralization in manure-amended soils. Information on the amount of N potentially mineralized from manure is needed when estimating rates of application of cattle manure containing CDW. The study presented in Chapter 3 characterized the availability, crop uptake, and residual soil concentrations of N following repeated application of manure from cattle fed either DDGS or a regular grain diet, with the manure mixed with peat or CDW. The study also sought to investigate the effects of nitrapyrin on  $\text{NO}_3$  leaching and N use efficiency using two different soil types.

#### **1.4. Nutrient Removal in Heavily Manured Soils**

Although land application of livestock manure recycles nutrients back to the soil, continuous application can result in the excessive build-up of N and P, which may lead to environmental degradation (Sharpley, 1995; Sharpley et al., 2001; Pote et al., 2003; Ferguson et al., 2005; Schröder et al., 2005). Agricultural fields in the vicinity of CAFOs have high nutrient and salt levels due to

repeated manure applications (Whalen and Chang, 2001; Hao and Chang, 2003; Pant et al., 2004; Larney et al., 2006). The high cost of hauling manure over long distances is one of the many factors that encourage manure application in fields close to livestock operations (Larney et al., 2006).

Phytoextraction using crops can be employed to reduce elevated levels of nutrients in soils (Novak and Chan, 2002; Pant et al., 2004). Nutrient phytoextraction has been demonstrated for forages in soils with a history of repeated manure application (Gaston et al., 2003; Rowe and Fairbrother, 2003; Pant et al., 2004; Read et al., 2007). Phytoextraction has also been successfully used to remove metals from heavily contaminated land (Schnoor et al., 1995; Ebbs and Kochian, 1998; Lasat, 2002; Nevel et al., 2007). The technique involves the use of plants to extract excess nutrients from the soil; the aboveground biomass is harvested so that the nutrients are not recycled back to the soil. The eventual outcome is the reduction in soil nutrient concentrations to more acceptable levels with successive annual cropping, hence minimizing nutrient loss to the environment (Brink et al., 2001; Pederson et al., 2002; Benke et al., 2013).

Nutrients phytoextraction from the soil depends not only on the nutritional requirements of the crop, but also on soil properties, including available soil moisture (Angle et al., 2003). Soil nutrient dynamics and the availability of nutrients for plant uptake are influenced by soil moisture. Studies have shown that soil organic matter decomposition and N mineralization increase as soil moisture content and temperature increase, thereby enhancing soil microbial activity (Sierra, 1997; Leirós et al., 1999; Agehara and Warncke, 2005). Sierra (1997) reported that N mineralization rate during a 35-d incubation study increased from  $0.52 \text{ mg N kg}^{-1} \text{ soil d}^{-1}$  at 35% water-filled pore space (WFPS) to  $0.72 \text{ mg N kg}^{-1} \text{ soil d}^{-1}$  at 60% WFPS. Nitrogen uptake by plants, therefore, increases as soil moisture content increases. A study by Angle et al. (2003) showed that metal uptake by hyperaccumulator plants was greater at higher soil moisture levels. The authors attributed the

increase to the greater plant biomass at higher soil moisture levels, which further enhanced metal phytoextraction from the soil.

While nutrient phytoextraction by various forages grown on manured soils has been extensively studied (Brink et al., 2001; Rowe and Fairbrother, 2003; Read et al., 2007), to our knowledge, there is a dearth of published research on the effectiveness of various agronomic crops in extracting excess soil nutrients from soils with a long history of manure application under contrasting moisture conditions. While the N and P utilization of most arable crops is known (Sieling et al., 2006; Barbazán et al., 2009; Slaton et al., 2013), the extent of N and P removal from previously heavily manured soils via multiple harvesting in mono-cropping systems is poorly understood. Therefore, the study presented in Chapter 4 of this thesis was conducted to investigate the phytoextraction of N and P from a heavily manured soil by various agronomic crops grown under two moisture regimes.

### **1.5. Modeling Crop Yield in a Heavily Manured Soil**

Plants rely on the soil for most of the nutrients necessarily for their physiological growth and development. Among other soil factors, soil organic matter (SOM) plays a critical role in determining the fertility of a soil (Reeves, 1997). Soils with high SOM are generally better able to sustain crop biomass production (Dick, 1992; Reeves, 1997). Under favorable field conditions, soil nutrients are released from SOM through the mineralization process. However, continuous cropping can result in the reduction of soil nutrients through the depletion of the SOM (Dick, 1992; Reeves, 1997).

Often, soil nutrients are supplemented with synthetic fertilizers or organic amendments such as livestock manure, crop residue or compost. These amendments increase microbial biological activity and also alter soil nutrient dynamics by increasing available nutrient concentrations (Dick,



1992; Gagnon et al., 1999; Marschner et al., 2003; Miller et al., 2010). These organic amendments are typically applied to agricultural fields at rates to minimize the excessive build-up of nutrients that can result in environmental degradation (Eghball, 2000; Stumborg and Schoenau, 2008; Olson et al., 2009). However, agricultural fields close to CAFOs have high levels of soil nutrients, especially N, P, and possibly salts due to annual application of manure (Hao and Chang, 2003; Ferguson et al., 2005; Olson et al., 2010).

Producers faced with elevated soil nutrient levels from repeated manure application on their fields may decide to discontinue manure application and utilize continuous cropping to reduce the nutrient concentrations (Eghball et al., 2003). Since no nutrients will be further added, successive crop production on such fields would be highly dependent on the soil's ability to maintain productivity over time and its nutrient reserves (McAndrews et al., 2006; Sieling et al., 2006). A good understanding of soil properties that might limit crop yields under such a system is needed in order to maximize crop production (Cox et al., 2006). This may involve relating crop yield data to soil parameters measured over multiple years. Studies have shown crop yield in agricultural fields to be highly influenced by varying soil properties, landscape characteristics and environmental conditions (Jiang and Thelen, 2004; McDonald, 2006; Noorbakhsh et al., 2008; Anthony et al., 2012). However, the cost of yearly soil and crop sampling and laboratory analysis could be quite high, and the question remains whether the initial soil properties measured prior to the annual manure application or immediately following the discontinuation of manure application on the field will be better able to predict crop yield seven years after the last manure application. While literature on the relationships between crop yields and soil and landscape properties is abundant, to our knowledge, there is no published information on the use of soil properties measured immediately following the discontinuation of long-term manure application to predict crop yield. The study

presented in Chapter 5 utilized partial least squares (PLS) regression to address this knowledge gap. Soil properties used in the modeling were assessed on samples from long-term study plots established in the fall of 1973 at the Agriculture and Agri-Food Canada Research and Development Centre in Lethbridge, Alberta (Sommerfeldt and Chang, 1985).

## **1.6. Thesis Outline**

The general layout of this thesis follows the thesis guidelines of the Department of Soil Science, University of Manitoba. The individual thesis research chapters (Chapter 2 through 5) were prepared in manuscript format and are as follows:

**Chapter 2:** Nitrogen mineralization in Chernozemic soils amended with manure from cattle fed dried distillers grains with solubles;

**Chapter 3:** A bioassay of nitrogen availability in soils amended with manure from cattle fed DDGS: Effects of construction/demolition waste and peat moss;

**Chapter 4:** Phytoextraction of nitrogen and phosphorus by crops grown in a heavily manured Dark Brown Chernozem under contrasting soil moisture conditions; and

**Chapter 5:** Modeling barley yield in a Dark Brown Chernozem following discontinuation of long-term manure application.

## **1.7. References**

**Agehara, S. and Warncke, D. D. 2005.** Soil moisture and temperature effects on nitrogen release from organic nitrogen sources. *Soil Sci. Soc. Am. J.* **69**: 1844-1855.

- Angle, J. S., Baker, A. J. M., Whiting, S. N. and Chaney, R. L. 2003.** Soil moisture effects on uptake of metal by *Thlaspi*, *Alyssum*, and *Berkheya*. *Plant Soil* **256**: 325-332.
- Anthony, P., Malzer, G., Sparrow, S. and Zhang, M. 2012.** Soybean yield and quality in relation to soil properties. *Agron. J.* **104**: 1443-1458.
- Bailey, L. D. 1990.** The effects of 2-chloro-6 (trichloromethyl)-pyridine ('N-Serve') and N fertilizers on productivity and quality of Canadian oilseed rape. *Can. J. Plant Sci.* **70**: 979-986.
- Barbazán, M. M., Mallarino, A.P. and Sawyer, J. E. 2009.** Liquid swine manure phosphorus utilization for corn and soybean production. *Soil Sci. Soc. Am. J.* **73**: 654-662.
- Beauchemin, K. A., Janzen, H. H., Little, S.M., McAllister, T. A. and McGinn, S. M. 2010.** Life cycle assessment of greenhouse gas emissions from beef production in western Canada: A case study. *Agric. Syst.* **103**: 371-379.
- Benke, M. B., Indraratne, S. P. and Hao, X. 2013.** Long-term manure applications impact on irrigated barley forage mineral concentrations. *Agron J.* **105**: 1441-1450.
- Brink, G. E., Pederson, G. A., Sistani, K. R. and Fairbrother, T. E. 2001.** Uptake of selected nutrients by temperate grasses and legumes. *Agron J.* **93**: 887-890.
- Calderón, F. J., McCarty, G. W. and Reeves III, J. B. 2005.** Nitrapyrin delays denitrification on manured soils. *Soil Sci.* **170**: 350-359.
- Calderón, F. J., McCarty, G. W., Van Kessel, J., Ann, S. and Reeves III, J. B. 2004.** Carbon and nitrogen dynamics during incubation of manured soil. *Soil Sci. Soc. Am. J.* **68**: 1592-1599.
- Chong, C. 2005.** Experiences with wastes and composts in nursery substrates. *HortTechnol.* **15**: 739-747.
- Choudhary, M., Bailey, L. D., and Grant, C. A. 1996.** Review of the use of swine manure in crop production: effect on yield and composition and on soil and water quality. *Waste Manage. Res.* **14**: 581-595.
- Cox, M. S., Gerard, P. D. and Abshire, M. J. 2006.** Selected soil properties' variability and their relationships with yield in three Mississippi fields. *Soil Sci.* **171**: 541-551.
- Crawford, D. M. and Chalk, P. M. 1993.** Sources of N uptake by wheat (*Triticum aestivum* L.) and N transformations in soil treated with a nitrification inhibitor (nitrapyrin). *Plant Soil* **149**: 59-72.
- Di, H. J. and Cameron, K. C. 2007.** Nitrate leaching losses and pasture yields as affected by different rates of animal urine nitrogen returns and application of a nitrification inhibitor-a lysimeter study. *Nutr. Cycl. Agroecosyst.* **79**: 281-290.

- Dick, R. P. 1992.** A review: long-term effects of agricultural systems on soil biochemical and microbial parameters. *Agric. Ecosyst. Environ.* **40**: 25-36.
- Ebbs, S. D. and Kochian, L. V. 1998.** Phytoextraction of zinc by oat (*Avena sativa*), barley (*Hordeum vulgare*), and indian mustard (*Brassica juncea*). *Environ. Sci. Technol.* **32**: 802-806.
- Ebeling, A. M., Bundy, L. G., Powell, J. M. and Andraski, T. W. 2002.** Dairy diet phosphorus effects on phosphorus losses in runoff from land-applied manure. *Soil Sci. Soc. Am. J.* **66**: 284-291.
- Eghball, B., Shanahan, J. F., Varvel, G. E. and Gilley, J. E. 2003.** Reduction of high soil test phosphorus by corn and soybean varieties. *Agron. J.* **95**: 1233-1239.
- Eghball, B. 2000.** Nitrogen mineralization from field-applied beef cattle feedlot manure or compost. *Soil Sci. Soc. Am. J.* **64**: 2024-2030.
- EPM [Ethanol Producer Magazine]. 2016.** Canadian ethanol industry operating at full capacity [Online]. Available:  
<http://www.ethanolproducer.com/articles/13646/report-canadian-ethanol-industry-operating-at-full-capacity> (verified 7 April 2017).
- Ferguson, R. B., Nienaber, J. A., Eigenberg, R. A. and Woodbury, B. L. 2005.** Long-term effects of sustained beef feedlot manure application on soil nutrients, corn silage yield, and nutrient uptake. *J. Environ. Qual.* **34**: 1672-1681.
- FOBI [Feed Opportunities from Biofuels Industries] Network. 2013.** Wheat dried distiller grains with solubles. Wheat DDGS feed guide [Online]. Available:  
[https://cigi.ca/wp-content/uploads/2013/02/DDGS-Feed-Guide\\_Revised\\_Jan.-2013.pdf](https://cigi.ca/wp-content/uploads/2013/02/DDGS-Feed-Guide_Revised_Jan.-2013.pdf) (verified 7 April 2017).
- Gagnon, B. and Simard, R. R. 1999.** Nitrogen and phosphorus release from on-farm and industrial composts. *Can. J. Soil Sci.* **79**: 481-489.
- Gagnon, B., Simard, R. R., Robitaille, R., Goulet, M. and Rioux, R. 1997.** Effect of composts and inorganic fertilizers on spring wheat growth and N uptake. *Can. J. Soil Sci.* **77**: 487-495.
- Gaston, L. A., Eilers, T. L., Kovar, J. L., Cooper, D. and Robinson, D. L. 2003.** Greenhouse and field studies on hay harvest to remediate high phosphorus soil. *Commun. Soil Sci. Plant Anal.* **34**: 2085-2097.
- Gibb, D. J., Hao, X. and McAllister, T. A. 2008.** Effect of dried distillers' grains from wheat on diet digestibility and performance of feedlot cattle. *Can. J. Anim. Sci.* **88**: 659-665.

**Hao, X., Hill, B., Caffyn, P., Travis, G., Olson, A. F., Larney, F. J., McAllister, T. A. and Alexander, T. 2014.** Co-composting of beef cattle feedlot manure with construction and demolition waste. *J. Environ Qual.* **43**: 1799-1808.

**Hao, X., Benke, M. B., Gibb, D. J., Stronks, A., Travis, G. and McAllister, T. A. 2009.** Effects of dried distillers' grains with solubles (wheat-based) in feedlot cattle diets on feces and manure composition. *J. Environ Qual.* **38**: 1709-1718.

**Hao, X. and Chang, C. 2003.** Does long-term heavy cattle manure application increase salinity of a clay loam soil in semi-arid southern Alberta? *Agric. Ecosyst. Environ.* **94**: 89-103.

**Helgason, B. L., Larney, F. J., Janzen, H. H. and Olson, B. M. 2007.** Nitrogen dynamics in soil amended with composted cattle manure. *Can. J. Soil Sci.* **87**: 43-50.

**Helgason, B. L., Larney, F. J. and Janzen, H. H. 2005.** Estimating carbon retention in soils amended with composted beef cattle manure. *Can. J. Soil Sci.* **85**: 29-46.

**Hristov, A. N. 2013.** Diet formulation as an effective tool for mitigating nitrogen excretion in dairy systems. *Adv. Anim. Biosci.* **4 s1**: 15-18.

**Hünerberg, M., McGinn, S. M., Beauchemin, K. A., Okine, E. K., Harstad, O. M. and McAllister, T. A. 2013a.** Effect of dried distillers' grains with solubles on enteric methane emissions and nitrogen excretion from finishing beef cattle. *Can. J. Anim. Sci.* **93**: 373-385.

**Hünerberg, M., McGinn, S. M., Beauchemin, K. A., Okine, E. K., Harstad, O. M. and McAllister, T. A. 2013b.** Effect of dried distillers' grains plus solubles on enteric methane emissions and nitrogen excretion from growing beef cattle. *J. Anim. Sci.* **91**: 2846-2857.

**International Grains Council. 2010.** Report for fiscal year 2009/10 [Online]. Available: <http://igc.int/downloads/publications/rfy/rfy0910.pdf> (verified 7 April 2017).

**Jayasinghe, G. Y., Liyana Arachchi, I. D. and Tokashiki, Y. 2010.** Evaluation of containerized substrates developed from cattle manure compost and synthetic aggregates for ornamental plant production as a peat alternative. *Resour. Conserv. Recy.* **54**: 1412-1418.

**Jiang, P. and Thelen, K. D. 2004.** Effect of soil and topographic properties on crop yield in a North-Central corn-soybean cropping system. *Agron. J.* **96**: 252-258.

**Kebreab, E., Hansen, A. V. and Leytem, A. B. 2013.** Feed management practices to reduce manure phosphorus excretion in dairy cattle. *Adv. Anim. Biosci.* **4 s1**: 37-41.

**Klopfenstein, T. J., Erickson, G. E. and Bremer, V. R. 2008.** Board-Invited Review: Use of distillers by-products in the beef cattle feeding industry. *J. Anim. Sci.* **86**: 1223-1231.

- Korcak, R. F., Meininger, R. and Yost, P. A. 2000.** . Beneficial reuse of aggregate mineral fines and scrap new construction wallboard. In: Power, J.F. and W.A. Dick (Eds.), Land application of agricultural, industrial, and municipal by-products. SSSA Book Series No. 6, SSSA, Madison, WI. p. 583-603.
- Larney, F., Olson, A. F., Miller, J. J., DeMaere, P. R., Zvomuya, F. and McAllister, T. A. 2008.** Physical and chemical changes during composting of wood-chip-bedded and straw-bedded beef cattle feedlot manure. *J. Environ. Qual.* **37**: 725-735.
- Larney, F. J., Sullivan, D. M., Buckley, K. E. and Eghball, B. 2006.** The role of composting in recycling manure nutrients. *Can. J. Soil Sci.* **86**: 597-611.
- Lasat, M. M. 2002.** Phytoextraction of toxic metals: A review of biological mechanisms. *J. Environ. Qual.* **31**: 109-120.
- Leirós, M. C., Trasar-Cepeda, C., Seoane, S. and Gil-Sotres, F. 1999.** Dependence of mineralization of soil organic matter on temperature and moisture. *Soil Biol. Biochem.* **31**: 327-335.
- Li, Y. L., McAllister, T. A., Beauchemin, K. A., He, M. L., McKinnon, J. J. and Yang, W. Z. 2011.** Substitution of wheat dried distillers grains with solubles for barley grain or barley silage in feedlot cattle diets: Intake, digestibility, and ruminal fermentation. *J. Anim. Sci.* **89**: 2491-2501.
- Liu, K. 2011.** Chemical composition of distillers grains, a review. *J. Agric. Food Chem.* **59**: 1508-1526.
- Marschner, P., Kandeler, E. and Marschner, B. 2003.** Structure and function of the soil microbial community in a long-term fertilizer experiment. *Soil Biol. Biochem.* **35**: 453-461.
- McAndrews, G. M., Liebman, M., Cambardella, C. A. and Richard, T. L. 2006.** Residual effects of composted and fresh solid swine (*Sus scrofa* L.) manure on soybean [*Glycine max* (L.) Merr.] growth and yield. *Agron. J.* **98**: 873-882.
- McDonald, G. K. 2006.** Effects of soil properties on variation in growth, grain yield and nutrient concentration of wheat and barley. *Aust. J. Exp. Agric.* **46**: 93-105.
- McGinn, S. M., Chung, Y. H., Beauchemin, K. A., Iwaasa, A. D. and Grainger, C. 2009.** Use of corn distillers' dried grains to reduce enteric methane loss from beef cattle. *Can. J. Anim. Sci.* **89**: 409-413.
- Meyer, U., Schwabe, A., Dänicke, S. and Flachowsky, G. 2010.** Effects of by-products from biofuel production on the performance of growing fattening bulls. *Anim. Feed Sci. Technol.* **161**: 132-139.

- Miller, J. J., Beasley, B. W., Drury, C. F., Larney, F. J. and Hao, X. 2015.** Influence of long-term (9 yr) composted and stockpiled feedlot manure application on selected soil physical properties of a clay loam soil in Southern Alberta. *Compost Sci. Util.* **23**: 1-10.
- Miller, J. J., Beasley, B. W., Drury, C. F. and Zebarth, B. J. 2010.** Available nitrogen and phosphorus in soil amended with fresh or composted cattle manure containing straw or wood-chip bedding. *Can. J. Soil Sci.* **90**: 341-354.
- Miller, J. J., Olson, Edith C. S., Chanasyk, D. S., Beasley, B. W., Yanke, L. J., Larney, F. J., McAllister, T. A., Olson, B. M. and Selinger, L. B. 2006.** Bedding and within-pen location effects on feedlot pen runoff quality using a rainfall simulator. *J. Environ. Qual.* **35**: 505-515.
- Miller, J. J., Beasley, B. W., Yanke, L. J., Larney, F. J., McAllister, T. A., Olson, B. M., Selinger, L. B., Chanasyk, D. S. and Hasselback, P. 2003.** Bedding and seasonal effects on chemical and bacterial properties of feedlot cattle manure. *J. Environ. Qual.* **32**: 1887-1894.
- Miller, J. J., Sweetland, N. J. and Chang, C. 2002.** Soil physical properties of a Chernozemic clay loam after 24 years of beef cattle manure application. *Can. J. Soil Sci.* **82**: 287-296.
- Naeth, M. A. and Wilkinson, S. R. 2013.** Can we build better compost? Use of waste drywall to enhance plant growth on reclamation sites. *J. Environ. Manage.* **129**: 503-509.
- N'Dayegamiye, A. N. and Isfan. D. 1991.** Chemical and biological changes in compost of wood shavings, sawdust and peat moss. *Can. J Soil Sci.* **71**: 475-484.
- Nevel, L. V., Mertens, J., Oorts, K. and Verheyen, K. 2007.** Phytoextraction of metals from soils: How far from practice? *Environ. Pollut.* **150**: 34-40.
- Noorbakhsh, S., Schoenau, J., Si, B., Zeleke, T. and Qian, P. 2008.** Soil properties, yield, and landscape relationships in South-central Saskatchewan Canada. *J. Plant Nutr.* **31**: 539-556.
- Novak, J. M. and Chan, A. S. K. 2002.** Development of P-Hyperaccumulator plant strategies to remediate soils with excess P concentrations. *Crit. Rev. Plant Sci.* **21**: 493-509.
- Olson, B. M., McKenzie, R. H., Larney, F. J. and Bremer, E. 2010.** Nitrogen-and phosphorus-based applications of cattle manure and compost for irrigated cereal silage. *Can. J. Soil Sci.* **90**: 619-635.
- Olson, B. M., Bennett, D. R., McKenzie, R. H., Ormann, T. D. and Atkins, R. P. 2009.** Nitrate leaching in two irrigated soils with different rates of cattle manure. *J. Environ. Qual.* **38**: 2218-2228.
- Pant, H. K., Adjei, M. B., Scholberg, J. M. S., Chambliss, C. G. and Rechcigl, J. E. 2004.** Forage production and phosphorus phytoremediation in manure-impacted soils. *Agron. J.* **96**: 1780-1786.

**Paul, J. W. and Beauchamp, E. G. 1994.** Short-term nitrogen dynamics in soil amended with fresh and composted cattle manures. *Can. J. Soil Sci.* **74**: 147-155.

**Paul, J. W. and Beauchamp, E. G. 1993.** Nitrogen availability for corn in soils amended with urea, cattle slurry, and solid and composted manures. *Can. J. Soil Sci.* **73**: 253-266.

**Pederson, G. A., Brink, G. E. and Fairbrother, T. E. 2002.** Nutrient uptake in plant parts of sixteen forages fertilized with poultry litter: Nitrogen, phosphorous, potassium, copper, and zinc. *Agron. J.* **94**: 895-904.

**Pote, D. H, Lory J. A. and Zhang, H. 2003.** Does initial soil P level affect water-extractable soil P response to applied P? *Adv. Environ. Res.* **7**: 503-509.

**Powell, J. M., Wattiaux, M. A., Broderick, G. A., Moreira, V. R. and Casler, M. D. 2006.** Dairy diet impacts on fecal chemical properties and nitrogen cycling in soils. *Soil Sci. Soc. Am. J.* **70**: 786-794.

**Peng, X., Maharjan, B., Yu, C., Su, A., Jin, V. and Ferguson, R. B. 2015.** A laboratory evaluation of ammonia volatilization and nitrate leaching following nitrogen fertilizer application on a coarse-textured soil. *Agron. J.* **107**: 871-879.

**Qian, P. and Schoenau, J. J. 2002.** Availability of nitrogen in solid manure amendments with different C:N ratios. *Can. J. Soil Sci.* **82**: 219-225.

**Randall, G. W. and Vetsch, J. A. 2005.** Corn production on a subsurface-drained Mollisol as affected by fall versus spring application of nitrogen and nitrapyrin. *Agron. J.* **97**: 472-478.

**Read, J. J., Sistani, K. R., Brink, G. E. and Oldham, J. L. 2007.** Reduction of high soil test phosphorous by Bermudagrass and Ryegrass-Bermudagrass following the cessation of broiler litter application. *Agron. J.* **99**: 1492-1501.

**Reeves, D. W. 1997.** The role of soil organic matter in maintaining soil quality in continuous cropping systems. *Soil Tillage Res.* **43**: 131-167.

**RFA [Renewable Fuels Association]. 2016.** Fueling a high octane future [Online]. Available: <http://www.ethanolrfa.org/wp-content/uploads/2016/02/Ethanol-Industry-Outlook-2016.pdf>

(verified 8 March 2017).

**Ronaghi, A., Soltanpour, P. N. and Mosier, A. R. 1993.** Nitrapyrin effect on corn N-15 uptake efficiency, denitrification and nitrate leaching. *Commun. Soil Sci. Plant Anal.* **24**: 2629-2639.

**Rowe, D. E. and Fairbrother, T. E. 2003.** Harvesting winter forages to extract manure soil nutrients. *Agron. J.* **95**: 1209-1212.



**Ruser, R. and Schulz, R. 2015.** The effect of nitrification inhibitors on the nitrous oxide (N<sub>2</sub>O) release from agricultural soils-a review. *J. Plant Nutr. Soil Sci.* **178**: 171-188.

**Saludes, R. B., Iwabuchi, K., Miyatake, F., Abe, Y. and Honda, Y. 2008.** Characterization of dairy cattle manure/wallboard paper compost mixture. *Bioresour. Technol.* **99**: 7285-7290.

**Sandler, K. 2003.** Analyzing what's recyclable in C&D debris. *Biocycle* **44**: 51-54.

**Saviozzi, A. and Cardelli, R. 2014.** Organic matter characteristics, biochemical activity and antioxidant capacity of soil amended with different organic materials. *Arch. Agron. Soil Sci.* **60**: 119-131.

**Schoenau, J. J. and Davis, J. G. 2006.** Optimizing soil and plant responses to land-applied manure nutrients in the Great Plains of North America. *Can. J. Soil Sci.* **86**: 587-595.

**Schnoor, J. L., Licht, L. A., McCutcheon, S. C., Wolfe, N. L. and Carreira, L. H. 1995.** Phytoremediation of organic and nutrient contaminants. *Environ. Sci. Technol.* **29**: 318-323.

**Schröder, J. J., Jansen, A. G. and Hilhorst, G. J. 2005.** Long-term nitrogen supply from cattle slurry. *Soil Use Manage.* **21**: 196-204.

**Sharpley, A. N., Kleinman, P. and McDowell, R. 2001.** Innovative management of agricultural phosphorus to protect soil and water resources. *Commun. Soil Sci. Plant Anal.* **32**: 1071-1100.

**Sharpley A. N. 1995.** Dependence of runoff phosphorus on extractable soil phosphorus. *J. Environ. Qual.* **24**: 920-926.

**Sieling, K., Brase, T. and Svib, V. 2006.** Residual effects of different N fertilizer treatments on growth, N uptake and yield of oilseed rape, wheat and barley. *Europ. J. Agron.* **25**: 40-48.

**Sierra, J. 1997.** Temperature and soil moisture dependence of N mineralization in intact soil cores. *Soil Biol. Biochem.* **29**: 1557-1563.

**Simard, R. R., Cluis, D., Gangbazo, G. and Beauchemin, S. 1995.** Phosphorus status of forest and agricultural soils from a watershed of high animal density. *J. Environ. Qual.* **24**: 1010-1017.

**Slaton, N. A., Roberts, T. L., Golden, B. R., Ross, W. J. and Norman, R. J. 2013.** Soybean response to phosphorus and potassium supplied as inorganic or poultry litter. *Agron. J.* **105**: 812-820.

**Sommerfeldt, T. G. and Chang, C. 1985.** Changes in soil properties under annual applications of feedlot manure and different tillage practices. *Soil Sci. Soc. Am. J.* **49**: 983-987.

**Statistics Canada. 2016.** Cattle Statistics [Online]. Available:

<http://www.statcan.gc.ca/daily-quotidien/160818/dq160818c-eng.pdf> (verified 7 March 2017).

**Statistics Canada. 2011.** Cattle Statistics [Online]. Available:

<http://www.statcan.gc.ca/pub/23-012-x/2010002/part-partie1-eng.htm> (verified 7 March 2017).

**Stumborg, C. and Schoenau, J. J. 2008.** Evaluating phosphorus loading from repeated manure applications to two Saskatchewan soils. *Can. J. Soil Sci.* **88**: 377-387.

**Subbarao, G.V., Ito, O., Sahrawat, K. L., Berry, W. L., Nakahara, K., Ishikawa, T., Watanabe, T., Suenaga, K., Rondon, M. and Rao, I. M. 2006.** Scope and strategies for regulation of nitrification in agricultural systems-challenges and opportunities. *Crit. Rev. Plant Sci.* **25**: 303-335.

**USDA Foreign Agricultural Service. 2016.** Canada: Annual biofuels. Global Agricultural Information Network GAIN report Number: CA16038 [Online]. Available:

[https://gain.fas.usda.gov/Recent%20GAIN%20Publications/Biofuels%20Annual\\_Ottawa\\_Canada\\_8-9-2016.pdf](https://gain.fas.usda.gov/Recent%20GAIN%20Publications/Biofuels%20Annual_Ottawa_Canada_8-9-2016.pdf) (verified 7 April 2017).

**Vergé, X. P. C., Dyer, J. A., Desjardins, R. L. and Worth, D. 2008.** Greenhouse gas emissions from the Canadian beef industry. *Agric. Syst.* **98**: 126-134.

**Volf, C. A., Ontkian, G. R., Bennett, D. R., Chanasyk, D. S. and Miller, J. J. 2007.** Phosphorus losses in simulated rainfall runoff from manured soils of Alberta. *J. Environ. Qual.* **36**: 730-741.

**Walter, L. J., Aalhus, J. L., Robertson, W. M., McAllister, T. A., Gibb, D. J., Dugan, M. E. R., Aldai, N. and McKinnon, J. J. 2010.** Evaluation of wheat or corn dried distillers grains with solubles on performance and carcass characteristics of feedlot steers. *Can. J. Anim. Sci.* **90**: 259-269.

**Whalen, J. K. and Chang, C. 2001.** Phosphorus accumulation in cultivated soils from long-term annual applications of cattle feedlot manure. *J. Environ. Qual.* **30**: 229-237.

**Wolt, J. D. 2004.** A meta-evaluation of nitrapyrin agronomic and environmental effectiveness with emphasis on corn production in the Midwestern USA. *Nutr. Cycling Agroecosyst.* **69**: 23-41.

**Wolt, J. D. 2000.** Nitrapyrin behavior in soils and environmental considerations. *J. Environ. Qual.* **29**: 367-379.

**Yeheyis, M., Hewage, K., Alam, M. S., Eskicioglu, C. and Sadiq, R. 2013.** An overview of construction and demolition waste management in Canada: a lifecycle analysis approach to sustainability. *Clean Technol. Environ. Policy* **15**: 81-91.

## **2. NITROGEN MINERALIZATION IN CHERNOZEMIC SOILS AMENDED WITH MANURE FROM CATTLE FED DRIED DISTILLERS GRAINS WITH SOLUBLES**

### **2.1. Abstract**

Inclusion of dried distillers grains with solubles (DDGS) in cattle diets, coupled with the increasing use of construction and demolition waste (CDW), particularly the wood and drywall fractions as bedding in beef cattle feedlots, may affect nitrogen (N) dynamics when the resulting manure is applied to soil. This laboratory incubation study was conducted to evaluate the mineralization of N in contrasting Chernozemic soils amended with regular manure (RM) from cattle fed a regular grain-based diet versus manure (DGM) from cattle fed a diet containing DDGS. The effect of adding CDW to DGM (DGM<sub>CDW</sub>) was also assessed. The soils (a Black Chernozem and a Brown Chernozem) were amended with manure (40 g kg<sup>-1</sup> soil, dry wt.), mixed and packed in leaching columns, and incubated at 15 and 25°C. Nitrogen mineralization in the manure-amended Brown Chernozem exhibited negative net mineralization. In the Black Chernozem, N mineralization was described by a mixed first order and zero order kinetic model. Potentially mineralizable N (N<sub>0</sub>) concentration in the manure-treated Black Chernozem ranged from 172 mg kg<sup>-1</sup> for DGM to 235 mg kg<sup>-1</sup> for RM and was greater at 25°C than 15°C. The first-order mineralization rate constant varied among manure treatments and decreased in the order DGM<sub>CDW</sub> > DGM > RM. The rate constants were not significantly affected by temperature, but the temperature sensitivity (Q<sub>10</sub>) of N mineralization was significantly greater for RM (1.0) and DGM (1.3) than DGM<sub>CDW</sub> (0.3). The percentages of total organic N mineralized from RM and DGM were greater than that for DGM<sub>CDW</sub>, with RM producing the greatest mineralization. This suggests that adding CDW to manure will affect

N dynamics by lowering the amount of N mineralized, which may necessitate either applying higher manure rates (and risking excess P build-up) or supplementing with inorganic fertilizers to minimize N deficiency in receiving crops.

## **2.2. Introduction**

The Canadian biofuels industry has recently seen a rapid expansion, producing approximately 1.36 billion liters of ethanol in 2010 alone, mostly from corn (*Zea mays* L.) (64%) and wheat (*Triticum aestivum* L.) (31%) (USDA FAS, 2010). Concomitant with this expansion has been an increased supply of valuable byproducts, including dried distillers grains with solubles (DDGS), which is a dried mixture of the condensed liquid fraction (solubles) remaining after ethanol extraction and the coarse ethanol-free solids (distillers grains). Approximately 0.26 million tonnes of wheat DDGS per year was produced by Canadian ethanol plants in 2009/2010, with an estimated value of \$51 million (International Grains Council, 2010). Nearly all wheat DDGS is used as an animal feedstuff (FOBI Network, 2013), which provides a concentrated source of nutrients.

The inclusion of DDGS in cattle diets may alter manure properties relative to manure from animals fed regular grain-based diets. This can have implications on manure nutrient release and plant nutrient uptake, as well as the potential for nutrient loss to the environment (Hao et al., 2014). Recent studies have shown higher concentrations of N and phosphorus (P) in manure from cattle fed DDGS (DGM) when compared to manure from cattle fed a regular grain diet (RM) (Klopfenstein et al., 2008; Hao et al., 2009; Hünenberg et al., 2013).

Manure properties may be further altered by bedding materials used in confined feeding operations. Recently, there has been a growing interest in western Canada in the use of construction and demolition waste (CDW), particularly the wood and drywall fractions, as bedding material in beef cattle feedlots (Hao et al., 2014). In Canada and the United States, CDW is largely buried in

landfills, with little or no effort made to recycle it (Sandler, 2003; Yeheyis et al., 2013). Approximately 9 million tons of CDW are generated by the construction industry in Canada annually, which is composed mainly of wood products, asphalt, drywall, concrete and masonry. Metals, plastics, cardboard and paper, earth and shingles are the other materials that are often present in CDW (Yeheyis et al., 2013). Nitrogen availability has been found to be lower in soils amended with beef cattle manure containing woodchips as compared to barley (*Hordeum vulgare* L.) straw as bedding (Miller et al., 2009, 2010). While the inclusion of DDGS in cattle diets may improve manure N availability, the use of CDW as bedding in feedlots may conceivably negate this because of the stable C in the woodchip fraction of CDW (Larney et al., 2008; Miller et al., 2010). Alternatively, the drywall fraction of CDW, which is mainly gypsum (calcium sulfate), may be beneficial since it supplies calcium (Ca) and sulfur (S) (Hao et al., 2014).

A good understanding of N availability for crop uptake, hence organic N mineralization during the growing season, is critical not only from the perspective of efficiency of fertilizer use, but also to ensure that loss of excess N to the environment is minimized (Zebarth et al., 2009; St. Luce et al., 2011). Nitrogen mineralization reactions in soils typically follow first-order kinetics, with the rate constants and potentially mineralizable N ( $N_0$ , a measure of soil mineralizable N concentration) usually derived from long-term incubation studies (Stanford and Smith, 1972; Ellert and Bettany, 1992; Curtin and Campbell, 2008). Others have estimated these parameters using either a double exponential (Bonde and Rosswall, 1987; Gil et al., 2011) or the mixed first-order and zero-order kinetic model (Bonde and Rosswall, 1987), suggesting that more than one fraction of soil organic N may be mineralized, each with a unique rate of decomposition (Benbi and Richter, 2002).

To the best of our knowledge, the mineralization of N from DGM mixed with CDW has previously not been characterized. Therefore, the objective of this study was to evaluate the mineralization of N in manure from cattle fed DDGS relative to manure from cattle fed a regular

grain-based diet. The study further examined the effects of CDW and temperature on DGM mineralization, and how these vary between two contrasting soils.

## **2.3. Materials and Methods**

### **2.3.1. Soil**

Soil samples (0-15 cm layer) were collected using a spade from an Orthic Black Chernozem (loam, Typic Haplocryoll) near Olds, Alberta, Canada (113° 57' 42" N, 51° 43' 46" W) and an Orthic Brown Chernozem (sandy clay loam, Aridic Haploboroll) near Cranford, Alberta (112° 20' 31" N, 49° 45' 51" W). These soils belong to the dominant Chernozem group in the cropland areas of Alberta and the western Canadian Prairies (Pennock et al., 2011). Their regional distribution is spatially correlated with the major climate zonation of the region, with the Brown Chernozems occupying areas of the prairies that have the greatest annual water deficit. During the season immediately preceding soil collection, the Cranford site was cropped to potatoes (*Solanum tuberosum* L.) while the Olds site was cropped to barley in a canola-wheat-barley rotation. The soils were air-dried, passed through a 4-mm mesh sieve, thoroughly mixed to ensure uniformity, and stored at room temperature (23-25°C) until the start of the experiment. Soil subsamples were ground to pass through a 2-mm sieve prior to laboratory analysis. Particle size distribution was determined using the hydrometer method (Kroetsch and Wang, 2008). Soil pH and electrical conductivity (EC) were determined in a 1:2 (mass/vol.) soil/water suspension using a pH/EC meter (Model SP2000, Skalar BV, Breda, Netherlands). Ammonium (NH<sub>4</sub>) N concentration in the soil was measured colorimetrically by the indophenol method following extraction with 2 M KCl. Soil nitrate (NO<sub>3</sub>) N concentration was measured colorimetrically by the cadmium-reduction method following extraction with 2 M KCl. Soil total N (TN) concentration was determined by the macro-Kjeldahl method using an automated Kjeldahl analyzer (Foss Kjeltac 8400, Höganäs, Sweden) following acid digestion with

H<sub>2</sub>SO<sub>4</sub> and TiO (catalyst). Total carbon (TC) concentration was determined using a CN analyzer (Elementar, Langenselbold, Germany). Total P (TP) concentration was measured with an inductively coupled optical emission spectrometer (Thermo Electron Corporation iCAP 6300, Cambridge, England) following digestion of a 1:10 (mass/vol.) soil/concentrated HNO<sub>3</sub> suspension with a MARS 5 microwave system (CEM Corporation, Matthews, NC).

**Table 2.1 Initial properties of soils and organic amendments used for the incubation study.**

Property <sup>†</sup>	Black Chernozem	Brown Chernozem	RM	DGM	DGM <sub>CDW</sub> <sup>‡</sup>
pH	7.6	7.2	8.7	8.6	8.4
EC, dS m <sup>-1</sup>	0.5	0.3	6.5	7.7	8.0
NO <sub>3</sub> <sup>-</sup> -N, mg kg <sup>-1</sup>	8.8	16.4	-	-	-
NH <sub>4</sub> <sup>+</sup> -N, mg kg <sup>-1</sup>	18.9	19.2	1564	1566	2262
Soluble P, mg kg <sup>-1</sup>	-	-	40	82	29
PO <sub>4</sub> -P, mg kg <sup>-1</sup>	38	31	-	-	-
Total C, g kg <sup>-1</sup>	50	21	106	114	111
Total N, g kg <sup>-1</sup>	4.7	2.0	6.8	9.2	8.4
Total organic N, g kg <sup>-1</sup>	-	-	5.3	7.6	6.2
C to N ratio	11	11	16	12	13
Total P, g kg <sup>-1</sup>	0.8	0.6	1.7	2.2	2.6
Dry matter content, kg kg <sup>-1</sup>	-	-	0.27	0.30	0.38
Clay, g kg <sup>-1</sup>	201	232	-	-	-
Silt, g kg <sup>-1</sup>	314	278	-	-	-
Sand, g kg <sup>-1</sup>	485	491	-	-	-

<sup>†</sup> All chemical analyses expressed on a dry weight basis.

<sup>‡</sup> RM, manure from cattle fed a regular grain diet; DGM, manure from cattle fed a diet containing DDGS; DGM<sub>CDW</sub>, DGM mixed with construction and demolition waste (CDW).

### 2.3.2. Manure

Two types of beef cattle (British cross heifers) manure were collected by scrapping from the pen floor of a feedlot at the Agriculture and Agri-Food Canada Research and Development Centre in Lethbridge, Alberta, Canada. Regular manure (RM) was collected from cattle fed a finishing grain-

based diet consisting of 85% barley grain, 10% barley silage, and 5% mineral supplement to provide trace minerals, vitamins and monensin. This diet is typical of that used in western Canadian feedlots (Hao et al., 2009). Manure (DGM manure) was also collected from cattle fed a diet composed of 45% barley grain, 40% wheat DDGS, and silage along with 5% of the same mineral supplement as in the grain-based diet. An additional manure treatment (DGM<sub>CDW</sub>) was prepared by mixing DGM with CDW (a mixture of drywall and woodchips, mainly wastage from new housing construction in southern Alberta) in a 5:1 ratio by mass to attain a C/N ratio < 20:1. Studies have shown that positive N mineralization from manure after application to soil occurs when manure C/N ratio is < 20:1 (Beauchamp and Paul, 1989; Qian and Schoenau, 2002). Manure samples were analyzed for pH, total N, total C, NH<sub>4</sub>-N, and NO<sub>3</sub>-N according to the methods of APHA et al. (2005). Initial soil and manure properties are presented in Table 1.

