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PREDICTING THE EFFECT OF WETLAND RESTORATION ON
PHOSPHORUS RETENTION, NORTHWEST MINNESOTA

by

Ryan J. Whittaker
Bachelor of Arts, University of Minnesota – Morris, 2006

A Thesis

Submitted to the Graduate Faculty

of the

University of North Dakota

in partial fulfillment of the requirements

for the degree of

Master of Science

Grand Forks, North Dakota
December
2009

This thesis, submitted by Ryan J. Whittaker in partial fulfillment of the requirements for the Degree of Master of Science from the University of North Dakota, has been read by the Faculty Advisory Committee under whom the work has been done and is hereby approved.

Chairperson

This thesis meets the standards for appearance, conforms to the style and format requirements of the Graduate School of the University of North Dakota, and is hereby approved.

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ABSTRACT

Water quality in the Red River Valley and downstream in Lake Winnipeg has been diminishing due to excess nutrients from agriculture. One method to reduce nutrient loading to surface water is to create or restore wetlands in agricultural areas that drain cropped fields. It is commonly believed that wetlands improve water quality, but research has shown that restoring wetlands can actually release phosphorus (P) into solution. Reducing conditions caused by soil flooding can release Fe-bound P, since ferrous Fe is more soluble than ferric Fe. Little is known concerning how the soils in the Red River Valley will be affected by wetland restoration. This research at the Judicial Ditch 66 watershed in northwestern Minnesota measured soil properties to predict the effect wetland restoration will have on P retention. Surface water and groundwater samples were collected to determine P mobility and indicated very little soluble P is transported either within or out of the watershed. Soil analyses indicated that the site contains mineral soils that are Ca-rich. A phosphorus sorption index (PSI) was used to determine the degree of P soil saturation. The mean PSI was 24.7, indicating that the soils at the site have a moderately high degree of P saturation. Spearman Rank correlation coefficients show soil organic matter and exchangeable calcium correlate highest with PSI. A three-part sequential extraction was performed to determine the dominant form of soil P. Organic P comprised nearly 80% of soil P with the remaining being nearly all Ca-bound P. Throughout the study area P is associated with compounds

that are not redox sensitive; therefore, it is predicted that wetland restoration will not release significant P into solution. In addition, wetlands store P by organic matter accumulation and the settling of particulate P. Wetland restoration at this site and at similar sites can be a long-term solution to improving water quality throughout the Red River Valley and in Lake Winnipeg.

CHAPTER I

INTRODUCTION

In many freshwater ecosystems phosphorus (P) is the limiting nutrient (Reddy et al., 1999). Agricultural inputs of nutrients to surface waters increase the risk of eutrophication, which is a significant water quality problem (Sharpley and Menzel, 1987). The P cycle, unlike the nitrogen cycle, does not contain a significant gaseous phase; therefore, excess P is not easily removed from a system (Mitsch and Gosselink, 2000; Richardson and Vepraskas, 2001). Excess nutrients are responsible for toxic algal blooms, decreased oxygen levels, fish kills, loss of biodiversity, and other problems (Carpenter et al., 1998). Unless current agricultural practices are changed, non-point source nutrient pollution of surface waters will increase (Carpenter et al., 1998).

Nutrient inputs to Lake Winnipeg have increased in the last three decades, and as a result, the lake is considered the most eutrophic of the world's 10 largest lakes (Barlow, 2006). This decade, large algal blooms have occurred throughout the lake. Research has shown that the Red River contributes 54% of the total P that enters the lake, but only 11% of the water entering the lake (Barlow, 2006). The Red River Valley has fertile, nutrient-rich soils with intense agricultural practices that are responsible for the high P contributions to Lake Winnipeg (Barlow, 2006). A goal of 10% reduction in P loading to Lake Winnipeg has been set to improve water quality (Barlow, 2006).

A common method to reduce P loss from small arable watersheds is to restore or create wetlands (Braskerud et al., 2005). Most P lost from agricultural lands is particulate P that, once settled out of the water column, is unlikely to be transported, except under high flow velocities (Sharpley and Menzel, 1987; Reddy et al., 1999). Wetlands increase the surface water residence time within a watershed, decreasing surface water velocities and allowing smaller particles to settle and be stored. Therefore, wetlands act like nutrient and sediment sinks. However, when soils are flooded, the redox potential decreases, conditions become more anaerobic, and P is released into solution (Richardson and Vepraskas, 2001). This P release is associated with the reduction of ferric iron (Fe^{3+}) to ferrous iron (Fe^{2+}), thereby releasing reductant-soluble P (Mitsch and Gosselink, 2000). Soil flooding can also lead to P release by the hydrolysis of ferric and Al phosphates and the release of P sorbed to clays and hydrous oxides by anion exchange (Mitsch and Gosselink, 2000). In addition, adsorbed P is held more tightly under oxidized conditions (Reddy et al., 1999). Consequently, soil flooding and subsequent anaerobic conditions can cause a significant release of P to solution.

Wetland restoration in the Judicial Ditch 66 (JD66) watershed of northwestern Minnesota (Figure 1) will alter soil and water chemistry, potentially affecting P retention. Any change in P retention will have a local effect, but could also affect water quality downstream, and in Lake Winnipeg. The objectives of this research are to:

- 1) determine P transport within the JD66 watershed in surface and groundwater;
- 2) determine which soil properties relate to P sorption;
- 3) determine the amounts of different forms of soil P; and, ultimately,

4) predict changes in P retention after wetland restoration.

The JD66 watershed is underlain by soils developed on carbonate-rich glacial till (Harris et al., 1974). Therefore, it is hypothesized that most of the P is stored within the watershed due to high levels of carbonates and clays under predominately oxidizing soil conditions. Soil P is thought to be mainly Ca-bound P and organic P, which have low bioavailability. Therefore, it is predicted that wetland restoration will release minimal amounts of P to solution.

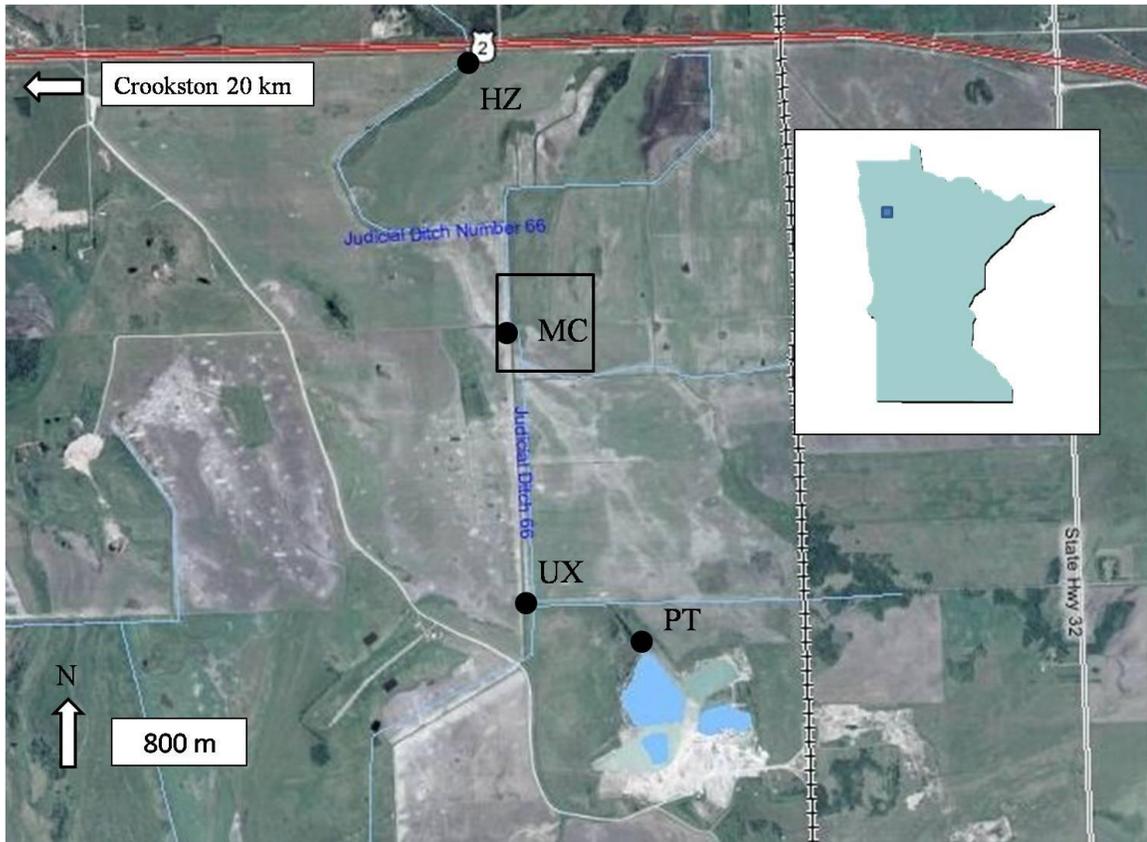


Figure 1. Location of JD66 watershed in northwestern Minnesota (inset). JD66 begins at the outlet of the pond within the gravel pit and flows north. The four surface water sampling sites are shown (PT, UX, MC, and HZ). The boxed region shows the location of the well field (see Figure 2).