### 2.3.3. Experimental Design and Setup

The experimental design was a randomized complete block with a split-plot treatment layout consisting of temperature (15 and 25°C, replicated three times) as the main plot and factorial combinations of the two soils and four amendment treatments (RM, DGM, DGM<sub>CDW</sub>, and a non-amended control) as the subplot. The temperatures were selected to closely mimic the mean daily soil temperatures at the 10-cm depth early in the spring and during the summer months, respectively.

Each organic amendment was thoroughly mixed with 30 g of air-dry soil to give a rate of 40 g kg<sup>-1</sup> soil. The manure rate approximates a field application rate of 60 Mg ha<sup>-1</sup> based on a soil bulk density of 1.0 Mg m<sup>-3</sup> for the Black Chernozem and 1.2 Mg m<sup>-3</sup> for the Brown Chernozem at a soil incorporation depth of 0.1 m. The 60 Mg ha<sup>-1</sup> rate is currently recommended in southern Alberta to supply 150 kg N ha<sup>-1</sup> for irrigated cereals (Benke et al., 2010).



Leaching column setup followed the technique described by Campbell et al. (1993). Briefly, PVC conduit pipes (2.5 cm i.d. × 15 cm long) were sealed at the base with a perforated #5 stopper of similar diameter. Before sealing the bottom of the column, a thin layer of bug screen mesh was placed on the end of the stopper going into the column, followed by a 2.4 cm Whatman glass microfiber filter and glass wool. Soil samples (30 g) were mixed with 30 g of quartz sand and packed into leaching columns to attain a bulk density of  $1.2 \text{ Mg m}^{-3}$ . A glass-wool pad (~5 mm) was placed over the contents of each column to prevent dispersion of the soil during addition of the leaching solution. The soil in each column was wetted to 60% water filled pore space (WFPS) (White and Mastalerz, 1966) and covered with a parafilm, after which the columns were placed in the incubators. The columns were weighed every 7 d and water was added to replace any water lost.

The columns were retrieved every 14 d during the first 12 wk of incubation and every 28 d during the remaining 24 wk of the study for extraction of inorganic N ( $\text{NH}_4^+\text{-N} + \text{NO}_3\text{-N}$ ). Inorganic N was extracted by leaching the columns with 100 mL of 0.01 M  $\text{CaCl}_2$  in five increments of 20 mL each, followed by 25 mL of a zero-N nutrient solution containing 0.002 M  $\text{CaSO}_4$ , 0.002 M  $\text{MgSO}_4$ , 0.005 M  $\text{Ca}(\text{H}_2\text{PO}_4)_2$ , and 0.0025 M  $\text{K}_2\text{SO}_4$  (Curtin and Campbell, 2008). Leachates were collected under a consistent suction of 78 kPa using a vacuum pump (Fisher Scientific MaximaDry, Ontario, Canada). Immediately after leaching, the top of each column was covered with parafilm on which three holes had been punched for aeration. The columns were placed back into the incubators in an upright position. Deionized water was added to the soil weekly to replenish any moisture lost through evaporation. Leachates collected on each sampling day were immediately stored in a freezer at  $-19^\circ\text{C}$  and analyzed within a week for inorganic N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) concentration using a SAN++ segmented flow analyzer (Skalar BV, Breda, Netherlands).

#### 2.3.4. Calculations and Statistical Analysis

Net N mineralization ( $N_{\min(t)}$ ) for amended soil, measured as soil inorganic N concentration at time  $t$  and corrected for mineralization in the unamended soil and for initial soil inorganic N concentration, was calculated from the cumulative amounts of leached N ( $\text{NO}_3\text{-N} + \text{NH}_4\text{-N}$ ) as follows:

$$N_{\min(t)} = \text{IN}_{\text{A}(t)} - \text{IN}_{\text{A}(0)} - [\text{IN}_{\text{C}(t)} - \text{IN}_{\text{C}(0)}] \quad [1]$$

where  $N_{\min(t)}$  ( $\text{mg kg}^{-1}$  soil) is net mineralized N concentration at sampling time  $t$  (d);  $\text{IN}_{\text{A}(t)}$  ( $\text{mg kg}^{-1}$ ) is amendment-derived inorganic N concentration at sampling time  $t$ ;  $\text{IN}_{\text{A}(0)}$  ( $\text{mg kg}^{-1}$ ) is inorganic N concentration in the amended soil at the start of the experiment ( $t = 0$ );  $\text{IN}_{\text{C}(t)}$  ( $\text{mg kg}^{-1}$ ) is inorganic N concentration in the unamended control at time  $t$ ; and  $\text{IN}_{\text{C}(0)}$  ( $\text{mg kg}^{-1}$ ) is inorganic N concentration at  $t = 0$ .

Cumulative net N mineralized ( $\%N_{\min}$ ), or the percentage of manure organic N ( $N_{\text{org}}$ ) mineralized between the start of the experiment and time  $t$ , was calculated as:

$$\%N_{\min} = [N_{\min(t)} / N_{\text{org}}] \times 100 \quad [2]$$

Three kinetic models that are commonly used to describe N mineralization in soils (Table 2.2) were fitted to  $N_{\min(t)}$  and  $\%N_{\min}$  using the NLIN procedure in SAS version 9.4 (SAS Institute, 2014). Model fits were compared using the corrected Akaike's Information Criterion ( $\text{AIC}_C$ ; Motulsky and Christopolous, 2003). The mixed first-order and zero-order kinetic model (Eq. 3), which had the lowest  $\text{AIC}_C$ , was selected as the most suitable for describing N mineralization kinetics for the Black Chernozem:

$$N_{\min(t)} = N_b[1 - \exp(-k_1 t)] + k_0 t \quad [3]$$

where  $N_b$  is potentially mineralizable N concentration ( $\text{mg N kg}^{-1}$  soil),  $k_1$  is the first-order mineralization rate constant ( $\text{d}^{-1}$ ) of the easily degradable organic N fraction, and  $k_0$  is the zero-order mineralization rate constant of the resistant N fraction ( $\text{mg kg}^{-1} \text{d}^{-1}$ ). Model parameter estimates were

**Table 2.2 Kinetic models used in describing N mineralization in a Black Chernozem amended with beef cattle manure.**

Model	Equation	Parameters†	Reference
Simple exponential	$N_{\min(t)} = N_0 [1 - \exp(-k_i t)]$	$N_0, k_i$	Stanford and Smith (1972)
Hyperbolic model	$N_{\min(t)} = N_0 t / (q N_0 + t)$	$N_0, q$	Juma et al. (1984)
Special model	$N_{\min(t)} = N_b [1 - \exp(-k_1 t)] + k_0 t$	$N_b, k_0, k_1$	Bonde and Rosswall (1987)

†  $N_{\min}$ , mineralized N concentration (mg kg<sup>-1</sup> soil) at time t;  $N_0$ , potentially mineralizable N (mg N kg<sup>-1</sup>);  $k_i$ , mineralization rate constant (d<sup>-1</sup>);  $N_b$ , potentially mineralizable N of the easily degradable organic nitrogen fraction (mg N kg<sup>-1</sup>);  $k_1$ , first-order mineralization rate constant (d<sup>-1</sup>);  $k_0$ , zero-order mineralization rate constant (mg kg<sup>-1</sup> d<sup>-1</sup>);  $q$ , constant.

compared using their 95% confidence intervals. The mixed first-order and zero-order model includes parameters for N released from both the resistant and the easily mineralizable organic fractions. Nitrogen mineralization from the resistant fraction ensures continued N release after the active fraction is depleted, which precludes the identification of a single substrate pool or mineralization mechanism (Benbi and Richter, 2002).

Based on the AIC<sub>C</sub>, the first-order kinetic model was selected as the most suitable for describing %N<sub>min</sub> kinetics for the Black Chernozem:

$$\%N_{\min} = N_0[1 - \exp(-k_I t)] \quad [4]$$

where  $N_0$  is potentially mineralizable N concentration (% of organic N mineralized) and  $k_I$  is the first-order mineralization rate constant (d<sup>-1</sup>). Model parameter estimates were compared using their 95% confidence intervals.

Analysis of variance was conducted on the model parameters (generated separately for each experimental unit with PROC NLIN), using the GLIMMIX procedure in SAS 9.4, with amendment and temperature as fixed effects and using the gamma distribution for the parameters. Analysis of variance was also performed on the cumulative net N mineralized (N<sub>min</sub>) at the end of the experiment (that is, on Day 245) using the GLIMMIX procedure in SAS 9.4, with amendment and temperature as fixed effects and using the lognormal distribution for N<sub>min</sub>.

For the Black Chernozem only, the temperature sensitivity coefficient (Q<sub>10</sub>, i.e., the factor by which the mineralization rate increased or decreased when the temperature was raised by 10°C) for each amendment was calculated separately for each replicate as:

$$Q_{10} = \left( \frac{K_2}{K_1} \right)^{[10/(T_2 - T_1)]} \quad [5]$$

where  $K_1$  and  $K_2$  are the mineralization first-order rate constants (d<sup>-1</sup>) at 15 and 25°C, respectively, and  $T_1$  and  $T_2$  are incubation temperatures of 15 and 25°C, respectively. Analysis of variance was

conducted on the  $Q_{10}$  data using the GLIMMIX procedure in SAS 9.4, with amendment as a fixed effect and using the gamma distribution for  $Q_{10}$ .

Mineralized N concentration and  $\%N_{\min}$  data for the Brown Chernozem were not suitably described by any of the kinetic models tested; these data were therefore analyzed with the GLIMMIX procedure for repeated measures, in conjunction with the first-order autoregressive [AR(1)] covariance structure, using the normal distribution for both  $N_{\min}$  and  $\%N_{\min}$ . The beta distribution could not be used for  $\%N_{\min}$ , because some of the values were negative. Treatment differences were tested at  $\alpha = 0.05$  using the Tukey method for multiple comparisons.

## **2.4. Results and Discussion**

### **2.4.1. Nitrogen Mineralization Kinetics for the Black Chernozem**

Nitrogen mineralization kinetics in the Black Chernozem followed the mixed first-order and zero-order model for all amendments at both temperatures, except for  $DGM_{CDW}$ , for which the model failed to converge. This is consistent with previous studies (e.g., Dou et al., 1996; Haer and Benbi, 2003; Gil et al., 2011), which demonstrated the superiority of the mixed first-order and zero-order model compared with other N mineralization kinetic models tested in the studies. For example, in Gil et al. (2011), the mixed first-order and zero-order model outperformed the simple exponential, the double exponential, the hyperbolic, and the parabolic kinetic models with respect to describing N mineralization kinetics in compost-amended soils.

The effect of temperature on the mixed first-order and zero-order model varied with manure type. Model fits for the two temperatures differed significantly for DGM but not for RM (Fig. 2.1). For DGM,  $N_0$  was greater ( $P < 0.001$ ) at 25°C (209 mg kg<sup>-1</sup> soil) than at 15°C (142 mg kg<sup>-1</sup> soil).

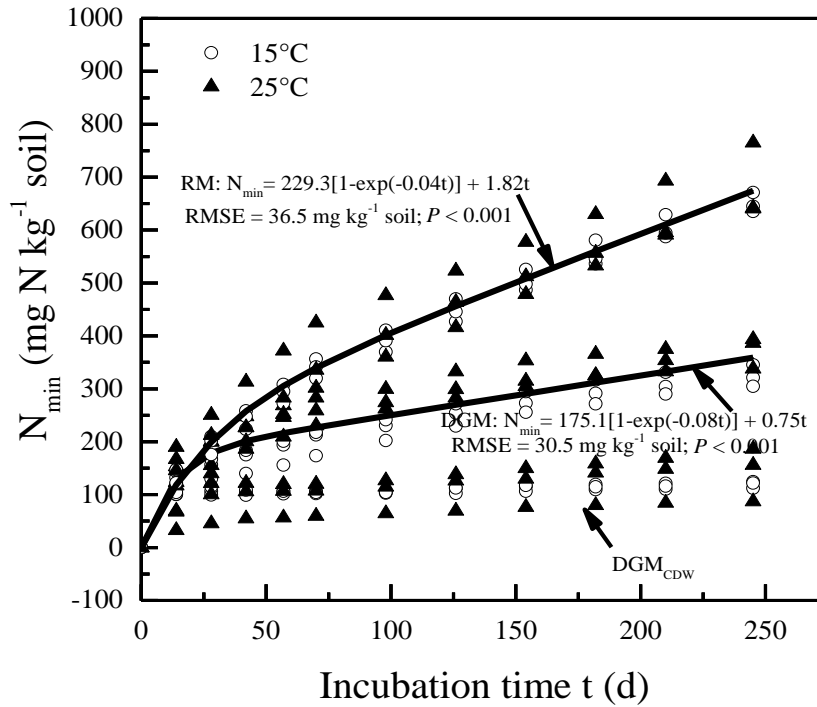


Figure 2.1 Mixed first-order plus zero-order kinetic model fits for cumulative N mineralization in a Black Chernozem amended with regular manure (RM) from cattle fed a typical beef cattle finishing diet and manure from cattle fed a DDGS diet (DGM).

Potentially mineralizable N concentration differed significantly between RM and DGM ( $P = 0.003$ ) and between temperatures ( $P = 0.004$ ) (Table 2.3), reflecting differences in organic N concentration between these manures. Averaged across temperatures,  $N_0$  was 27% greater for RM ( $235 \text{ mg kg}^{-1} \text{ soil}$ ) than DGM ( $172 \text{ mg kg}^{-1} \text{ soil}$ ). Averaged across amendments,  $N_0$  was greater at 25°C ( $234 \text{ mg kg}^{-1} \text{ soil}$ ) than at 15°C ( $172 \text{ mg kg}^{-1} \text{ soil}$ ). The zero-order rate constant ( $k_0$ ) was greater ( $P < 0.001$ ) for RM than DGM whereas the first-order ( $k_1$ ) rate constant was greater ( $P = 0.001$ ) for DGM than RM. Temperature had no significant effect on the two rate constants. The larger  $k_1$  for DGM indicates that the easily decomposable organic N pool was mineralized more rapidly for DGM than RM. By comparison, the large resistant N pool was mineralized at a lower rate ( $k_0$ ) for DGM than for RM.

These results suggest that it is the mineralizable N pool ( $N_0$ ) which is temperature dependent instead of the rate constants ( $k_0$  and  $k_1$ ). This corroborates findings by MacDonald et al. (1995), who observed no temperature effect on the first order rate constant in a 32-wk incubation study utilizing surface soils from a hardwood forest, but observed an increase in the mineralizable N pool as the temperature was increased from 5°C to 25°C. The authors attributed the increase in the mineralizable N pool with increasing temperature to a shift in the microbial community, changes in the biochemical composition of the fraction mineralized, and / or changes in transport processes such as diffusion. However, other studies have shown temperature-dependency of rate constants (Ellert and Bettany, 1992; Sierra, 1997; Dessureault-Rompré et al., 2010).

**Table 2.3 Amendment and temperature effects on mixed first-order and zero-order kinetic model parameters for net N mineralization ( $N_{\min}$ ) in a manure-amended Black Chernozem.**

Effect	$N_0^\dagger$ mg kg <sup>-1</sup> soil	$k_0^\ddagger$ mg kg <sup>-1</sup> d <sup>-1</sup>	$k_1^\S$ d <sup>-1</sup>
Amendment (A) <sup>¶</sup>			
DGM	172b	0.74b	0.1a
RM	235a	1.80a	0.04b
Temperature (T, °C)			
15	172b	1.20a	0.08a
25	234a	1.11a	0.05a
	————— <i>P</i> value —————		
Amendment	0.003	<0.001	0.001
Temperature	0.004	0.45	0.25
A × T	0.1	0.53	0.12

<sup>†</sup>  $N_0$ , potentially mineralizable N.

<sup>‡</sup>  $k_0$ , zero-order mineralization rate constant.

<sup>§</sup>  $k_1$ , first-order mineralization rate constant.

<sup>¶</sup> RM, manure from cattle fed a regular grain diet; DGM, manure from cattle fed a diet containing DDGS.

# Means in the same column followed by the same letter are not significantly different at  $\alpha = 0.05$  according to the Tukey multiple comparison procedure.

In the Black Chernozem, temporal changes in %N<sub>min</sub> followed first-order kinetics for all amendments at both temperatures (Fig. 2.2). However, temperature effects on the first-order rate constant ( $k_I$ ) varied with amendment, as indicated by an amendment  $\times$  temperature interaction (Table 2.4;  $P = 0.001$ ). For DGM<sub>CDW</sub>,  $k_I$  was significantly greater at 15°C (0.18 d<sup>-1</sup>) than at 25°C (0.04 d<sup>-1</sup>), but there were no significant differences in  $k_I$  between the two temperatures for RM (mean = 0.009 d<sup>-1</sup>) and DGM (0.02 d<sup>-1</sup>) (Fig. 2.3). At 15°C,  $k_I$  was significantly greater for DGM<sub>CDW</sub> (0.18 d<sup>-1</sup>) than RM (0.009 d<sup>-1</sup>) and DGM (0.02 d<sup>-1</sup>) but did not differ significantly between RM and DGM. At 25°C, DGM<sub>CDW</sub> also had the largest  $k_I$  (0.05 d<sup>-1</sup>), which was significantly greater than that for RM (0.009 d<sup>-1</sup>), while  $k_I$  was also significantly greater for DGM (0.03 d<sup>-1</sup>) than RM at this temperature. The larger  $k_I$  for DGM and DGM<sub>CDW</sub> at 25°C further indicates that the easily decomposable organic N pool was mineralized more rapidly for DGM and DGM<sub>CDW</sub> than RM.

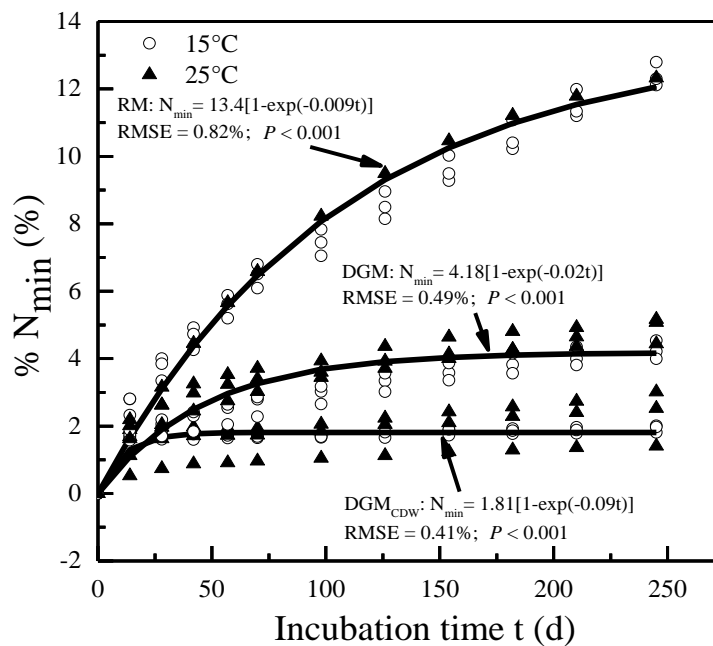


Figure 2.2 Temperature effects on first-order kinetic model fits for cumulative N mineralized as percent of organic N applied (%N<sub>min</sub>) in a Black Chernozem amended with regular manure (RM) from cattle fed a typical beef cattle finishing diet, manure from cattle fed a DDGS diet (DGM), and DGM mixed with CDW (DGM<sub>CDW</sub>).



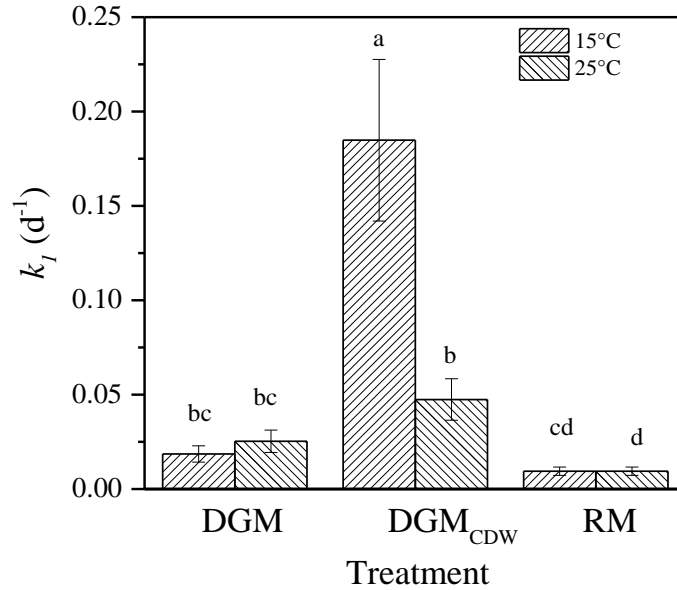


Figure 2.3 Effects of temperature on the first-order N mineralization rate constant in a Black Chernozem amended with manure from cattle fed a typical beef cattle finishing diet (RM), manure from cattle fed a DDGS diet (DGM), and DGM mixed with construction and demolition waste (DGM<sub>CDW</sub>). Error bars represent standard errors of the mean, and bars followed by different letters differ at  $P < 0.05$ .

Potentially mineralizable N concentration (% of initial organic N concentration) differed significantly among amendments ( $P < 0.001$ ) and between temperatures ( $P = 0.03$ ) (Table 2.4). Averaged across temperatures,  $N_0$  was significantly greater for RM (14%) than DGM (4.2%) and DGM<sub>CDW</sub> (1.9%) and greater for DGM than DGM<sub>CDW</sub>.

Cumulative N mineralization concentration ( $N_{min}$ ) on day 245 was greater ( $P < 0.001$ ) for RM (670 mg kg<sup>-1</sup>) than for DGM<sub>CDW</sub> (135 mg kg<sup>-1</sup>) and DGM (352 mg kg<sup>-1</sup>) (Table 2.5). This is consistent with the greater % $N_{min}$  observed for RM than for DGM and DGM<sub>CDW</sub>.

Our results for % $N_{min}$  are within the range reported by Chiyoka et al. (2014) for a Black Chernozem that received fresh beef cattle feedlot manure. The lower  $N_{min}$  with DGM<sub>CDW</sub> than DGM could possibly be due to the more recalcitrant C or higher lignin content of the woodchip fraction of

the CDW, which could have resulted in greater N immobilization when  $DGM_{CDW}$  was applied (Miller et al., 2009, 2010).

**Table 2.4 Amendment and temperature effects on first-order kinetic model parameters for net N mineralization as a percentage of initial organic N concentration ( $\%N_{min}$ ) in a manure-amended Black Chernozem and the  $Q_{10}$  function.**

Effect	$N_0^\dagger$	$k_1^\ddagger$	$Q_{10}^\S$
	%	$d^{-1}$	
Amendment (A) <sup>¶</sup>			
DGM	4.24b	0.02	1.31a
$DGM_{CDW}$	1.86c	0.09	0.3b
RM	13.62a	0.009	1.0a
Temperature (T, °C)			
15	4.49a	0.03	-
25	5.03a	0.02	-
	<hr/> <i>P</i> value <hr/>		
Amendment	<0.001	<0.001	0.01
Temperature	0.03	0.44	-
A × T	0.64	0.001	-

<sup>†</sup>  $N_0$ , potentially mineralizable N as a percentage of initial organic N concentration in the manure applied.

<sup>‡</sup>  $k_1$ , first-order mineralization rate constant.

<sup>§</sup>  $Q_{10}$ , the factor by which the mineralization rate increased per 10°C increase in temperature.

<sup>¶</sup> RM, manure from cattle fed a regular grain diet; DGM, manure from cattle fed a diet containing DDGS;  $DGM_{CDW}$ , DGM mixed with construction and demolition waste (CDW).

# Means in the same column followed by the same letter are not significantly different at  $\alpha = 0.05$  according to the Tukey multiple comparison procedure.

**Table 2.5 Amendment, temperature, and incubation time effects on cumulative net N mineralized in Black and Brown Chernozems.**

Effect	N <sub>min</sub> on day245†	N <sub>min</sub> ‡	%N <sub>min</sub> §
	mg kg <sup>-1</sup> soil Black Chernozem	mg kg <sup>-1</sup> soil Brown Chernozem	%
Amendment (A)¶			
DGM	352b	-208a	-2.72a
DGM <sub>CDW</sub>	135c	-208a	-3.38ab
RM	670a	-182a	-3.48b
Temp (T, °C )			
15	362a	-198a	-3.16a
25	409a	-201a	-3.22a
	<hr/> P value <hr/>		
Amendment	<0.001	0.21	0.02
Temp	0.37	0.82	0.78
Time (D)	-	<0.0001	<0.0001
D × T	-	<0.0001	<0.0001
D × A	-	<0.0001	<0.0001
T × A	0.73	0.64	0.65
D × T × A	-	0.73	0.67

† N<sub>min</sub> on day 245, cumulative net mineralized N at the end of the incubation on day 245.

‡ N<sub>min</sub>, cumulative N mineralized from N sources in a Brown Chernozem.

§ %N<sub>min</sub>, cumulative net mineralized N from manure organic N expressed as a percentage of the organic N applied with the manure in a Brown Chernozem.

¶ RM, manure from cattle fed a regular grain diet; DGM, manure from cattle fed a diet containing DDGS; DGM<sub>CDW</sub>, DGM mixed with construction and demolition waste (CDW).

#Means in the same column followed by the same letter are not significantly different at  $\alpha = 0.05$  according to the Tukey multiple comparison procedure.

†† Time main effect for N<sub>min</sub> and %N<sub>min</sub> is not shown in the ANOVA table because of the many levels of time effect; however, it is plotted in a graph when time × amendment interaction is significant.

The differences in  $N_{\min}$  between DGM and  $DGM_{CDW}$  could also possibly be due to the dilution effect resulting from mixing the CDW with DGM before it was applied to soil on a weight basis. The addition of CDW increased manure C:N ratio from 12:1 for DGM to 13:1 for  $DGM_{CDW}$ . Other studies have reported lower N mineralization at C:N > 15 (Qian and Schoenau, 2002; Helgason et al., 2007). Miller et al. (2009, 2010) have reported slow N mineralization rates in soils that received organic amendments with high C:N ratios (> 25:1) or high recalcitrant C contents from woodchip bedding. This suggests that a greater proportion of the organic N in DGM and RM will be readily converted to plant available forms ( $NH_4^+-N$ ) while lower amounts will be available with the application of  $DGM_{CDW}$ .

The temperature sensitivity,  $Q_{10}$ , of mineralization varied among amendments ( $P = 0.01$ ) and was significantly lower for  $DGM_{CDW}$  (0.3) than RM (1.0) and DGM (1.3) (Table 2.4). These results indicate that the N mineralization rate of RM and, to a lesser extent, DGM, is not affected by temperature within the range of temperatures tested, and that the addition of CDW to DGM results in a decrease in the mineralization rate when the temperature is increased by 10°C. The  $Q_{10}$  values for RM and DGM fall within the range of values reported by Whalen et al (2001) for a Dark Brown Chernozem in which beef cattle manure was applied. The authors reported that the N mineralization rate did not double in response to a 10°C increase in temperature ( $Q_{10}$ ). However, they found that soils incubated at 75% of field moisture capacity had lower  $Q_{10}$  values than soils incubated at 50% of field moisture capacity. This further indicates that temperature effect on N mineralization is greater in oxic soils than in soils close to anoxic or suboxic conditions.

The lower  $Q_{10}$  for  $DGM_{CDW}$  could be partly due to the high C:N ratio and high recalcitrant C content of the woodchip fraction of CDW, which is more resistant to decomposition. Studies have shown that  $Q_{10}$  will vary depending on soil characteristics such as pH, texture, and water content,

which influence microbial activity, coupled with the quantity and quality of the substrate that is being mineralized (Kirschbaum, 1995; Dessureault-Rompré et al., 2010).

#### 2.4.2. Mineralized Nitrogen Concentration in the Brown Chernozem

All organic amendments resulted in net immobilization ( $N_{\min} < 0$ ) in the Brown Chernozem on all sampling days, except on Day 14 when a spike was observed for DGM (Table 2.5; Fig. 2.4). Averaged across temperatures and incubation times,  $\%N_{\min}$  was lower for DGM than RM, whereas there was no significant difference between DGM<sub>CDW</sub> and the other two amendments. The negative mineralization observed in the manure-amended Brown Chernozem has previously been reported for a Dark Brown Chernozem amended with cattle manure (Chiyoka et al., 2014). They attributed the greater immobilization effect in the amended soil to the lower initial inorganic N concentration and organic C of the Dark Brown compared to the Black Chernozem.

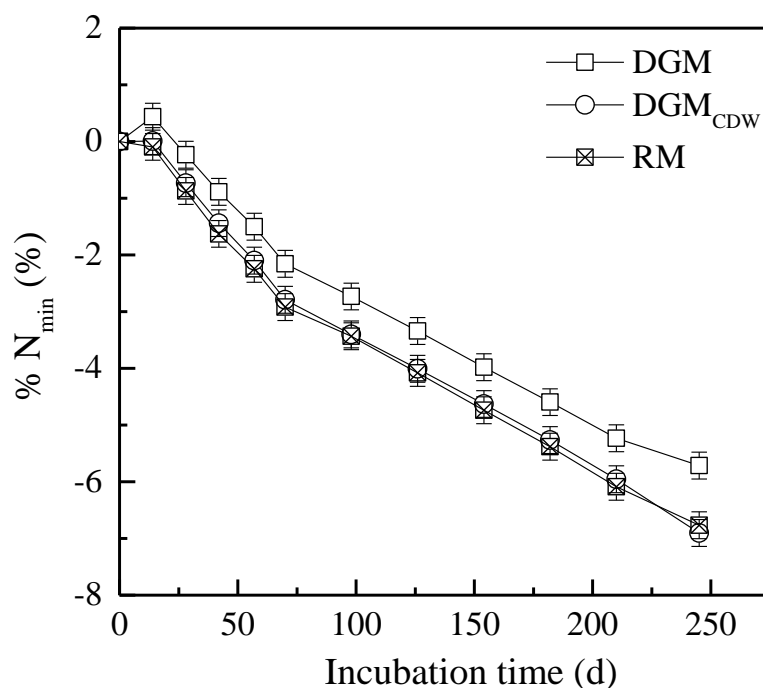


Figure 2.4 Cumulative N mineralized as a percentage of initial organic N concentration ( $\%N_{\min}$ ) of beef cattle manure applied to a Brown Chernozem during the 240-d incubation period.

In the present study, the Brown Chernozem had lower concentrations of total C and total N than the Black Chernozem, which may explain the observed apparent immobilization. It is also likely that the 60% WFPS maintained during incubation may have produced enough anaerobic conditions to cause some denitrification in the Brown Chernozem (Sierra, 1997; Drury et al., 2003; Coyne, 2008). However, other studies have reported that denitrification is favored when the soil water content is high, e.g., 70-100% WFPS (Linn and Doran, 1984; Weir et al., 1993). Nonetheless, our results suggest that manure N may not be readily available for crop uptake when applied to the Brown Chernozem under the conditions of the present study. A greenhouse bioassay in which five growth cycles of canola were grown in the same soils amended with the same amendments (DGM, DGM<sub>CDW</sub>, and RM) also showed negative mineralization ( $N_{\min} < 0$ ) in the Brown Chernozem (Agomoh et al., 2017). Nitrogen uptake was consistently lower for all amendments in the Brown Chernozem compared with the Black Chernozem even though the Brown Chernozem had a greater initial inorganic N concentration. Additional research is needed to further elucidate the factors limiting N mineralization from organic N sources applied to the Brown Chernozem and to determine the optimum moisture content for N mineralization in this soil.

## **2.5. Conclusions**

The N mineralization pattern of manure differed between the Black Chernozem and the Brown Chernozem, with the Brown Chernozem exhibiting negative net mineralization (i.e., net immobilization) throughout the entire incubation period, whereas net mineralization occurred in the Black Chernozem. Nitrogen mineralization in the DGM- and RM-amended Black Chernozem followed mixed first-order and zero-order kinetics while the cumulative net N mineralized expressed as a percentage of initial organic N concentration ( $\%N_{\min}$ ) followed first-order kinetics. In general, cumulative N mineralization was greater in the RM- than the DGM-amended Black Chernozem.

More organic N was mineralized from RM and DGM at the higher temperature than the lower temperature, a reflection of greater microbial activity at higher temperature. However, the mineralization rate constants were not affected by temperature. The rate constant for the easily mineralizable N pool was greater for DGM than RM, whereas the rate of N mineralization for the slowly mineralizable N pool was greater for RM. Addition of CDW to DGM resulted in a decrease in N release compared with DGM and RM in the amended Black Chernozem. In addition, the  $Q_{10}$  (i.e., the factor by which the mineralization rate increased per 10°C increase in temperature) decreased from 1.31 to 0.30 with the addition of CDW to DGM. The slow N release pattern of DGM<sub>CDW</sub> could be beneficial since a large proportion of N is not mineralized and therefore less subject to loss via NO<sub>3</sub>-N leaching or denitrification. However, application of DGM<sub>CDW</sub> to crops may require supplementing with synthetic fertilizer to prevent N deficiency. This should be based on the crop's nutrient requirement and the soil test to minimize the risk of excess P build-up because of the high P concentration of DGM<sub>CDW</sub>.

## 2.6. References

- Agomoh, I., Zvomuya, F., Hao, X. and McAllister, T. A. 2017.** A bioassay of nitrogen availability in soils amended with manure from cattle fed DDGS: Effects of construction/demolition waste and peat moss. *Can. J. Soil Sci.* doi:10.1139/cjss-2017-0031.
- APHA, AWWA, and WEF. 2005.** Standard methods for the examination of water and wastewater, 21st ed. American Public Health Association, American Water Works Association, and Water Environment Federation, New York, NY.
- Beauchamp, E. G. and Paul, J. W. 1989.** A simple model to predict manure N availability to crops in the field. P. 140-149. In J.A. Hansen and K. Henriksen (ed.) *Nitrogen in organic wastes applied to soils*. Harcourt Brace Jovanovich Publ., Boston.
- Benbi, D. K. and Richter, J. 2002.** A critical review of some approaches to modelling nitrogen mineralization. *Biol. Fertil. Soils* **35**: 168-183.

**Benke, M. B., Hao, X., Caffyn, P., Schoenau, J. J. and McAllister, T. A. 2010.** Using manure from cattle fed dried distillers' grains with solubles (DDGS) as fertilizer: Effects on nutrient accumulation in soil and uptake by barley. *Agri. Ecosyst. Environ.* **139**: 720-727.

**Bonde, T. A. and Rosswall, T. 1987.** Seasonal variation of potentially mineralizable nitrogen in four cropping systems. *Soil Sci. Soc. Am. J.* **51**: 1508-1514.

**Campbell, C. A., Ellert, B. H. and Jame, Y. W. 1993.** Nitrogen mineralization potential in soils. In: M.R. Carter, editor, *Soil sampling and methods of analysis*. Chapter 33. Canadian Society of Soil Science. Taylor & Lewis Publishers.

**Chiyoka, W. L., Zvomuya, F. and Hao, X. 2014.** Changes in nitrogen availability in Chernozemic soils amended with anaerobically digested cattle manure. *Soil Sci. Soc. Am. J.* **78**: 843-851.

**Coyne, M. S. 2008.** Biological denitrification. In: J.S. Schepers and W.R. Raun, editors, *Nitrogen in agricultural systems*. Agronomy Monograph 49. ASA, CSSA, and SSSA, Madison, WI. p. 201-254.

**Curtin, D. and Campbell, C. A. 2008.** Mineralizable nitrogen. In: In M.R. Carter and E.G. Gregorich, editors, *Soil sampling and methods of analysis*. Chapter 46. Canadian Society of Soil Science. Taylor & Francis Group.

**Dessureault-Rompré, J., Zebarth, B. J., Georgallas, A., Burton, D. L., Grant, C. A. and Drury, C. F. 2010.** Temperature dependence of soil nitrogen mineralization rate: Comparison of mathematical models, reference temperatures and origin of soils. *Geoderma* **157**: 97-108.

**Dou, Z., Toth, J. D., Jabro, J. D., Fox, R. H. and Fritton, D. D. 1996.** Soil nitrogen mineralization during laboratory incubation: Dynamics and model fitting. *Soil Biol. Biochem.* **28**: 625-632.

**Drury, C. F., Zhang, T. Q. and Kay, B. D. 2003.** The non-limiting and least limiting water ranges for soil nitrogen mineralization. *Soil Sci. Soc. Am. J.* **67**: 1388-1404.

**Ellert, B. H. and Bettany, J. R. 1992.** Temperature dependence of net nitrogen and sulfur mineralization. *Soil Sci. Soc. Am. J.* **56**: 1133-1141.

**FOBI Network. 2013.** Wheat dried distiller grains with solubles. Wheat DDGS feed guide. Feed Opportunities from Biofuels Industries. [Online]. Available: [https://cigi.ca/wp-content/uploads/2013/02/DDGS-Feed-Guide\\_Revised\\_Jan.-2013.pdf](https://cigi.ca/wp-content/uploads/2013/02/DDGS-Feed-Guide_Revised_Jan.-2013.pdf) (verified 7 April 2017).

**Gil, M. V., Carballo, M. T. and Calvo, L. F. 2011.** Modelling N mineralization from bovine manure and sewage sludge composts. *Bioresour. Technol.* **102**: 863-871.

**Haer, H. S. and Benbi, D. K. 2003.** Modeling nitrogen mineralization kinetics in arable soils of semiarid India. *Arid Land Res. Manag.* **17**: 153-168.



**Hao, X., Hill, B., Caffyn, P., Travis, G., Olson, A. F., Larney, F. J., McAllister, T. A. and Alexander, T. 2014.** Co-composting of beef cattle feedlot manure with construction and demolition waste. *J. Environ. Qual.* **43**: 1799-1808.

**Hao, X., Benke, M. B., Gibb, D. J., Stronks, A., Travis, G. and McAllister, T. A. 2009.** Effects of dried distillers' grains with solubles (wheat-based) in feedlot cattle diets on feces and manure composition. *J. Environ. Qual.* **38**: 1709-1718.

**Helgason, B. L., Larney, F. J., Janzen, H. H. and Olson, B. M. 2007.** Nitrogen dynamics in soil amended with composted cattle manure. *Can. J. Soil Sci.* **87**: 43-50.

**Hünerberg, M., McGinn, S. M., Beauchemin, K. A., Okine, E. K., Harstad, O. M. and McAllister, T. A. 2013.** Effect of dried distillers' grains with solubles on enteric methane emissions and nitrogen excretion from finishing beef cattle. *Can. J. Anim. Sci.* **93**: 373-385.

**International Grains Council. 2010.** Report for fiscal year 2009/10. [Online]. Available: <http://igc.int/downloads/publications/rfy/rfy0910.pdf> (verified 7 April 2017).

**Juma, N. G., Paul, E. A. and Mary, B. 1984.** Kinetic analysis of nitrogen mineralization in soil. *Soil Sci. Soc. Am. J.* **48**: 753-757.

**Kirschbaum, M. U. F. 1995.** The temperature dependence of soil organic matter decomposition, and the effect of global warming on soil organic storage. *Soil Biol. Biochem.* **27**: 753-760.

**Klopfenstein, T. J., Erickson, G. E. and Bremer, V. R. 2008.** Use of distillers by-products in the beef cattle feeding industry. *J. Anim. Sci.* **86**: 1223-1231.

**Kroetsch, D. and Wang, C. 2008.** Particle size-distribution. In: M.R. Carter and E.G. Gregorich editors, *Soil sampling and methods of analysis*. Chapter 55. Canadian Society of Soil Science. Taylor & Francis Group.

**Larney, F., Olson, A. F., Miller, J. J., DeMaere, P. R., Zvomuya, F. and McAllister, T. A. 2008.** Physical and chemical changes during composting of wood chip-bedded and straw-bedded beef cattle feedlot manure. *J. Environ. Qual.* **37**: 725-735.

**Linn, D. M. and Doran, J. W. 1984.** Effect of water-filled pore space on carbon dioxide production in tilled and nontilled soils. *Soil Sci. Soc. Am. J.* **48**: 1267-1272.

**MacDonald, N. W., Zak, D. R. and Pregitzer, K. S. 1995.** Temperature effects on kinetics of microbial respiration and net nitrogen and sulfur mineralization. *Soil Sci. Soc. Am. J.* **59**: 233-240.

**Miller, J. J., Beasley, B. W., Drury, C. F. and Zebarth, B. J. 2010.** Available nitrogen and phosphorus in soil amended with fresh or composted cattle manure containing straw or wood-chip bedding. *Can. J. Plant Sci.* **90**: 341-354.

**Miller, J. J., Beasley, B. W., Drury, C. F. and Zebarth, B. J. 2009.** Barley yield and nutrient uptake for soil amended with fresh and composted cattle manure. *Agron. J.* **101**: 1047–1059.

**Motulsky, H. and Christopolous, A. 2003.** Comparing models using Akaike's Information Criterion (AIC). In: *Fitting models to biological data using linear and nonlinear regression. A practical guide to curve fitting.* GraphPad Software Inc., San Diego, CA. p. 143-148.

**Pennock, D., Bedard-Haughn, A. and Viaud, V. 2011.** Chernozemic soils of Canada: Genesis, distribution, and classification. *Can. J. Soil Sci.* **91**: 719-747.

**Qian, P. and Schoenau, J. J. 2002.** Availability of nitrogen in solid manure amendments with different C:N ratios. *Can. J. Soil Sci.* **82**: 219-225.