CHAPTER II

BACKGROUND INFORMATION

Phosphorus Chemistry

In natural systems P exists as both soluble and insoluble complexes in organic and inorganic forms (Mitsch and Gosselink, 2000). Organic P is associated with living organisms and consists of easily decomposable P compounds (nucleic acids, phospholipids, and sugar phosphates) and slowly decomposable P compounds (inositol phosphates-phytin) (Reddy et al., 1999). The principal form of inorganic P is orthophosphate which exists as the anion H_2PO_4^- , HPO_4^{2-} , or PO_4^{3-} ; at pHs of 2 to 7, 8 to 12, and >13, respectively (Richardson and Vepraskas, 2001; Mitsch and Gosselink, 2000). Soluble inorganic P is considered bioavailable, while transformations must occur to organic, fixed mineral, and particulate P forms to make them bioavailable (Reddy et al., 1999; Mitsch and Gosselink, 2000).

Phosphorus transformations within a system effect P availability. Bioavailable P forms can be transformed into nonavailable forms and vice versa (Reddy et al., 1999; Richardson and Vepraskas, 2001). Processes that make P unavailable to plants and microorganisms include the following (Mitsch and Gosselink, 2000):

- 1) The adsorption of phosphate onto clay particles, organic peat, and ferric Fe and Al hydroxides and oxides,

- 2) The precipitation of insoluble phosphates with ferric Fe, Ca, and Al under aerobic conditions,
- 3) The binding of P in organic matter as a result of its incorporation into the living biomass.

Phosphorus is sorbed onto clay particles by chemical bonding of the negatively charged phosphates to the positively charged broken edges of the clay (Mitsch and Gosselink, 2000). Phosphate ions can also replace a structural hydroxyl, thereby becoming part of the clay matrix (Brady and Weil, 2002). Precipitation of P by Al, Fe, or Mn is more common in acidic soils due to higher levels of these elements. This process involves Al, Fe, or Mn as dissolved ions, oxides, or hydrous oxides (Brady and Weil, 2002). In alkaline solutions adsorption of P with Ca may result in precipitation as calcium phosphate (Reddy et al., 1999). These adsorption and precipitation processes form compounds that are soluble at certain pHs.

The mobility and fixation of P is controlled by pH and redox conditions (Richardson, 1999). Phosphorus is most bioavailable at neutral to slightly acidic pH (Mitsch and Gosselink, 2000; Reddy et al., 1999). At low pH, P is fixed as Al and Fe phosphates, while P is bound to Ca and Mg at high pH. Consequently, Ca or Mg bound P becomes more soluble with decreasing pH, and Al or Fe bound P becomes more soluble with increasing pH. Soil P has a constant valence of +5 and is unaffected by redox processes; however, P is associated with compounds, particularly Fe, that are influenced by redox processes (Richardson and Vepraskas, 2001). Redox processes control P solubility by dissolving or decomposing the P-bearing compounds.

Phosphorus fixation results in removal of dissolved P ions from solution, lowering soluble P concentrations (Brady and Weil, 2002). The amount of P fixated to mineral surfaces is related to the amount of amorphous, or oxalate-extractable, Al and Fe (Al_{ox} , Fe_{ox}) oxides and hydroxides (Richardson, 1985). Since there is a limited amount of Al and Fe oxides and hydroxides in a system, P adsorption with these compounds is limited (Richardson and Vepraskas, 2001). Organic uptake can also fixate P; however, the amount of organic material is not as good an indicator of the amount of P fixation as amorphous Al and Fe content (Richardson and Vepraskas, 2001). Soils high in organic matter generally have low P fixation capacity because humic molecules adhere to P fixation sites (Brady and Weil, 2002). In soils with similar pH and mineralogy, P fixation tends to be greater in the soil with higher clay content (Brady and Weil, 2002). Clays that have a higher anion exchange capacity fix more P by having a greater affinity for phosphate ions. For soil components, the degree of P fixation, in order of increasing fixation, follows (Brady and Weil, 2002): 2:1 clays \ll 1:1 clays < carbonate crystals < crystalline Al, Fe, Mn oxides < amorphous Al, Fe, Mn oxides, allophane.

Previous Research

Phosphorus field studies involve determining retention time, measuring P concentrations after flooding, and comparing constructed wetlands to natural wetlands. Laboratory studies have focused on measuring various soil properties and relating them to P sorption and performing sequential extractions to determine the size of different P pools (bioavailable, mineral bound, organic, etc.). Batch incubation experiments have been used to determine P sorption characteristics under various conditions, in particular,

anaerobic versus aerobic conditions. These experiments can be performed over extended time periods, thus showing long term effects of soil flooding on P sorption.

Retention time studies have investigated the relationship between residence times within a wetland to P retention. In constructed wetlands, the most important factor for P retention is the ratio of wetland area to catchment area (Koskiaho and Puustinen, 2005). Similarly, Reinhardt et al. (2005) calculated that to retain half of the soluble reactive P (SRP) in an agricultural wetland, the surface area of the wetland must be at least 4% of the catchment area. Mitsch et al. (1995) studied four wetlands in northeastern Illinois and compared them to previous studies on both natural and constructed wetlands in the Midwest. They found that constructed wetlands retain the same order of magnitude of P per unit area as natural wetlands. Therefore, constructed wetlands of appropriate size can function like natural wetlands in retaining P in a watershed. The age of the wetland may also play an important role in P retention. Braskerud et al. (2005) studied 17 wetlands in temperate and boreal climatic zones. They found that particulate P retention increased with wetland age, while SRP retention was highest in younger wetlands.

Numerous soil properties have been measured that relate to P sorption capacity (Table 1). Bruland and Richardson (2006) studied 15 wetland sites in Minnesota and determined that soil organic matter (SOM), exchangeable calcium (Ca_{ex}), and Fe_{ox} best predict P sorption, whereas Richardson (1985) found Al_{ox} to be the best predictor. At a proposed wetland construction site in Florida, Al_{ox} and citrate-dithionite-bicarbonate extractable Al were correlated to P sorption (Pant et al., 2002). In flooded soils,

Table 1. Soil properties commonly used to correlate with P sorption capacity, with literature references.

Soil Property	Reference
Bulk Density	Bruland and Richardson, 2006; Hogan et al., 2004
pH	Bruland and Richardson, 2006; Hogan et al., 2004; Pant et al., 2002; Richardson, 1985
Soil organic matter	Bruland and Richardson, 2006; Hogan et al., 2004; Richardson, 1985
Total carbon	Hogan et al., 2004; Pant et al., 2002
Clay content	Hogan et al., 2004
Exchangeable Ca	Bruland and Richardson, 2006; Hogan et al., 2004; Pant et al., 2002; Richardson, 1985
Magnesium	Pant et al., 2002
Oxalate-extractable Fe	Bruland and Richardson, 2006; Hogan et al., 2004; Khalid et al., 1977; Pant et al., 2002; Richardson, 1985
Oxalate-extractable Al	Bruland and Richardson, 2006; Hogan et al., 2004; Pant et al., 2002; Richardson, 1985
Citrate-dithionite-bicarbonate extractable Fe	Hogan et al., 2004; Pant et al., 2002
Citrate-dithionite-bicarbonate extractable Al	Pant et al., 2002

Khalid et al. (1977) found Fe_{ox} most important to P sorption capacity. Phosphorus sorption correlated with residual Al, Al_{ox} , Fe_{ox} , clay, HCl-extractable Fe, and pyrophosphate-extractable Fe in restored herbaceous wetlands (Hogan et al., 2004). There is little agreement in the literature as to which soil property best predicts P sorption; however, Al_{ox} and Fe_{ox} seem to be the most common P sorption predictors.

Field and laboratory studies have shown that P is often released when soils are flooded. Newman and Pietro (2001) studied cropland conversion to wetland. Once flooded, SRP increased, suggesting that constructed wetlands may act as a P source. Similarly, in dairy soils SRP may be released during the first 28 days after flooding (Pant and Reddy, 2003). However, this P release may not necessarily reach surface waters.

Young and Ross (2001) flooded 14 soils from New York and found that porewater P increased from 2.2 to 27 times the initial concentration, but the floodwater P only increased a maximum of 3.6 times. The soils released P, but not all of it entered surface water. They attributed this phenomenon to a redox interface at the soil/sediment surface. Therefore, as long as the upper part of the soil/sediment remains oxidized, P release will be minimized.