**Sandler, K. 2003.** Analyzing what's recyclable in C&D debris. *Biocycle* **44**: 51-54.

**SAS Institute. 2014.** SAS/STAT 13.2 User's guide. Version 9.4. Cary (NC): SAS Institute, Inc.

**Sierra, J. 1997.** Temperature and soil moisture dependence of N mineralization in intact soil cores. *Soil Biol. Biochem.* **29**: 1557-1563.

**Stanford, G. and Smith, S. J. 1972.** Nitrogen mineralization potentials of soils. *Soil Sci. Soc. Am. Proc.* **36**: 465-472.

**St. Luce, M., Whalen, J. K., Ziadi, N. and Zebarth, B. J. 2011.** Nitrogen dynamics and indices to predict soil nitrogen supply in humid temperate soils. *Adv. Agron.* **112**: 55-102.

**USDA Foreign Agricultural Service. 2010.** Canada: Annual biofuels. Global Agricultural Information Network (GAIN) report CA0023. [Online]. Available:

[https://gain.fas.usda.gov/Recent%20GAIN%20Publications/Biofuels%20Annual\\_Ottawa\\_Canada\\_7-13-2010.pdf](https://gain.fas.usda.gov/Recent%20GAIN%20Publications/Biofuels%20Annual_Ottawa_Canada_7-13-2010.pdf) (verified 7 April 2017).

**Weir, K. L., Doran, J. W., Power, J. F. and Walters, D. T. 1993.** Denitrification and the dinitrogen/nitrous oxide ratio as affected by soil water, available carbon, and nitrate. *Soil Sci. Soc. Am. J.* **57**: 66-72.

**Whalen, J. K., Chang, C. and Olson, B. M. 2001.** Nitrogen and phosphorus mineralization potentials of soils receiving repeated annual cattle manure applications. *Biol. Fertil. Soils* **34**:334-341.

**White, J. W. and Mastalerz, J. W. 1966.** Soil moisture as related to container capacity. *Proc. Amer. Hort. Sci.* **89**: 758-765.

**Yeheyis, M., Hewage, K., Alam, M. S., Eskicioglu, C. and Sadiq, R. 2013.** An overview of construction and demolition waste management in Canada: a lifecycle analysis approach to sustainability. *Clean Technol. Environ. Policy* **15**: 81-91.

**Zebarth, B. J., Drury, C. F., Tremblay, N. and Cambouris, A. N. 2009.** Opportunities for improved fertilizer nitrogen management in production of arable crops in eastern Canada: a review. *Can. J. Soil Sci.* **89**: 113-132.

### **3. A BIOASSAY OF NITROGEN AVAILABILITY IN SOILS AMENDED WITH MANURE FROM CATTLE FED DDGS: EFFECTS OF CONSTRUCTION/DEMOLITION WASTE AND PEAT MOSS**

#### **3.1. Abstract**

The use of construction and demolition waste (CDW) and peat moss as bedding in beef cattle feedlots may affect the amount of plant-available nitrogen (N) in manure. Such an effect, however, may differ between manure from cattle fed a regular grain diet (RM) and those fed dried distillers grains with solubles (DDGS) (DGM). We used five 40-day crop cycles in a greenhouse bioassay to determine the effects of RM and DGM manure containing CDW or peat moss on canola (*Brassica napus* L.) growth and N uptake in a Black Chernozem (loam, Typic Hapocryoll) and a Brown Chernozem (sandy clay loam, Aridic Haploboroll) with or without the addition of a nitrification inhibitor (nitrapyrin) in each cycle to minimize nitrate (NO<sub>3</sub>) leaching. Our results showed that the presence of CDW in DGM and RM manure depressed ( $P < 0.05$ ) cumulative dry matter yield (DMY) and N uptake in canola relative to manure without CDW, while the presence of peat increased ( $P < 0.05$ ) DMY and N uptake. Results suggest that it may be necessary to supplement CDW-amended DGM and RM manure with synthetic N fertilizer to supply adequate available N for plant uptake, since the addition of CDW decrease organic N mineralization.

#### **3.2. Introduction**

The steadily growing bioethanol industry generates large amounts of dried distillers grains with solubles (DDGS) as a by-product, which is fed to animals (Meyer et al., 2010; Salim et al., 2010). Most of the starch in the grain is utilized for ethanol production, resulting in a by-product with low starch and crude protein and mineral contents that are almost three times those in the original grain (Liu, 2011; Hünnerberg et al., 2013). Previous studies have shown that inclusion of

DDGS in cattle diets results in increased N and P concentrations in manure, which may pose a risk to the environment when land applied (Benke et al., 2010; Hünérberg et al., 2013). Often, the amount of DDGS added to the diet exceeds the crude protein requirements of the animal, resulting in increased excretion of both fecal and urinary N relative to cattle that are fed barley grain diets (Hao et al., 2009). Benke et al. (2010) reported greater soil total N, total P, and soil test P concentrations and greater N and P uptake by barley grown in a Dark Brown Chernozemic soil that received manure from cattle fed DDGS compared with manure from cattle fed a barley grain diet.

The type and quantity of bedding material used in confined animal feeding operations influence the nutrient content of manure (Choudhary et al., 1996; Miller et al., 2003). In cattle feedlots in western Canada, high volumes of bedding material are typically used in the winter months to keep animals warm by providing insulation from snow or the frozen ground (Miller et al., 2006; Larney et al., 2008). Recently, in southern Alberta, there has been a gradual shift from the conventional use of cereal (barley) straw as bedding material to wood shavings and sometimes woodchips mixed with drywall from construction and demolition waste (CDW) (Miller et al., 2006; Larney et al., 2008; Hao et al., 2014).

Studies have shown a five-fold increase in Ca content and an eighteen-fold increase in sulfate ( $\text{SO}_4^{2-}$ ) content of dairy manure when drywall was added to compost as compared to when it was absent (Naeth and Wilkinson, 2013). Hao et al. (2014) similarly reported that the addition of CDW to cattle manure prior to composting resulted in higher concentrations of water-extractable Ca and  $\text{SO}_4$ -S, but lower pH, water-soluble N,  $\text{NH}_4$ -N, and  $\text{PO}_4^{3-}$ . They attributed these differences to the high calcium sulfate content of the drywall.

Peat moss is another material that has been used as a soil conditioner in agriculture, horticulture, and forestry (N'Dayegamiye and Isfan, 1991; Jayasinghe et al., 2010). In a composting

experiment in Quebec, Canada, sphagnum peat moss was mixed with cattle manure (2:1 manure to peat moss ratio by volume) and composted for 36 mo (N'Dayegamiye and Isfan, 1991). The authors reported that peat moss and sawdust produced composts with similar qualities but that these composts did not produce as high a temperature as wood shavings.

Although the effects of CDW as a compost bulking agent are well documented (Saludes et al., 2008; Naeth and Wilkinson, 2013; Hao et al., 2014), to our knowledge, no published studies have examined the agronomic and environmental risks associated with land-application of manure containing CDW. Therefore, the overall objective of this study was to characterize the availability, crop uptake, and residual soil concentrations of N following repeated application of manure from cattle fed either DDGS or a regular grain diet, with the manure mixed with peat or CDW.

### **3.3. Materials and Methods**

#### **3.3.1. Organic Amendment**

Two types of beef cattle feedlot manure [regular manure (REG) and DDGS manure] were collected from barley straw-bedded pens at the Lethbridge Research and Development Centre feedlot in southern Alberta, Canada (Lat. 49°42'N; Long. 112°48'W). The REG manure was collected from cattle (British cross heifers) fed a typical western Canadian beef finishing diet consisting of 85% barley grain, 10% barley silage, and 5% mineral supplement (Hao et al., 2009). The DDGS manure was collected from cattle fed a diet consisting of 45% barley grain, 40% wheat DDGS, 10% barley silage, and 5% mineral supplement. Prior to soil application, each manure type was mixed, in a 5:1 ratio by mass, with either construction waste (a mixture of drywall and woodchips) or peat moss to attain a C:N ratio < 20:1. The C:N ratio < 20:1 has been used as a bench mark for positive N mineralization from manure after application to soil (Beauchamp and Paul, 1989; Qian and Schoenau, 2002).

### 3.3.2. Soils

Soils from the A horizon (0-15 cm) were collected from two sites in Alberta, Canada: a Black Chernozem (loam, Typic Haplocryoll) from a site near Olds (113° 57' 42" N, 51° 43' 46" W) and a Brown Chernozem (sandy clay loam, Aridic Haploboroll) from a site near Cranford (112° 20' 31" N, 49° 45' 51" W). The Olds site was under a canola-wheat-barley rotation and had been cropped to barley in the growing season preceding soil collection while the Cranford site was cropped to potatoes in the preceding growing season. Soils were air-dried, passed through a 7-mm mesh sieve, and stored at room temperature until the start of the experiment.

### 3.3.3. Experimental Design and Setup

The REG and DDGS manure were tested in separate experiments. The experimental layout for each manure type was a completely randomized design with a  $5 \times 2 \times 2$  factorial treatment layout and three replicates per treatment. The three factors were amendment [manure only, manure mixed with peat, manure mixed with CDW, inorganic fertilizer (FERT, 125 mg N kg<sup>-1</sup> soil as urea and 18 mg P kg<sup>-1</sup> soil as triple super phosphate) and an unamended control (ConT)], soil type (Brown Chernozem and Black Chernozem), and nitrapyrin (N-Serve®, 2-chloro-6-(trichloromethyl)-pyridine) rate (0 and 0.56 g kg<sup>-1</sup> soil). Sixty grams of each manure were thoroughly mixed with 1 kg of air-dried soil to approximate an application rate of 60 Mg ha<sup>-1</sup> (based on a bulk density of 1.1 Mg m<sup>-3</sup> for the Black Chernozem and 1.2 Mg m<sup>-3</sup> for the Brown Chernozem and a soil incorporation depth of 0.1 m), which is currently recommended in southern Alberta to supply approximately 150 kg N ha<sup>-1</sup> for irrigated cereal production (Benke et al., 2010). This manure rate supplied approximately 0.4 g N kg<sup>-1</sup> soil from REG manure and 0.5 g N kg<sup>-1</sup> soil from DDGS manure.

Amended soils were placed in 2-L plastic pots (17 cm wide at the top × 13 cm wide at the bottom × 13 cm deep) lined with doubled plastic bags. Canola (*Brassica napus* L. cv. InVigor L150)

seeds were pre-germinated by placing them on wet paper towels for 4 d. Seven uniform seedlings were then transplanted into each pot and thinned to three per pot after 7 d. The pots were placed in a greenhouse maintained at a photoperiod of 16 h with day/night temperatures of 23/17°C. The moisture levels in the pots were maintained at approximately 65% of water-filled pore space (WFPS, approximately 260 g kg<sup>-1</sup>). Five crop growth cycles, each 40 d long, were tested.

#### **3.3.4. Leaching Events**

One leaching event per growth cycle was conducted 15 d after amendment application and planting. Holes were punched in the plastic bags at the bottom of each pot. Approximately 300 to 650 mL of reverse osmosis (RO) water were used to leach the soils, and the leachate volume was recorded. The soil moisture content was measured gravimetrically immediately after each leaching event to estimate the volume of retained water. The holes in the plastic bags were sealed with tape after each leaching event to prevent further loss of water from the pots. Nitrate- and NH<sub>4</sub>-N concentrations were measured in the leachate samples as described below. The residual solution volume was added to the leachate volume to estimate the total volume containing NO<sub>3</sub>-N.

#### **3.3.5. Harvesting**

Canola was harvested 40 d after planting by cutting aboveground biomass at the soil surface using pruning shears. Research has shown that maximum canola biomass accumulation and N uptake rates occur at 21 to 42 d after emergence (Malhi et al., 2007). Biomass samples from each growth cycle were oven-dried at 60°C for 48 h to estimate dry matter yield (DMY). Samples were fine-ground (< 0.15 mm) with a Cyclone plant tissue grinder (Udy Corp., Fort Collins, CO) prior to analysis for total N concentration as described below for the organic amendments.

Soil from each pot was emptied into an aluminum pan, mixed, and subsampled for laboratory analysis. Roots in the remaining soil were chopped and mixed back into the soil, followed by



reapplication of the same amendment applied in the preceding cycle 2 d after harvest. The soils were reseeded to canola to start the next crop cycle. This was repeated four times, resulting in five crop cycles over a 200-d period.

### **3.3.6. Laboratory Analysis**

#### **3.3.6.1. Manure**

Manure pH was measured in a 1:4 manure :water (mass/volume) suspension using a pH meter (Model 290A, Orion, Boston, MA), after which suspensions were filtered for measurement of electrical conductivity (EC) using an EC meter (Model 125A, Orion, Boston, MA). Immediately after EC measurement, the solution was filtered through a 0.45- $\mu\text{m}$  Whatman filter. Water-extractable  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$  concentrations in the supernatant were then measured using an ion chromatograph (Model Dx-600, Dionex, Sunnyvale, CA). Inorganic N ( $\text{IN} = \text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) was extracted from fresh manure (1 g) using 25 mL of 2 M KCl and determined using an Auto-analyzer III (Bran + Luebbe, Germany). Manure samples were freeze-dried and fine-ground ( $< 0.15$  mm) and then analyzed for total N (TN) and total C (TC) concentrations using an automated CN analyzer (Carlo Erba NA 1500, Carlo Erba, Milan, Italy). Percent inorganic N (PIN) concentration was calculated as  $\text{IN}/\text{TN} \times 100$ . Inorganic P (IP) was extracted with 25 mL of 0.5 M  $\text{NaHCO}_3$  from 0.25 g of fresh manure. Phosphorus concentration in the extract was measured using an EasyChem Pro discrete analyzer (Systea Analytical Technologies, Anagni, Italy). Total P (TP) concentration was measured using the EasyChem Pro discrete analyzer following acid digestion with  $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$ .

#### **3.3.6.2. Soil**

Subsamples (40 g) of the soil ( $< 2$  mm) were analyzed for particle size distribution by the hydrometer method (Kroetsch and Wang, 2006). Soil pH and EC were measured using the same

method as described above for manure samples. Soil inorganic P concentration was analyzed with an EasyChem Pro discrete analyzer following extraction of soil (2.5 g dry wt.) with 25 mL of 0.5 M NaHCO<sub>3</sub> (Schoenau and O'Halloran, 2006). Soil IN, TN, TC, and TP concentrations were determined as described above for manure. Total inorganic C (TIC) concentration was measured by the method of Amundson et al. (1988). Total organic C was estimated as the difference between TC and TIC.

### 3.3.7. Calculations

For each treatment, dry matter yields from all five growth cycles were summed to give cumulative dry matter yield (CDMY, g kg<sup>-1</sup> soil).

Cumulative N uptake (CNU) over the five growth cycles was calculated for each treatment by summing the uptake value from each of the five harvests. Amendment-derived N uptake (AmNU) (mg kg<sup>-1</sup> soil) was calculated as:

$$\text{AmNU} = (\text{NU}_A - \text{NU}_C) / M_s \quad [1]$$

where  $M_s$  is the mass of soil (kg) in each pot,  $\text{NU}_A$  is N uptake from the amended soil, and  $\text{NU}_C$  is N uptake from the unamended (control) soil.

Apparent N recovery (ANR, %) was calculated as

$$\text{ANR} = (\text{AmNU} / \text{total N applied}) \times 100 \quad [2]$$

Potentially leachable NO<sub>3</sub>-N (PLN) content in each pot (mg kg<sup>-1</sup> soil) for each growth cycle was calculated as the product of NO<sub>3</sub>-N concentration (mg L<sup>-1</sup> soil solution) in the total soil solution volume (leached plus residual):

$$\text{NO}_3\text{-N (mg kg}^{-1}\text{ soil)} = \text{NO}_3\text{-N} \times \text{total volume} \quad [3]$$

Several leachate samples from the Black Chernozem soil collected in Cycle 3 were lost before soluble NO<sub>3</sub>-N measurement. The missing leachate NO<sub>3</sub>-N concentrations for the Black

Chernozem in Cycle 3 were estimated by expressing leachate NO<sub>3</sub>-N concentrations for Cycles 1, 2, 4, and 5 as proportions of the corresponding total mineralized N concentrations (that is, NO<sub>3</sub>-N in leachate plus mineralized N in the soil) and regressing the proportions against the cumulative time (cycle) since the start of the experiment. Any significant linear regression detected was then used to estimate the amount of NO<sub>3</sub>-N in the leachate collected in Cycle 3. If the proportion did not vary significantly with cycle, the mean proportion of the four cycles was used to estimate leachate NO<sub>3</sub>-N concentration in Cycle 3. Cumulative potentially leachable NO<sub>3</sub>-N (CPLN) content for each treatment was calculated by summing the NO<sub>3</sub>-N contents for the 5 growth cycles.

The concentration of N mineralized from organic amendments (N<sub>min</sub>, mg kg<sup>-1</sup>) in the first growth cycle was calculated as

$$N_{\min} = \text{AmNU} + (\text{IN}_A - \text{IN}_C) - \text{AmN}_A + (\text{NL}_A - \text{NL}_C) \quad [4]$$

where IN<sub>A</sub> and IN<sub>C</sub> are inorganic N concentrations (mg kg<sup>-1</sup> soil) in the amended and unamended soils, respectively, after the first growth cycle, AmN<sub>A</sub> is the amendment inorganic N added (mg kg<sup>-1</sup> soil), and NL<sub>C</sub> and NL<sub>A</sub> are NO<sub>3</sub>-N concentrations (mg kg<sup>-1</sup> soil) in the leachate in the unamended and amended soils, respectively, on Day 14 of the cycle. For each of the subsequent growth cycles, N<sub>min</sub> was calculated as

$$N_{\min} = \text{AmNU}_f + [\text{IN}_{Af} - \text{IN}_{Ai} - (\text{IN}_{Cf} - \text{IN}_{Ci})] - \text{AmN}_{Af} + (\text{NL}_A - \text{NL}_C) \quad [5]$$

where subscripts *i* and *f* represent the start and the end of the growth cycle, respectively.

### 3.3.8. Statistical Analysis

Statistical analysis was conducted separately for the DDGS and REG manure treatments, consistent with the experimental layout described above. Analysis of variance for all dependent variables was performed using the GLIMMIX procedure for repeated measures in SAS version 9.4 (SAS Institute, 2014), with soil, amendment, and nitrapyrin as fixed effects and growth cycle as the

repeated measures factor. Various covariance structures were compared and the one with the lowest corrected Akaike Information Criterion (Littell et al., 2006) was selected as the best fit for the repeated measures analysis. When treatment effects were significant, means were compared using the Tukey-Kramer adjustment for multiple comparisons. Contrasts were performed using the ESTIMATE statement in SAS to compare combinations of amendment treatments. Effects were considered significant at  $\alpha = 0.05$ .

### **3.4. Results**

#### **3.4.1. Manure and Soil Properties**

Chemical properties differed considerably among the DDGS and REG manures used in this study (Table 3.1). Electrical conductivity was higher for DDGS (mean  $7.4 \text{ dS m}^{-1}$ ) than REG manure (mean  $6.8 \text{ dS m}^{-1}$ ). While total N concentrations were similar for DDGS (mean  $25 \text{ g kg}^{-1}$ ) and REG manure (mean  $23 \text{ g kg}^{-1}$ ), total available N ( $\text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) concentration was higher for DDGS (mean  $3891 \text{ mg kg}^{-1}$ ) than REG manure (mean  $2364 \text{ mg kg}^{-1}$ ). Similarly, the percentage of total N in the available form was greater for DDGS (16%) than REG manure (10%). Total P concentration was lower for REG manure (mean  $5.4 \text{ g kg}^{-1}$ ) than DDGS manure (mean  $7.0 \text{ g kg}^{-1}$ ). Addition of CDW and peat moss to the manures altered their properties to different degrees (Table 3.2).

Soils used in the experiment differed in their initial chemical properties (Table 3.1). The Black Chernozem had higher initial EC ( $0.59$  vs.  $0.34 \text{ dS m}^{-1}$ ), total C concentration ( $56$  vs.  $23 \text{ g kg}^{-1}$ ), and total N concentration ( $4.8$  vs.  $2.0 \text{ g kg}^{-1}$ ) than the Brown Chernozem.

#### **3.4.2. Cumulative Dry Matter Yield**

For both manure types, there was a significant soil  $\times$  amendment treatment interaction for cumulative dry matter yield (CDMY) from the five canola harvests (Tables 3.3 and 3.4). Averaged

across nitrapyrin levels, CDMY in the Black Chernozem was significantly greater for non-amended manure (14 and 15 g kg<sup>-1</sup> soil for RM and DGM, respectively) than for REG and DDGS manure amended with CDW (11 and 13 g kg<sup>-1</sup> soil for RM<sub>CDW</sub> and DGM<sub>CDW</sub>, respectively), FERT (11 g kg<sup>-1</sup> soil), and ConT (6 g kg<sup>-1</sup> soil), but did not differ significantly between the manure amended with peat moss (RMp and DGMp) (Figs. 3.1a and 3.1b). In the Brown Chernozem, CDMY was significantly greater for the peat-amended manures (10 and 12 g kg<sup>-1</sup> soil for RMp and DGMp, respectively) than for all the other treatments.

**Table 3.1 Initial properties of soils and organic amendments used in the experiment.**

Property‡	Black Chernozem	Brown Chernozem	REG†			DDGS		
			RM	RM <sub>p</sub>	RM <sub>CDW</sub>	DGM	DGM <sub>p</sub>	DGM <sub>CDW</sub>
pH	7.72 ± 0.01§	8.05 ± 0.02	8.7 ± 0.11	8.4 ± 0.16	8.2 ± 0.1	8.6 ± 0.1	8.3 ± 0.2	8.4 ± 0.1
EC, dS m <sup>-1</sup>	0.59 ± 0.01	0.34 ± 0.003	6.5 ± 0.1	6.1 ± 0.02	7.9 ± 0.1	7.7 ± 0.1	6.5 ± 0.2	8.1 ± 0.9
NO <sub>3</sub> <sup>-</sup> -N, mg kg <sup>-1</sup>	19 ± 1.7	30 ± 2.1	2.29 ± 0.35	1.18 ± 0.45	0.89 ± 0.57	4.57 ± 3.5	2.89 ± 0.9	2.17 ± 0.16
NH <sub>4</sub> <sup>+</sup> -N, mg kg <sup>-1</sup>	4 ± 0.3	2.38 ± 0.49	2592 ± 84	2616 ± 73	1881 ± 216	4219 ± 305	3996 ± 156	3447 ± 524
IN, mg kg <sup>-1</sup>	24 ± 1.9	32 ± 1.9	2594 ± 84	2617 ± 73	1882 ± 217	4224 ± 308	3999 ± 157	3449 ± 525
IP, mg kg <sup>-1</sup>	31 ± 2.4	27 ± 2.4	1715 ± 180	1364 ± 318	925 ± 74	1960 ± 236	1524 ± 125	896 ± 127
Total C, g kg <sup>-1</sup>	56 ± 0.4	23 ± 0.4	365 ± 6.6	381 ± 5.7	338 ± 16	337 ± 18	385 ± 5.6	330 ± 1.9
Organic C, g kg <sup>-1</sup>	56 ± 1.1	20 ± 1.2	-	-	-	-	-	-
Total N, g kg <sup>-1</sup>	4.77 ± 0.02	2.04 ± 0.04	26 ± 0.32	24 ± 0.7	18 ± 0.65	28 ± 1.3	25 ± 0.61	21 ± 0.94
PIN, %	-	-	9.9 ± 0.21	10.9 ± 0.06	10.7 ± 1.6	15 ± 1.6	15.9 ± 0.95	16.4 ± 2.6
Total P, g kg <sup>-1</sup>	0.2 ± 0.01	0.12 ± 0.04	6.71 ± 0.4	5.21 ± 0.7	4.29 ± 0.02	8.29 ± 0.53	6.48 ± 0.13	6.12 ± 0.19
C:N ratio	12 ± 0.04	11 ± 0.05	14 ± 0.41	16 ± 0.5	19 ± 0.79	12 ± 0.23	15 ± 0.47	16 ± 0.75
Total Ca, g kg <sup>-1</sup>	8.9 ± 0.04	13 ± 0.32	14 ± 1.4	13 ± 0.6	40 ± 1.8	13 ± 0.19	14 ± 0.41	35 ± 1.3
Dry matter Content, g kg <sup>-1</sup>	-	-	271 ± 6.2	315 ± 8.7	340 ± 18	303 ± 11	354 ± 13	375 ± 5.4
Clay, g kg <sup>-1</sup>	204 ± 11	227 ± 19	-	-	-	-	-	-
Silt, g kg <sup>-1</sup>	337 ± 12	273 ± 35	-	-	-	-	-	-
Sand, g kg <sup>-1</sup>	460 ± 20	500 ± 53	-	-	-	-	-	-
Total N applied, g kg <sup>-1</sup> soil	-	-	0.42	0.45	0.37	0.51	0.53	0.47

†REG, manure from beef cattle fed regular diets; RM, unamended REG; RM<sub>p</sub>, peat-amended REG; RM<sub>CDW</sub>, construction and demolition waste-amended REG; DGM, manure from beef cattle fed DDGS; DGM<sub>p</sub>, peat-amended DGM; DGM<sub>CDW</sub>, construction and demolition waste-amended DGM.

‡IN, inorganic N; IP, inorganic P; PIN, percent inorganic nitrogen. All chemical analysis expressed on a dry weight basis.

§Mean ± standard deviation (n = 3).

**Table 3.2 Chemical properties of peat moss and construction and demolition waste mixed with manure before applying to soil.**

Property	CDW†	Peat Moss
pH	6.24 ± 0.06‡	4.64 ± 0.07
EC, dS m <sup>-1</sup>	2.39 ± 0.02	0.12 ± 0.001
NO <sub>3</sub> <sup>-</sup> -N, mg kg <sup>-1</sup>	< 0.27	28 ± 4.7
NH <sub>4</sub> <sup>+</sup> -N, mg kg <sup>-1</sup>	1.11 ± 1.05	61 ± 6.3
Inorganic N, mg kg <sup>-1</sup>	2.36 ± 0.99	90 ± 1.6
Total C, g kg <sup>-1</sup>	264 ± 11	494 ± 4.1
Total N, g kg <sup>-1</sup>	1.44 ± 0.04	12 ± 0.76
C to N ratio	183 ± 6.9	40 ± 2.3
Total P, g kg <sup>-1</sup>	0.09 ± 0.02	0.24 ± 0.09
Inorganic P, mg kg <sup>-1</sup>	4.35 ± 1.56	6.59 ± 0.64
Water-extractable Ca, mg kg <sup>-1</sup>	12259 ± 272	708 ± 65
Water-extractable SO <sub>4</sub> <sup>2-</sup> , mg kg <sup>-1</sup>	29473 ± 222	790 ± 54

†Construction and demolition waste (CDW) bedding.

‡Mean ± standard deviation (n = 3).

**Table 3.3 Treatment effects on canola cumulative dry matter yield (CDMY), cumulative crop N uptake (CNU), amendment-derived N uptake (AmNU), apparent nitrogen recovery (ANR), soil available N (SAN), organic N mineralized (N<sub>min</sub>), potentially-leachable NO<sub>3</sub>-N (PLN), and cumulative PLN in Chernozems amended with REG manure.**

Effect	CDMY	CNU	AmNU	ANR	SAN	N <sub>min</sub>	PLN	CPLN
	g kg <sup>-1</sup> soil	mg kg <sup>-1</sup> soil		%		mg kg <sup>-1</sup> soil		
Soil								
Black Chernozem	11	224	195	42	51	85	89	2376
Brown Chernozem	7.6	157	154	31	45	5.76	73	1865
Amendment <sup>†</sup>								
RM	11	262	193	44	58	103	76	1808
RM <sub>CDW</sub>	9.7	181	112	26	43	-16	38	891
RM <sub>P</sub>	12	263	194	43	53	50	68	1623
FERT	9.4	291	216	35	70	-	209	4503
ConT	5.0	64	-	-	18	-	15	328
Nitrapyrin								
Applied	9.51	190	195	41	47	62	72	1853
No nitrapyrin	9.39	184	154	32	49	29	91	2303
Growth cycle								
1	-	-	-	-	37	-26	47	-
2	-	-	-	-	36	6.7	62	-
3	-	-	-	-	55	66	43	-
4	-	-	-	-	55	75	135	-
5	-	-	-	-	59	105	119	-
ANOVA	P value							
Soil (S)	<0.001	<0.001	<0.001	<0.001	0.03	<0.001	<0.001	<0.001
Amendment (A)	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Nitrapyrin (N)	0.49	0.19	<0.001	<0.001	0.46	0.001	0.22	0.06
Cycle (C)	<0.001	0.001	0.002	0.002	<0.001	<0.001	<0.001	-



$S \times A$	0.02	0.05	0.001	0.001	0.18	0.01	0.32	0.01
$S \times N$	0.003	<0.001	0.05	0.04	0.84	0.04	0.88	0.67
$A \times N$	-	-	-	-	0.06	0.55	0.27	0.02
$S \times C$	-	-	-	-	0.002	<0.001	0.03	-
$A \times C$	-	-	-	-	<0.001	<0.001	<0.001	-
$N \times C$	-	-	-	-	0.62	0.01	0.12	-
$S \times A \times N$	0.12	0.08	0.65	0.56	0.11	0.96	0.99	0.42
$S \times A \times C$	-	-	-	-	<0.001	0.02	<0.001	-
$S \times N \times C$	-	-	-	-	0.33	<0.001	0.23	-
$A \times N \times C$	-	-	-	-	0.03	0.11	0.02	-
$S \times A \times N \times C$	-	-	-	-	0.74	0.98	0.53	-
Contrasts					-	-	-	-
ConT vs. REG	<0.001	<0.001	-	-	<0.001	-	<0.001	<0.001
FERT vs. REG	<0.001	<0.001	<0.001	-	<0.001	-	0.05	<0.001

†RM, unamended manure from beef cattle fed regular diet (REG); RMp, peat-amended REG; RM<sub>CDW</sub>, construction and demolition waste-amended REG.

**Table 3.4 Treatment effects on canola cumulative dry matter yield (CDMY), cumulative crop N uptake (CNU), amendment-derived N uptake (AmNU), apparent nitrogen recovery (ANR), soil available N (SAN), organic N mineralized (N<sub>min</sub>), potentially-leachable NO<sub>3</sub>-N (PLN), and cumulative PLN in soils amended with DDGS manure.**

Effect	CDMY	CNU	AmNU	ANR	SAN	N <sub>min</sub>	PLN	CPLN
	g kg <sup>-1</sup> soil	mg kg <sup>-1</sup> soil		%		mg kg <sup>-1</sup> soil		
Soil								
Black Chernozem	12	275	289	53	71	198	154	4197
Brown Chernozem	8.6	200	222	40	64	33	102	2683
Amendment <sup>†</sup>								
DGM	12	365	296	57	103	198	164	3880
DGM <sub>CDW</sub>	12	266	199	40	64	5.33	83	1999
DGM <sub>p</sub>	13	392	323	56	83	144	168	3956
FERT	9.4	291	216	35	70	-	209	4493
ConT	5.0	64	-	-	18	-	15	327
Nitrapyrin								
Applied	10	239	280	51	68	146	121	3213
No nitrapyrin	10	230	229	41	68	86	135	3399
Growth cycle								
1	-	-	-	-	39	-31	52	-
2	-	-	-	-	48	50	93	-
3	-	-	-	-	79	162	98	-
4	-	-	-	-	78	158	189	-
5	-	-	-	-	94	239	208	-
ANOVA	<i>P</i> value							
Soil (S)	<0.001	<0.001	<0.001	<0.001	0.06	<0.001	<0.001	<0.001
Amendment (A)	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Nitrapyrin (N)	0.98	0.13	<0.001	<0.001	0.97	<0.001	0.36	0.12
Cycle (C)	<0.001	0.01	0.003	0.002	<0.001	<0.001	<0.001	-
S × A	0.58	0.24	0.02	0.02	0.86	0.001	0.01	0.004
S × N	0.03	<0.001	0.14	0.13	0.65	0.01	0.22	0.24

A × N	-	-	-	-	0.02	0.07	0.21	0.003
S × C	-	-	-	-	0.13	<0.001	0.42	-
A × C	-	-	-	-	<0.001	<0.001	<0.001	-
N × C	-	-	-	-	0.32	<0.001	0.62	-
S × A × N	0.56	0.11	0.32	0.33	0.35	0.29	0.62	0.13
S × A × C	-	-	-	-	0.01	0.13	0.004	-
S × N × C	-	-	-	-	0.001	0.02	0.03	-
A × N × C	-	-	-	-	0.03	0.40	0.08	-
S × A × N × C	-	-	-	-	0.002	0.81	0.23	-
Contrasts					-	-	-	-
ConT vs. DDGS	<0.001	<0.001	-	-	<0.001	-	<0.001	<0.001
FERT vs. DDGS	<0.001	<0.001	<0.001	-	0.01	-	0.001	0.01

†DGM, manure from beef cattle fed DDGS; DGMp, peat-amended DGM; DGM<sub>CDW</sub>, construction and demolition waste-amended DGM.

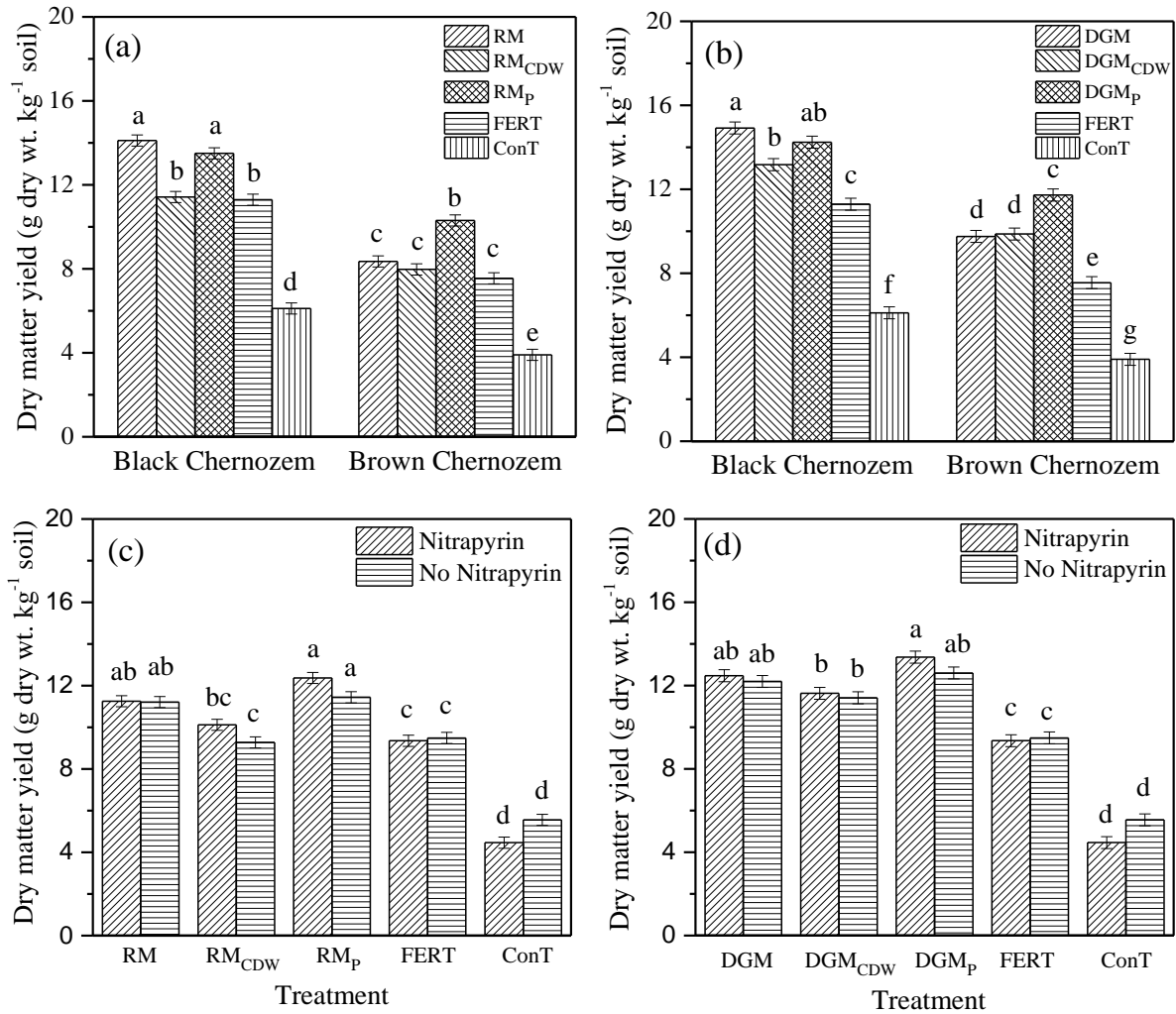


Figure 3.1 Cumulative dry matter yield, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across soils for each amendment and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM<sub>P</sub>, RM<sub>CDW</sub>, DGM, DGM<sub>P</sub>, DGM<sub>CDW</sub>, and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at P < 0.05.

Cumulative dry matter yield for FERT was similar to that for RM and RM<sub>CDW</sub> in the Brown Chernozem while it was only similar to RM<sub>CDW</sub> in the Black Chernozem. By comparison, CDMY for the FERT treatment was lower than for the DDGS manure treatments in both soils. For all

amendment treatments, including the control, CDMY was significantly greater in the Black Chernozem than the Brown Chernozem. Orthogonal contrast analysis indicated that, in both soils, manure treatments (RM, RM<sub>CDW</sub>, RMp, DGM, DGM<sub>CDW</sub>, and DGMp) significantly increased CDMY compared with the control (Tables 3.3 and 3.4). Cumulative dry matter yield was greater for the manure treatments than the FERT treatment regardless of nitrapyrin level.

There was also a significant ( $P = 0.003$  for both regular and DDGS manures) amendment  $\times$  nitrapyrin interaction for CDMY (Tables 3.3 and 3.4). While nitrapyrin application did not significantly affect CDMY from any of the amendments, it altered the difference in CDMY among some of the amendments. For regular manure, the treatment RM produced significantly greater CDMY than RM<sub>CDW</sub> in the absence of nitrapyrin, whereas these differences were not significant in the presence of nitrapyrin (Fig. 3.1c). By contrast, RMp significantly outyielded RM<sub>CDW</sub> with or without nitrapyrin application. These results for regular manure contrast with those for DDGS manure, for which CDW addition (DGM<sub>CDW</sub> treatment) did not significantly reduce CDMY relative to unamended DDGS manure (DGM treatment). Similarly, while peat moss addition (DGMp treatment) also produced greater CDMY than the DGM<sub>CDW</sub> treatment, this was significant only in the presence of nitrapyrin (Fig. 3.1d). Regardless of nitrapyrin level, the FERT treatment produced significantly lower CDMY than all organic amendments except RM<sub>CDW</sub>.

#### **3.4.3. Cumulative Nitrogen Uptake by Canola**

For both manure types, all amendment treatments significantly increased CNU relative to the unamended control. However, this effect varied with soil type, as indicated by the significant soil  $\times$  amendment interaction (Tables 3.3 and 3.4).

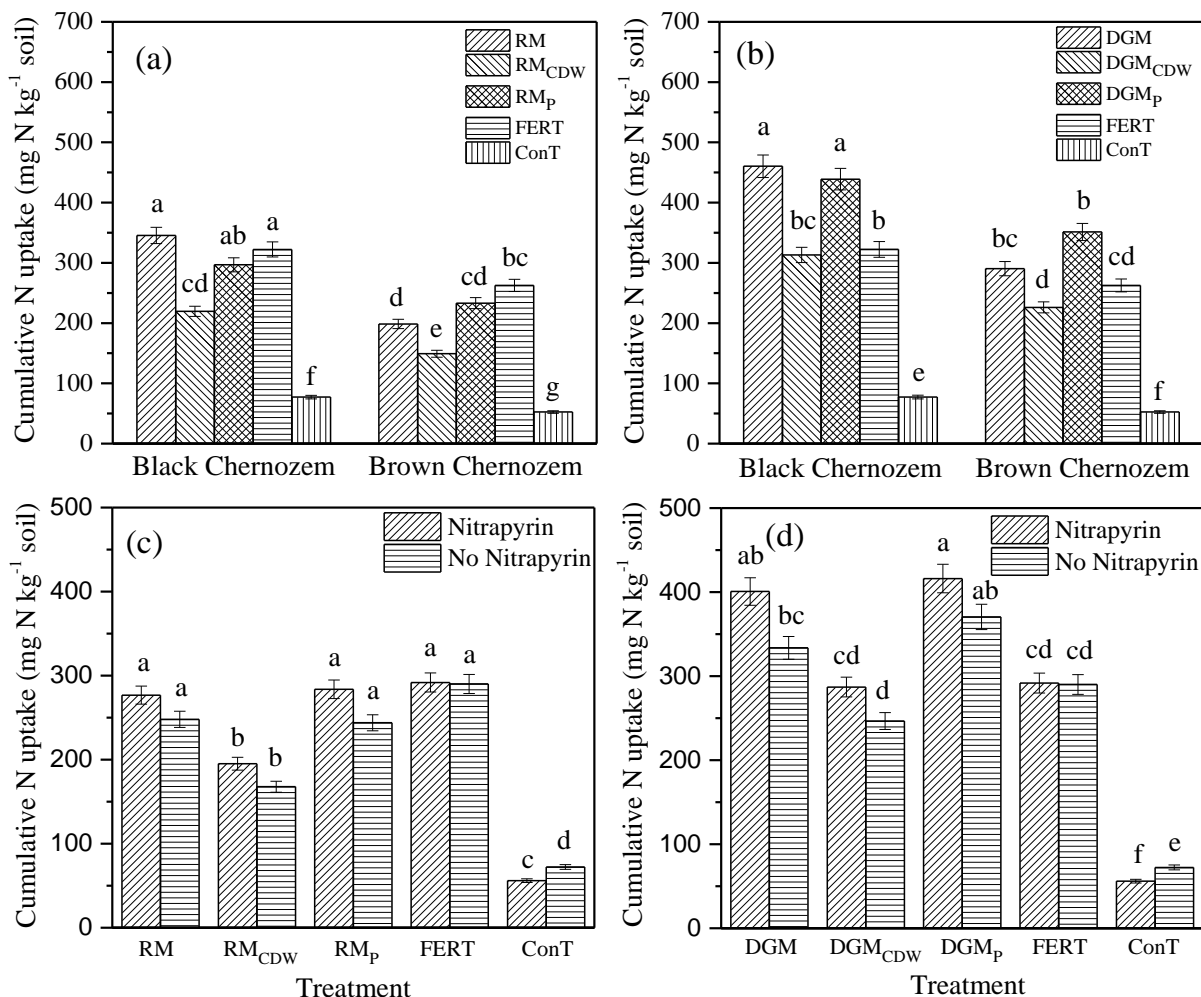


Figure 3.2 Cumulative N uptake, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across soils for each amendment and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM<sub>p</sub>, RM<sub>CDW</sub>, DGM, DGM<sub>p</sub>, DGM<sub>CDW</sub>, and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at  $P < 0.05$ .