Incubation experiments along with field studies have shown that the release of SRP after flooding occurs rapidly. Several studies have determined that the maximum SRP concentration in soil porewater and surface water is attained within one month (Szilas et al., 1998; Scalenghe et al., 2002; Pant and Reddy, 2003; Surridge et al., 2007). Newman and Pietro (2001) measured the maximum SRP within 2-3 months of flooding. Once a steady reduced state is reached, i.e. constant $p_e + pH$, SRP concentrations level off or decrease (Scalenghe et al., 2002; Newman and Pietro, 2001). Therefore, soil flooding will quickly cause reducing conditions, potentially leading to the rapid release of P to soil porewater and surface water.

These studies show that P is potentially released following wetland restoration or construction. Little is known concerning how soils in the Red River Valley will be affected by restoring wetlands and how restoration and construction of wetlands may influence downstream water quality. The research presented in this report characterizes the soils at the study site and predicts the impact that wetland restoration will have on P retention. Future monitoring at the site will determine if these predictions are correct.

CHAPTER III
MATERIALS AND METHODS

Site Description

The approximately 3,500 ha JD66 watershed lies within the Glacial Ridge National Wildlife Refuge (GRNWR) approximately 20 km east of Crookston, Minnesota, south of U.S. Highway 2 (Figure 1). The 10,000 ha GRNWR is owned by The Nature Conservancy and the U.S. Fish and Wildlife Service. Early in the 20th century this area was extensively ditched to help drain the wet meadows and prairies, thus allowing agriculture in the area (Cowdery et al., 2007). The Nature Conservancy and its partners are restoring wetlands and native prairies to their original condition, with work scheduled for completion by 2011 (Cowdery et al., 2007). Wetland restoration includes blocking, modifying, and removing ditches, along with recreating original wetlands and reintroducing original native plant communities (Cowdery et al., 2007).

JD66 begins at a large pond within a gravel pit, which provides the main source of baseflow to the ditch, and leaves GRNWR through a culvert underneath U.S. Highway 2. Water within the ditch eventually reaches the Red Lake River, the Red River, and Lake Winnipeg. The JD66 watershed drains land between beach ridges that formed on the eastern edge of Glacial Lake Agassiz, approximately 13,000 years ago. Between beach ridges the land has a low slope, with the average gradient of JD66 being 1.5 m/km (Cowdery et al., 2007).

Beach ridges are composed of sand and gravel, which are well drained. The western slopes of beach ridges often have seepage zones that form calcareous fens. Between beach ridges are finer grained soils, which are more poorly drained, forming organic-rich wetlands (Cowdery et al., 2007). Soils at the site are formed on the wave-modified Red Lake Falls Formation (Harris et al., 1974). Surface exposures of this till unit extend from the Canadian border to the Wild Rice River, approximately 50 kilometers south of the study area. The till extends in the subsurface westward into North Dakota. Texturally, the till is approximately 40% sand, 40% silt, and 20% clay (Harris et al., 1974). The composition of the sand is approximately 50% igneous and metamorphic, 40% carbonate, and 10% shale (Harris et al., 1974).

Soils at the study site were formed from parent material of glacial origin, including glaciolacustrine, till, and beach deposits (Saari and Heschke, 1996) (Table 2). The geologic setting of the soil reflects the glacial topography, with soils being formed on lake plains, outwash plains, beach plains, and moraines (Saari and Heschke, 1996). Due to the glacial complexity of the site, soil composition ranges between organic-rich mucks, loams, fine sands, and gravelly coarse sands.

Overview

A primary research area was established by installation of shallow groundwater sampling wells near the center of the watershed where an unpaved road crosses JD66. By November 2008, there were 11 wells located within this research area (Figure 2). To determine groundwater flow patterns, wells were surveyed and the depth to water below the top of the risers was measured periodically to obtain relative water table elevations.

Table 2. Names and descriptions of the nine NRCS soil series sampled at the study site, along with the typical profile.