For regular manure, CNU decreased in the order:  $RM \approx FERT \approx RM_p > RM_{CDW} > ConT$  in both soils (Fig. 3.2a). For DDGS manure, CNU decreased in the order:  $DGM \approx DGM_p > DGM_{CDW} \approx FERT > ConT$  in both soils (Fig. 3.2b). For both manure types, there was generally a significant

positive response in N uptake to all amendments compared with the control. For all amendment treatments, including the control, CNU was significantly greater in the Black Chernozem than the Brown Chernozem.

There was also a significant amendment  $\times$  nitrapyrin interaction for CNU ( $P < 0.001$  for both manure types; Tables 3 and 4). While nitrapyrin application had no significant effect on CNU for manured treatments, it significantly reduced CNU in the unmanured soils (treatment (ConT) (Figs. 3.2c and 3.2d).

#### **3.4.4. Amendment-derived Nitrogen Uptake**

Amendment-derived N uptake (AmNU) differed significantly among amendments ( $P < 0.001$ ), but amendment differences varied with soil type, as indicated by the significant ( $P = 0.002$  for regular manure and  $P = 0.003$  for DDGS manure) soil  $\times$  amendment interaction (Tables 3.3 and 3.4). For regular manure, amendment-derived N uptake decreased in the order: RM  $\approx$  FERT  $\approx$  RMp  $>$  RM<sub>CDW</sub> in both soils (Fig. 3.3a). Treatment rankings were slightly different for DDGS manure, for which AmNU decreased in the order: DGM  $\approx$  DGMp  $>$  DGM<sub>CDW</sub>  $\approx$  FERT in the Black Chernozem, whereas it decreased in the order: DGMp  $>$  DGM  $\approx$  FERT  $\approx$  DGM<sub>CDW</sub> in the Brown Chernozem (Fig. 3.3b). For all amendments except FERT and RMp, AmNU was greater in the Black Chernozem than the Brown Chernozem.

The soil  $\times$  nitrapyrin interaction was significant ( $P = 0.001$  for regular manure and  $P = 0.02$  for DDGS manure) for AmNU. For both manure types, nitrapyrin application significantly increased AmNU, averaged across amendment treatments, in the Brown Chernozem, whereas the increase in AmNU in the Black Chernozem was not significant (Fig. 3.3c and 3.3d). Amendment-derived N uptake was greater in the Black Chernozem than the Brown Chernozem regardless of nitrapyrin level.

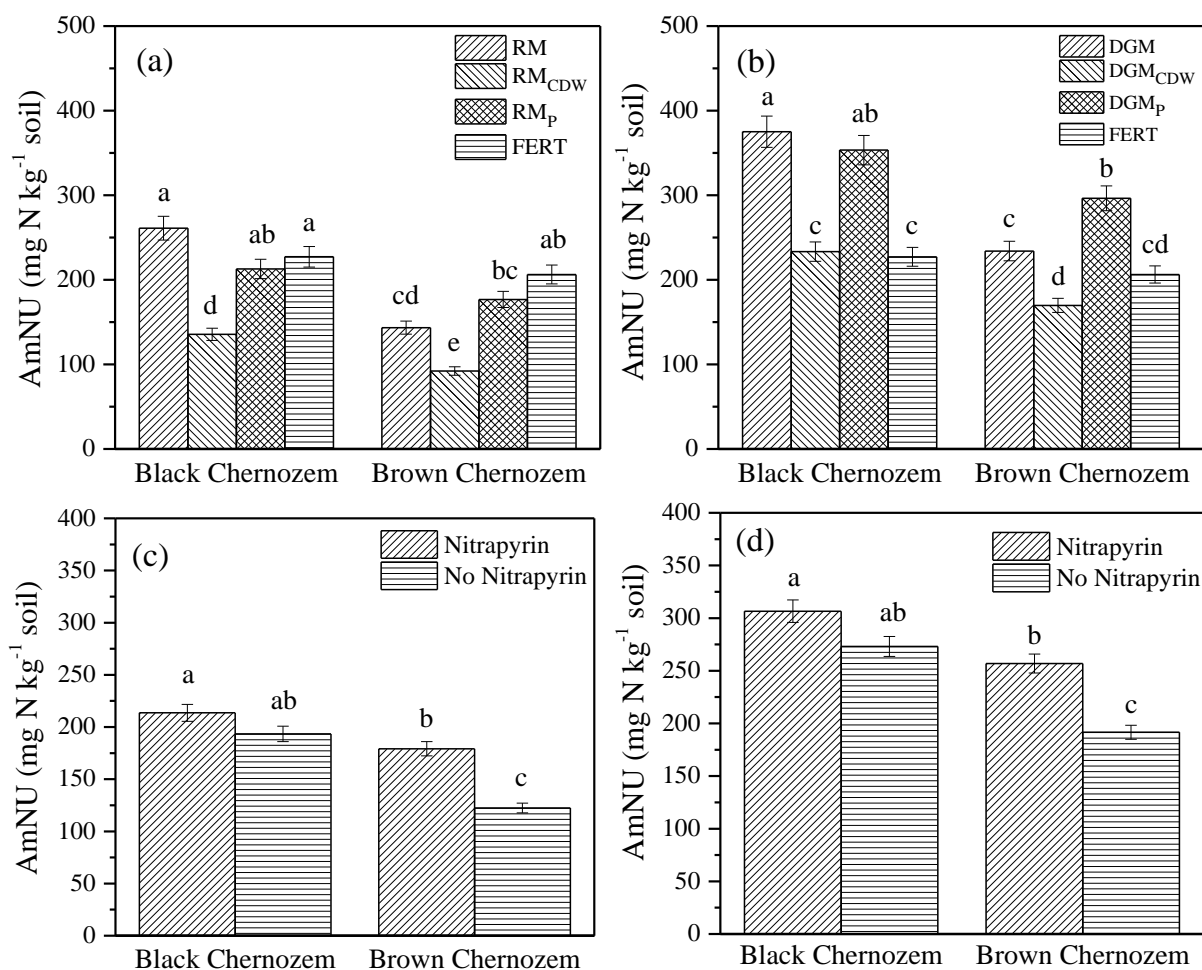


Figure 3.3 Amendment-derived N uptake, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across amendment for each soil and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM<sub>P</sub>, RM<sub>CDW</sub>, DGM, DGM<sub>P</sub>, DGM<sub>CDW</sub>, and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at P < 0.05.

### 3.4.5. Apparent Nitrogen Recovery

Apparent N recovery differed significantly among amendment treatments (P < 0.001 for both manure types), but treatment differences varied with soil type, as indicated by the significant (P = 0.002 for both regular and DDGS manures) soil × amendment interaction (Tables 3.3 and 3.4).



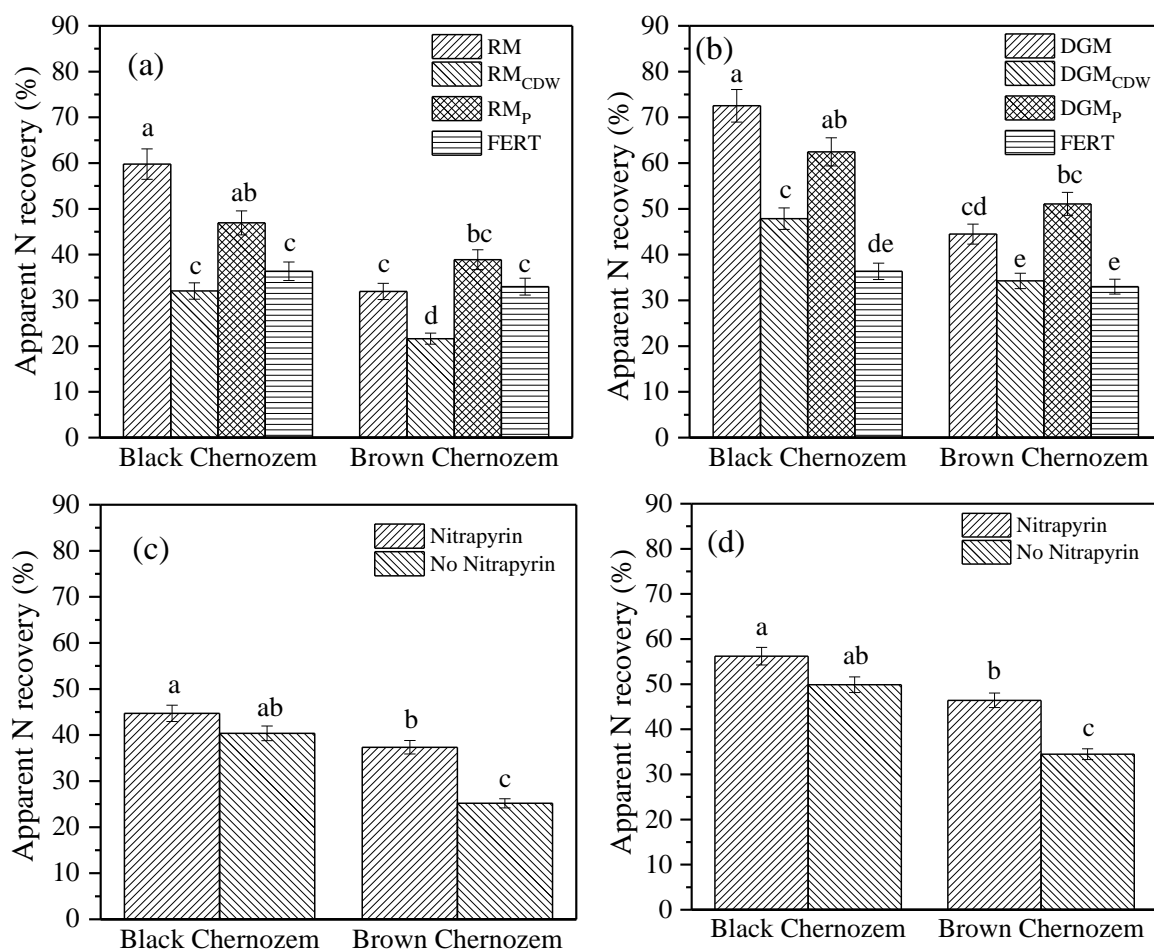


Figure 3.4 Apparent N recovery, averaged across nitrpyrin rates for each amendment and soil [(a) and (b)] and averaged across amendment for each soil and nitrpyrin application [(c) and (d)] for five growth cycles of canola grown in Chernozemic soils amended with RM,  $RM_{CDW}$ ,  $RM_P$ , DGM,  $DGM_{CDW}$ ,  $DGM_P$ , and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet; and subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively. Error bars represent standard errors of the mean, and bars followed by different letters differ at  $P < 0.05$ .

In both soils, ANR was significantly lower for  $RM_{CDW}$  than RM and  $RM_P$  (Fig. 3.4a), and lower for  $DGM_{CDW}$  than DGM and  $DGM_P$  (Fig. 3.4b). The soil  $\times$  nitrpyrin interaction was significant ( $P = 0.001$  for regular manure and  $P = 0.02$  for DDGS manure). For both manure types, averaged across amendments, ANR was significantly greater with nitrpyrin addition in the Brown Chernozem

compared with treatments receiving no nitrapyrin, whereas nitrapyrin had no significant effect on ANR in the Black Chernozem (Figs. 3.4c and 3.4d). Overall, ANR was significantly greater in the Black Chernozem than in the Brown Chernozem regardless of nitrapyrin level.

#### **3.4.6. Soil Available Nitrogen**

For regular manure, there was a significant ( $P = 0.03$ ) amendment  $\times$  nitrapyrin  $\times$  cycle interaction for SAN concentration (Table 3.3). With nitrapyrin applied, SAN concentration, averaged across soils, was significantly greater for FERT than  $RM_{CDW}$  and  $RMp$  in Cycles 2 and 5 and ConT in Cycles 2, 3, and 5 (Fig 3.5a). Soil available N was significantly lower in  $RM_{CDW}$  than  $RM$  in Cycle 3. Soil available N in  $RM$  amended soils increased significantly ( $P < 0.001$ ) from  $34 \text{ mg kg}^{-1}$  in Cycle 2 to  $92 \text{ mg kg}^{-1}$  in Cycle 3 after which there was no significant change. There was also a significant increase in SAN for  $RMp$  from  $31 \text{ mg kg}^{-1}$  in Cycle 2 to  $60 \text{ mg kg}^{-1}$  in Cycle 3, after which there was no significant change. Soil available N increased significantly from  $58 \text{ mg kg}^{-1}$  in Cycle 4 to  $111 \text{ mg kg}^{-1}$  in Cycle 5 for FERT amended soils. In the absence of nitrapyrin, SAN concentration, averaged across soils, was significantly lower in ConT than FERT except in Cycle 4, and  $RM$  and  $RMp$  in Cycles 3, 4, and 5, and  $RM_{CDW}$  in Cycle 4 (Fig 3.5b). There was a significant decrease in SAN for FERT from  $75 \text{ mg kg}^{-1}$  in Cycle 3 to  $34 \text{ mg kg}^{-1}$  in Cycle 4, followed by a significant increase to  $85 \text{ mg kg}^{-1}$  in Cycle 5.

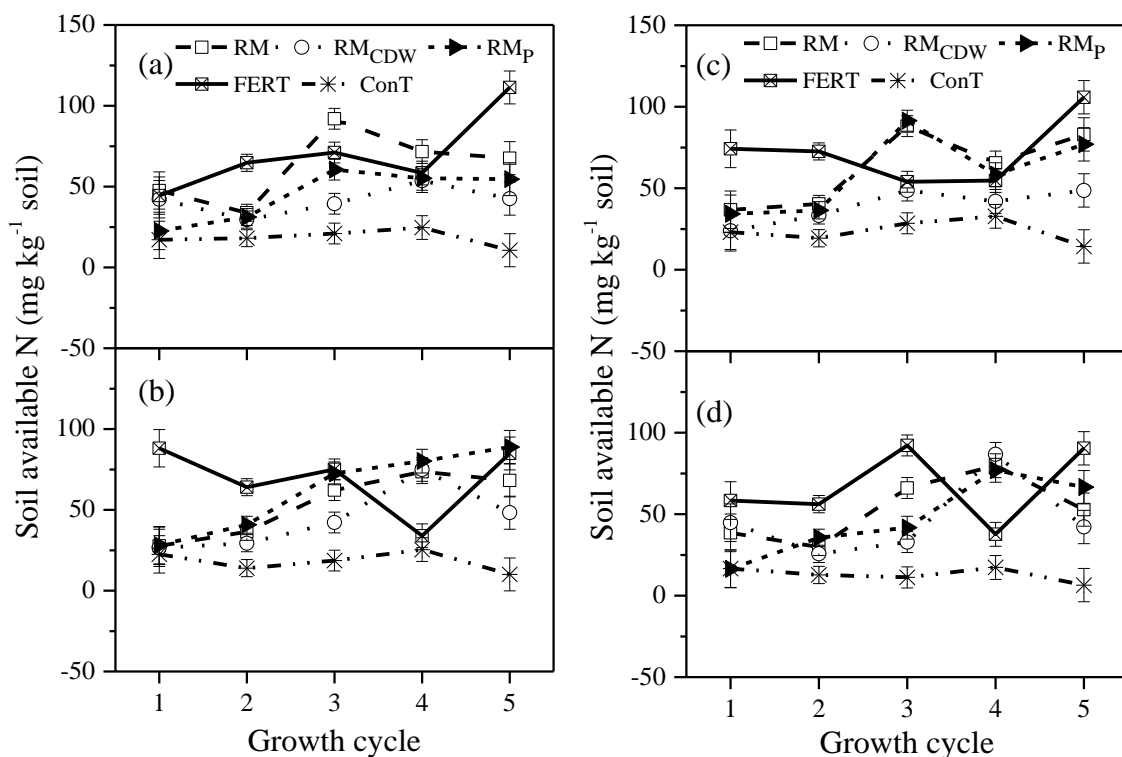


Figure 3.5 Soil available N (i) averaged across soils with (a) and without (b) nitrapyrin addition; (ii) averaged across nitrapyrin in the Black Chernozem (c) and in the Brown Chernozem (d) amended with RM, RM<sub>P</sub>, RM<sub>CDW</sub>, and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, the subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is a mineral fertilizer. Vertical bars represent standard errors of the mean.

There was also a significant ( $P < 0.001$ ) soil  $\times$  amendment  $\times$  cycle interaction (Table 3.3). In the Black Chernozem, SAN concentration, averaged across nitrapyrin levels, was significantly greater for FERT than ConT in Cycles 2 and 5 and RM, RM<sub>CDW</sub> and RM<sub>P</sub> in Cycle 2 (Fig. 3.5c). Soil available N was significantly lower in RM<sub>CDW</sub> than RM and RM<sub>P</sub> in Cycle 3. There was a significant increase in SAN for RM<sub>P</sub> from 36 mg kg<sup>-1</sup> in Cycle 2 to 91 mg kg<sup>-1</sup> in Cycle 3, after which there was no significant change. In the Brown Chernozem, SAN concentration (mean of the two nitrapyrin levels) was significantly lower for ConT than RM in Cycles 3 and 4, while it was also lower for ConT than RM<sub>CDW</sub> and RM<sub>P</sub> in Cycle 4 (Fig. 3.5d).

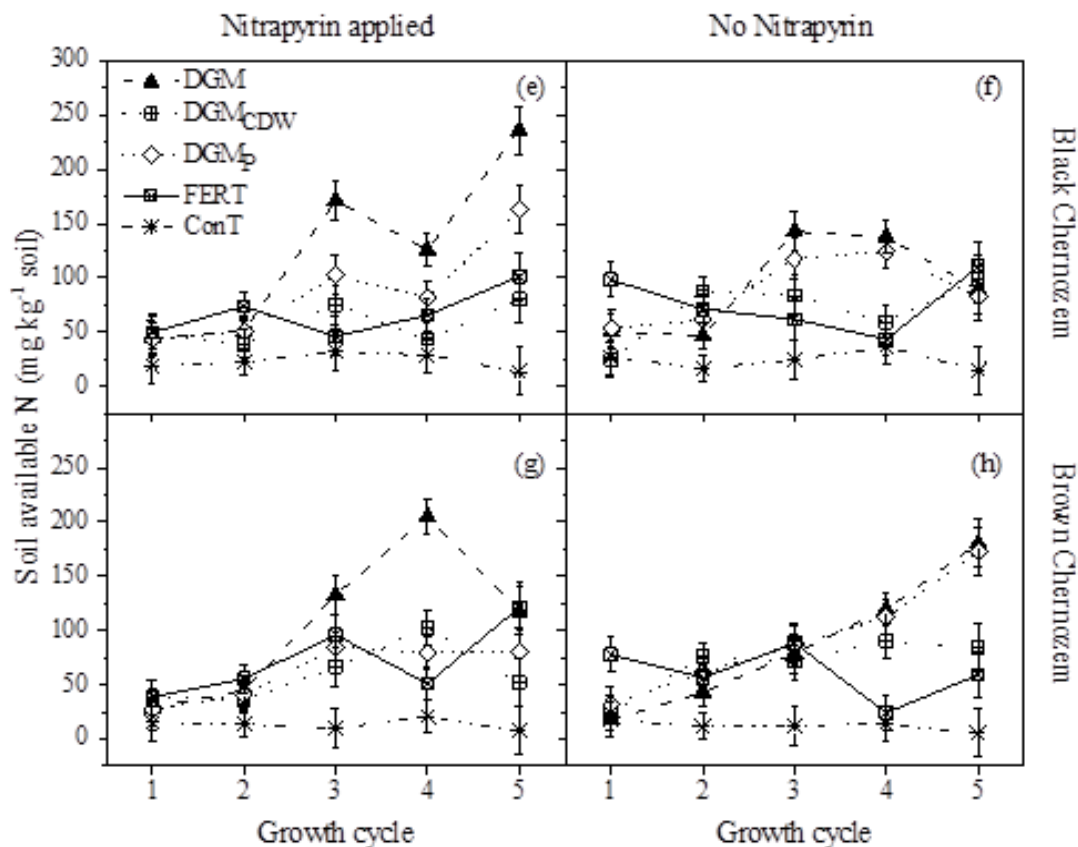


Figure 3.6 Soil available N in a Black Chernozem [(a) and (b)] and a Brown Chernozem [(c) and (d)] amended with DGM, DGM<sub>P</sub>, DGM<sub>CDW</sub>, and FERT, with [(a) and (c)] or without [(b) and (d)] nitrapyrin application, where DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively, and FERT is a mineral fertilizer. Vertical bars represent standard errors of the mean.

There was a significant increase in SAN for RM<sub>CDW</sub> from 33 mg kg<sup>-1</sup> in Cycle 3 to 88 mg kg<sup>-1</sup> in Cycle 4, followed by a significant decrease to 42 mg kg<sup>-1</sup> in Cycle 5.

For DDGS manure, there was a significant ( $P = 0.002$ ) cycle  $\times$  soil  $\times$  amendment  $\times$  nitrapyrin interaction for SAN concentration (Table 3.4). In the Black Chernozem treated with nitrapyrin, SAN was significantly greater for DGM than DGM<sub>CDW</sub> in Cycle 5, while it was greater for DGM than FERT in Cycles 3 and 5 (Fig. 3.6a). Soil available N concentration in the Black Chernozem amended

with DGM decreased from 51 mg kg<sup>-1</sup> in Cycle 2 to 17 mg kg<sup>-1</sup> in Cycle 3, with an increase from 125 mg kg<sup>-1</sup> in Cycle 4 to 236 mg kg<sup>-1</sup> in Cycle 5. With no nitrapyrin applied in the Black Chernozem, DGM produced significantly greater SAN than ConT in Cycles 3 and 4. Soil available N increased significantly from 46 mg kg<sup>-1</sup> in Cycle 2 to 143 mg kg<sup>-1</sup> in Cycle 3 (Fig. 3.6b).

In the Brown Chernozem treated with nitrapyrin, SAN was significantly greater for DGM than ConT in Cycles 3 and 4, while DGM produced greater SAN than all the other amendment treatments in Cycle 4 (Fig. 3.6c). Soil available N concentration increased from 45 mg kg<sup>-1</sup> in Cycle 2 to 133 mg kg<sup>-1</sup> in Cycle 3 in the DGM-amended Brown Chernozem treated with nitrapyrin. Similarly, with no nitrapyrin applied in the Brown Chernozem, SAN was significantly greater for DGM and DGMp than FERT (Fig. 3.6d).

#### **3.4.7. Apparent Organic Nitrogen Mineralization**

For regular manure, there was a significant ( $P = 0.02$ ) soil  $\times$  amendment  $\times$  cycle interaction for apparent organic N mineralization ( $N_{\min}$ ) (Table 3.3). In the Black Chernozem,  $N_{\min}$ , averaged across nitrapyrin levels, was significantly greater for RM and RM<sub>P</sub> than RM<sub>CDW</sub> in Cycles 3 and 4 (Fig. 3.7a). Similarly,  $N_{\min}$  was significantly greater for RM than RM<sub>CDW</sub> and RM<sub>P</sub> in Cycle 5. Net N immobilization was observed for RM<sub>P</sub> in Cycle 1, followed by a significant increase in  $N_{\min}$  from 6 mg kg<sup>-1</sup> in Cycle 2 to 147 mg kg<sup>-1</sup> in Cycle 3, after which there was no significant change. There was also a significant increase in  $N_{\min}$  for RM from 57 mg kg<sup>-1</sup> in Cycle 2 to 201 mg kg<sup>-1</sup> in Cycle 3, followed by a significant increase from 217 mg kg<sup>-1</sup> in Cycle 5 to 302 mg kg<sup>-1</sup> in Cycle 5.

In the Brown Chernozem, apparent organic N mineralization (mean of the two nitrapyrin levels) was significantly greater for RM than RM<sub>CDW</sub> in Cycles 2 and 3 (Fig. 3.7b). For RM,  $N_{\min}$  was greater in the Black Chernozem than in the Brown Chernozem in Cycles 3, 4 and 5. Similarly,  $N_{\min}$  for RM<sub>CDW</sub> and RM<sub>P</sub> were greater in the Black Chernozem than in the Brown Chernozem in Cycle 3.

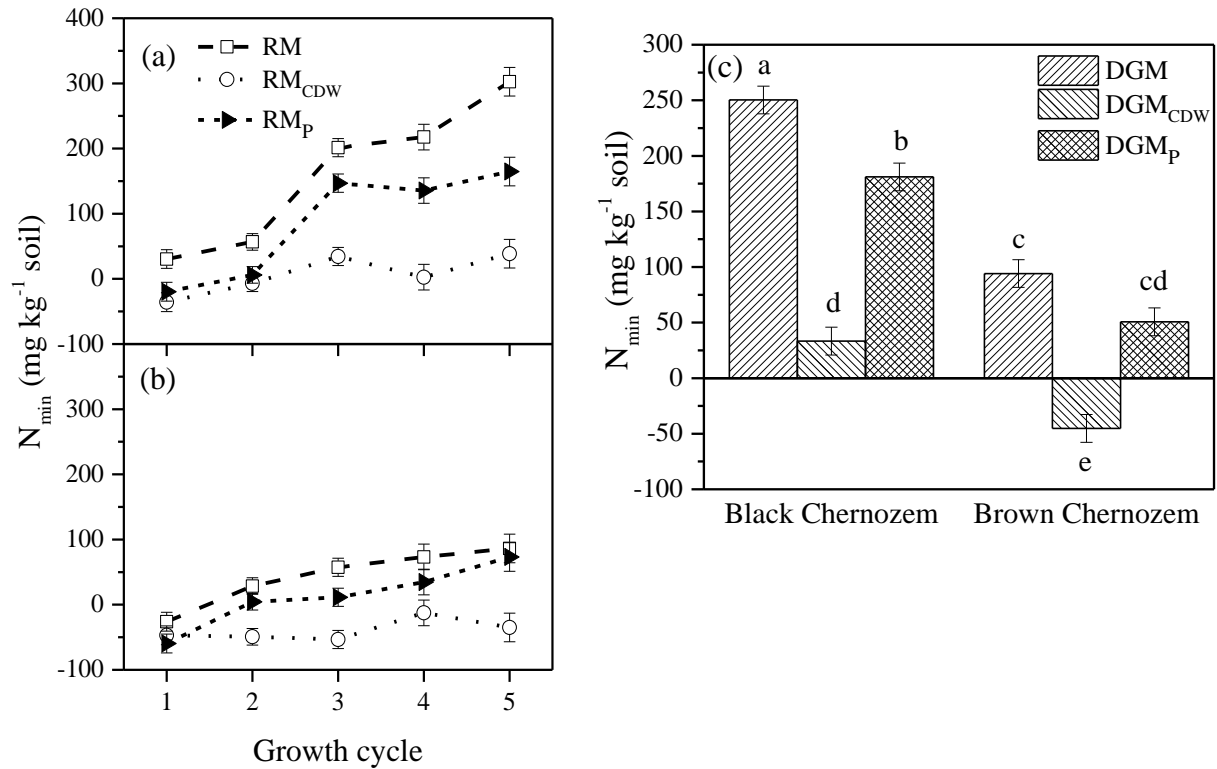


Figure 3.7 Cumulative organic N mineralization ( $N_{min}$ ) averaged across nitrapyrin in a Black Chernozem (a) and a Brown Chernozem (b) and averaged across nitrapyrin application and growth cycles for each amendment and soil (c) following amendment with RM,  $RM_p$ ,  $RM_{CDW}$ , DGM,  $DGM_p$ , and  $DGM_{CDW}$ , where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, and subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively. Error bars represent standard errors of the mean, and bars followed by different letters differ at  $P < 0.05$ .

For DDGS manure, amendment effects on  $N_{min}$  varied with soil type, as indicated by the significant ( $P = 0.001$ ) soil  $\times$  amendment treatment interaction (Table 3.4). In the Black Chernozem,  $N_{min}$  concentration, averaged across nitrapyrin levels, decreased in the order: DGM  $>$   $DGM_p$   $>$   $DGM_{CDW}$ , while it decreased in the order: DGM =  $DGM_p$   $>$   $DGM_{CDW}$  in the Brown Chernozem (Fig 3.7c). The  $DGM_{CDW}$  amended Brown Chernozem resulted in the net immobilization of inorganic N (i.e., negative mineralization). Net organic N mineralization for treatment  $DGM_{CDW}$  was lower in the

Brown Chernozem than the Black Chernozem. Generally, application of DDGS manure treatments resulted in greater mineralization in the Black Chernozem than the Brown Chernozem.

#### **3.4.8. Potentially Leachable Nitrate Nitrogen**

For regular manure, there was a significant soil  $\times$  amendment  $\times$  cycle interaction ( $P < 0.001$ ) for potentially-leachable  $\text{NO}_3\text{-N}$  (PLN) concentration (Table 3.3). In both soils, averaged across nitrapyrin levels, PLN was significantly greater for FERT than all amendment treatments including ConT in all the Cycles except in Cycle 3 (Figs. 3.8a and 3.8b). Potentially-leachable  $\text{NO}_3\text{-N}$  concentration for FERT in the Black Chernozem increased from  $97 \text{ mg kg}^{-1}$  in Cycle 3 to  $347 \text{ mg kg}^{-1}$  in Cycle 4 after which there was no significant change. Similarly, in the Brown Chernozem, PLN concentration for FERT increased from  $78 \text{ mg kg}^{-1}$  in Cycle 3 to  $516 \text{ mg kg}^{-1}$  in Cycle 4 after which there was no significant change.

For the DDGS manure, there was a significant ( $P = 0.004$ ) soil  $\times$  amendment  $\times$  cycle interaction for PLN concentration (Table 3.4). In the Black Chernozem, averaged across nitrapyrin levels, PLN concentration was greater for FERT than all amendment treatments including ConT in Cycle 1 (Fig 3.8c). Potentially leachable  $\text{NO}_3\text{-N}$  concentration was significantly greater for DGM, DGMp, and FERT than ConT in Cycles 3 and 5. In the Brown Chernozem, PLN concentration (mean of the two nitrapyrin levels) was significantly greater for FERT than all amendment treatments and ConT (Fig 3.8d). Potentially leachable  $\text{NO}_3\text{-N}$  concentration was greater for DGM, DGMp and FERT than ConT in Cycle 5. For DGMp, PLN was greater in the Black Chernozem ( $298 \text{ mg kg}^{-1}$ ) than in the Brown Chernozem ( $89 \text{ mg kg}^{-1}$ ) in Cycle 3.

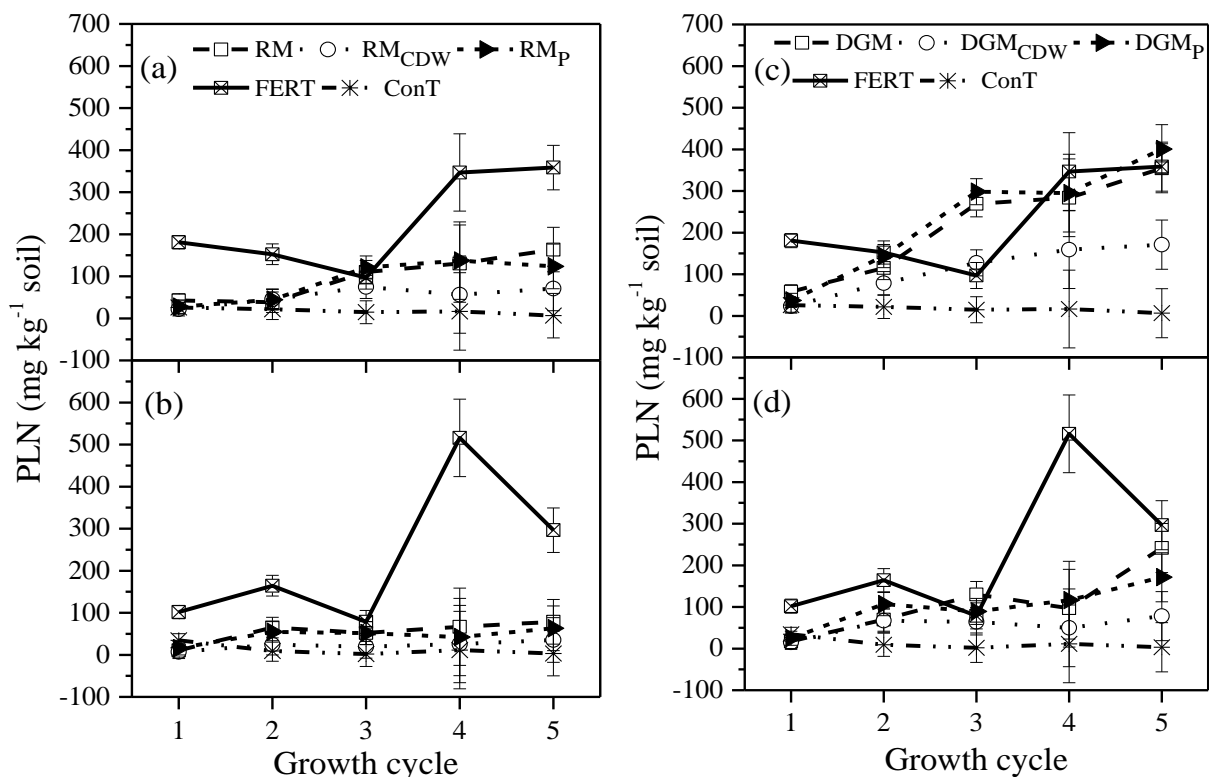


Figure 3.8 Potentially leachable NO<sub>3</sub>-N (PLN), averaged across nitrapyrin rate in a Black Chernozem [(a) and (c)] and a Brown Chernozem [(b) and (d)] amended with RM, RM<sub>p</sub>, RM<sub>CDW</sub>, DGM, DGM<sub>p</sub>, and DGM<sub>CDW</sub>, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet; and subscripts p and CDW represent the bedding materials peat and construction and demolition waste, respectively. Vertical bars represent standard errors of the mean.

Cumulative PLN (CPLN) differed significantly among amendments ( $P < 0.001$ ), but amendment differences varied with soil type as indicated by the significant ( $P = 0.01$  for regular manure and  $P = 0.004$  for DDGS manure) soil  $\times$  amendment interactions (Tables 3.3 and 3.4). For both manure types, all amendment treatments significantly increased CPLN relative to non-amended control (Figs. 3.9a and 3.9b). For the regular manure, CPLN was significantly greater for FERT than RM<sub>CDW</sub> in the Black Chernozem. In the Black Chernozem, CPLN was significantly greater for FERT than RM, RM<sub>CDW</sub> and RM<sub>p</sub>. Cumulative PLN was greater for RM than RM<sub>CDW</sub> but was not different



compared to RMp in the Black Chernozem. For RM<sub>CDW</sub>, CPLN was significantly greater in the Black Chernozem than the Brown Chernozem. Similarly for the DDGS manure, CPLN was significantly greater in the Black Chernozem than the Brown Chernozem for DGMp.

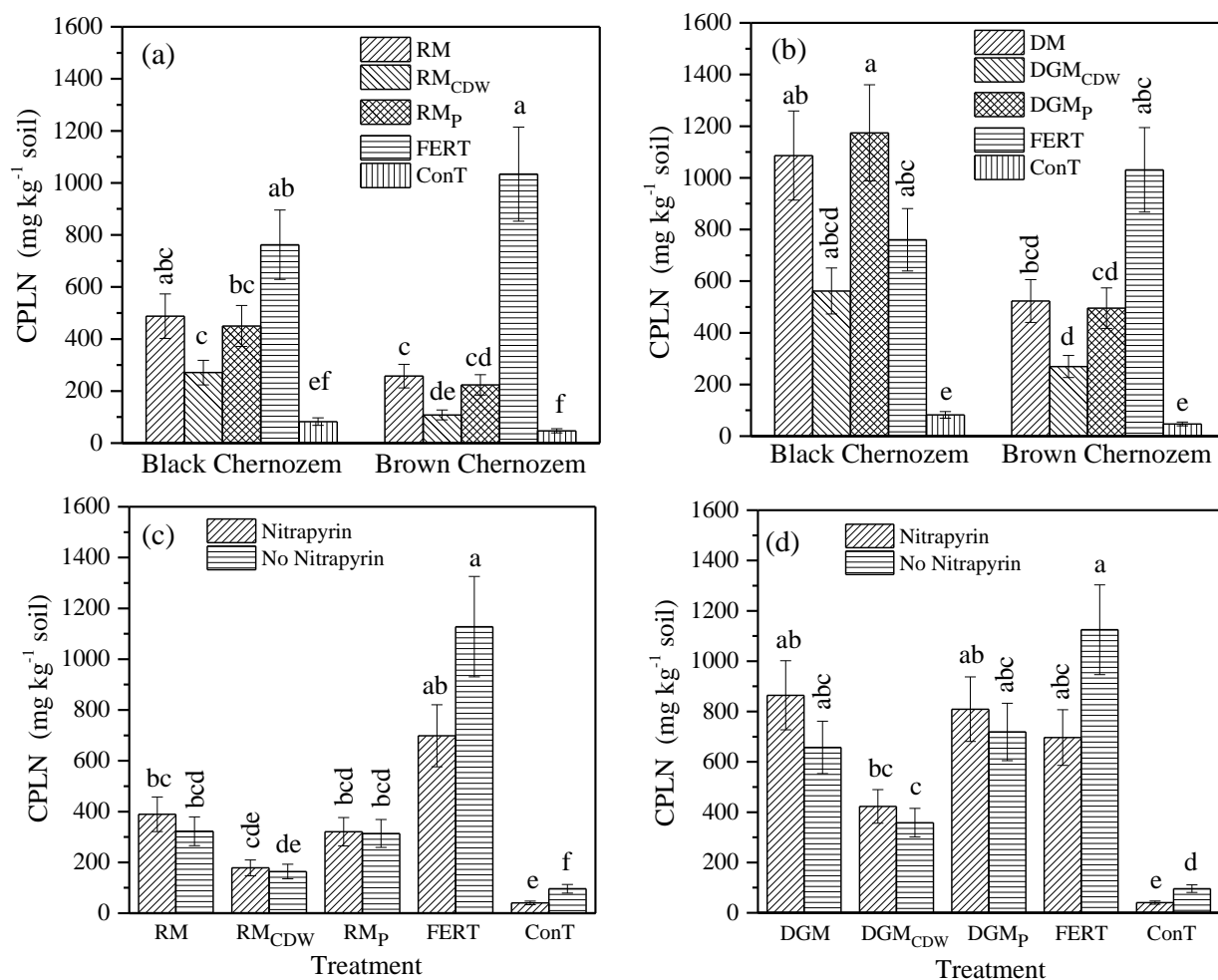


Figure 3.9 Cumulative PLN, averaged across nitrapyrin application for each amendment and soil [(a) and (b)] and averaged across soils for each amendment and nitrapyrin application [(c) and (d)], for five growth cycles of canola grown in a Black and a Brown Chernozem amended with RM, RM<sub>p</sub>, RM<sub>CDW</sub>, DGM, DGM<sub>p</sub>, DGM<sub>CDW</sub>, and FERT, where RM indicates regular manure from a typical beef cattle finishing diet, DGM is manure from cattle fed a DDGS diet, subscripts p and CDW represent the bedding materials peat and construction waste, and FERT is mineral fertilizer. Error bars represent standard errors of the mean, and bars followed by different letters differ at P < 0.05.

There was also a significant amendment  $\times$  nitrapyrin interaction ( $P = 0.02$  for regular manure and  $P = 0.003$  for DDGS manure) for CPLN. For both manure CPLN was significantly greater for all amendment treatments than the ConT in the absence of nitrapyrin and for all amendments except RM<sub>CDW</sub> in the presence of nitrapyrin (Figs 3.9c and 3.9d). For the regular manure, regardless of nitrapyrin treatment, FERT had significantly greater CPLN than RM<sub>CDW</sub> (Fig. 9c). For the regular manure, regardless of nitrapyrin treatment, FERT had significantly greater CPLN than RM<sub>CDW</sub> (Fig. 9c). Similarly, CPLN was significantly greater for FERT than RM and RM<sub>P</sub> in the absence of nitrapyrin, but the differences were not significant in the presence of nitrapyrin. Similarly for the DDGS manure, CPLN was significantly greater for FERT than DGM<sub>CDW</sub> in the absence of nitrapyrin, but the differences were not significant in the presence of nitrapyrin (Fig. 9d).

### 3.5. Discussion

In this study, manure N rates were approximately  $0.41 \text{ g N kg}^{-1}$  soil for regular manure and  $0.5 \text{ g N kg}^{-1}$  soil for DDGS (Table 3.1) in each growth cycle. Because of this difference in the N rates, data for the two manure types were therefore analyzed separately.