Soil Series	Sample Sites ^a	Name	Setting	Parent Material	Slope (%)	Typical Profile (cm.)
15	4	Flaming loamy sand	Flats and rises on lake plains	Glacio-lacustrine	0 – 3	0 – 30 30 – 203 Loamy fine sand Fine sand
296	2	Fram loam	Rises and flats on moraines	Till	1 – 3	0 – 203 Loam
439	12	Strathcona fine sandy loam	Flats on lake plains	Glacio-lacustrine over till	0 – 2	0 – 43 43 – 76 76 – 203 Fine sandy loam Fine sand Fine sandy loam
547	6	Deerwood muck	Depressions on lake and outwash plains	Organic over glacio-lacustrine	0 – 1	0 – 36 36 – 89 89 – 203 Muck Loamy fine sand Fine sand
704	1	Wyrene sandy loam	Rises and flats on beach plains	Beach deposits	0 – 2	0 – 53 53 – 203 Sandy loam Gravelly coarse sand
712	8	Rosewood fine sandy loam	Flats and swales on lake plains	Glacio-lacustrine	0 – 2	0 – 43 43 – 76 76 – 203 Fine sandy loam Fine sand Sand
1117	5	Hedman loam	Flats and swales on moraines	Till	0 – 2	0 – 28 28 – 203 Loam Fine sandy loam
1142	1	Hedman - Fram Complex	Swales and flats on moraines	Till	0 – 2	0 – 25 25 – 203 Loam Fine sandy loam
1278	1	Rosewood - Venlo Complex	Swales and flats on lake plains	Glacio-lacustrine	0 – 2	0 – 43 43 – 76 76 – 203 Fine sandy loam Fine sand Sand

^an = 40

This research uses both water and soil samples. Water samples were used to determine phosphorus concentration and transport within the JD66 watershed. Water level data were used to determine groundwater flow and the interaction between groundwater and ditch water. Soil samples were collected to determine soil characteristics relating to P and P adsorption capacity. To determine which soil properties are related, the Spearman Rank correlation was performed. Combined, the data are used to predict the likelihood of P release after wetland reconstruction and determine which areas pose the greatest risk.



Figure 2. Location of wells used for groundwater sampling and water level measurements. The black square on Figure 1 outlines the area shown above. Coordinates of wells can be found in Appendix A.

Water Sampling

During the 2008 field season, water samples were collected five times for analysis. Surface water was collected using a dip sampler. Samples were analyzed in the field for conductivity, pH, and temperature using an Extech Instruments ExStik EC500 (Waltham, Massachusetts), oxidation-reduction potential (ORP) using an Extech Instruments ExStik RE300 (Waltham, Massachusetts), and turbidity using a HF Scientific Inc. MicroTPI turbidimeter (Fort Myers, Florida). Groundwater samples were collected

using a bailer after the wells were thoroughly purged and allowed to recover. The same field analyses were performed for the groundwater samples, excluding turbidity. A peristaltic pump was used to filter the sample using disposable 0.45 µm cartridges. Samples were placed in an appropriate container depending on the analyses to be performed (Table 3), and transported immediately on ice to the Environmental Analytical Research Laboratory (EARL) at the University of North Dakota (UND).

At the EARL, samples were analyzed for total and inorganic carbon by combustion catalytic oxidation using a Shimadzu TOC-Vcsn analyzer (Columbia, Maryland), reactive P by ascorbic acid colorimetry using a HACH DREL/2010 spectrometer (Loveland, Colorado), total P by acid persulfate digestion and ascorbic acid colorimetry, nitrite and nitrate using a Dionex DX-120 Ion Chromatograph (Sunnyvale, California), and ammonia using an ORION Model 95-12 ammonia selective electrode (Waltham, Massachusetts).

Table 3. Sample collection and preservation information for water samples.

Analysis	Container	Volume (mL)	Filtration	Preservation
Total P	Plastic	250	Unfiltered	H ₂ SO ₄ , pH < 2
Reactive P, Nitrite	Plastic	250	Filtered	None
Nitrate, Ammonia	Plastic	250	Filtered	H ₂ SO ₄ , pH < 2
Total Suspended Solids	Plastic	250	Unfiltered	None
Carbon	Borosilicate Glass	125	Filtered	No head space

Soil Sampling

Soil samples were collected in November 2008 at 40 sites within the study area using a hand auger (Figure 3). The 40 soil samples sites represent nine Natural Resources Conservation Service (NRCS) soil series, ranging texturally between gravels, fine sands, sandy loams, and organic-rich mucks (Saari and Heschke, 1996) (Table 2).

Sampling sites were selected along profiles that transect soil series throughout the study area. Additional samples were collected within the well field, where the most water data were collected and groundwater flow was mapped. Samples were collected at two depth intervals at each site, 10-18 cm and 25-33 cm, in order to determine how soil properties change with depth. Samples were not collected at the surface in order to avoid roots, which might interfere with P analyses. At each depth interval two samples were collected, one for laboratory analysis and one for bulk density (BD). For BD analysis a sample of known volume was collected using the auger. The hand auger may have caused some compaction of the BD sample, but correlation should still be possible. Samples were stored in Ziploc bags and frozen prior to analysis.