For both manure types, the addition of CDW (treatments DGM<sub>CDW</sub> and RM<sub>CDW</sub>) produced significantly lower N uptake and AmNU than all the other organic amendments in both soil types. When compared to peat moss, CDW had a higher C:N ratio (183:1 vs. 40:1), which likely explains the lower total N and NH<sub>4</sub>-N concentrations in soils amended with DGM<sub>CDW</sub> and RM<sub>CDW</sub> relative to DGM and DGM<sub>P</sub>, and RM and RM<sub>P</sub>, respectively. The high C:N ratio of CDW may also explain why the addition of CDW also resulted in significantly lower N uptake, AmNU, and ANR in both regular manure (RM<sub>CDW</sub> vs. RM and RM<sub>P</sub>) and DDGS manure treatments (DGM<sub>CDW</sub> vs. DGM and DGM<sub>P</sub>), whereas the addition of peat moss did not produce any negative effects on these N indices.

In fact, N uptake, AmNU, and ANR for DGMp and RMp were similar to those for DGM and RM, respectively. Gagnon et al. (1997) found that commercial composts with peat moss improved wheat yield and N uptake relative to farm compost. The authors attributed the improved N uptake to the greater concentration of stable C and the higher cation exchange capacity of the peat moss in the commercial compost, which increased N availability by retaining  $\text{NH}_4\text{-N}$  on its exchange sites.

The lower  $N_{\min}$  values in soils amended with  $\text{DGM}_{\text{CDW}}$  and  $\text{RM}_{\text{CDW}}$  relative to those amended with DGM and RM, respectively, were also likely due to the high C:N ratio of CDW added to the manure, which increased manure C/N ratios from 12:1 for DGM to 16:1 for  $\text{DGM}_{\text{CDW}}$  and from 14:1 for RM to 19:1 for  $\text{RM}_{\text{CDW}}$ . Lower N mineralization at C:N > 15:1 has also been reported in other studies (Qian and Schoenau, 2002; Helgason et al., 2007). Other studies have reported lower DMY and lower N uptake in woodchip-based cattle manure compared with straw-based manure (Miller et al., 2004, 2009).

Amendment-derived N uptake was consistently greater for DGMp than DGM in the Brown Chernozem but similar for the two amendments in the Black Chernozem. The greater percentage of TN in the inorganic form in DGMp (16 vs. 15%) may explain the higher AmNU observed for DGMp in the Brown Chernozem.

The greater N uptake observed for DDGS manure (DGM,  $\text{DGM}_{\text{CDW}}$ , DGMp) relative to regular manure (RM,  $\text{RM}_{\text{CDW}}$ , RMp) treatments in both soils was likely due to greater  $\text{NH}_4\text{-N}$  and total N concentration in DDGS manure. Nitrogen uptake results from the present study are consistent with those reported by Benke et al. (2010), which showed greater N uptake by barley for DDGS manure than for regular manure. In the present study, the greater N uptake with DDGS did not translate to greater dry matter yield relative to regular manure in either soil, except for  $\text{RM}_{\text{CDW}}$ , which produced lower CDMY than the other DDGS manure treatments. This can be explained by the lower initial  $\text{NH}_4\text{-N}$  concentration of  $\text{RM}_{\text{CDW}}$ . Generally, the  $\text{NH}_4\text{-N}$  concentration in regular manure was lower

than in DDGS manure. Mixing regular manure with CDW further reduced the  $\text{NH}_4\text{-N}$  concentration in  $\text{RM}_{\text{CDW}}$  relative to the other organic amendments likely due to immobilization.

The greater N uptake from fertilizer than from manure application observed in both soils has previously been reported (Gagnon et al., 1997; Helgason et al., 2007). However, others have generally found similar or greater N uptake from organic amendments compared with fertilizers (Miller et al., 2009). For all amendments, N uptake was consistently greater in the Black Chernozem than the Brown Chernozem. This was likely due to the greater  $\text{NH}_4\text{-N}$  ( $4 \text{ mg kg}^{-1}$ ) and total C ( $56 \text{ g kg}^{-1}$ ) concentrations in the Black Chernozem than in the Brown Chernozem ( $2.4 \text{ mg NH}_4\text{-N kg}^{-1}$  and  $23 \text{ g TOC kg}^{-1}$ ). The greater N uptake in the Black Chernozem reflects greater N mineralization associated with the higher organic matter content of this soil. The difference in N uptake could also be due to the lower net N mineralization observed for all amendments in the Brown Chernozem than in the Black Chernozem. Our results are consistent with those reported by Chiyoka et al. (2014b), which showed greater N uptake in a Black Chernozem than in a Dark brown Chernozem following cattle manure application. The authors attributed the difference in N uptake to differences in the soil organic matter and initial N status of the soils, with the Black Chernozem having a higher total N ( $71 \text{ vs. } 22 \text{ mg kg}^{-1}$ ) and inorganic N ( $42 \text{ vs. } 13 \text{ mg kg}^{-1}$ ) concentration than the Dark Brown Chernozem.

The greater AmNU with nitrapyrin application could be due to greater N concentration in the soil as a result of slower conversion of  $\text{NH}_4\text{-N}$  to  $\text{NO}_3\text{-N}$ . Although nitrapyrin had no influence on  $\text{NO}_3\text{-N}$  leaching, the amount of  $\text{NO}_3\text{-N}$  that was leached on day 15 in each growth cycle was generally lower when compared to the total amount of available N (residual soil N plus manure N applied) at the start of each cycle (data not presented). The slower conversion of  $\text{NH}_4\text{-N}$  to  $\text{NO}_3\text{-N}$  from the amendments with nitrapyrin application resulted in greater N availability, which may have enhanced root growth and, as a result, N uptake (Wolt, 2000; Havlin et al., 2014).

Greater residual soil inorganic N concentration for DGM compared with the other DDGS amendment treatments (DGMp and DGM<sub>CDW</sub>) likely reflects the greater total N and inorganic N concentrations in DDGS manure. In contrast to our results, Benke et al. (2010) reported no differences in soil inorganic N concentration among soils that received six repeated applications of DDGS and REG manure, regardless of rate, indicating that DDGS manure generally performed as well as regular manure in maintaining soil inorganic N concentration. They attributed the lack of difference to the greater N uptake of soil inorganic N by barley from DDGS manure than from regular manure and also to the possibility of greater ammonia volatilization since DDGS manure contained more NH<sub>4</sub>-N than REG manure. Nitrogen uptake by canola in the present study was not consistently greater for DGM and RM than the other amendment treatments in both soils.

Soil inorganic N concentration in both soils continued to increase throughout the study with DGM and RM relative to DGM<sub>CDW</sub> and RM<sub>CDW</sub> applications, respectively. This could reflect excessive nitrate build-up and its eventual loss to the environment through leaching, as seen with the greater PLN and CPLN with DGM and RM compared to DGM<sub>CDW</sub> and RM<sub>CDW</sub>, respectively.

The lack of a nitrapyrin effect on soil inorganic N concentration and PLN contradicts results from other studies, which showed greater soil inorganic N concentration and reduced NO<sub>3</sub>-N leaching with nitrapyrin application in soils amended with manure or fertilizer (Ronaghi et al., 1993; Calderón et al., 2005; Peng et al., 2015). Other studies have reported that the effects of nitrapyrin on soil mineral N concentration in manured soils differs with soil type because of the interaction between nitrification, denitrification, and N immobilization (Wolt, 2004; Calderón et al., 2005). The effectiveness of nitrapyrin has also been reported to decrease with increasing soil organic matter, volatilization, soil sorption, and soil pH (Hendrickson and Keeney, 1978, 1979; Wolt, 2000). Other studies (Bailey, 1990; Randall and Vetsch, 2005) have reported no effect of nitrapyrin on crop yield and N accumulation when applied with fertilizer. Randall and Vetsch (2005) found nitrapyrin to be

effective in only two of six years of application under extremely wet conditions. In the present study, the effectiveness of nitrapyrin might have been reduced due to its volatilization during mixing with the organic amendment, the high soil pH and organic matter content of the soils used, and the high  $\text{NH}_4\text{-N}$  concentration of the organic amendments (Bailey, 1990; Wolt, 2000).

### 3.6. Conclusions

Manure application improved dry matter yield and nutrient uptake relative to the non-amended control. There was a continual increase in the soil residual N concentration with repeated manure application, which was greater with DDGS than REG manure. Results from this study demonstrate that at the rates of manure N tested, CDW in manure reduced available N concentrations compared with manure containing no CDW. Further investigation is required to establish the mixing ratio of CDW and manure that will not negatively impact N uptake while minimizing the environmental risk associated with repeated manure application under field conditions. Nitrapyrin was not effective in reducing  $\text{NO}_3\text{-N}$  leaching but increased mineralized N concentration and the uptake of N derived from amendments.

### 3.7. References

- Amundson, R. G., Trask, J. and Pendall, E. 1988.** A rapid method of soil carbonate analysis using gas chromatography. *Soil Sci. Soc. Am. J.* **52**: 880-883.
- Bailey, L. D. 1990.** The effects of 2-chloro-6 (trichloromethyl)-pyridine ('N-Serve') and N fertilizers on productivity and quality of Canadian oilseed rape. *Can. J. Plant Sci.* **70**: 979-986.
- Beauchamp, E. G. and Paul, J. W. 1989.** A simple model to predict manure N availability to crops in the field. P. 140-149. In J.A. Hansen and K. Henriksen (ed.) *Nitrogen in organic wastes applied to soils*. Harcourt Brace Jovanovich Publ., Boston.

- Benke, M. B., Hao, X., Caffyn, P., Schoenau, J. J. and McAllister, T. A. 2010.** Using manure from cattle fed dried distillers' grains with solubles (DDGS) as fertilizer: Effects on nutrient accumulation in soil and uptake by barley. *Agri. Ecosyst. Environ.* **139**: 720-727.
- Calderón, F. J., McCarty, G. W. and Reeves III, J. B. 2005.** Nitrapyrin delays denitrification on manured soils. *Soil Sci.* **170**: 350-359.
- Chiyoka, W., Zvomuya, F. and Hao, X. 2014b.** A bioassay of nitrogen availability in soils amended with solid digestate from anaerobically digested beef cattle feedlot manure. *Soil Sci. Soc. Am. J.* **78**: 1291-1300.
- Choudhary, M., Bailey, L. D. and Grant, C. A. 1996.** Review of the use of swine manure in crop production: effect on yield and composition and on soil and water quality. *Waste Manage. Res.* **14**: 581-595.
- Gagnon, B., Simard, R. R., Robitaille, R., Goulet, M. and Rioux, R. 1997.** Effect of composts and inorganic fertilizers on spring wheat growth and N uptake. *Can. J. Soil Sci.* **77**: 487-495.
- Hao, X., Hill, B., Caffyn, P., Travis, G., Olson, A. F., Larney, F. J., McAllister, T. A. and Alexander, T. 2014.** Co-composting of beef cattle feedlot manure with construction and demolition waste. *J. Environ. Qual.* **43**: 1799-1808.
- Hao, X., Benke, M. B., Gibb, D. J., Stronks, A., Travis, G. and McAllister, T. A. 2009.** Effects of dried distillers' grains with solubles (wheat-based) in feedlot cattle diets on feces and manure composition. *J. Environ. Qual.* **38**: 1709-1718.
- Havlin, J. L., Tisdale, S. L., Nelson, W. L. and Beaton, J. D. 2014.** Soil fertility and fertilizers: An introduction to nutrient management. 8th ed. Pearson, Inc, Upper Saddle River, NJ.
- Helgason, B. L., Larney, F. J., Janzen, H. H. and Olson, B. M. 2007.** Nitrogen dynamics in soil amended with composted cattle manure. *Can. J. Soil Sci.* **87**: 43-50.
- Hendrickson, L. L. and Keeney, D. R. 1979.** Effect of some physical and chemical factors on the rate of hydrolysis of nitrapyrin (N-Serve). *Soil Biol. Biochem.* **11**: 47-50.
- Hendrickson, L. L. and Keeney, D. R. 1978.** A bioassay to determine the effect of organic matter and pH on the effectiveness of nitrapyrin (N-Serve) as a nitrification inhibitor. *Soil Biol. Biochem.* **11**: 51-55.
- Hünerberg, M., McGinn, S. M., Beauchemin, K. A., Okine, E. K., Harstad, O. M. and McAllister, T. A. 2013.** Effect of dried distillers' grains with solubles on enteric methane emissions and nitrogen excretion from finishing beef cattle. *Can. J. Anim. Sci.* **93**: 373-385.

- Jayasinghe, G. Y., Liyana Arachchi, I. D. and Tokashiki, Y. 2010.** Evaluation of containerized substrates developed from cattle manure compost and synthetic aggregates for ornamental plant production as a peat alternative. *Resour. Conserv. Recy.* **54**: 1412-1418.
- Kroetsch, D. and Wang, C. 2006.** Particle size distribution. In: M.R. Carter and E.G. Gregorich editors, *Soil sampling and methods of analysis*. Chapter 55. Canadian Society of Soil Science. Taylor & Francis Group.
- Larney, F., Olson, A. F., Miller, J. J., DeMaere, P. R., Zvomuya, F. and McAllister, T. A. 2008.** Physical and chemical changes during composting of wood-chip-bedded and straw-bedded beef cattle feedlot manure. *J. Environ. Qual.* **37**: 725-735.
- Littell, R. C., Milliken, G. A., Stroup, W. W., Wolfinger, R. D. and Schabenberger, O. 2006.** SAS system for mixed models. SAS Institute. Inc., Cary, NC.
- Liu, K. 2011.** Chemical composition of distillers grains, a review. *J. Agric. Food Chem.* **59**: 1508-1526.
- Malhi, S. S., Johnston, A. M., Schoenau, J. J., Wang, Z. H. and Vera, C. L. 2007.** Seasonal biomass accumulation and nutrient uptake of canola, mustard, and flax on a Black Chernozem soil in Saskatchewan. *J. Plant Nutr.* **30**: 641-658.
- Meyer, U., Schwabe, A., Danicke, S. and Flachowsky, G. 2010.** Effects of by-products from biofuel production on the performance of growing fattening bulls. *Anim. Sci. Technol.* **161**: 132-139.
- Miller, J. J., Beasley, B. W., Drury, C. F., and Zebarth, B. J. 2009.** Barley yield and nutrient uptake for soil amended with fresh and composted cattle manure. *Agron. J.* **101**: 1047-1059.
- Miller, J. J., Olson, Edith C. S., Chanasyk, D. S., Beasley, B. W., Yanke, L. J., Larney, F. J., McAllister, T. A., Olson, B. M. and Selinger, L. B. 2006.** Bedding and within-pen location effects on feedlot pen runoff quality using a rainfall simulator. *J. Environ. Qual.* **35**: 505-515.
- Miller, J. J., Beasley, B. W., Larney, F. J. and Olson, B. M. 2004.** Barley dry matter yield, crop uptake, and soil nutrients under fresh and composted manure containing straw or wood-chip bedding. *Can. J. Plant Sci.* **84**: 987-999.
- Miller, J. J., Beasley, B. W., Yanke, L. J., Larney, F. J., McAllister, T. A., Olson, B. M., Selinger, L. B., Chanasyk, D. S. and Hasselback, P. 2003.** Bedding and seasonal effects on chemical and bacterial properties of feedlot cattle manure. *J. Environ. Qual.* **32**: 1887-1894.
- Naeth, M. A. and Wilkinson, S. R. 2013.** Can we build better compost? Use of waste drywall to enhance plant growth on reclamation sites. *J. Environ. Manage.* **129**: 503-509.



**N'Dayegamiye, A. and Isfan, D. 1991.** Chemical and biological changes in compost of wood shavings, sawdust and peat moss. *Can. J. Soil Sci.* **71**: 475-484.

**Peng, X., Maharjan, B., Yu, C., Su, A., Jin, V. and Ferguson, R. B. 2015.** A laboratory evaluation of ammonia volatilization and nitrate leaching following nitrogen fertilizer application on a coarse-textured soil. *Agron. J.* **107**: 871-879.

**Qian, P. and Schoenau, J. J. 2002.** Availability of nitrogen in solid manure amendments with different C:N ratios. *Can. J. Soil Sci.* **82**: 219-225.

**Randall, G. W. and Vetsch, J. A. 2005.** Corn production on a subsurface-drained Mollisol as affected by fall versus spring application of nitrogen and nitrapyrin. *Agron. J.* **97**: 472-478.

**Ronaghi, A., Soltanpour, P. N. and Mosier, A. R. 1993.** Nitrapyrin effect on corn N-15 uptake efficiency, denitrification and nitrate leaching. *Commun. Soil Sci. Plant Anal.* **24**: 2629-2639.

**Salim, H. M., Kruk, Z. A. and Lee, B. D. 2010.** Nutritive value of corn distillers dried grains with solubles as an ingredient of poultry diets: A review. *World's Poult. Sci. J.* **66**: 411-432.

**Saludes, R. B., Iwabuchi, K., Miyatake, F., Abe, Y. and Honda, Y. 2008.** Characterization of dairy cattle manure/wallboard paper compost mixture. *Bioresour. Technol.* **99**: 7285-7290.

**SAS Institute. 2014.** SAS/STAT 13.2 User's guide. Version 9.4. Cary (NC): SAS Institute, Inc.

**Schoenau, J. J. and O'Halloran, I. P. 2006.** Sodium bicarbonate-extractable phosphorus. In: M.R. Carter and E.G. Gregorich editors, *Soil sampling and methods of analysis*. Chapter 8. Taylor & Francis Group., Boca Raton, FL.

**Wolt, J. D. 2004.** A meta-evaluation of nitrapyrin agronomic and environmental effectiveness with emphasis on corn production in the Midwestern USA. *Nutr. Cycling Agroecosyst.* **69**: 23-41.

**Wolt, J. D. 2000.** Nitrapyrin behavior in soils and environmental considerations. *J. Environ. Qual.* **29**: 367-379.

## **4. PHYTOEXTRACTION OF NITROGEN AND PHOSPHORUS BY CROPS GROWN IN A HEAVILY MANURED DARK BROWN CHERNOZEM UNDER CONTRASTING SOIL MOISTURE CONDITIONS**

### **4.1. Abstract**

Repeated uptake of excess nutrients by crops in soils with a long history of manure application may be a viable option for reducing the nutrient levels. In this greenhouse study, we examined the effectiveness of six 40-day cycles of barley, canola, corn, oat, pea, soybean, and triticale, all grown under two moisture regimes (100% and 50% soil field capacity, SFC), at extracting nitrogen (N) and phosphorus (P) from a Dark Brown Chernozem that had received 180 Mg ha<sup>-1</sup> (wet wt.) of beef cattle feedlot manure annually for 38 years. Repeated cropping resulted in an overall decrease in dry matter yield (DMY). The decrease in N and P uptake relative to Cycle 1 was fastest for the cereal grains and less pronounced for the two legumes. However, cumulative N and P uptake values were significantly greater for corn than the other crops under both moisture regimes. The reduction in soil N and P was greater under the 100% than the 50% SFC. These results indicate that repeated cropping can be a useful management practice for reducing N and P in a heavily manured soil. The extent of reduction will be greater for crops with high biomass production under adequate moisture supply.

### **4.2. Introduction**

Continuous application of livestock manure on agricultural land can result in the excessive build-up of N and P, which may lead to nutrient loss to the environment (Ferguson et al., 2005; Schröder et al., 2005). Among other problems, eutrophication of lakes and streams due to P transport by surface runoff and nitrate contamination of groundwater via leaching are the two major environmental challenges associated with livestock manure application on agricultural land (Sharpley, 1995; Pote et al., 2003).

Extraction of nutrients from soils with high levels of nutrients through repeated biomass harvesting could be an effective means of reducing nutrient concentrations to more acceptable levels, hence minimizing nutrient loss to the environment (Brink et al., 2001; Pederson et al., 2002; Benke et al., 2013). This has been demonstrated for forages in soils with a history of repeated livestock manure application (Rowe and Fairbrother, 2003; Read et al., 2007). The amounts of nutrients extracted from soil depend not only on the nutritional requirement of the crop grown, but also on soil properties, including available soil moisture. Soil moisture influences nutrient dynamics and invariably the availability of nutrients for plant uptake. Studies have shown that soil organic matter decomposition and N mineralization increase as soil moisture content and temperature increase, thereby enhancing soil microbial activity (Sierra, 1997; Leirós et al., 1999; Agehara and Warncke, 2005). Sierra (1997) reported that N mineralization rate during a 35-d incubation study increased from 0.52 mg N kg<sup>-1</sup> soil d<sup>-1</sup> at 35% water-filled pore space (WFPS) to 0.72 mg N kg<sup>-1</sup> soil d<sup>-1</sup> at 60% WFPS. Nitrogen uptake by plants therefore increases as soil moisture content increases. Nutrient uptake by plants is also influenced by management practices such as manure application rates and timing, and crop maturity at harvest (Pederson et al., 2002).

Although the ability of various forages to remove excess nutrients from soils receiving livestock manure application has been extensively studied (Brink et al., 2001; Rowe and Fairbrother, 2003; Read et al., 2007), to our knowledge, there is a dearth of published research on the effectiveness of various agronomic crops in extracting excess soil nutrients from soils with a long history of manure application under contrasting moisture conditions. While the N and P utilization of most arable crops is known (Sieling et al., 2006; Barbazán et al., 2009; Slaton et al., 2013), the extent of N and P removal from previously heavily manured soils via multiple harvesting in mono-cropping systems is poorly understood. The overall objective of this study was, therefore, to investigate the ability of

various agronomic crops to effectively extract excess N and P from a Dark Brown Chernozemic soil that had received annual manure applications for 38 years.

### **4.3. Materials and Methods**

#### **4.3.1. Soil Collection and Analysis**

Surface soil (0-15 cm) was collected from long-term manured experimental plots at the Agriculture and Agri-Food Canada Research and Development Centre in Lethbridge, Alberta, Canada (Lat. 49°42' N; Long. 112°48' W). The soil was a calcareous, Dark Brown Chernozem (Typic Haplustoll) with a sandy clay loam texture. The soil had received annual solid feedlot cattle manure applications ( $180 \text{ Mg ha}^{-1} \text{ yr}^{-1}$  wet wt.) under irrigation since 1973 (38 yr), with the manure applied each fall following harvest. The soil was collected immediately after barley harvest in 2012 and before manure was applied that year. The soil was air-dried, passed through a 7-mm mesh sieve, and stored at room temperature until the start of the experiment.

#### **4.3.2. Experimental Setup**

The experimental design was a strip plot with an  $8 \times 2$  factorial treatment layout and three replications. The factors were crop [barley (*Hordeum vulgare* L. cv. Spring Kasota), triticale (*Triticosecale wittmack* X. cv. Luoma Winter), oat (*Avena sativa* L. cv. A.C. Morgan), soybean (*Glycine max* L. cv. Heather), canola (*Brassica napus* L. cv. InVigor L150), corn (*Zea mays* L. cv. Pioneer 39H84), pea (*Pisum sativum* L. cv. CDC Golden), and a control (uncropped)] and moisture regime [100% and 50% soil field capacity (SFC)]. These crops were chosen because they are representative of the annual crops grown in the Canadian prairies (Zentner et al., 2002; Blackshaw et al., 2011; Nadler and Bullock, 2011). Approximately 1.5 kg of air-dry soil was placed in 2-L plastic pots (17 cm wide at the top  $\times$  13 cm wide at the bottom  $\times$  13 cm deep) and reverse osmosis (RO)

water was added to maintain soil moisture at 100% or 50% SFC, which roughly correspond to moisture conditions under irrigated and dryland conditions in semi-arid southern Alberta (Alberta Agriculture and Forestry, 2016). To ensure uniform seedling establishment for all crops, seeds were pre-germinated by placing them on wet paper towels for four days. Seven seedlings of each crop were transplanted into each pot and thinned to three per pot after one week. Pea was seeded after a nitrogen-fixing inoculant (Cell-Tech<sup>®</sup> Granular Pea and Lentil, supplied by Novozymes Bio Ag) was applied to the soil. The pots were placed in a greenhouse maintained at a day/night temperature regime of 23/17 °C and a corresponding photoperiod of 16 h. The pots were watered regularly to maintain soil moisture at 100% or 50% SFC. Plants were harvested 40 d after planting by cutting aboveground biomass at the soil surface. Research has shown that maximum N and P uptake rates occur at 22 to 36 d after emergence for barley and oat, and at 21 to 42 d after emergence for canola (Malhi et al., 2006, 2007).

Dry biomass yields were determined after each growth cycle following oven-drying at 60°C for 48 h. The biomass was fine-ground (< 0.15 mm) in a plant tissue grinder (Cyclone, Udy Corp., Fort Collins, CO) for determination of total N and P concentrations as described below. The soil in each pot was emptied into an aluminum pan and the roots chopped and mixed properly with the soil after sub-soil samples were taken for inorganic N (NO<sub>3</sub>-N and NH<sub>4</sub>-N) and Olsen P determination. After each growth cycle, the same soil in each pot was reseeded to the same crop. This was repeated five times, resulting in six crop cycles over a 240-d period.

#### **4.3.3. Laboratory Analysis**

Soils (< 2 mm) were analyzed for particle size distribution by the hydrometer method (Kroetsch and Wang, 2006). Soil pH was measured in a 1:2 soil/water (mass/volume) suspension using a pH meter (Model 290A, Orion, Boston, MA), after which suspensions were filtered for measurement of

electrical conductivity (EC) using an EC meter (Model 125A, Orion, Boston, MA). Soil total carbon (TC) and total nitrogen (TN) were determined using an automated CN analyzer (Carlo Erba NA 1500, Carlo Erba, Milan, Italy). Total P concentration was determined using an EasyChem Pro discrete analyzer (Systea Analytical Technologies, Anagni, Italy) following acid digestion with  $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$  (Parkinson and Allen, 1975). Total K, Ca, and Mg concentrations were determined using an atomic absorption spectrometer (Varian Model AA240, Palo Alto, CA) following digestion with  $\text{HNO}_3 + 30\% \text{H}_2\text{O}_2$ . For soil inorganic N determination, the soil was extracted with 2 M KCl (1:5 soil to solution ratio) (Maynard et al., 2006) and the concentrations of  $\text{NO}_3\text{-N}$  and  $\text{NH}_4\text{-N}$  were measured using an Auto-analyzer III (Bran + Luebbe, Germany). For soil test (Olsen) P determination, the soil was extracted (1:10 soil to solution ratio) with 0.5 M  $\text{NaHCO}_3$  at a pH of 8.5, followed by measurement of orthophosphate P concentration using an EasyChem Pro discrete analyzer (Systea Analytical Technologies, Anagni, Italy). Total N and P concentrations in the finely ground plant tissue samples were determined as described above for soil.

#### 4.3.4. Calculations

Nitrogen and P uptake by the various crops is a function of the dry matter yield, which inherently varied among the crops. Therefore, the data for DMY and nutrient uptake were normalized relative to DMY and N and P uptake in growth cycle 1.

The percent change in dry matter yield relative to growth cycle 1  $[(\Delta\text{DMY})_1, \%]$  for each crop was estimated as:

$$(\Delta\text{DMY})_1 = [(\text{DMY}_t - \text{DMY}_1) / \text{DMY}_1] \times 100 \quad [1]$$

where  $\text{DMY}_t - \text{DMY}_1$  is the relative DMY ( $\text{DMY}_{\text{rel}}$ ),  $\text{DMY}_t$  is the dry matter yield ( $\text{g kg}^{-1}$  soil) from Cycle t and  $\text{DMY}_1$  is the dry matter yield ( $\text{g kg}^{-1}$  soil) in Cycle 1.

Changes in N uptake  $[(\Delta NU)_1]$  and P uptake  $[(\Delta PU)_1]$  relative to Cycle 1 were calculated by replacing DMY with NU or PU in Eq. [1]. For each crop, total N and P uptake values from all six growth cycles were summed to give cumulative N uptake (CNU,  $\text{mg kg}^{-1}$  soil) and cumulative P uptake (CPU,  $\text{mg kg}^{-1}$  soil) over the 240-d period, as illustrated for CNU:

$$\text{CNU} = \sum_{x=1}^n (\text{DMY} \times \text{N}) \quad [2]$$

where DMY is plant dry matter yield ( $\text{g kg}^{-1}$  soil), N is the biomass N concentration, x is the crop cycle number, and n (= 6) is the total number of crop cycles.

#### 4.3.5. Statistical Analysis

The GLIMMIX procedure in SAS version 9.4 (SAS Institute, 2014) was used for the analysis of variance for all dependent variables, with crop and moisture regime as fixed effects and growth cycle as the repeated measure. Various covariance structures were compared and the one with the lowest corrected Akaike Information Criterion was selected as the best fit for the repeated measures analysis. When treatment effects were significant ( $\alpha = 0.05$ ), means were compared using the Tukey adjustment for multiple comparisons.

### 4.4. Results

#### 4.4.1. Initial Soil Properties

The soil had a long history of beef cattle manure application, which is reflected in the high EC ( $5.17 \text{ dS m}^{-1}$ ), high soil available N (SAN) ( $575 \text{ mg kg}^{-1}$ ), high Olsen P concentration ( $555 \text{ mg kg}^{-1}$ ), and high organic C concentration ( $130 \text{ g kg}^{-1}$ ) (Table 4.1).

**Table 4.1 Selected initial physicochemical properties of the soil used in the greenhouse study.**

Property†	Dark Brown Chernozem
pH	7.23
EC, dS m <sup>-1</sup>	5.17
Available N, mg kg <sup>-1</sup>	575
Olsen P, mg kg <sup>-1</sup>	555
Total C, g kg <sup>-1</sup>	137
Organic C, g kg <sup>-1</sup>	130
Total N, g kg <sup>-1</sup>	13
Total P, g kg <sup>-1</sup>	1.24
Total K, g kg <sup>-1</sup>	7.80
Total Ca, g kg <sup>-1</sup>	34
Total Mg, g kg <sup>-1</sup>	7.92
Clay, g kg <sup>-1</sup>	21
Silt, g kg <sup>-1</sup>	23
Sand, g kg <sup>-1</sup>	56

† Concentrations are on a dry weight basis.

#### 4.4.2. Change in Dry Matter Yield

The change in DMY relative to Cycle 1 was not significantly affected by moisture regime ( $P = 0.26$ ) for all crops. However, there was a significant temporal trend in the  $\Delta\text{DMY}_1$ , which varied with crop, as indicated by the significant ( $P < 0.0001$ ) growth cycle  $\times$  crop interaction (Table 4.2). Averaged across moisture regimes,  $\Delta\text{DMY}_1$  was negative (i.e., DMY decreased relative to Cycle 1) for all crops and followed the order: soybean (21%) < pea (31%) < canola (38%) < corn (55%) < oat (66%) < triticale (67%) < barley (80%). However, crop differences in  $\Delta\text{DMY}_1$  varied with cycle. The decrease in DMY was significantly greater for barley (-63%) and triticale (-56%) than for corn (-23%) in growth Cycle 2 (Fig. 4.1). Soybean showed a lower decrease in  $\Delta\text{DMY}_1$  (-10%) than barley (-79%), corn (-69%), pea (-43%), oat (-67%), and triticale (-62%) in Cycle 3. Similarly, in Cycle 3, the decrease in  $\Delta\text{DMY}_1$  was greater for barley, corn, and oat than canola.



**Table 4.2 Effects of moisture on soil available nitrogen (SAN), soil available phosphorus (P), percent change in dry matter yield ( $\Delta\text{DMY}_1$ ), nitrogen ( $\Delta\text{NU}_1$ ), and phosphorus ( $\Delta\text{PU}_1$ ), and cumulative dry matter yield (CDMY), cumulative nitrogen (CNU) and phosphorus uptake (CPU) of crops grown in a previously heavily manured soil.**

Effect	$\Delta\text{DMY}_1^\dagger$	$\Delta\text{NU}_1$	$\Delta\text{PU}_1$	CDMY	CNU	CPU	SAN	Available P
	———— % ————	———— % ————		g kg <sup>-1</sup> soil		———— mg kg <sup>-1</sup> soil ————		
Moisture Regime (M) <sup>‡</sup>								
100% SFC	-54	-21	-36	36	1024	146	350	424
50% SFC	-48	-26	-40	22	831	95	806	424
Crop (C)								
Control	-	-	-	-	-	-	962	426
Barley	-80	-65	-66	18	651	90	856	446
Canola	-38	-9.14	-29	37	1326	168	159	396
Corn	-55	-53	-54	81	1785	238	105	406
Oat	-66	-44	-61	25	917	140	477	409
Pea	-31	3.47	16	18	612	58	794	454
Soybean	-21	54	-19	6.99	335	39	789	444
Triticale	-67	-50	-52	19	864	111	484	410
Growth Cycles (GC)								
1	-	-	-	-	-	-	468	536
2	-44	-16	-24	-	-	-	520	522
3	-52	-19	-32	-	-	-	524	424
4	-62	-37	-30	-	-	-	669	369
5	-46	-15	-19	-	-	-	674	372
6	-52	-29	-84	-	-	-	615	321
	<i>P</i> value							
Moisture Regime	0.26	0.55	0.51	<0.001	0.01	0.01	<0.0001	0.92

Crop	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.0001	0.001
Growth Cycle	0.001	<0.001	<0.001	-	-	-	<0.0001	<0.0001
GC $\times$ C	<0.001	<0.001	<0.001	-	-	-	<0.0001	0.04
GC $\times$ M	0.98	0.67	0.86	-	-	-	0.22	0.14
M $\times$ C	0.91	0.21	0.55	<0.001	<0.001	<0.001	<0.0001	0.30
GC $\times$ M $\times$ C	0.41	0.52	0.16	-	-	-	0.0002	0.62

†  $\Delta\text{DMY}_1$ , percent change in dry matter yield relative to Cycle 1;  $\Delta\text{NU}_1$ , percent change in N uptake relative to Cycle 1;  $\Delta\text{PU}_1$ , percent change in P uptake relative to Cycle 1;  $\text{CDMY}$ , cumulative dry matter yield;  $\text{CNU}$ , cumulative N uptake;  $\text{CPU}$ , cumulative P uptake;  $\text{SAN}$ , soil available nitrogen.

‡ 100% and 50% soil field capacity.

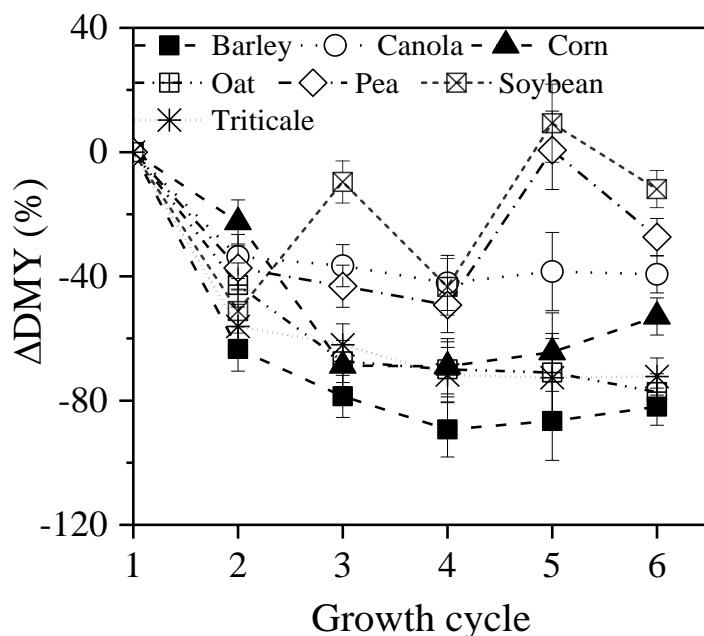


Figure 4.1 Changes in dry matter yield relative to Cycle 1 ( $\Delta\text{DMY}_1$ ) for crops grown in a previously heavily manured soil. Averaged across moisture regimes, the mean dry matter yields in Cycle 1 were 9.32, 9.21, 26.2, 9.13, 4.32, 1.49, and 7.15 g kg<sup>-1</sup> soil for barley, canola, corn, oat, pea, soybean, and triticale, respectively. Vertical bars represent standard errors of the mean.

Dry matter yield reductions increased for corn and oat but decreased for soybean from Cycle 2 to 3. Soybean showed a significant decrease in  $\Delta\text{DMY}_1$  from Cycle 3 to 4 and an increase from Cycle 4 to 5. The decrease in DMY relative to Cycle 1 was greater for barley (-89%) than canola (-42%), pea (-49%), and soybean (-43%) in Cycle 4 and for barley and triticale than soybean and pea in Cycles 5 and 6. Similarly, soybean showed a significantly lower decrease in DMY, relative to Cycle 1, than corn and oat in Cycles 5 and 6. The decrease was significantly greater for corn than pea in Cycle 5 and greater for triticale than pea in Cycles 5 and 6. In Cycle 6, the decrease in DMY relative to Cycle 1 was significantly greater for barley, oat, and triticale than canola, for barley than corn, and for oat than pea.

#### 4.4.3. Change in Nitrogen Uptake

Averaged across growth cycles and moisture regimes,  $\Delta NU_1$  decreased in the order: soybean (54%) > pea (3%) > canola (-9%) > oat (-44%) > triticale (-50%) > corn (-53%) > barley (-65%). However, crop differences varied with growth cycle, as indicated by the significant ( $P < 0.0001$ ) growth cycle  $\times$  crop interaction (Table 4.2). While there were no significant differences in  $\Delta NU_1$  among crops in Cycle 2, relative N uptake ( $NU_{rel} = N$  uptake in Cycle  $t - N$  uptake in Cycle 1) was positive and significantly greater for soybean than all the other crops in Cycles 3, 5, and 6 (Fig. 4.2).

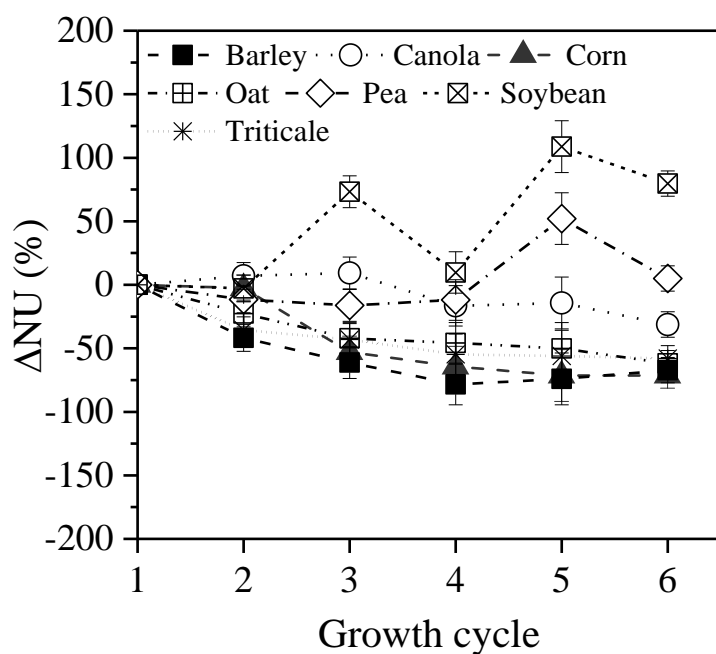


Figure 4.2 Changes in N uptake relative to Cycle 1 ( $\Delta NU_1$ ) for crops grown in a previously heavily manured soil. Averaged across moisture regimes, the mean N uptake in Cycle 1 were 220, 242, 503, 230, 94, 38, and 230  $\text{mg kg}^{-1}$  soil for barley, canola, corn, oat, pea, soybean, and triticale, respectively. Vertical bars represent standard errors of the mean.

The only other crops that showed increased N uptake relative the Cycle 1 were pea in Cycle 5 and, to a lesser extent, canola in Cycles 2 and 3. Reduction in N uptake relative to Cycle 1 was

highest for barley and corn, followed by triticale and oat. The percent change in N uptake of all crops relative to Cycle 1  $[(\Delta NU)_1]$  was not significantly affected by moisture regime ( $P = 0.55$ ).

#### 4.4.4. Soil Available Nitrogen

Soil available N (SAN) concentration differed significantly ( $P < 0.0001$ ) among crops; however, crop differences varied with moisture regime and growth cycle ( $P < 0.001$  for the 3-way interaction; Table 4.2). At 100% SFC, SAN was lower under canola and corn than under pea in Cycle 1 and was significantly greater under barley than canola and corn in all but the second growth cycle (Fig. 4.3a). Barley produced a significantly greater SAN than all the other crops in Cycle 5 and than canola, corn, oat, and triticale in Cycle 6.

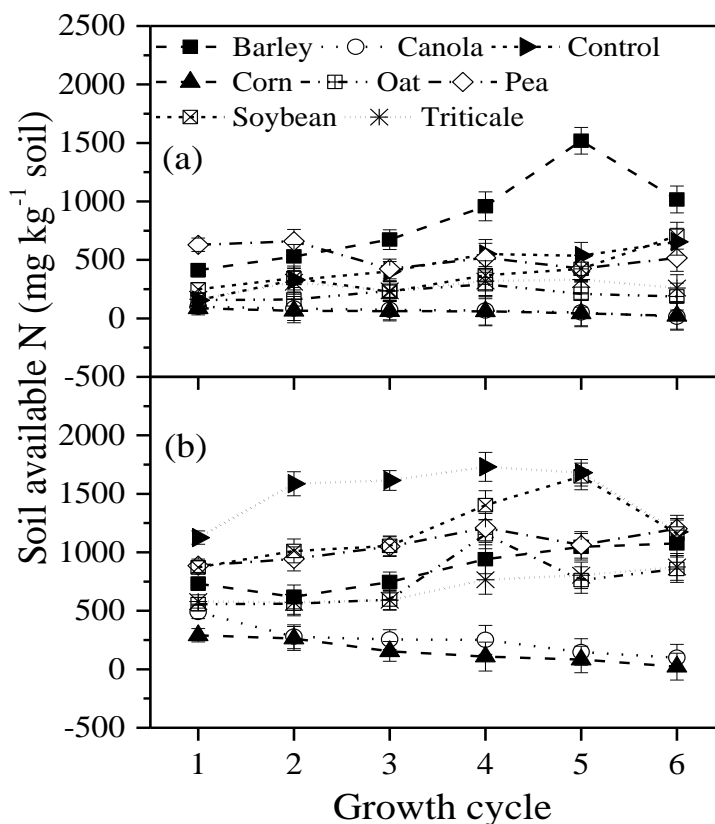


Figure 4.3 Soil residual N by various crops grown repeatedly in a previously heavily manured soil under (a) 100% and (b) 50% soil field capacity. Vertical bars represent standard errors of the mean.

At 50% SFC, SAN was significantly lower for canola and corn than the control, soybean, and pea in all growth cycles (Fig. 4.3b). In Cycle 6, SAN was lower for canola and corn than the control and the rest of the crops. By the end of Cycle 6, SAN under corn and canola had decreased to 21 and 98 mg kg<sup>-1</sup>, respectively, compared with 1075 mg kg<sup>-1</sup> for barley, 858 mg kg<sup>-1</sup> for oat, 1202 mg kg<sup>-1</sup> for pea, 1161 mg kg<sup>-1</sup> for soybean, and 875 mg kg<sup>-1</sup> for triticale.

Soil available N under soybean and the control was significantly lower at 100% than at 50% SFC in all but the sixth growth cycle. Similarly, SAN was lower at 100% than at 50% SFC for pea in Cycle 3 and for oat in Cycles 1 and 4. A greater reduction in SAN at 100% compared with 50% SFC was also observed for canola and triticale in Cycle 1.

#### **4.4.5. Change in Phosphorus Uptake**

There was a significant ( $P = 0.0001$ ) crop  $\times$  growth cycle interaction for the percent change in P uptake relative to Cycle 1 ( $\Delta PU_1$ ) (Table 4.2). Only the legumes (pea in Cycles 2, 4, and 5, and, to a lesser extent, soybean in Cycles 3 and 5) showed an increase in P uptake relative to Cycle 1, whereas the four grain crops (corn, barley, oat, and triticale) recorded the largest reductions in  $\Delta PU_1$  (Fig. 4a). While pea showed a significantly greater increase in  $\Delta PU_1$  than the other six crops in Cycles 4 and 5, than barley in Cycle 2, and than corn in Cycle 3, the decrease in  $\Delta PU_1$  was significantly greater for barley than for soybean in all but the second growth cycle and for corn than soybean in Cycles 3 and 5.

While  $\Delta PU_1$  decreased progressively with growth cycle for the four grain crops,  $\Delta PU_1$  for pea increased from -1% in Cycle 3 through 51% in Cycle 4 to peak at 93% in Cycle 5, while that for soybean increased significantly from -35% in Cycle 2 to peak at 21% in Cycle 5. The  $\Delta PU_1$  for all crops was lowest in Cycle 6 and did not differ significantly among crops in that cycle. Moisture regime had no significant effect ( $P = 0.51$ ) on  $\Delta PU_1$  regardless of crop or growth cycle.

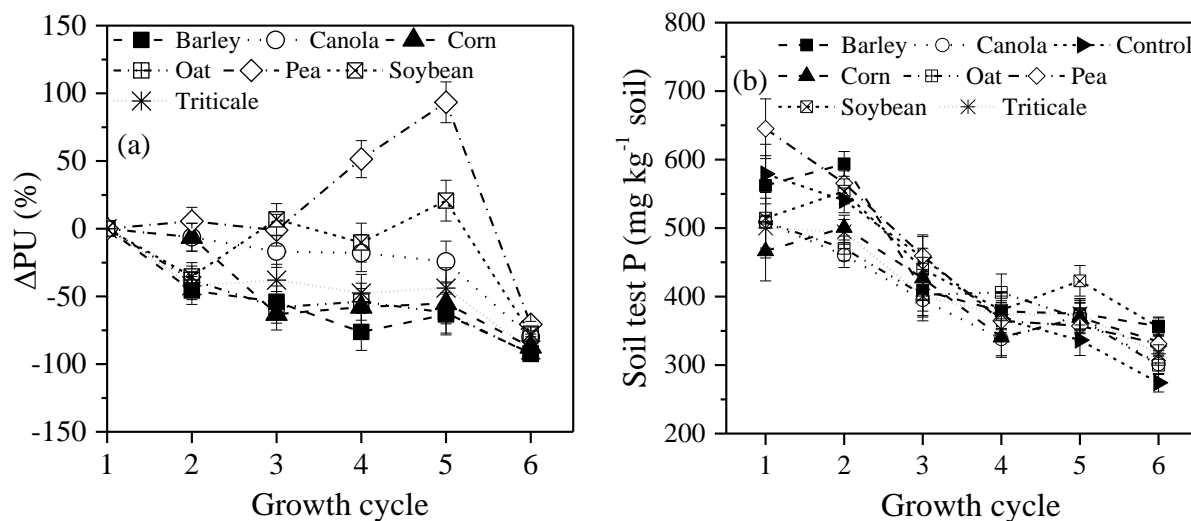


Figure 4.4 Change in P uptake (a) relative to Cycle 1 ( $\Delta PU_1$ ) and soil test (Olsen) P concentration (b) for various crops grown in a previously heavily manured soil. Averaged across moisture regimes, mean P uptake values in Cycle 1 were 31.3, 35.6, 69.4, 44.3, 8.02, 7.54, and 30.5  $\text{mg kg}^{-1}$  soil for barley, canola, corn, oat, pea, soybean, and triticale, respectively. Vertical bars represent standard errors of the mean.

#### 4.4.6. Soil Available Phosphorus

There was a significant ( $P = 0.04$ ) crop  $\times$  growth cycle interaction for Olsen P concentration (Table 4.2). Averaged across moisture regimes, Olsen P concentration in Cycle 2 was significantly greater for barley (594  $\text{mg kg}^{-1}$  soil) than canola (460  $\text{mg kg}^{-1}$  soil) and oat (470  $\text{mg kg}^{-1}$  soil) (Fig. 4.4b). Soil available P concentration decreased significantly from 594  $\text{mg kg}^{-1}$  soil in Cycle 2 to 395  $\text{mg kg}^{-1}$  soil in Cycle 3 for barley. Soil available P concentration was significantly lower for the control (274  $\text{mg kg}^{-1}$  soil) than barley (357  $\text{mg kg}^{-1}$  soil) and soybean (355  $\text{mg kg}^{-1}$  soil) in Cycle 6.

#### 4.4.7. Cumulative Nitrogen Uptake

There was a significant crop  $\times$  moisture regime interaction ( $P = 0.01$ ) for cumulative N uptake (CNU) (Table 4.2). Cumulative N uptake was greater under 100% than under 50% SFC for oat and triticale, but did not differ significantly with moisture regime for barley, canola, corn, pea and

soybean (Fig. 4.5a). Under both moisture regimes, CNU was greatest with corn followed by canola. Under the 100% SFC moisture regime, CNU was similar for barley (722 mg kg<sup>-1</sup>) and pea (682 mg kg<sup>-1</sup>). However, barley produced a lower CNU than oat (1131 mg kg<sup>-1</sup>) and triticale (1048 mg kg<sup>-1</sup>). Soybean showed a lower CNU compared with the rest of the crops. Under the 50% SFC, CNU did not differ significantly among barley (581 mg kg<sup>-1</sup>), oat (702 mg kg<sup>-1</sup>), pea (541 mg kg<sup>-1</sup>), and triticale (679 mg kg<sup>-1</sup>). Cumulative N uptake was lower with soybean (287 mg kg<sup>-1</sup>) than the rest of the crops.

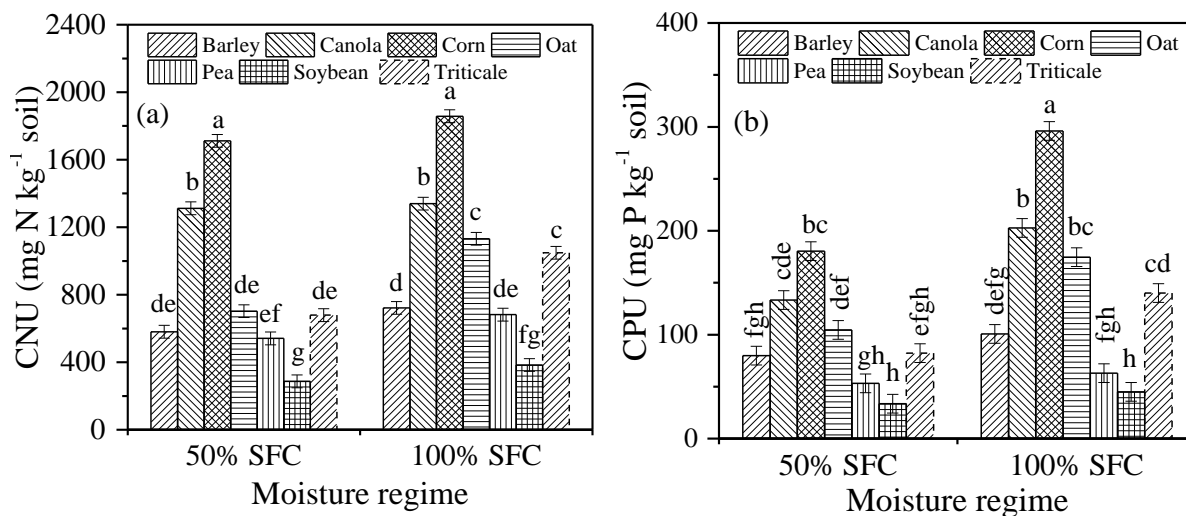


Figure 4.5 Cumulative N (CNU), and P uptake (CPU) by six cycles of barley, canola, corn, oat, pea, soybean, and triticale grown in a heavily manured soil at 50% and 100% soil field capacity (SFC). Error bars represent standard errors of the mean. Bars with the same letter are not significantly different ( $\alpha = 0.05$ , Turkey's test).

#### 4.4.8. Cumulative Phosphorus Uptake

There was a significant moisture regime  $\times$  crop interaction ( $P < 0.001$ ) for CPU (Table 4.2). Cumulative P uptake was greater under 100% compared with 50% SFC for corn, canola, oat, and triticale but did not differ significantly between the moisture regimes for barley, soybean, and pea



(Fig. 4.5b). Under both moisture regimes, CPU was greater with corn than the rest of the crops. Under 100% SFC, CPU was similar for canola (203 mg kg<sup>-1</sup>) and oat (175 mg kg<sup>-1</sup>) but significantly greater for these two crops than barley (101 mg kg<sup>-1</sup>). Pea (63 mg kg<sup>-1</sup>) and soybean (45 mg kg<sup>-1</sup>) showed a lower CPU than the rest of the crops, while CPU was similar for barley (101 mg kg<sup>-1</sup>) and triticale (140 mg kg<sup>-1</sup>). Under 50% SFC, CPU for soybean (33 mg kg<sup>-1</sup>) was not significantly different from that for barley (80 mg kg<sup>-1</sup>), oat (105 mg kg<sup>-1</sup>), and triticale (82 mg kg<sup>-1</sup>).

#### **4.5. Discussion**

Available N (575 mg kg<sup>-1</sup>) and Olsen P (555 mg kg<sup>-1</sup>) concentrations at the start of the experiment were well above the critical levels for optimum yield in western Canada (Howard, 2006). The high soil test P concentration presents a major risk of surface water contamination with soluble or particulate P (Whalen and Chang, 2001). While, overall, reductions in DMY and, correspondingly, N and P uptake relative to Cycle 1 were greater for the four grain crops (corn, barley, oat, and triticale) than the two legumes (pea and soybean), this did not translate to superior N and P uptake for the legumes across the 6 growth cycles. In fact, nutrient uptake was generally greater for corn, oat, and triticale, reflecting their much higher DMY and CDMY even after substantial decreases in DMY in later growth cycles. Reductions in DMY and N and P uptake were also lower for canola than the grain crops, and unlike the legumes, canola accumulated significantly more N and P as a result of its greater DMY compared with pea and soybean. Overall, the order of nutrient accumulation matched that for crop DMY and CDMY. The progressive decline in DMY observed for all crops in successive growth cycles was likely due to the restrictions imposed by the volume of the potted soil, which could have resulted in limited amounts of nutrients available to sustain crop growth with repeated harvesting. Other studies have reported similar reductions in biomass yields of crops with repeated harvesting in pot experiments, and attributed this yield

decrease to either nutrient (especially N) deficiency in the soil or root infection resulting from growing the same crop in successive growth cycles (Helgason et al., 2007; Benke et al., 2010). Agricultural management practices that ensure that crop yield is maximized will definitely increase N and P removal from the soil.

The decrease in DMY with repeated cropping for corn and canola could possibly be due to nutrient (particularly N) depletion since nutrients were not applied during the study. Repeated cropping resulted in a decrease in soil available N for corn and canola at the end of the sixth harvest cycle compared to the initial level prior to the start of the experiment. The lower DMY observed in subsequent harvest cycles suggests that the depletion of soil nutrients could have reduced the yield potential of corn and canola. Without further fertilization of the soil, the soil nutrient reserve gets depleted due to the continuous extraction of the soil nutrients by the crops.

It seems that soil N not available for plant utilization in the first harvest cycle was gradually released in subsequent crop cycles through the mineralization process in the presence of plant roots and under optimum environmental conditions. However, the nutrients released in subsequent growth cycles were not enough to sustain higher biomass production and therefore resulted in lower nutrient removal from the soil. However, soil available N was high even at the end of the sixth harvest cycle for the other grain crops (barley, triticale, and oat), and the legumes (pea and soybean). The greater soil available N observed under both moisture regimes at the end of the cycle 6 for barley, oat, triticale, pea, and soybean indicates that some factors may have limited crop growth, and hence nutrient uptake by these crops, resulting in the greater residual N and P observed. In addition, the ability of pea and soybean to fix N from the atmosphere possibly reduced the N requirement for optimum yield, resulting in greater amounts of soil available N (Grant et al., 2002). Overall, N removal by barley, canola, corn, oat, pea, soybean, and triticale after six growth cycles resulted in the removal of 5, 10.2, 13.7, 7.1, 4.7, 2.6, and 6.6%, respectively, of TN initially present in the soil.

Dry matter yield and N uptake declined with repeated cropping for almost all crops, with the decreasing trends greater for grain crops followed by canola. Sieling et al. (2006) reported a reduction in DMY and N uptake of barley, wheat, and winter oilseed rape during a 3-yr field study when these crops were grown in a soil that had received a single application of pig slurry. They attributed this reduction to a decrease in available N concentration in subsequent years relative to the first year of the experiment. In our study, soil residual N concentration was lowest under corn and canola, a result which was consistent with the greater CNU of the two crops, suggesting that these crops will readily deplete elevated levels of N in soil when harvested. The lower residual soil N under 100% SFC reflects greater N uptake by plants under the optimum moisture conditions, which produced greater biomass yields.

In the Dark Brown soil zone and for medium and fine-textured soils in Alberta where the soil used in this study was collected, the maximum recommended agronomic limit of  $\text{NO}_3\text{-N}$  (0-60cm) in manured cropland is  $170 \text{ kg ha}^{-1}$ , which is equivalent to  $21 \text{ mg N kg}^{-1}$ , assuming a bulk density of  $1.33 \text{ g cm}^{-3}$  for the soil (0-60 cm) (Miller et al., 2011). At the end of the Cycle 6, soil available N of  $14 \text{ mg kg}^{-1}$  for canola under 100% SFC was lower than the agronomic limit while the  $22 \text{ mg kg}^{-1}$  measured for corn matched the agronomic limit. Soil available N concentrations for the other crops were greater than the agronomic limit even at the end of Cycle 6.

The four cereal crops showed a gradual decrease in  $\Delta\text{PU}_1$  across all growth cycles and generally produced greater reductions in P uptake (relative to Cycle 1) than the legumes and, to a lesser extent, canola. However, similarly to N uptake, corn, oat, and canola, and to a lesser extent, barley and triticale, produced greater P uptake than the legumes. After six growth cycles, the amount of P removed by barley, canola, corn, oat, pea, soybean, and triticale was 7.3, 13.5, 19.2, 11.3, 4.7, 3.2, and 9%, respectively, of TP initially present in the soil. Eghball et al. (2003) reported that, based on

the amount of P removed in the grain, corn was more effective than soybean in reducing P levels in a soil testing high for soil test P. Phosphorus uptake was, on average, lower at 50% SFC than at 100% SFC, reflecting the reduced DMY at the lower moisture level. The soil test P at the end of the six crop cycles for all the crops was greater than the maximum agronomic limit of  $60 \text{ mg kg}^{-1}$  allowed in cropland in Alberta (Olson et al., 2010; Miller et al., 2011).

Feeding high N crop biomass to cattle in a feedlot operation could result in increased excretion of N and P in the manure. Repeated application of the manure in fields in the vicinity of the feedlot could result in N and P loading, which may pose a risk to the environment. The best practice may be to export harvested crops with high nutrient accumulation to farms that have low soil nutrient levels. This way, the excessive soil nutrients are not returned to the heavily manured soil.

Long term studies are needed to examine the nutrient phytoextraction effectiveness of the crops tested under field conditions. It is possible that the N and P removal rates under field condition will be greater than those observed in this pot experiment which imposed restrictions on root growth. Under favourable conditions in the field, barley and oat roots, for example, can extend to a depth of 2 m (Hoad et al., 2001). Past results have shown a large proportion of root biomass for barley and soybean in the top 15 cm, while the bulk of the corn rooting system was concentrated in the top 45 cm (Dwyer et al., 1988; Qin et al., 2006). López-Castañeda and Richards, (1994) reported that the root length density for barley cultivars in the 0-15 cm soil depth was greater than triticale, with barley exhibiting greater roots at all depths down to 90 cm. Cutforth et al. (2013) found that although pea and canola root systems extended down to a depth of 120-cm, a greater portion of the rooting systems for both crops were concentrated in the surface 0-20 cm soil depth. In another study, Gan et al. (2009) reported that approximately 77 to 85% of the root mass for canola and pea were found in the 0-40 cm depth. While these differences rooting depth profiles of the crops tested may result in differences in N phytoextraction, P removal rates in this study likely closely mimic those under field

conditions since soil P is immobile and is confined mostly within the 0-15-cm depth (Miller et al., 2011).

#### **4.6. Conclusions**

Overall, N and P phytoextraction effectiveness in the heavily manured soil was directly related to DMY, with the highest yielding crop, corn, being the most effective. Nitrogen and P uptake by the cereal grains and canola were highest in Cycle 1 and decreased progressively through Cycle 3, after which there was little change. The decrease in nutrient uptake was fastest for the cereal grains and less pronounced for the two legumes. Nutrient phytoextraction was generally greater at 100% than 50% SFC, reflecting optimum growing conditions at 100% SFC. Results from this study show that phytoextraction rates after six growth cycles were greater for P than N. The results indicate that continuous cropping with crops such as corn and canola can be a useful management practice to reduce nutrient levels in heavily manured soils. However, it will take several harvest cycles to reduce nutrient concentrations to environmentally acceptable levels.

#### **4.7. References**

**Agehara, S. and Warncke, D. D. 2005.** Soil moisture and temperature effects on nitrogen release from organic nitrogen sources. *Soil Sci. Soc. Am. J.* **69**: 1844-1855.

**Alberta Agriculture and Forestry. 2016.** Alberta irrigation management manual. [Online]. Available:

[http://www1.agric.gov.ab.ca/\\$department/deptdocs.nsf/all/irr14310/\\$file/2016\\_irr\\_manag\\_manual.pdf?OpenElement](http://www1.agric.gov.ab.ca/$department/deptdocs.nsf/all/irr14310/$file/2016_irr_manag_manual.pdf?OpenElement) (verified 25 January 2017).

**Barbazán, M. M., Mallarino, A. P. and Sawyer, J. E. 2009.** Liquid swine manure phosphorus utilization for corn and soybean production. *Soil Sci. Soc. Am. J.* **73**: 654-662.

**Benke, M. B., Indraratne, S. P. and Hao, X. 2013.** Long-term manure applications impact on irrigated barley forage mineral concentrations. *Agron. J.* **105**: 1441-1450.

- Benke, M. B., Hao, X., Caffyn, P., Schoenau, J. J. and McAllister, T. A. 2010.** Using manure from cattle fed dried distillers' grains with solubles (DDGS) as fertilizer: Effects on nutrient accumulation in soil and uptake by barley. *Agric. Ecosyst. Environ.* **139**: 720-727.
- Blackshaw, R. E., Johnson, E. N., Gan, Y., May, W. E., McAndrew, D. W., Barthet, V., McDonald, T. and Wispinski, D. 2011.** Alternative oilseed crops for biodiesel feedstock on the Canadian prairies. *Can. J. Plant Sci.* **91**: 889-896.
- Brink, G. E., Pederson, G. A., Sistani, K. R. and Fairbrother, T. E. 2001.** Uptake of selected nutrients by temperate grasses and legumes. *Agron. J.* **93**: 887-890.
- Cutforth, H. W., Angadi, S. V., McConkey, B. G., Miller, P. R., Ulrich, D., Gulden, R., Volkmar, K. M., Entz, M. H. and Brandt, S. A. 2013.** Comparing rooting characteristics and soil water withdrawal patterns of wheat with alternative oilseed and pulse crops growth in the semiarid Canadian prairie. *Can. J. Soil Sci.* **93**: 147-160.
- Dwyer, L. M., Stewart, D. W. and Balchin, D. 1988.** Rooting characteristics of corn, soybeans and barley as a function of available water and soil physical characteristics. *Can. J. Soil Sci.* **68**: 121-132.
- Eghball, B., Shanahan, J. F., Varvel, G. E. and Gilley, J. E. 2003.** Reduction of high soil test phosphorus by corn and soybean varieties. *Agron. J.* **95**: 1233-1239.
- Ferguson, R. B., Nienaber, J. A., Eigenberg, R. A. and Woodbury, B. L. 2005.** Long-term effects of sustained beef feedlot manure application on soil nutrients, corn silage yield, and nutrient uptake. *J. Environ. Qual.* **34**: 1672-1681.
- Gan, Y. T., Campbell, C. A., Janzen, H. H., Lemke, R., Liu, L. P., Basnyat, P. and McDonald, C. L. 2009.** Root mass for oilseed and pulse crops: Growth and distribution in the soil profile. *Can. J. Plant Sci.* **89**: 883-893.
- Grant, C. A., Peterson, G. A. and Campbell, C. A. 2002.** Nutrient considerations for diversified cropping systems in Northern Great Plains. *Agron. J.* **94**: 186-198.
- Helgason, B. L., Larney, F. J., Janzen, H. H. and Olson, B. M. 2007.** Nitrogen dynamics in soil amended with composted cattle manure. *Can. J. Soil Sci.* **87**: 43-50.
- Hoad, S. P., Russell, G., Lucas, M. E. and Bingham, I. J. 2001.** The management of wheat, barley, and oat root systems. *Adv. Agron.* **74**: 193-246.
- Howard, A. E. 2006.** Agronomic thresholds for soil phosphorus in Alberta: a review. In: Alberta soil phosphorus limits projects. Vol. 5. Background information and reviews. Alberta Agriculture, Food and Rural Development, Lethbridge, Alberta, Canada. [Online]. Available:

[http://www1.agric.gov.ab.ca/\\$department/deptdocs.nsf/all/sag11864/\\$FILE/vol-5-agronomic-thresholds.pdf](http://www1.agric.gov.ab.ca/$department/deptdocs.nsf/all/sag11864/$FILE/vol-5-agronomic-thresholds.pdf) (verified 18 February 2016).

**Kroetsch, D. and Wang, C. 2006.** Particle size distribution. In: M.R. Carter and E.G. Gregorich editors, Soil sampling and methods of analysis. Chapter 55. Canadian Society of Soil Science. Taylor & Francis Group., Boca Raton, FL.

**Leirós, M. C., Trasar-Cepeda, C., Seoane, S. and Gil-Sotres, F. 1999.** Dependence of mineralization of soil organic matter on temperature and moisture. *Soil Biol. Biochem.* **31**: 327-335.

**López-Castañeda, C. and Richards, R. A. 1994.** Variation in temperate cereals in rainfed environments I. Grain yield, biomass and agronomic characteristics. *Field Crops Res.* **37**: 51-62.

**Malhi, S. S., Johnston, A. M., Schoenau, J. J., Wang, Z. H. and Vera, C. L. 2007.** Seasonal biomass accumulation and nutrient uptake of canola, mustard, and flax on a Black Chernozem soil in Saskatchewan. *J. Plant Nutr.* **30**: 641-658.

**Malhi, S. S., Johnston, A. M., Schoenau, J. J., Wang, Z. H. and Vera, C. L. 2006.** Seasonal biomass accumulation and nutrient uptake of wheat, barley and oat on a Black Chernozem soil in Saskatchewan. *Can. J. Plant Sci.* **86**: 1005-1014.

**Maynard, D. G., Kalra, Y. P. and Crumbaugh, J. A. 2006.** Nitrate and exchangeable ammonium nitrogen. In: M.R. Carter and E.G. Gregorich editors, Soil sampling and methods of analysis. Chapter 6. Canadian Society of Soil Science. Taylor & Francis Group., Boca Raton, FL.

**Miller, J. J., Chanasyk, D. S., Curtis, T. W. and Olson, B. M. 2011.** Phosphorus and nitrogen in runoff after phosphorus-or nitrogen-based manure applications. *J. Environ. Qual.* **40**: 949-958.

**Nadler, A. J. and Bullock, P. R. 2011.** Long-term changes in heat and moisture related to corn production on the Canadian prairies. *Clim. Change* **104**: 339-352.

**Olson, B. M., Bremer, E., McKenzie, R. H. and Bennett, D. R. 2010.** Phosphorus accumulation and leaching in two irrigated soils with incremental rates of cattle manure. *Can. J. Soil Sci.* **90**: 355-362.

**Parkinson, J. A. and Allen, S. E. 1975.** A wet oxidation procedure suitable for the determination of nitrogen and mineral nutrients in biological material. *Commun. Soil Sci. Plant Anal.* **6**: 1-11.

**Pederson, G. A., Brink, G. E. and Fairbrother, T. E. 2002.** Nutrient uptake in plant parts of sixteen forages fertilized with poultry litter: Nitrogen, phosphorous, potassium, copper, and zinc. *Agron. J.* **94**: 895-904.

**Pote, D. H., Lory, J. A. and Zhang, H. 2003.** Does initial soil P level affect water-extractable soil P response to applied P? *Adv. Environ. Res.* **7**: 503-509.

**Qin, R., Stamp, P. and Richner, W. 2006.** Impact of tillage on maize rooting in a Cambisol and Luvisol in Switzerland. *Soil Tillage Res.* **85**: 50-61.

**Rowe, D. E. and Fairbrother, T. E. 2003.** Harvesting winter forages to extract manure soil nutrients. *Agron. J.* **95**: 1209-1212.

**Read, J. J., Sistani, K. R., Brink, G. E. and Oldham, J. L. 2007.** Reduction of high soil test phosphorous by Bermudagrass and Ryegrass-Bermudagrass following the cessation of broiler litter application. *Agron. J.* **99**: 1492-1501.

**SAS Institute. 2014.** SAS/STAT 13.2 User's guide. Version 9.4. Cary (NC): SAS Institute, Inc.

**Schröder, J. J., Jansen, A. G. and Hilhorst, G. J. 2005.** Long-term nitrogen supply from cattle slurry. *Soil Use Manage.* **21**: 196-204.

**Sharpley, A. N. 1995.** Dependence of runoff phosphorus on extractable soil phosphorus. *J. Environ. Qual.* **24**: 920-926.

**Sieling, K., Brase, T. and Svib, V. 2006.** Residual effects of different N fertilizer treatments on growth, N uptake and yield of oilseed rape, wheat and barley. *Europ. J. Agron.* **25**: 40-48.

**Sierra, J. 1997.** Temperature and soil moisture dependence of N mineralization in intact soil cores. *Soil Biol. Biochem.* **29**: 1557-1563.

**Slaton, N. A., Roberts, T. L., Golden, B. R., Ross, W. J. and Norman, R. J. 2013.** Soybean response to phosphorus and potassium supplied as inorganic or poultry litter. *Agron. J.* **105**: 812-820.

**Whalen, J. K. and Chang, C. 2001.** Phosphorus accumulation in cultivated soils from long-term annual applications of cattle feedlot manure. *J. Environ. Qual.* **30**: 229-237.

**Zentner, R. P., Wall, D. D., Nagy, C. N., Smith, E. G., Young, D. L., Miller, P. R., Campbell, C. A., McConkey, B. G., Brandt, S. A., Lafond, G. P., Johnston, A. M. and Derksen, D. A. 2002.** Economics of crop diversification and soil tillage opportunities in the Canadian prairies. *Agron. J.* **94**: 216-230.



## **5. MODELING BARLEY YIELD IN A DARK BROWN CHERNOZEM FOLLOWING DISCONTINUATION OF LONG-TERM MANURE APPLICATION**

### **5.1. Abstract**

Fertilizer application on soils that have received long-term manure application is often not necessary and typically not advised for environmental reasons. Soil properties at the discontinuation of manure application may assist in modeling crop yield trends on such soils in subsequent years. The data used in this study were from a long-term field experiment initiated in 1973 at the Agriculture and Agri-Food Canada Research and Development Centre in Lethbridge, Alberta. Soil and plant samples were collected annually from 1973 through 2010 from irrigated plots that were cropped to barley (*Hordeum vulgare* L.) every year. Soil properties measured at the start of the experiment in 1973 were used to predict yield in the control plots while soil properties measured at the end of annual manure applications in 2003 were used as predictors in partial least squares (PLS) regression models to predict barley yield in subsequent years in plots in which manure application was discontinued. Soil total N, organic carbon, P, and nitrate-N ( $\text{NO}_3\text{-N}$ ) concentrations were the most important soil properties for predicting annual barley grain yield in manured soils using PLS regression. Our results indicate that initial measured soil properties can be a useful tool in modeling yearly crop yield. During the 7 yr following discontinuation of manure application, there was no evidence of convergence in grain yield among amendment treatments, reflecting that the soil nutrient levels which were above agronomic thresholds for optimum yields. Longer-term monitoring of yield will assist in the development of models for predicting when nutrient application will become necessary.

## **5.2. Introduction**

Livestock manure is routinely applied to crop land to replenish essential plant nutrients in the soil (Schoenau and Davis, 2006) and improve soil physical properties such as bulk density, porosity, permeability, and hydraulic conductivity (Miller et al., 2002). Caution is usually taken to minimize the excessive build-up of nutrients that can result in environmental degradation (Eghball, 2000; Stumborg and Schoenau, 2008; Olson et al., 2009). Most agricultural soils near concentrated animal feeding operations often have a long history of manure application, with high levels of nitrogen (N), phosphorus (P), and possibly salts (Hao and Chang, 2003; Ferguson et al., 2005; Olson et al., 2010b).

Producers often discontinue manure application on cropland after an extended period of repeated manure application to allow for the phytoextraction of excess soil nitrogen (N) and phosphorus (P) through repeated cropping (Eghball et al., 2003). Successive crop production on such soils would be highly dependent upon the soil's ability to maintain its productivity and nutrient reserves with time (McAndrews et al., 2006; Sieling et al., 2006). To maximize crop production without any nutrient addition to the soil, the producer does require an understanding of soil properties that might limit yield (Cox et al., 2006). This can be accomplished by relating yield data to measured soil variables in the same field for many years. However, yearly data (soil and biomass yield) collection and laboratory analysis could be costly and labor-intensive, and the questions remain whether initial soil properties measured prior to the start of repeated manure application or immediately following cessation of manure application can be used to predict crop yields in subsequent years.

A variety of statistical techniques have been widely employed in studies examining the relationship between crop yield and soil and landscape properties. Many of the studies have

attempted to understand this relationship using methods such as stepwise multiple linear regression and correlation analysis (Majchrzak et al., 2001; Redulla et al., 2002; Iqbal et al., 2005). Others have employed multivariate statistical methods such as canonical correlation analysis, principal component analysis, factor analysis, and partial least squares (PLS) regression analysis (Ping et al., 2004; McDonald, 2006; Anthony et al., 2012). Partial least squares analysis offers a major advantage over ordinary least squares regression techniques in that it is not affected by the multicollinearity arising from the intercorrelation among soil properties (Ping et al., 2004).

In many jurisdictions, increasingly stringent environmental regulations dictate that no N and/or P fertilizers should be applied on soils that have received repeated manure applications and are enriched in these nutrients. In addition to these key nutrients, other soil properties are also critical to the sustenance of high crop yields. However, while many studies have examined relationships between soil properties and crop yields, to the best of our knowledge, no published study has attempted to explore the long-term trends in the yields of crops grown without supplemental fertilizer application in previously manured fields based on key soil properties at the start or cessation of manure application.

The overall objective of this study was to model irrigated barley yield using routinely measured soil properties determined at the establishment of long-term manure field plots in 1973 and at the cessation of manure application in 2003. The study also sought to investigate if the barley grain and soil properties measured during the period following discontinuation of manure application were becoming more similar (i.e., converging) among the discontinued manure treatments.

### 5.3. Materials and Methods

The study description and the analytical procedures used have previously been described in detail (Sommerfeldt and Chang, 1985; Chang et al., 2007; Indraratne et al., 2009; Benke et al., 2013) and are summarized here briefly.

#### 5.3.1. Site Description

The long-term experiment, established in 1973 was located at the Agriculture and Agri-Food Canada Research and Development Centre in Lethbridge, Alberta, Canada (Lat. 49°42'N; Long. 112°48'W). The soil at the site was a Calcareous Dark Brown Chernozemic clay loam (Typic Haploboroll). The clay mineralogy of the Chernozems located in this area is mainly dominated by mica and smectites, with small fractions of vermiculite, kaolinite and chlorite (Pennock et al., 2011).

The experiment was initially set up under irrigation and laid out as a split plot design with three replicates, with tillage (plow, rototiller, cultivator plus disk) as the whole plot (7.5 m by 60 m) and cattle (*Bos taurus*) feedlot manure rate (0, 60, 120, and 180 Mg ha<sup>-1</sup> yr<sup>-1</sup> wet weight) as the subplot (7.5 m by 15 m). The 60 Mg ha<sup>-1</sup> yr<sup>-1</sup> (wet weight) was the recommended manure application rate for irrigated soils in this area at the initiation of the experiment (Alberta Agriculture, 1980). Manure containing little or no straw bedding was collected from a commercial cattle feedlot operation (Chang et al., 2007) and was surface applied and then incorporated immediately each fall using one of the above tillage methods (Sommerfeldt and Chang, 1985). The control (zero rate) plots did not receive any fertilizer or manure application.

Since there was no significant effect of tillage on crop yield and soil properties, the tillage treatment was discontinued in 1986 after which a cultivator was used to incorporate manure in all subplots to a depth of 10 to 15 cm (Chang et al., 2007). This meant that the effective number of

replicates per manure treatment was now nine. Three of the nine replicates were discontinued in fall 1987 and another three replicates were discontinued in fall 2003, that is, after 14 and 30 annual manure applications, respectively. The remaining three replicates of each manure rate continued receiving manure applications until 2010. For the current study, data from 2003 to 2010 from treatments that had 30 yr of annual manure application followed by 8 yr of no manure application were used.

Barley (*Hordeum vulgare* L. cv. Galt) was the major crop grown in most years (1974-1995) on the irrigated plots except in 1996 when the plots were cropped to canola (*Brassica napus* L.) and 1997 to 2000 when corn (*Zea mays* L. cv. Pioneer hybrid 3957) was grown for silage. The barley cultivars used from 2003 to 2010 were ‘Duke’, ‘Kasota’, and ‘Vivar’ whereas those prior to 2003 were varieties commonly grown by local farmers. The site was generally seeded in May, and harvested for forage (the total above ground biomass at soft dough stage for silage making) (Alberta Agriculture and Forestry, 2015b) in late August or early September of each year using a 50 cm × 50 cm quadrat while grain was harvested with a small plot harvester from a 4-m<sup>2</sup> area of each plot in October of each year (Chang et al., 1993). The harvested crop biomass samples were oven dried at 60°C to estimate aboveground biomass yield. Crop residue was removed from all plots after grain harvest except for a 5 to 15 cm stubble layer which was left in the field (Indraratne et al., 2009; Benke et al., 2013).

### **5.3.2. Soil Collection**

Soil samples (two cores per plot) were collected from the 0- to 150-cm layer before fall application of manure at the establishment of the long-term plots in 1973 and also at the cessation of manure application in 2003. The soil cores were sectioned into six segments corresponding to the 0-15, 15-30, 30-60, 60-90, 90-120, and 120-150 cm depth intervals. Soil samples were also similarly

collected from each plot in 2004, 2006, and 2008 immediately after harvest and before fall manure application. The samples were air-dried and passed through a 2-mm sieve before laboratory analysis. Subsamples were fine-ground ( $< 0.15\text{mm}$ ) prior to analysis for soil total C (TC), total organic C (TOC), total N (TN) and total P (TP) determination. In this study, soil analysis results for samples from the 0-15 cm depth interval were used, except for the mobile nutrients  $\text{NO}_3$ ,  $\text{SO}_4$ , and  $\text{Cl}$ , which were measured in samples from the 0-60 cm layer.

### **5.3.3. Laboratory Analysis**

Soil total C (TC) and total N (TN) concentrations were determined using an automated CN analyzer (NA 1500, Carlo Erba, Milan, Italy). Total inorganic C (TIC) concentration was measured by the method of Amundson et al. (1988). Soil TOC was estimated as the difference between TC and TIC. Soil inorganic N ( $\text{IN} = \text{NH}_4\text{-N} + \text{NO}_3\text{-N}$ ) was extracted with 25 mL of 2 M KCl and measured using an Auto-analyzer III (Bran + Luebbe, Germany). For extractable inorganic P or soil-test P (STP) determination, the soil was extracted (1:10 soil to solution ratio) with 0.5 M  $\text{NaHCO}_3$  (Olsen et al., 1954). Total P (TP) concentration was measured using an EasyChem Pro discrete analyzer (Systea Analytical Technologies, Anagni, Italy) following digestion with  $\text{H}_2\text{SO}_4 + \text{H}_2\text{O}_2$  (Parkinson and Allen, 1975). Soil electrical conductivity (EC) was determined in saturated paste extracts (Janzen, 1993). Immediately after EC measurement, concentrations of  $\text{Mg}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{K}^+$ ,  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$  in the extract were measured using an ion chromatograph (Dionex Corporation, Sunnyvale, CA).

### **5.3.4. Weather Data**

Daily precipitation and temperature data were obtained from a meteorological station at the study site. Growing season potential evapotranspiration (PET) was calculated according to Baier and Robertson, (1965).

### **5.3.5. Statistical Analysis**

#### **5.3.5.1. Descriptive Statistics and Analysis of Variance**

Descriptive statistics (mean, variance, standard deviation, and coefficient of variation) were performed for all soil variables measured in 1973 and 2003 using the MEANS procedure in SAS version 9.4 (SAS Institute, 2014).

Analysis of variance (ANOVA) was performed using the GLIMMIX procedure for repeated measures in SAS 9.4 (SAS Institute, 2014) to determine the effects of manure rates on barley grain yield and soil parameters during the 8-yr period (2003-2010) following discontinuation of manure application. Manure rate was modeled as a fixed factor with year as the repeated measure. The covariance structures selected for the repeated measures analysis based on the corrected Akaike Information Criterion (Littell et al., 2006) were compound symmetry [CS] for TN, unstructured [UN] for TP, banded main diagonal [UN(1)] for barley grain yield, STP, NO<sub>3</sub>-N, and NH<sub>4</sub>-N, and first-order autoregressive [AR(1)] for TOC. When treatment effects were significant, means were compared using the Tukey-Kramer adjustment for multiple comparisons. Treatment effects were considered significant at  $\alpha = 0.05$ .

#### **5.3.5.2. Test of Homogeneity of Coefficients of Variation**

Tests of homogeneity of coefficients of variation (CVs) (Zar, 1999) were performed to determine if barley grain yields during the period (2004-2010) following discontinuation of manure application were becoming more similar among the discontinued manure treatments. The tests were also performed on soil NO<sub>3</sub>-N, NH<sub>4</sub>-N, STP, TN, TOC, and TP concentrations in 2003, 2004, 2006, and 2008. The MEANS procedure in SAS was used to estimate the CV used to calculate the test

statistic, which approximates the  $\chi^2$  distribution with  $k-1$  degrees of freedom, where  $k$  is the number of years. The z-test (Zar, 1999) was used for pairwise comparison of CVs between years.

#### **5.3.5.3. Partial Least Squares Regression**

Partial least squares (PLS) regression was performed using PROC PLS in SAS 9.4 to identify soil parameters (predictor variables) that best explain the variability in barley grain yield (response variable) over the years. Soil parameters measured in 2003 were included as explanatory variables in the initial model for the manured plots in which manure application was discontinued in 2003. For the control plots, the initial soil parameters measured at the establishment of the long-term plots in 1973 were included as explanatory variables.

The contribution of each explanatory variable to the PLS model was assessed based on the variable importance in the projection (VIP). Only predictor variables with a  $VIP > 0.8$  (Wold, 1995) were retained in the model. In PLS regression analysis, latent variables are generated to explain the variation in both the response and the predictor variables. The split cross validation method (CV = SPLIT) was used to determine the number of PLS extracted factors (latent variables) with the minimum predicted residual sum of squares (PRESS). The CVTEST option of PROC PLS was used to compare the PRESS for a model with fewer factors to that for a model with an optimum or minimizing number of factors. The model with fewer factors was selected as the final model if there was no significant difference between the two. The measured values in the response variables were regressed against the predicted values obtained from cross-validation using PROC REG in SAS to determine the predictive strength of the model.



## **5.4. Results and Discussion**

### **5.4.1. Precipitation and Irrigation**

Growing season (May 1 through August 31) precipitation ranged from 120 mm in 2007 to 375 mm in 2005 (Fig. 5.1). Growing season precipitation in 2007 was 43% lower than the long-term (1981-2010) mean of 220 mm. The greatest amounts of irrigation water were applied in 2008 (241 mm) followed by 229 mm in 2003 and 178 mm in 2006 and 2007. The PET values were 554, 492, 500, 574, 575, 516, 511, and 468 mm in 2003 through 2010, respectively. Growing season precipitation was lowest and the PET highest in 2007 compared to the other years. The highest growing season mean air temperature of 17.4°C was recorded in 2006 and 2007, and this which may have resulted in the high PET in 2007. Growing season precipitation plus irrigation water applied were generally less than PET except in 2008. The growing season precipitation plus irrigation applied in 2007 were 48% less than PET.