Laboratory Soil Analyses

Bulk density was determined by oven drying the soil sample of known volume at 105°C for 24 hours (Blake and Hartge, 1986). The samples for chemical analysis were placed on paper towels and allowed to air dry. The dried samples were placed in a plastic bucket and mixed with a soil mixer. After mixing, a majority of the soil sample was ground using a mortar and pestle. This pulverized portion was used for the remaining analyses. Soil pH was determined with a 1:1 soil to water ratio (Thomas, 1996). Particle size distribution was determined by sieve and hydrometer measurements (see Appendix B). Soil Fe_{ox} was extracted with acid ammonium oxalate adjusted to pH 3.0 (Ross and Wang, 1993). The extraction was performed in duplicate and analyzed using a Thermo Scientific (Waltham, Massachusetts) atomic absorption spectrometer (AAS).

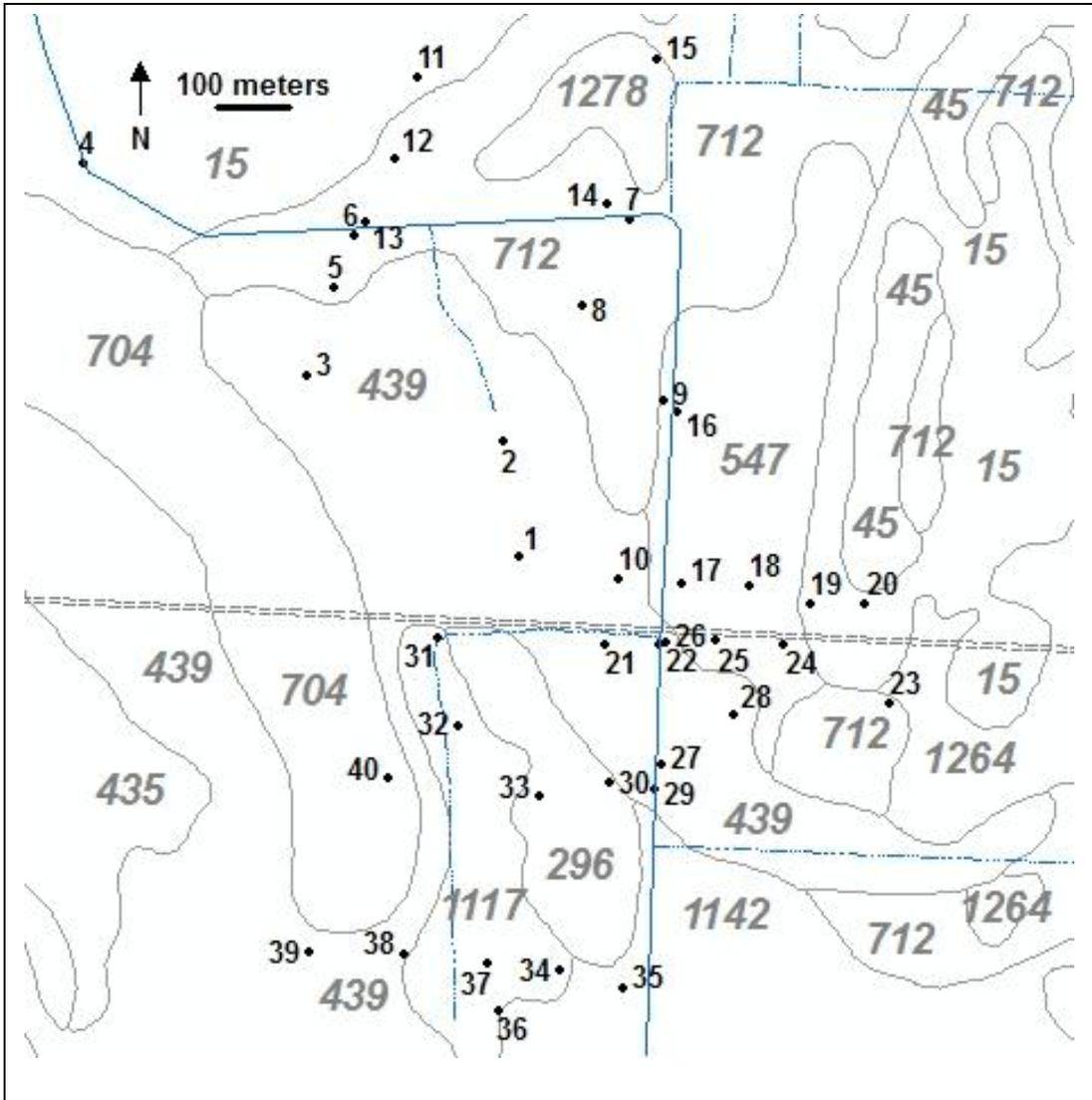


Figure 3. Soil sample locations (black) and soil series (gray) map from the NRCS soil survey. Soil series are described in Table 2. Sampling location coordinates can be found in Appendix A.