### **5.4.2. Soil Properties**

Soil properties varied widely among plots prior to treatment application in 1973 and also following discontinuation of manure application in 2003 (Table 5.1). Electrical conductivity in manured plots in 2003 ranged from 4-13 dS m<sup>-1</sup> compared with 1-2 dS m<sup>-1</sup> in the control plots. This indicates the accumulation of salts in the plots receiving repeated manure application. Generally, the mean and range of soil nutrients measured in 2003 in plots in which manure application was discontinued in fall 2002 were greater than those from the same plots measured in 1973 and in the control plots in 1973 and 2003. The mean STP concentration (1895 mg kg<sup>-1</sup>) in the 0-15 cm depth interval in plots in which manure was discontinued in 2003 exceeded the maximum recommended agronomic threshold of 60 mg kg<sup>-1</sup>, above which crops seldom respond to added P in Alberta soils (Howard, 2006).

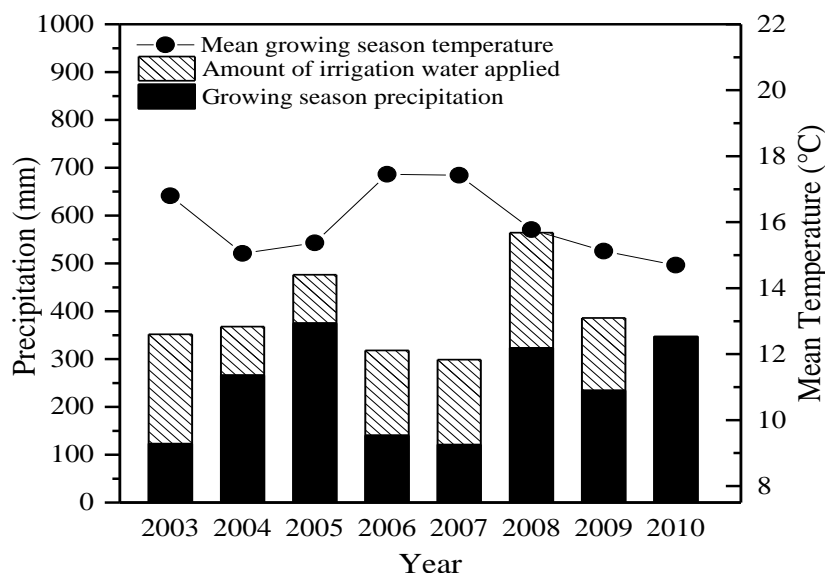


Figure 5.1 Annual growing season precipitation, amount of irrigation water applied, and average growing season temperature during the eight years (2003-2010) following discontinuation of 30 yr of manure application. The 30-yr (1981-2010) average growing season precipitation is 220 mm and the average growing season temperature is 16°C (Environment Canada 2016).

Also, the mean  $\text{NO}_3\text{-N}$  concentration ( $409 \text{ mg kg}^{-1}$ ) in the 0-60 cm depth interval in plots in which manure was discontinued exceeded the maximum recommended  $\text{NO}_3\text{-N}$  limit of  $34 \text{ mg kg}^{-1}$  ( $270 \text{ kg ha}^{-1}$ ) for manured, irrigated Dark Brown Chernozems and for medium and fine-textured soils (Province of Alberta, 2017). The excess soil N and P may be subject to loss to the environment via leaching of  $\text{NO}_3$  to groundwater and P surface runoff to water bodies, resulting in environmental degradation. The high mean STP ( $200 \text{ mg kg}^{-1}$ ), EC ( $1.4 \text{ dS m}^{-1}$ ), and Cl concentration ( $75 \text{ mg kg}^{-1}$ ) in the control plots in 2003 relative to measurements in 1973 could possibly be due to soil drifting and/or soil movement from manured plots resulting from tillage operations, which may have contaminated the control plots.

**Table 5.1 Descriptive statistics of soil properties measured at the start of the experiment in 1973 and in 2003 after discontinuation of manure application.**

Variable†	Control (1973)‡					Manured plots (1973)§					Control (2003) ¶					Manured plots (2003)#				
	Min.	Max.	Mean	SD	CV (%)	Min.	Max.	Mean	SD	CV (%)	Min.	Max.	Mean	SD	CV (%)	Min.	Max.	Mean	SD	CV (%)
pH	7.6	7.8	7.7	0.1	1	7.7	7.8	7.7	0.03	0.4	7.2	7.7	7.4	0.2	2.3	7	7.4	7.3	0.1	2
EC, dSm <sup>-1</sup>	0.5	0.9	0.7	0.1	18	0.5	1.2	0.7	0.2	28	1.1	2.3	1.4	0.4	28	4.2	13	7.7	2.6	34
STP, mg kg <sup>-1</sup>	8.8	19	11	3.3	30	3	16	9.6	3.6	38	58	659	200	185	92	1160	2628	1895	591	31
NO <sub>3</sub> -N, mg kg <sup>-1</sup>	7.8	26.2	14.1	5.9	42	7.6	24	15	4.8	32	15	129	35	37	106	137	732	409	172	42
NH <sub>4</sub> -N, mg kg <sup>-1</sup>	-	-	-	-	-	-	-	-	-	-	15	30	20	5.1	25	20	41	30	6.6	22
TN, g kg <sup>-1</sup>	0.3	2.1	1.6	0.5	34	1.5	2	1.8	0.2	10	2.1	4.2	2.7	0.6	24	8.6	19	13.8	4.3	31
TP, g kg <sup>-1</sup>	0.6	0.7	0.7	0.1	9	0.6	0.7	0.6	0.04	7	0.7	1.8	1	0.3	33	2	6.4	4.1	1.5	36
TC, g kg <sup>-1</sup>	-	-	-	-	-	-	-	-	-	-	22	42	29	5.7	20	56	136	89	27	30
TOC, g kg <sup>-1</sup>	11	15	12	1.4	12	9.2	16	13	1.9	15	17	38	23	6.5	28	55	135	88	26	30
SO <sub>4</sub> , mg kg <sup>-1</sup>	178	742	312	181	58	220	886	413	198	48	80	225	127	55	43	213	1483	706	349	49
Cl, mg kg <sup>-1</sup>	0	29	10	8.6	85	3.5	19	10	6.5	63	33	179	75	49	65	244	1575	898	424	47
Ca, mg kg <sup>-1</sup>	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	59.8	179	101	43	43
K, mg kg <sup>-1</sup>	-	-	-	-	-	-	-	-	-	-	14	59	32	14	45	182	832	429	246	57
Mg, mg kg <sup>-1</sup>	-	-	-	-	-	-	-	-	-	-	16	51	27	10	39	46	148	78	35	45
Na, mg kg <sup>-1</sup>	6.6	12	9	1.7	19	7	13	10	1.8	18	12	37	18	7.9	44	32	212	96	60	62

† EC, electrical conductivity; STP, soil-test phosphorus; NO<sub>3</sub>-N, soil nitrate-nitrogen; NH<sub>4</sub>-N, soil ammonium-nitrogen; TN, total nitrogen; TP, total phosphorus; TC, total carbon; TOC, total organic C; SO<sub>4</sub>, soluble sulphate; Cl, soluble chloride; Ca, soluble calcium; K, soluble potassium; Mg, soluble magnesium; Na, soluble sodium. Sample size was nine for the control and manured plots, respectively. For soil parameters, the soil analyses for samples from the 0-15 cm depth interval were used, except for NO<sub>3</sub>-N, SO<sub>4</sub>, and Cl, which were measured in samples from the 0-60 cm depth interval.

‡ Soil parameters measured in 1973 for the control plots.

§ Soil parameters measured in 1973 for the plots in which manure was applied until 2002.

¶ Soil parameters measured in 2003 for the control plots.

# Soil parameters measured in 2003 for plots in which manure application was discontinued in 2002.

### 5.4.3. Grain Yield Trends and Changes in Soil Properties after Discontinuation of Manure

#### Application

Repeated measures ANOVA for grain yield indicated a significant ( $P = 0.01$ ) year  $\times$  rate interaction (Table 5.2). In 2004 through 2007, the grain yield each year generally decreased with rate of manure application (Fig. 5.2). The yield in 2005 was in fact significantly greater for the 60 Mg ha<sup>-1</sup> yr<sup>-1</sup> rate than the 180 Mg ha<sup>-1</sup> yr<sup>-1</sup> rate and greater for the control than the 180 Mg ha<sup>-1</sup>, likely reflecting the high EC associated with the high manure rate.

**Table 5.2 Soil properties as affected by historical (30 yr) manure application and the number of years elapsed since discontinuation of manure application.**

Effect	Yield†	NH <sub>4</sub> -N	NO <sub>3</sub> -N	STP	TN	TP	TOC
	kg ha <sup>-1</sup>	————	mg kg <sup>-1</sup>	————	————	g kg <sup>-1</sup>	————
Rate (Mg ha <sup>-1</sup> yr <sup>-1</sup> )							
0	4852	20	22	153	3.07c‡	1.16c	28c
60	5132	65	144	840	6.25b	2.85b	52bc
120	4496	59	270	1225	8.48ab	3.69a	77ab
180	3904	76	330	1615	11a	4.36a	97a
Year							
2003	-	31	312	1471	7.45	3.35	67a
2004	4228	138	179	1414	7.51	3.26	69a
2005	4906	-	-	-	-	-	-
2006	5113	26	172	476	7.18	2.78	-
2007	3267	-	-	-	-	-	-
2008	6275	25	103	472	6.21	2.67	55b
2009	5006	-	-	-	-	-	-
2010	3377	-	-	-	-	-	-
ANOVA				<i>P</i> value			
Rate ( R )	0.01	0.61	<0.001	<0.001	<0.001	<0.001	<0.001
Year (Y)	<0.001	0.18	<0.001	<0.001	0.21	0.1	0.004
Y x R	0.01	0.23	0.01	<0.001	0.4	0.1	0.25

† Yield, barley grain yield; NH<sub>4</sub>-N, soil ammonium-nitrogen; NO<sub>3</sub>-N, soil nitrate-nitrogen; STP, soil-test phosphorus; TN, total nitrogen; TP, total phosphorus; TOC, total organic carbon. For soil parameters, soil samples from the 0-15 cm depth interval were used, except for NO<sub>3</sub>-N, which was measured in samples from the 0-60 cm depth interval.

‡ Values in the same column followed by the same letter are not significantly different at  $P < 0.05$ .

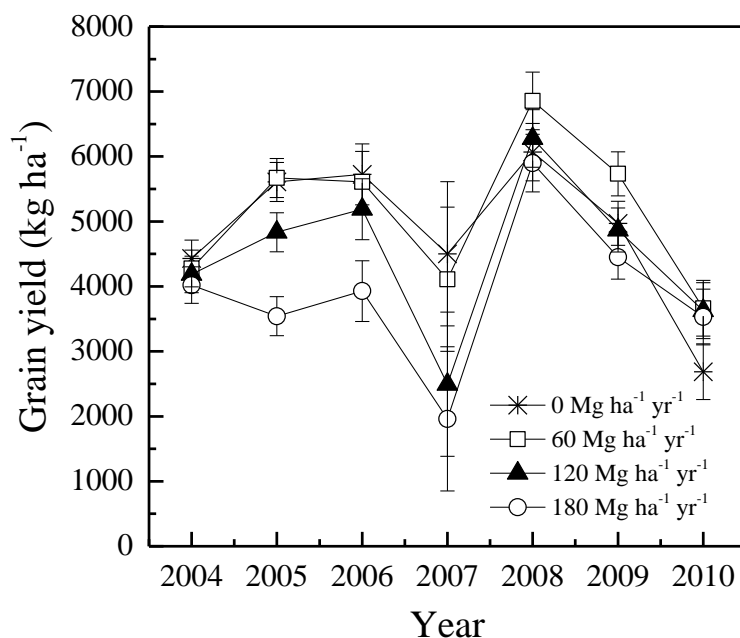


Figure 5.2 Barley grain yield during the 7 yr following discontinuation of 30 yr of annual beef cattle manure applications at different rates.

During the post-manuring period, grain yield showed no consistent overall trend regardless of historical manure application rate. However, yield from the last 3 yr (2008 through 2010) showed a nearly linear decrease with year for all treatments, which was likely due to the gradual decline in soil nutrient concentrations.

Tests for homogeneity of CVs for grain yield during the post-treatment phase (that is, 2004-2010) showed significant ( $P < 0.05$ ) differences in CVs among the years, indicating that treatment differences were not consistent across the years (Fig. 5.3a). This difference is due to the unusually high CVs observed in 2007 as a result of the reduced barley grain yield due to the low growing-season precipitation in 2007 (121 mm) compared to the other years, which ranged from 140 mm in 2006 to 375 mm in 2005. Most of the rainfall observed in 2007 was in May (86 mm), with much

smaller amounts in June (23 mm) and August (12 mm) and none in July (Fig. S1). Throughout much of Alberta, the Feekes Large Growth Stages 6 to 10 for barley, which correspond to Zadoks stages 30 to 45, occur in late June or early July, and possibly represent phenological stages when the crop has its highest moisture and nutrient requirements needs for dry matter accumulation (Alberta Agriculture and Forestry, 2015). When the CV for 2007 was excluded from the analysis, the test for homogeneity of CVs for grain yield indicated no significant difference in the CVs, suggesting no evidence that treatment effects were becoming more similar (Fig. 5.3b).

There was a significant year  $\times$  rate interaction ( $P = 0.01$ ) for  $\text{NO}_3\text{-N}$  concentration (Table 5.2). Soil  $\text{NO}_3\text{-N}$  concentration was significantly lower in the control than in plots receiving 120 and 180  $\text{Mg ha}^{-1} \text{ yr}^{-1}$  manure in 2003 and 2006 (Fig. 5.4). Similarly,  $\text{NO}_3\text{-N}$  concentration was significantly greater for the 120 and 180  $\text{Mg ha}^{-1} \text{ yr}^{-1}$  rates than the 60  $\text{Mg ha}^{-1} \text{ yr}^{-1}$  rate and the control in 2008. The  $\text{NO}_3\text{-N}$  concentration for the 60  $\text{Mg ha}^{-1} \text{ yr}^{-1}$  treatment was similar to that for the control in 2008. Apart from crop N uptake, the decrease in  $\text{NO}_3\text{-N}$  concentration may have been a result of nitrate leaching and denitrification following irrigation.

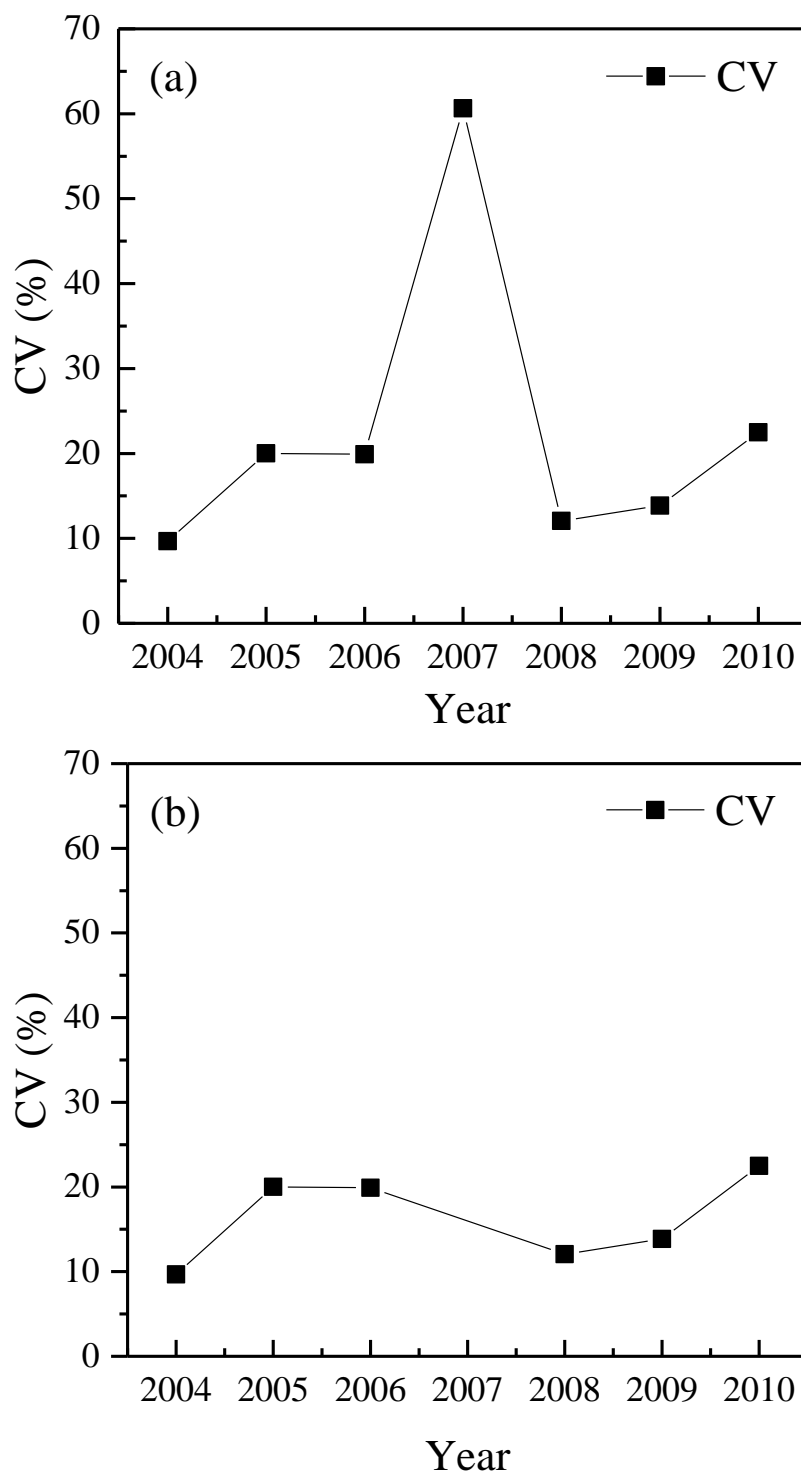


Figure 5.3 Trends in coefficient of variation (CV) of barley grain yield during the 7 yr following discontinuation of 30 yr of annual beef cattle manure application.

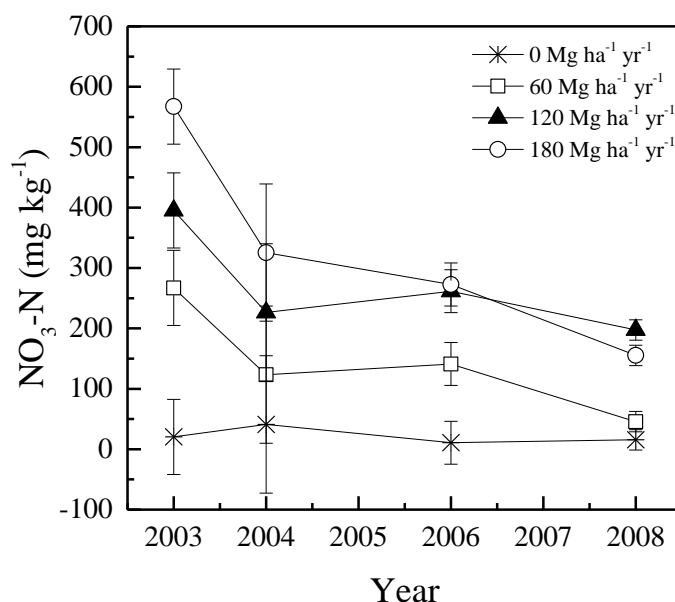


Figure 5.4 Soil nitrate N ( $\text{NO}_3\text{-N}$ ) concentration in the 0-60 cm soil layer during the 5 yr following discontinuation of 30 yr of annual beef cattle manure application at different rates.

There was a significant ( $P < 0.001$ ) year  $\times$  manure rate effect on STP (Table 5.2). In 2003 through 2008, STP generally decreased with rate of manure application, with the STP significantly greater for the  $180 \text{ Mg ha}^{-1} \text{ yr}^{-1}$  rate than the  $0$ ,  $60$ , and  $120 \text{ Mg ha}^{-1} \text{ yr}^{-1}$  rates in 2003 and 2004. For all manure treatments, STP decreased significantly from 2004 to 2006 and remained statistically unchanged in 2008 (Fig. 5.5). Studies have shown that the stabilization of freshly applied P and water soluble P in the soil results in the reduction of extractable soil P over time (Spratt et al., 1980; Sheppard and Racz, 1984a; Kashem et al., 2004). Dark Brown Chernozems are known to have high content of base cations ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$ , and  $\text{Na}^+$ ) in the A horizon, with  $\text{Ca}^{2+}$  as the dominant cation (Pennock et al., 2011). The decline in STP concentration from 2004 to 2006 could be due to a combination of crop uptake, dilution of P resulting from tillage operations, leaching, and precipitation of the inorganic P by the exchangeable Ca and Mg in the soil, transforming the P to



more stable forms over time (Hao et al., 2008). In an incubation study, Kashem et al. (2004) reported that most of the labile P ( $H_2O$ -P) in Chernozemic soils amended with hog, cattle manure, and synthetic P fertilizer moved into the less labile fraction ( $NaHCO_3$ -P) with increasing incubation time. The authors concluded that P sorption was the main retention mechanism for added P. By the fall of 2006 in our study, manure P remaining in the soil probably consisted of a relatively greater proportion of less soluble forms, and a correspondingly lower proportion of Olsen P. Pizzeghello et al. (2016) reported that the amount of P sorbed increased with soil organic matter content and Ca and Mg concentrations in the soil.

In addition to the organic matter content and precipitation reactions, P availability and hence STP may have been influenced by soil moisture content and temperature at the time of soil sampling (Sheppard and Racz, 1984b; Siebers et al., 2017). Soil samples were collected after harvest (crop nutrient uptake generally completed by end of July) and immediately before fall manure application in early November. The cumulative precipitation and mean air temperature between August and October in 2006 were 68.7 mm and 12.9°C, respectively, compared with 97.6 mm and 12.2°C, respectively, in 2004 (Table S1). The low precipitation in 2006, coupled with the high temperature that year, may have enhanced the formation (via precipitation) of more stable forms of P relative to 2004, resulting in the observed decrease in Olsen P in 2006 (Sheppard and Racz, 1984b). Nonetheless and importantly, STP concentrations were still above agronomic thresholds even for plots previously receiving the lowest manure rate, which had an STP concentration of 403 mg kg<sup>-1</sup> soil in 2008.

Results show that soil NO<sub>3</sub>-N and STP concentrations declined gradually with repeated harvesting with no further fertilization following the discontinuation of manure application. However, it will take a much longer time for the STP to decrease to acceptable agronomic levels

when compared to N. This could have environmental implications in that, the P is still susceptible to loss to the environment. The plots therefore need to be managed carefully to minimize this risk of loss from occurring.

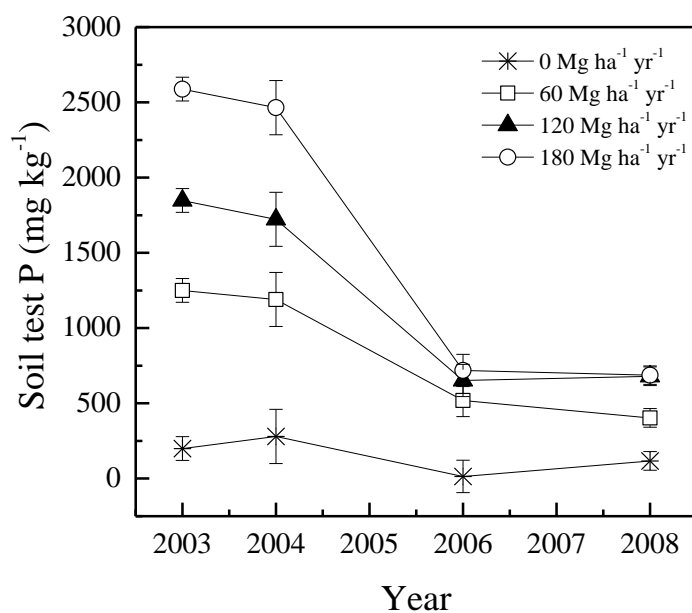


Figure 5.5 Soil test P concentration in the 0-15 cm soil layer during the 5 yr following discontinuation of 30 yr of annual beef cattle manure applications at different rates.

Tests of homogeneity of CVs for the soil parameters during the years following discontinuation of manure application indicated no significant difference in the CVs among the six years for NH<sub>4</sub>-N, TN, TP, and TOC concentrations, reflecting the high accumulation of N, P, and C from 30 yr of manure application.

#### 5.4.4. Predicting Grain Yield following Discontinuation of Long-term Manure Application

A one-latent variable PLS model explained 25 to 88% of the total variability in grain yield during the 7 yr (2004-2010) following discontinuation of manure application (Table 5.3). A two-latent variable PLS model improved the predictive power, explaining 90% of the variability in grain

yield 4 yr since the last manure application and 94% of the variability in cumulative grain yield (2003-2010). Nine to 13 explanatory variables had a significant ( $VIP > 0.8$ ) contribution to the model explaining yield each year and cumulative yield during the 7 yr following discontinuation of manure application. Four variables (TOC, TP, TN, and  $NO_3$ -N) were critical factors ( $VIP > 1.0$ ) for barley grain yield in almost all years following discontinuation of manure application. However, the model containing these four variables had a lower predictive power ( $R^2$ ) than models containing more variables selected using the criterion  $VIP > 0.8$ . For example,  $R^2$  decreased from 87% for the model based on  $VIP > 0.8$  to 85% in the model with just TOC, TP, TN, and  $NO_3$ -N in the model (4-parameter model) for grain yield 3 yr since the last manure application. On the other hand, the model based on  $VIP > 1$  improved  $R^2$  relative to the model based on  $VIP > 0.8$  and, for the most part, the four-parameter model, except in 2009. Hence, the final model selected was the one based on  $VIP > 1$ .

The four critical soil variables (TOC, TP, TN, and  $NO_3$ -N) were generally negatively correlated with barley grain yield. This apparently anomalous correlation may reflect in part the negative effect of EC, which increased concomitantly with the critical nutrients from a baseline range of 0.5-0.9 dS  $m^{-1}$  prior to manure application in 1973 to 4.2-12.7 dS  $m^{-1}$  in 2003 following discontinuation of manure application. Positive response to nutrient concentrations was not expected in the manured plots since concentrations remained very high even 6 yr after the end of manure application. On the other hand, negative effects from high  $NO_3$ -N concentrations (137-732 mg  $kg^{-1}$  soil in manured plots) may also have negatively affected barley yields. McDonald, (2006) reported that  $NO_3$ -N concentration in the subsoil was negatively related with both the grain yield and total dry matter yield of barley and wheat at maturity. Redulla et al. (2002) reported a similar negative correlation between total tuber count of potato (*Solanum tuberosum* L.) and soil  $NO_3$ -N concentration.

**Table 5.3 Variable importance in the projection (VIP) and regression coefficients (b) for soil variables measured in 2003 and used in models for predicting barley grain yield during the 8 yr (2003-2010) following discontinuation of 30 yr of manure application.**

Variable†	2004‡		2005		2006		2007		2009		Cumulative	
	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b
TOC, g kg <sup>-1</sup>	0.74	-0.03	1.18	-0.09	1.23	-0.09	1.13	-0.07	1.17	-0.09	1.17	-0.09
TP, g kg <sup>-1</sup>	0.80	-0.04	1.18	-0.09	1.29	-0.09	1.24	-0.08	1.19	-0.09	1.24	-0.09
TN, g kg <sup>-1</sup>	0.65	-0.03	1.16	-0.09	1.24	-0.09	1.20	-0.08	1.18	-0.09	1.19	-0.09
STP, mg kg <sup>-1</sup>	0.44	-0.02	1.15	-0.09	1.05	-0.08	0.91	-0.06	1.01	-0.08	1.06	-0.08
NO <sub>3</sub> -N, mg kg <sup>-1</sup>	1.32	-0.06	1.01	-0.08	1.09	-0.08	1.11	-0.07	1.16	-0.09	1.09	-0.09
NH <sub>4</sub> -N, mg kg <sup>-1</sup>	0.36	-0.02	0.61	-0.05	1.30	-0.08	1.45	-0.09	0.94	-0.07	1.01	-0.07
K, mg kg <sup>-1</sup>	0.86	-0.04	1.14	-0.09	0.96	-0.07	0.85	-0.05	0.98	-0.08	1.00	-0.08
Ca, mg kg <sup>-1</sup>	1.41	-0.07	0.98	-0.08	0.61	-0.05	0.34	-0.02	0.69	-0.05	0.71	-0.06
Mg, mg kg <sup>-1</sup>	0.99	-0.05	1.12	-0.09	0.81	-0.06	0.59	-0.04	0.89	-0.07	0.84	-0.07
Na, mg kg <sup>-1</sup>	1.14	-0.05	1.11	-0.09	0.94	-0.07	0.81	-0.05	0.99	-0.08	0.96	-0.08
Cl, mg kg <sup>-1</sup>	1.05	-0.05	0.84	-0.07	0.61	-0.05	0.79	-0.05	0.92	-0.07	0.79	-0.06
SO <sub>4</sub> , mg kg <sup>-1</sup>	1.37	-0.06	0.30	-0.02	0.73	-0.05	1.11	-0.07	0.93	-0.07	0.83	-0.06
pH	1.32	0.06	0.82	0.07	0.89	0.07	1.13	0.07	0.97	0.08	1.08	0.08
EC, dSm <sup>-1</sup>	0.75	-0.03	0.98	-0.08	0.85	-0.06	0.77	-0.05	0.84	-0.07	0.83	-0.06
PLS factors§	1		1		1		1		1		1	
Overall model PV ¶	25		88		71		48		80		80	
R <sup>2</sup> (VIP > 0.8) #	0.23		0.87		0.69		0.47		0.79		0.80	
R <sup>2</sup> (VIP > 1) ††	0.29		0.9		0.80		0.57		0.75		0.84	
R <sup>2</sup> (Four parameters) ‡‡	-		0.85		0.72		0.36		0.79		0.79	

† EC, electrical conductivity; STP, soil-test phosphorus; NO<sub>3</sub>-N, soil nitrate-nitrogen; NH<sub>4</sub>-N, soil ammonium-nitrogen; TN, total nitrogen; TP, total phosphorus; TOC, total organic carbon; SO<sub>4</sub>, soluble sulphate; Cl, soluble chloride; Ca, soluble calcium; K, soluble potassium; Mg, soluble magnesium; Na, soluble sodium.

‡ 2004, 2005, 2006, 2007, and 2009 represent 1, 2, 3, 4 and 6 yr following discontinuation of 30 yr of manure application; cumulative, predicting cumulative by summing barley grain yield from 2004 to 2010.

§ Number of partial least square (PLS) factors determined by cross-validation.

¶ Percentage of variation explained by the PLS factor in the cross-validation step.

# Predictive strength assessed by linear regression of measured values of response variables versus predicted values obtained in the cross-validation step using predictors with  $VIP > 0.8$ .

†† Predictive strength assessed by linear regression of measured values of response variables versus predicted values obtained in the cross-validation step using predictors with  $VIP > 1$  (preferred model).

‡‡ Predictive strength assessed by linear regression of measured values of response variables versus predicted values obtained in the cross-validation step with TOC, TP, TN, and  $NO_3-N$  as predictors.

The authors suggested that this negative correlation was likely due to the large amount of N applied, which was greater than the optimal for potato production.

The 2007 growing season was the driest of all years following discontinuation of manure application, receiving just 121 mm of rainfall compared with 139-375 mm for the other six seasons. Clearly, supplemental irrigation did not do much to mitigate the effects of the drought, as indicated by the generally lower yields in 2007 compared to the other years (Fig. 5.1). The dry conditions may also have resulted in the high yield variability in 2007, which showed a CV of 61% compared to 10-22% for the other years (Fig. 5.3). This likely explains the low  $R^2$  (48%) for the predictive model in 2007 compared to 0.8-0.94 for the other years. The low predictive power (25%) of the model observed for 2004 was likely due to poor seedling establishment resulting from delayed seeding early in the spring of 2004 (Personal communication with Dr. Xiying Hao) coupled with the low temperatures at the time of seeding in 2004 (7°C) compared with 10-19°C for the other six growing seasons (Fig. S2), which resulted in low barley grain yields for all treatments. Although, the average monthly temperature for May of 2004 was 15°C, there were days with as low as < 5°C.

Overall, our results suggest that soil parameters measured in the year following discontinuation of manure application accounted for a great percentage of the variability in grain yield and were therefore effective in predicting the yield even 7 yr after manure application was discontinued. In fact, inclusion of only TOC, TP, TN, and NO<sub>3</sub>-N concentration in the PLS model provided adequate predictive power in the subject plots that had a long history of manure application.

#### **5.4.5. Predicting Grain Yield for the Control Plot**

Although the seven-latent variable model provided the greatest  $R^2$  for grain yield prediction in 1988, as did the four-latent variable model in 1992, the one-factor model provided adequate

predictive power for the majority of the years (Table 5.4). The model did not converge when the multiple latent variable equivalents were replaced by their one latent variable in 1988 and 1992. The model containing fewer variables selected based on  $VIP > 1$  improved the predictive power relative to the model containing more variables selected using the criterion  $VIP > 0.8$  in 1976, 1979, 1984, 1992, 1993, 1994, and 1995. However, the  $R^2$  for both models were similar in 1977, 1978, 1985, 2009, 2010, and cumulative yield during the 37 yr of continuous cropping. Generally, the model based on  $VIP > 1$  had a greater predictive power than the four-parameter model for almost all the years, except in 1975, 2006, 2007 and 2010. Hence, the model based on  $VIP > 1$  was selected as the preferred model compared to the others.

Initial TOC, TN, TP, and  $\text{NO}_3\text{-N}$  concentrations were the most critical soil variables in the models and explained most of the variability in annual and cumulative grain yield. As expected for these control plots, there was a generally positive correlation between grain yield and the four soil variables. In contrast, the same predictors were generally negatively correlated with grain yield in plots that had previously received 30 yr of manure application, reflecting the negative effects of high EC and soil nutrient concentrations.

**Table 5.4 Variable importance in projection (VIP) and regression coefficients (b) values for soil parameters measured in 1973 and used to predict barley grain yield in the control plots.**

Variable†	1974‡		1975		1976		1977		1978		1979		1980	
	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b
TOC, g kg <sup>-1</sup>	0.19	-0.04	1.57	0.26	1.62	0.23	1.38	0.19	1.26	0.17	0.91	0.14	1.33	0.18
TP, g kg <sup>-1</sup>	1.00	-0.21	1.42	0.23	1.40	0.20	1.40	0.19	1.22	0.17	0.59	0.09	0.77	0.10
TN, g kg <sup>-1</sup>	0.43	0.1	1.52	0.25	1.69	0.24	1.36	0.19	1.51	0.21	1.35	0.21	1.71	0.23
Soil test P, mg kg <sup>-1</sup>	1.14	-0.24	0.57	0.09	0.12	0.02	0.42	0.06	0.27	0.04	0.63	-0.09	0.03	0.004
NO <sub>3</sub> -N, mg kg <sup>-1</sup>	0.84	0.18	1.13	0.18	0.94	0.14	1.29	0.18	1.11	0.15	0.89	0.14	1.21	0.17
Na, mg kg <sup>-1</sup>	1.30	0.27	0.41	0.07	0.24	0.03	0.34	0.05	0.2	-0.03	0.78	0.12	0.01	0.001
Cl, mg kg <sup>-1</sup>	1.28	0.27	0.19	0.03	0.37	0.05	1.03	0.14	1.35	0.19	1.57	0.24	1.17	0.16
SO <sub>4</sub> , mg kg <sup>-1</sup>	0.35	0.1	0.41	0.07	0.48	0.07	0.14	-0.02	0.37	-0.05	0.22	0.03	0.93	-0.13
EC, dSm <sup>-1</sup>	1.52	0.32	0.47	0.08	0.45	0.06	0.52	0.07	0.59	0.08	1.29	0.19	0.10	0.01
PLS factors§	1		1		1		1		1		1		1	
Overall model PV¶	70		82		60		63		60		54		57	
R <sup>2</sup> (VIP > 0.8) #	0.67		0.70		0.49		0.56		0.54		0.47		0.57	
R <sup>2</sup> (VIP > 1)††	0.58		0.70		0.55		0.56		0.54		0.48		0.53	
R <sup>2</sup> (Four parameters)‡‡	-		0.70		0.49		0.47		0.39		-		0.38	



**Table 5.4 (continued)**

	1981		1984		1985		1988		1992		1993		1994	
	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b
TOC, g kg <sup>-1</sup>	1.58	0.29	1.66	0.24	1.32	0.2	0.96	0.14	0.99	-0.01	1.18	0.18	1.22	0.18
TP, g kg <sup>-1</sup>	0.81	0.15	1.58	0.23	1.12	0.17	0.98	0.79	1.06	0.22	0.86	0.13	0.94	0.14
TN, g kg <sup>-1</sup>	1.30	0.24	1.56	0.22	1.57	0.24	1.11	-0.77	0.99	0.003	1.41	0.21	1.29	0.2
Soil test P, mg kg <sup>-1</sup>	0.90	-0.17	0.06	0.01	0.26	0.04	1.31	-0.81	1.57	0.79	0.28	-0.04	0.87	0.13
NO <sub>3</sub> -N, mg kg <sup>-1</sup>	0.89	0.16	0.88	0.13	1.25	0.19	0.94	0.69	1.05	0.01	1.25	0.19	0.89	0.13
Na, mg kg <sup>-1</sup>	1.45	0.27	0.26	0.04	0.19	0.03	0.99	0.73	0.59	0.02	0.26	-0.04	0.79	-0.12
Cl, mg kg <sup>-1</sup>	0.44	-0.08	1.24	0.03	1.15	0.17	0.63	0.04	0.46	0.03	1.32	0.19	0.2	0.03
SO <sub>4</sub> , mg kg <sup>-1</sup>	0.43	0.08	0.59	0.09	0.26	-0.04	0.87	-0.68	0.89	0.31	1.16	-0.17	1.23	-0.18
EC, dSm <sup>-1</sup>	0.25	-0.05	0.32	0.05	0.70	0.11	1.07	-0.51	0.98	0.42	0.04	-0.01	1.1	-0.16
PLS factors§	1		1		1		7		4		1		1	
Overall model PV¶	61		60		75		99		98		65		87	
R <sup>2</sup> (VIP > 0.8) #	0.48		0.47		0.69		-		0.98		0.56		0.82	
R <sup>2</sup> (VIP > 1) ††	0.46		0.54		0.69		-		0.99		0.61		0.97	
R <sup>2</sup> (Four parameters) ‡‡	-		0.47		0.56		-		0.49		0.32		0.54	

**Table 5.4 (continued)**

	1995		2006		2007		2008		2009		2010		Cumulative	
	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b	VIP	b
TOC, g kg <sup>-1</sup>	0.63	0.09	1.42	-0.2	1.48	-0.22	1.51	0.17	1.02	0.17	1.39	0.21	1.29	0.15
TP, g kg <sup>-1</sup>	0.77	0.11	1.13	-0.16	1.40	-0.21	0.58	0.09	0.70	0.12	1.05	0.16	1.11	0.13
TN, g kg <sup>-1</sup>	0.86	0.12	1.51	-0.21	1.45	-0.22	1.51	0.25	1.42	0.23	1.33	0.2	1.65	0.19
Soil test P, mg kg <sup>-1</sup>	0.94	0.14	0.53	-0.08	0.99	-0.15	0.17	-0.03	0.07	0.01	0.71	0.11	0.31	0.03
NO <sub>3</sub> -N, mg kg <sup>-1</sup>	1.56	0.22	1.11	-0.16	1.28	-0.19	1.45	0.24	1.53	0.25	1.66	0.25	1.04	0.12
Na, mg kg <sup>-1</sup>	0.45	-0.07	0.67	-0.09	0.11	-0.02	0.19	0.03	0.05	-0.01	0.31	0.05	0.19	-0.02
Cl, mg kg <sup>-1</sup>	1.64	0.24	0.4	0.06	0.14	-0.02	1.44	0.24	1.45	0.24	0.61	0.09	1.31	0.15
SO <sub>4</sub> , mg kg <sup>-1</sup>	0.71	-0.10	0.76	-0.11	0.24	0.04	0.72	-0.12	0.78	-0.13	0.69	-0.1	0.18	-0.02
EC, dSm <sup>-1</sup>	0.74	0.11	0.85	-0.12	0.20	-0.02	0.74	0.12	0.63	0.10	0.02	-0.003	0.63	0.07
PLS factors§	1		1		1		1		1		1		1	
Overall model PV¶	59		51		74		76		81		75		41	
R <sup>2</sup> (VIP > 0.8) #	0.62		0.29		0.71		0.69		0.80		0.66		0.32	
R <sup>2</sup> (VIP > 1)††	0.68		0.22		0.66		0.39		0.80		0.66		0.32	
R <sup>2</sup> (Four parameters)‡‡	0.19		0.22		0.66		0.29		0.47		0.66		0.22	

† EC, electrical conductivity; STP, soil-test phosphorus; NO<sub>3</sub>-N, soil nitrate-nitrogen; TN, total nitrogen; TP, total phosphorus; TOC, total organic carbon; SO<sub>4</sub>, soluble sulphate; Cl, soluble chloride; Na, soluble sodium.

‡ 1974, 1975, 1976, 1977, 1978, 1979, 1980, 1981, 1984, 1985, 1988, 1992, 1993, 1994, 1995, 2006, 2007, 2008, 2009 and 2010 represent 1, 2, 3, 4, 5, 6, 7, 8, 11, 12, 15, 19, 20, 21, 22, 33, 34, 35, 36 and 37 yr after the start of the experiment; cumulative, predicting cumulative by summing barley grain yield from 1974 to 2010.

§ Number of partial least square (PLS) factors determined by cross-validation.

¶ Percentage of variation explained by the PLS factor by cross-validation step.

# Predictive strength assessed by linear regression of measured values of response variables versus predicted values obtained in the cross-validation step using predictors with VIP > 0.8.

†† Predictive strength assessed by linear regression of measured values of response variables versus predicted values obtained in the cross-validation step using predictors with VIP > 1 (preferred model).

‡‡ Predictive strength assessed by linear regression of measured values of response variables versus predicted values obtained in the cross-validation step with TOC, TP, TN, and NO<sub>3</sub>-N as predictors.

## 5.5. Conclusions

Partial least squares models using soil variable measured at the end of a 30-yr annual manure application period adequately predicted barley grain yield for up to 7 yr following discontinuation of manure application. Similarly, annual grain yield in control plots that did not receive any manure application was adequately predicted, for up to 37 yr since the start of the experiment, by PLS models utilizing soil properties measured in 1973. In both cases, the four soil variables, TOC, TP, TN, and  $\text{NO}_3\text{-N}$  concentrations were the most influential and most consistent in explaining the variability in yield. The predictive power of the models was very good, ranging from 41 to 88%. Overall, a one-factor model with variables having  $\text{VIP} > 1$  was selected as the most suitable for predicting grain yield in both the manured and unmanured plots in all years. Our results showed no evidence of convergence in grain yield among the discontinued manure treatments 7 yr after discontinuation of manure application, which reflects the high soil nutrient concentrations and the adverse and persistent EC effects. This indicates that there is no need to supplement soil nutrients through the addition of synthetic fertilizers or manure within this period since the nutrient concentrations are still above the agronomic thresholds for optimum yield production. There is a need for long term measurements of barley yield to determine when barley yield convergence across treatments will occur and when nutrient addition should begin. Yields were greater for the 120 and  $180 \text{ Mg ha}^{-1} \text{ yr}^{-1}$  manure rates than the control in 2010, unlike in the earlier years following discontinuation of manure application, when yields were lower in the manured plots. Because the STP and  $\text{NO}_3\text{-N}$  levels were still greater than the maximum agronomic limits of 60 and  $34 \text{ mg kg}^{-1}$ , respectively, even 7 yr after manure application was discontinued, these plots still need to be managed with care to ensure that excess nutrients are not lost to the environment.

## 5.6. References

- Alberta Agriculture. 1980.** Land application of animal manure. Agdex 538-1. Alberta Agriculture, Edmonton, AB, Canada.
- Alberta Agriculture and Forestry. 2015a.** Barley production in Alberta: Plant development. [Online]. Available: [http://www1.agric.gov.ab.ca/\\$department/deptdocs.nsf/all/crop4933](http://www1.agric.gov.ab.ca/$department/deptdocs.nsf/all/crop4933) (verified 5 June 2017).
- Alberta Agriculture and Forestry. 2015b. Barley production in Alberta: Harvesting. [Online]. Available: [http://www1.agric.gov.ab.ca/\\$department/deptdocs.nsf/all/crop1256](http://www1.agric.gov.ab.ca/$department/deptdocs.nsf/all/crop1256) (verified 26 November 2017).
- Amundson, R. G., Trask, J. and Pendall, E. 1988.** A rapid method of soil carbonate analysis using gas chromatography. *Soil Sci. Soc. Am. J.* **52**: 880-883.
- Anthony, P., Malzer, G., Sparrow, S. and Zhang, M. 2012.** Soybean yield and quality in relation to soil properties. *Agron. J.* **104**: 1443-1458.
- Baier, W. and Robertson, G. W. 1965.** Estimation of latent evaporation from simple weather observations. *Can. J. Plant Sci.* **45**: 276-284.
- Benke, M. B., Indraratne, S. P. and Hao, X. 2013.** Long-term manure applications impact on irrigated barley forage mineral concentrations. *Agron. J.* **105**: 1441-1450.
- Chang, C., Ellert, B., Hao, X. and Clayton, G. 2007.** Elevation-based soil sampling to assess temporal changes in soil constituents. *Soil Sci. Soc. Am. J.* **71**: 424-429.
- Chang, C., Sommerfeldt, T. G. and Entz, T. 1993.** Barley performance under heavy applications of cattle feedlot manure. *Agron. J.* **85**: 1013–1018.
- Cox, M. S., Gerard, P. D. and Abshire, M. J. 2006.** Selected soil properties' variability and their relationships with yield in three Mississippi fields. *Soil Sci.* **171**: 541-551.
- Eghball, B. 2000.** Nitrogen mineralization from field-applied beef cattle feedlot manure or compost. *Soil Sci. Soc. Am. J.* **64**: 2024-2030.

**Eghball, B., Shanahan, J. F., Varvel, G. E. and Gilley, J. E. 2003.** Reduction of high soil test phosphorus by corn and soybean varieties. *Agron. J.* **95**: 1233-1239.

**Environment Canada. 2016.** Canadian climate normals or averages 1981-2010. [Online]. Available:

[http://climate.weather.gc.ca/climate\\_normals/results\\_1981\\_2010\\_e.html?searchType=stnProv&lstProvince=AB&txtCentralLatMin=0&txtCentralLatSec=0&txtCentralLongMin=0&txtCentralLongSec=0&stnID=2263&dispBack=0](http://climate.weather.gc.ca/climate_normals/results_1981_2010_e.html?searchType=stnProv&lstProvince=AB&txtCentralLatMin=0&txtCentralLatSec=0&txtCentralLongMin=0&txtCentralLongSec=0&stnID=2263&dispBack=0) (verified 15 December 2016).

**Ferguson, R. B., Nienaber, J. A., Eigenberg, R. A. and Woodbury, B. L. 2005.** Long-term effects of sustained beef feedlot manure application on soil nutrients, corn silage yield, and nutrient uptake. *J. Environ. Qual.* **34**:1672-1681.

**Hao, X., Godlinski, F. and C. Chang. 2008.** Distribution of phosphorus forms in soil following long-term continuous and discontinuous cattle manure applications. *Soil Sci. Soc. Am. J.* **70**: 90-97.

**Hao, X. and Chang, C. 2003.** Does long-term heavy cattle manure application increase salinity of a clay loam soil in semi-arid southern Alberta? *Agri. Ecosyst. Environ.* **94**: 89-103.

**Howard, A. E. 2006.** Agronomic thresholds for soil phosphorus in Alberta: a review. In: Alberta soil phosphorus limits projects. Vol. 5: Background information and reviews. Alberta Agriculture, Food and Rural Development, Lethbridge, Alberta, Canada. [Online]. Available:

[http://www1.agric.gov.ab.ca/\\$department/deptdocs.nsf/all/sag11864/\\$FILE/vol-5-agronomic-thresholds.pdf](http://www1.agric.gov.ab.ca/$department/deptdocs.nsf/all/sag11864/$FILE/vol-5-agronomic-thresholds.pdf) (Verified 20 November. 2017).

**Indraratne, S. P., Hao, X., Chang, C. and Godlinski, F. 2009.** Rate of soil recovery following termination of long-term cattle manure applications. *Geoderma* **150**: 415-423.

**Iqbal, J., Read, J. J., Thomasson, A. J. and Jenkins, J. N. 2005.** Relationships between soil-landscape and dryland cotton lint yield. *Soil Sci. Soc. Am. J.* **69**: 872-882.

**Janzen, H. H. 1993.** Soluble salts. In: M.R. Carter editor, *Soil sampling and methods of analysis*. Lewis Publ., Boca Raton, FL. p. 161-166.

- Kashem, Md. A., Akinremi, O. O. and Racz, G. J. 2004.** Extractable phosphorus in alkaline soils amended with high rates of organic and inorganic phosphorus. *Can. J. Soil Sci.* **84**: 459-467.
- Littell, R. C., Milliken, G. A., Stroup, W. W., Wolfinger, R. D. and Schabenberger, O. 2006.** SAS system for mixed models. SAS Institute. Inc., Cary, NC.
- Majchrzak, R. N., Olson, K. R., Bollero, G. and Nafziger, E. D. 2001.** Using soil properties to predict wheat yields on Illinois soils. *Soil Sci.* **166**: 267-280.
- McAndrews, G. M., Liebman, M., Cambardella, C. A. and Richard, T. L. 2006.** Residual effects of composted and fresh solid swine (*Sus scrofa* L.) manure on soybean [*Glycine max* (L.) Merr.] growth and yield. *Agron. J.* **98**: 873-882.
- McDonald, G. K. 2006.** Effects of soil properties on variation in growth, grain yield and nutrient concentration of wheat and barley. *Aust. J. Exp. Agric.* **46**: 93-105.
- Miller, J. J., Sweetland, N. J. and Chang, C. 2002.** Soil physical properties of a Chernozemic clay loam after 24 years of beef cattle manure application. *Can. J. Soil Sci.* **82**: 287-296.
- Olsen, S. R., Cole, C. V., Watanabe, F. S. and Dean, L. A. 1954.** Estimation of available phosphorus in soils by extraction with sodium bicarbonate. Circular 939. USDA, Washington, DC.
- Olson, B. M., McKenzie, R. H., Larney, F. J. and Bremer, E. 2010b.** Nitrogen- and phosphorus-based applications of cattle manure and compost for irrigated cereal silage. *Can. J. Soil Sci.* **90**: 619-635.
- Olson, B. M., Bennett, D. R., McKenzie, R. H., Ormann, T. D. and Atkins, R. P. 2009.** Nitrate leaching in two irrigated soils with different rates of cattle manure. *J. Environ. Qual.* **38**: 2218-2228.
- Parkinson, J. A. and Allen, S. E. 1975.** A wet oxidation procedure suitable for the determination of nitrogen and mineral nutrients in biological material. *Commun. Soil Sci. Plant Anal.* **6**:1-11.
- Pennock, D., Bedard-Haughn, A. and Viaud, V. 2011.** Chernozemic soils of Canada: Genesis, distribution, and classification. *Can. J. Soil Sci.* **91**: 719-747.
- Ping, J. L., Green, C. J., Bronson, K. F., Zartman, R. E. and Dobermann, A. 2004.** Identification of relationships between cotton yield, quality, and soil properties. *Agron. J.* **96**: 1588-1597.

**Pizzeghello, D., Berti, A., Nardi, S. and Morari, F. 2016.** Relationship between soil test phosphorus and phosphorus release to solution in three soils after long-term mineral and manure application. *Agri. Ecosyst. Environ.* **233**: 214-223.

**Province of Alberta. 2017.** Agricultural operation practices acts: Standards and administration regulation. Alberta regulations 267/2001. Queen's Printer, Edmonton, AB. [Online]. Available:

[http://www.qp.alberta.ca/documents/Regs/2001\\_267.pdf](http://www.qp.alberta.ca/documents/Regs/2001_267.pdf) (accessed 20 Nov. 2017).

**Redulla, C. A., Davenport, J. R., Evans, R. G., Hattendorf, M. J., Alva, A. K. and Boydston, R. A. 2002.** Relating potato yield and quality to field scale variability in soil characteristics. *Amer. J. of Potato Res.* **79**: 317-323.

**SAS Institute, 2014.** SAS/STAT 13.2 User's guide. Version 9.4. Cary (NC): SAS Institute, Inc.

**Schoenau, J. J. and Davis, J. G. 2006.** Optimizing soil and plant responses to land-applied manure nutrients in the Great Plains of North America. *Can. J. Soil Sci.* **86**: 587-595.

**Sheppard, S. C. and Racz, G. J. 1984a.** Effects of soil temperature on phosphorus extractability. I. Extractions and plant uptake of soil and fertilizer phosphorus. *Can. J. Soil Sci.* **64**: 241-254.

**Sheppard, S. C. and Racz, G. J. 1984b.** Effects of soil temperature on phosphorus extractability. II. Soil phosphorus in six carbonated and six non-carbonated soils. *Can. J. Soil Sci.* **64**: 255-263.

**Siebers, N., Sumann, M., Kaiser, K. and Amelung, W. 2017.** Climatic effects on phosphorus fractions of native and cultivated north American grassland soils. *Soil Sci. Soc. Am. J.* **81**: 299-309.

**Sieling, K., Brase, T. and Svib, V. 2006.** Residual effects of different N fertilizer treatments on growth, N uptake and yield of oilseed rape, wheat and barley. *Europ. J. Agronomy* **25**: 40-48.

**Sommerfeldt, T. G. and Chang, C. 1985.** Changes in soil properties under annual applications of feedlot manure and different tillage practices. *Soil Sci. Soc. Am. J.* **49**: 983-987.

**Spratt, E. D., Warder, F. G., Bailey, L. D. and Read, D. W. L. 1980.** Measurement of fertilizer phosphorus residues and its utilization. *Soil Sci. Soc. Am. J.* **44**: 1200-1204.



**Stumborg, C. and Schoenau, J. J. 2008.** Evaluating phosphorus loading from repeated manure applications to two Saskatchewan soils. *Can. J. Soil Sci.* **88**: 377-387.

**Wold, S. 1995.** PLS for multivariate linear modeling. In: H. Van de Waterbeemd (ed.) *QSAR. Chemometric methods in molecular design: Methods and principles in medicinal chemistry.* Verlag Chemie, Weinheim, Germany. p. 195-218.

**Zar, J. H. 1999.** *Biostatistical analysis.* 3 rd ed. Prentice Hall, Upper Saddle River, NJ.

## **6. OVERALL SYNTHESIS**

### **6.1. Relevant Findings and Implications of the Research**

The beef cattle feedlot industry in North America has seen an increase in the use of dried distillers grains with solubles (DDGS) as animal feed (Li et al., 2011; FOBI Network, 2013). This is partly due to the increased supply of DDGS with the rapid expansion of the biofuel industry in the United States and Canada (Liu, 2011; FOBI Network, 2013). Studies have shown that inclusion of DDGS in cattle diet alters manure properties by increasing the excretion of nutrients, especially nitrogen (N) and phosphorus (P), relative to manure from animals fed a regular grain diet (Hao et al., 2009; McGinn et al., 2009; Hünérberg et al., 2013a). Greater N and P uptake, soil total N, total P, and soil test P concentrations have been reported in soil following application of manure from cattle fed DDGS compared with manure from cattle fed a barley grain diet (Benke et al., 2010).

Manure properties are affected not only by the type of diet fed to the animal but also by the bedding material used in confined feeding operations (Miller et al., 2003, 2010). The increasing use of construction and demolition waste (CDW), especially the wood and drywall fractions, as bedding material in beef cattle feedlots in the Canadian Prairies may further reduce N availability in manure from animals receiving DDGS in their diet. This is because the stable C in the woodchip fraction can alter nutrient release dynamics when the manure is applied to soil (Larney et al., 2008; Miller et al., 2010; Hao et al., 2014).

Information on the agronomic effectiveness and environmental risk associated with repeated land application of manure from cattle fed either DDGS or regular grain diet, and the effects of mixing the manure with peat or CDW, were reported in Chapter 3 of this thesis. The addition of CDW to DGM and RM manure lowered canola cumulative dry matter yield (CDMY) and N uptake

relative to manure without CDW, while CDMY and N uptake increased in the presence of peat. The lower dry matter yield and N uptake resulting from reduced organic N mineralization indicate that CDW-amended DGM and RM manure may have to be supplemented with synthetic N fertilizer to meet crop N requirements. In both the N mineralization (Chapter 2) and the bioassay (Chapter 3) experiments, the presence of CDW in manure resulted in net immobilization of N compared with manure without CDW, suggesting that CDW affects N dynamics by lowering the amount of N that is mineralized. In the bioassay, all organic amendment treatments resulted in lower net N mineralization in the Brown Chernozem compared to the Black Chernozem. This is consistent with results from the N mineralization study (Chapter 2) in which negative mineralization occurred in the Brown Chernozem with the same amendments. This further highlights the relevance of the combined effect of soil and amendment properties on soil N mineralization-immobilization processes, and hence the eventual release of N for crop uptake.

Nitrogen mineralization kinetics for the Black Chernozem (Chapter 2) was best described by the mixed first-order and zero-order kinetic model. Our results showed that potentially mineralizable N ( $N_o$ ) was the only temperature-dependent model parameter. Averaged across organic amendment treatments,  $N_o$  was greater at 25°C than at 15°C. This indicates that a greater amount of N is mineralized as the temperature of incubation is increased possibly due to enhanced microbial activity. Averaged across incubation temperatures,  $N_o$  was lower for DGM than RM. The lower  $N_o$  for DGM in the Black Chernozem possibly reflects the rapid mineralization of the easily decomposable organic N pool (i.e., larger  $k_l$ ) and the slower mineralization of the large resistant N pool (i.e., lower  $k_o$ ) compared to RM. It is possible that a greater fraction of N is made available in soil when the large resistant N pool gets mineralized compared to the N released from the easily decomposable organic N pool. This is consistent with the results from the bioassay study (Chapter

3), in which the cumulative organic N mineralized in the Black Chernozem was greater for RM than DGM.

Although, manure containing CDW reduced biomass yield, N uptake and organic N mineralization relative to manure without CDW, our results showed reduced soil inorganic N concentration in both soils with DGM<sub>CDW</sub> and RM<sub>CDW</sub> compared to DGM and RM applications, respectively (Chapter 3). The result is the excessive buildup of nitrate, which is susceptible to loss to the environment through leaching, as seen with the greater PLN and CPLN with DGM and RM compared to DGM<sub>CDW</sub> and RM<sub>CDW</sub>, respectively. Our results further showed that nitrapyrin application had no significant effect on nitrate leaching. In general, the results showed that both RM and DGM resulted in greater residual N concentrations at the end of the five growth cycles compared to the initial soil inorganic N concentration prior to the experiment, with the increase greater with DDGS manure (Chapter 3).

Agricultural fields in the vicinity of concentrated animal operations (e.g., cattle feedlots in southern Alberta) have a long history of livestock manure application, which results in elevated levels of N and P, which are susceptible to loss to the environment (Pant et al., 2004; Larney et al., 2006). On such fields, further fertilization with synthetic fertilizer or manure is undesirable. In many cases, the fields are repeatedly cropped to forage to reduce soil nutrient concentrations, a technique known as phytoextraction (Pederson et al., 2002; Pant et al., 2004; Read et al., 2007). Chapter 4 of this thesis provides information on the phytoextraction of N and P from a heavily manured Dark Brown Chernozem by various agronomic crops [four cereal crops (barley, corn, oat and triticale), canola and legumes (pea and soybean)] grown under two moisture regimes (100% and 50% SFC).

The results showed that dry matter yield, N and P uptake were on average lower at 50% SFC than at 100% SFC, reflecting the reduced biomass yields at the lower moisture level. However, soil residual N concentration was lowest under 100% SFC an indication of greater N uptake by plants under optimum moisture conditions through increased biomass yields. The implication is that phytoextraction of N in a heavily manured soil is maximized when the soil moisture is optimum, resulting in increased soil organic matter decomposition and N mineralization through enhanced soil microbial activity (Sierra, 1997; Leirós et al., 1999; Agehara and Warncke, 2005). Our results also showed that soil available P and percent change in dry matter ( $\Delta\text{DMY}_1$ ), nitrogen ( $\Delta\text{NU}_1$ ) and phosphorus ( $\Delta\text{PU}_1$ ) relative to Cycle 1 were all unaffected by the moisture regimes.

Results from the study indicate that P phytoextraction rates after six growth cycles for most crops except pea were greater than those for N. This suggests that a greater fraction of P that was initially present in the heavily manured soil was removed with repeated harvesting. This is important since the common practice is to apply manure to meet crop N requirements, which usually results in accumulation of excess P in soil, which is a major environmental concern (Sharpley et al., 2001; Eghball et al., 2003).

Results showed that repeated harvesting of canola and corn resulted in the lowest soil residual N and available P concentrations compared to the other grain crops and legumes, which were consistent with the greater CNU and CPU of both canola and corn. This indicates that under the conditions tested in this study, canola and corn are the appropriate crop choices for phytoextraction of N and P in a heavily manured soil. Although N and P phytoextraction rates after six growth cycles were greatest for corn than canola, however, we recommend that canola would be the ideal crop to use since our results showed that it maintained a consistent uptake of N and P rates with repeated

cropping compared to corn that initially outyielded all the other crops, with yields increasing little with repeated harvests, resulting in lower nutrient uptake at the later growth cycles.

Apart from seasonal and yearly variability in weather, landscape characteristics of a field, management practices adopted on the farm and the occurrence of crop disease, crop biomass production and the eventual nutrient uptake on any particular agricultural field are highly influenced by soil properties. The soil's ability to supply nutrients required for optimum growth and development by crops is critical in sustaining productivity even after application of nutrient sources such as synthetic fertilizer and livestock manure is discontinued. Chapter 5 of this thesis provides information on the prediction, using PLS modeling, of barley grain yield in years following discontinuation of long term manure application in a Dark Brown Chernozem using soil properties measured at the cessation of manure application. Our results (Chapter 5) showed that a one-latent variable PLS model with variables having  $VIP > 1$  explained between 29-99% of the variability in grain yield in the manured plots and between 22-99% in the unmanured plots. Results from the study showed that the four soil variables, TOC, TP, TN, and  $NO_3$ -N concentration, were the most influential in explaining the variability in yield. The consideration of TN and  $NO_3$ -N as parts of key critical factors goes further to confirms the generally accepted fact that the N demand by most arable crops is generally greater compared to crop P requirement including other essential soil nutrients (Malhi et al., 2006, 2007; Meisinger et al., 2008). The relatively high and constant predictive power of the model during the years following discontinuation of manure application suggest that under the same condition as in this study, yearly soil sampling and measurement may not be necessary, especially in a field where manure application was discontinued since the initial soil properties were able to explain the variability in the barley grain yield years after the last manure application. Hence,

analysis of soil samples collected from such a field should be limited to just TOC, TP, TN and  $\text{NO}_3\text{-N}$  for routine soil testing if the need arises.

## **6.2. Recommendations**

Our study provides information on crop N uptake and N dynamics (Chapter 2 and 3) in Chernozems as influenced by CDW and peat in manure from beef cattle fed either a regular grain diet or DDGS. This is of great importance as there is growing interest in the use of CDW as bedding in cattle feedlots in the Canadian Prairies, along the increasing inclusion of DDGS in animal diets. Findings from this study will help beef producers and policy makers to make informed decisions on manure nutrient management, especially with the use of CDW and peat, by ensuring that negative effects to the environment that are associated with manure application are minimized while aiming towards a more sustainable crop production using cattle manure. Hence, future studies should be focused on further improving our understanding of N dynamics in the field using cattle manure containing CDW or peat by using manure in which the bedding material is actually added into the animal pen instead of mixing manure from pens with CDW and peat as done in this study. It is possible that the CDW and peat added with manure used in this study did not have enough time to react as would be the situation in feedlots. In the Canadian prairies, manure in cattle feedlots is usually removed from the pens once a year, usually in September to November, which coincides with the fall application of manure, which is widely practiced (Miller et al., 2003; Sheppard et al., 2011; Sheppard and Bittman, 2012). The extended contact time allows for a greater interaction between the bedding material and the manure. Under such conditions, manure N dynamics may differ due to dilution in the presence of the bedding. Furthermore, manure nutrients, particularly N, will have been susceptible to loss to the environment through various pathways (e.g., volatilization,

denitrification and leaching) while in the feedlot pen and during outdoor storage (e.g. stockpiling) following the cleaning of the pen (Sheppard and Bittman, 2012).

The rates of fresh manure application used in this study were based on the field application rate of 60 Mg ha<sup>-1</sup> recommended in southern Alberta to supply 150 kg N ha<sup>-1</sup> for irrigated cereal production. However, the use of the same manure weight for both DDGS and RM manure in our study resulted in slightly different amounts of N being applied to the soils, which precluded a combined analysis of the data. Manure application to meet crop requirement based on the nutrient content of manure and rate of manure application will ensure the same N rate for all amendments, hence allowing a comparison of manure types.

Results from the N mineralization studies showed that N immobilization was predominant in the Brown Chernozem for all the organic amendments. Under similar controlled conditions as was used in our study, Chiyoka et al. (2014) reported a negative N mineralization for a Dark Brown Chernozem amended with cattle manure. There is the need to further our understanding on soil factors and organic amendment properties that may be responsible for the N immobilization in the Brown Chernozem and to confirm if the same negative mineralization will occur under field conditions.

Our phytoextraction study (Chapter 4) was conducted under controlled conditions and provides relevant information on crops that are more efficient in reducing soil N and P concentrations in a heavily manured soil under varying soil moisture conditions. This research is the first step towards providing information on N and P removal in a heavily manured soil by various agronomic crops. Our understanding of the phytoextraction rates of these crops could be further extended by conducting field research to examine rooting depth effects on nutrient extraction since



root growth of the various crops used in our study may have been restricted by the size of the experimental pots.

The prediction of barley grain yield in the study used only soil chemical properties such as NO<sub>3</sub>-N, pH, TOC, TN (Chapter 5). However, soil physical (e.g., hydraulic conductivity, porosity, aeration status, soil strength, and temperature) and biological (e.g., microbial biomass, organic matter) properties also influence crop growth and yield. Inclusion of all these factor in the models will improve their predictive power.

### **6.3. References**

**Agehara, S. and Warncke, D. D. 2005.** Soil moisture and temperature effects on nitrogen release from organic nitrogen sources. *Soil Sci. Soc. Am. J.* **69**: 1844-1855

**Benke, M. B., Hao, X., Caffyn, P., Schoenau, J. J. and McAllister, T. A. 2010.** Using manure from cattle fed dried distillers' grains with solubles (DDGS) as fertilizer: Effects on nutrient accumulation in soil and uptake by barley. *Agri. Ecosyst. Environ.* **139**: 720-727.

**Chiyoka, W. L., Zvomuya, F. and Hao, X. 2014.** Changes in nitrogen availability in Chernozemic soils amended with anaerobically digested cattle manure. *Soil Sci. Soc. Am. J.* **78**: 843-851.

**Eghball, B., Shanahan, J. F., Varvel, G. E. and Gilley, J. E. 2003.** Reduction of high soil test phosphorus by corn and soybean varieties. *Agron. J.* **95**: 1233-1239.

**FOBI [Feed Opportunities from Biofuels Industries] Network. 2013.** Wheat dried distiller grains with solubles. Wheat DDGS feed guide [Online]. Available:

[https://cigi.ca/wp-content/uploads/2013/02/DDGS-Feed-Guide\\_Revised\\_Jan.-2013.pdf](https://cigi.ca/wp-content/uploads/2013/02/DDGS-Feed-Guide_Revised_Jan.-2013.pdf) (Verified 7 April 2017).

**Hao, X., Hill, B., Caffyn, P., Travis, G., Olson, A. F., Larney, F. J., McAllister, T. A. and Alexander, T. 2014.** Co-composting of beef cattle feedlot manure with construction and demolition waste. *J. Environ. Qual.* **43**: 1799-1808.

**Hao, X., Benke, M. B., Gibb, D. J., Stronks, A., Travis, G. and McAllister, T. A. 2009.** Effects of dried distillers' grains with solubles (wheat-based) in feedlot cattle diets on feces and manure composition. *J. Environ. Qual.* **38**: 1709-1718.

- Hünerberg, M., McGinn, S. M., Beauchemin, K. A., Okine, E. K., Harstad, O. M. and McAllister, T. A. 2013a.** Effect of dried distillers' grains with solubles on enteric methane emissions and nitrogen excretion from finishing beef cattle. *Can. J. Anim. Sci.* **93**: 373-385.
- Larney, F., Olson, A. F., Miller, J. J., DeMaere, P. R., Zvomuya, F. and McAllister, T. A. 2008.** Physical and chemical changes during composting of wood-chip-bedded and straw-bedded beef cattle feedlot manure. *J. Environ. Qual.* **37**: 725-735.
- Larney, F. J., Sullivan, D. M., Buckley, K. E. and Eghball, B. 2006.** The role of composting in recycling manure nutrients. *Can. J. Soil Sci.* **86**: 597-611.
- Leirós, M. C., Trasar-Cepeda, C., Seoane, S. and Gil-Sotres, F. 1999.** Dependence of mineralization of soil organic matter on temperature and moisture. *Soil Biol. Biochem.* **31**: 327-335.
- Li, Y. L., McAllister, T. A., Beauchemin, K. A., He, M. L., McKinnon, J. J. and Yang, W. Z. 2011.** Substitution of wheat dried distillers grains with solubles for barley grain or barley silage in feedlot cattle diets: Intake, digestibility, and ruminal fermentation. *J. Anim. Sci.* **89**: 2491-2501.
- Liu, K. 2011.** Chemical composition of distillers grains, a review. *J. Agric. Food Chem.* **59**: 1508-1526.
- Malhi, S. S., Johnston, A. M., Schoenau, J. J., Wang, Z. H. and Vera, C. L. 2007.** Seasonal biomass accumulation and nutrient uptake of canola, mustard, and flax on a Black Chernozem soil in Saskatchewan. *J. Plant Nutr.* **30**: 641-658.
- Malhi, S. S., Johnston, A. M., Schoenau, J. J., Wang, Z. H. and Vera, C. L. 2006.** Seasonal biomass accumulation and nutrient uptake of wheat, barley and oat on a Black Chernozem soil in Saskatchewan. *Can. J. Plant Sci.* **86**: 1005-1014.
- McGinn, S. M., Chung, Y. H., Beauchemin, K. A., Iwaasa, A. D. and Grainger, C. 2009.** Use of corn distillers' dried grains to reduce enteric methane loss from beef cattle. *Can. J. Anim. Sci.* **89**: 409-413.
- Meisinger, J. J., Schepers, J. S. and Raun, W. R. 2008.** Crop nitrogen requirement and fertilization. In: *Nitrogen in Agricultural Systems*. J. S. Schepers and W. R. Raun (ed), ASA-CSSA-SSSA., Madison, WI. Chapter 14.
- Miller, J. J., Beasley, B. W., Drury, C. F. and Zebarth, B. J. 2010.** Available nitrogen and phosphorus in soil amended with fresh or composted cattle manure containing straw or wood-chip bedding. *Can. J. Soil Sci.* **90**: 341-354.
- Miller, J. J., Beasley, B. W., Yanke, L. J., Larney, F. J., McAllister, T. A., Olson, B. M., Selinger, L. B., Chanasyk, D. S. and Hasselback, P. 2003.** Bedding and seasonal effects on chemical and bacterial properties of feedlot cattle manure. *J. Environ. Qual.* **32**: 1887-1894.

**Pant, H. K., Adjei, M. B., Scholberg, J. M. S., Chambliss, C. G. and Rechcigl, J. E. 2004.** Forage production and phosphorus phytoremediation in manure-impacted soils. *Agron. J.* **96**: 1780-1786.

**Pederson, G. A., Brink, G. E. and Fairbrother, T. E. 2002.** Nutrient uptake in plant parts of sixteen forages fertilized with poultry litter: Nitrogen, phosphorous, potassium, copper, and zinc. *Agron. J.* **94**: 895-904.

**Read, J. J., Sistani, K. R., Brink, G. E. and Oldham, J. L. 2007.** Reduction of high soil test phosphorous by Bermudagrass and Ryegrass-Bermudagrass following the cessation of broiler litter application. *Agron. J.* **99**: 1492-1501.

**Sharpley, A. N., Kleinman, P. and McDowell, R. 2001.** Innovative management of agricultural phosphorus to protect soil and water resources. *Commun. Soil Sci. Plant Anal.* **32**: 1071-1100.

**Sheppard, S. C. and Bittman, S. 2012.** Farm practices as they affect NH<sub>3</sub> emissions from beef cattle. *Can. J. Anim. Sci.* **92**: 525-543.

**Sheppard, S. C., Bittman, S., Donohoe, G., Flaten, D., Wittenberg, K. M., Small, J. A., Berthiaume, R., McAllister, T. A., Beauchemin, K. A., McKinnon, J., Amiro, B. D., MacDonald, D., Mattos, F. and Ominski, K. H. 2011.** Beef cattle husbandry practices across ecoregions of Canada in 2011. *Can. J. Anim. Sci.* **95**: 305-321.

**Sierra, J. 1997.** Temperature and soil moisture dependence of N mineralization in intact soil cores. *Soil Biol. Biochem.* **29**: 1557-1563.

## APPENDICES

### Supplementary Information: Chapter 5

**Table S1. Cumulative precipitation and average air temperature before soil sampling during the eight years (2003-2010) following discontinuation of 30 yr of manure application in a Dark Brown Chernozem at the AAFC Research and Development Centre in Lethbridge, Alberta, Canada.**

Year	Cumulative Precip.	Average temp.
	Aug - Oct.	Aug - Oct.
	mm	°C
2003	40.4	14.3
2004	97.6	12.2
2005	243	12.5
2006	68.7	12.9
2007	70.7	13
2008	98.3	13.1
2009	134.3	12.8
2010	108.1	12.8

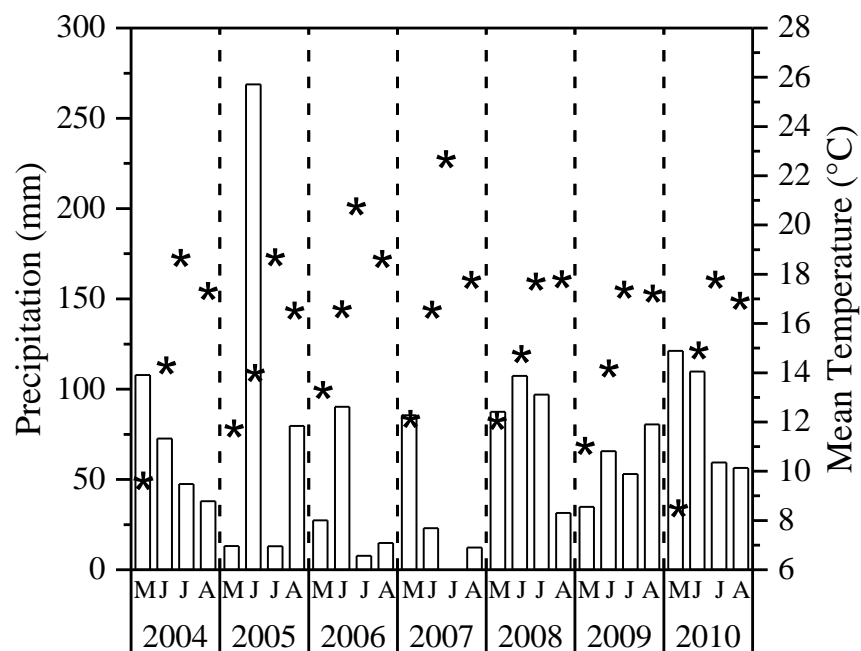


Figure 5S1. Growing season monthly precipitation (white bars) and mean air temperatures (black asterisk) during the seven years (2004-2010) following discontinuation of 30 yr of manure application in a Dark Brown Chernozem at the AAFC Research and Development Centre in Lethbridge, Alberta, Canada.

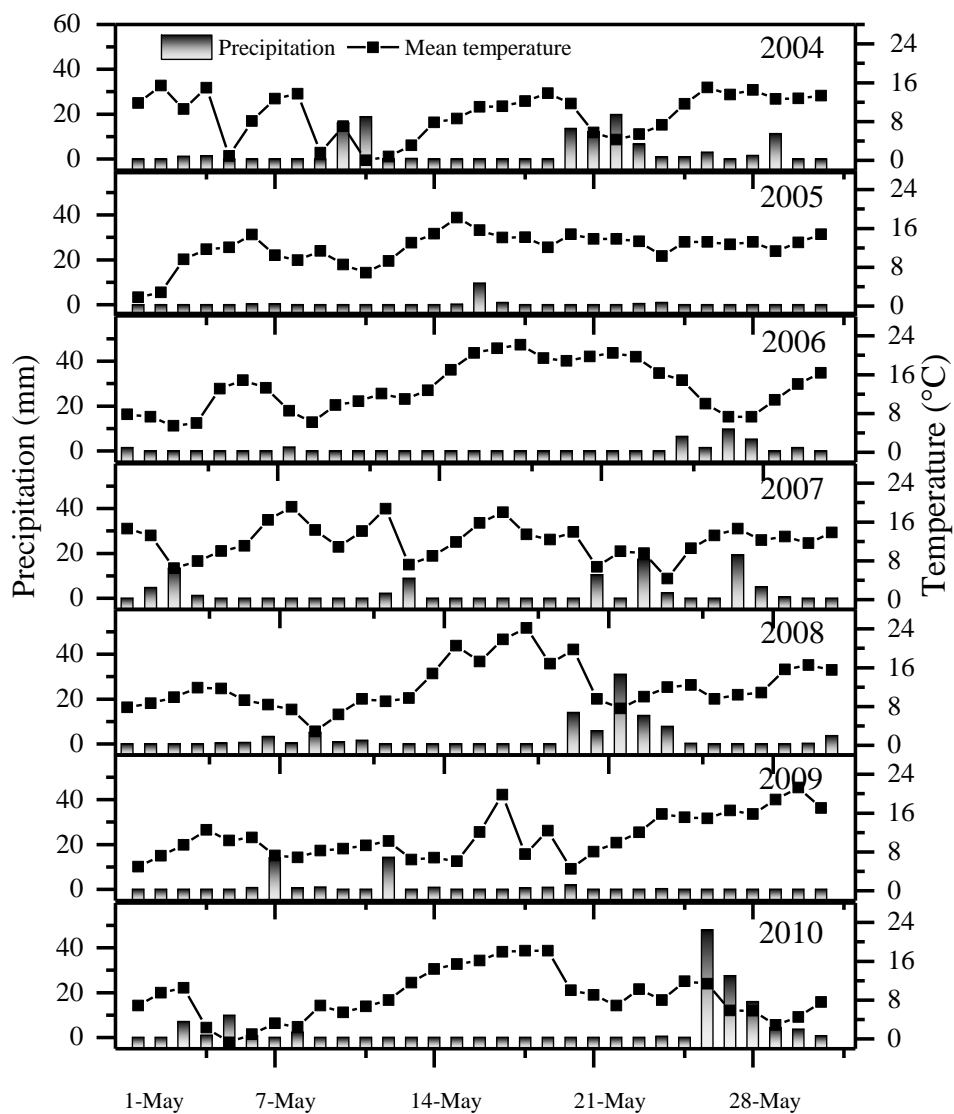


Figure 5S2 Daily precipitation and mean air temperatures in May during the seven years (2004-2010) following discontinuation of 30 yr of manure application in a Dark Brown Chernozem at the AAFC Research and Development Centre in Lethbridge, Alberta, Canada.

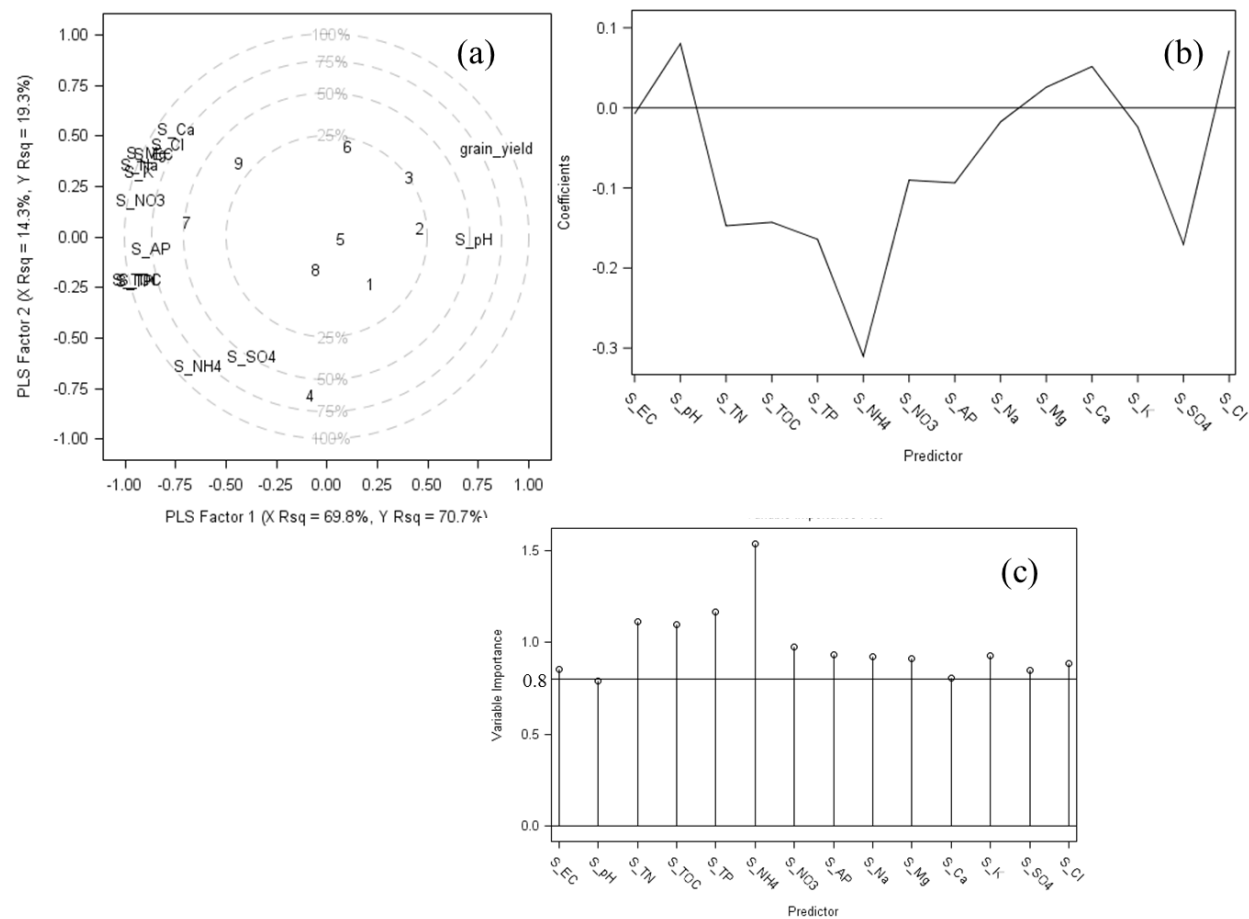


Figure 5S3 Correlation loading plot (a), profiles of centered and scaled parameters (b), and variable importance of the projection plots (c) of the PLS model for predicting barley grain yield 4 yr following discontinuation of 30 yr of manure application.