Analyses that were unable to be performed at UND’s EARL were sent to other laboratories for analysis. Along with the 80 samples, 10 blind, duplicate samples were sent to each laboratory for quality control. Agvise Laboratories (Northwood, ND) measured SOM by loss-on-ignition and total P by acid digestion (see Appendix B). Exchangeable Ca and Al_{ox} analyses were performed at the North Dakota State University

Soil Testing Laboratory (Fargo, ND). Oxalate-extractable Al was extracted with acid ammonium oxalate adjusted to pH 3.0, and analyzed by ion chromatography (Ross and Wang, 1993). The extractant for Ca_{ex} was 1 N ammonium acetate at pH 7.0, with the extraction analyzed by AAS (Suarez, 1996).

The previous analyses provide the various soil properties that can be correlated to P sorption. To quantify the P sorption capacity of the soil sample, a P sorption index (PSI) was used. The PSI is a single point isotherm that has been shown to serve as a reliable gauge of the P sorption potential of a wetland soil and to aid in comparison with soil properties (Bruland and Richardson, 2006). To determine the PSI, 2 g of dry soil were added to a 25 mL solution of 130 mg/L $\text{PO}_4\text{-P}$ and shaken for 24 hours (Bache and Williams, 1971; Richardson, 1985). Samples were then filtered with Whatman 42 filter paper and analyzed for $\text{PO}_4\text{-P}$ by ion chromatography (Dionex, Sunnyvale, CA). The amount of P sorbed by the soil is the difference between initial and final concentrations. The PSI is equal to $X / (\log C)$, where X is the amount of P sorbed (mg P/100 g soil) and C is the final inorganic P concentration in solution (mg $\text{PO}_4\text{-P/L}$).

A sequential extraction was performed to determine the amount of each form of soil P. The procedure involved sequential extraction with 0.5 M NaHCO_3 , followed by 0.1 M NaOH , and finally 1.0 M HCl , based on the procedure of Tiessen and Moir (1993). The NaHCO_3 extraction corresponds to bioavailable P, the NaOH extraction to Al and Fe-bound P, and the HCl extraction to Ca-bound P. Mineral-bound P is the sum of the Al, Fe, and Ca-bound P. To determine organic P the three extractions were added together and subtracted from total P.

Statistical Analysis

Mean and standard deviations were calculated for each of the soil properties for all samples, and at each depth interval. To correlate soil properties to PSI, Spearman's rank correlation coefficients were calculated (Davis, 2002). Samples were broken into two data sets, depth and soil series, to determine which provided the highest correlation. Critical values were used to determine which correlation coefficients are significant (Ramsey, 1989).

CHAPTER IV

RESULTS

Phosphorus Transport in Surface and Groundwater

Within the study area, groundwater flow is affected by precipitation (Figure 4). Under baseflow conditions, groundwater on the east side of the ditch flows toward a water table depression near and roughly parallel to the ditch (Figure 4a). Water from within the ditch also flows toward the depression, indicating that this reach of the ditch is losing under baseflow conditions. After a period of unusually heavy rainfall, when 4.52 cm of rain fell October 11-13, 2008 (recorded at a weather station within the watershed), the groundwater flow dynamics changed. The water table depression disappeared and flow was into the ditch, therefore the ditch was gaining (Figure 4b).

Water sample analyses show changing chemical conditions throughout the year (Appendix C). Reactive P in the ditch was never higher than the detection limit of 0.01 mg/L P (Table 4a). Total P in the ditch was not above the detection limit until August (Table 4a). When detected, the highest concentration of total P was at the pit outlet. The October 3, 2008 sampling had the highest concentration of total P leaving the study area (0.03 mg/L P), but bioavailable reactive P was undetected. Groundwater samples show similar results to surface water samples (Table 4b). The highest concentration of reactive P in groundwater is at the detection limit of 0.01 mg/L P. However, unlike surface water, the total P concentrations are high, ranging from no detection to 1.38 mg/L P.

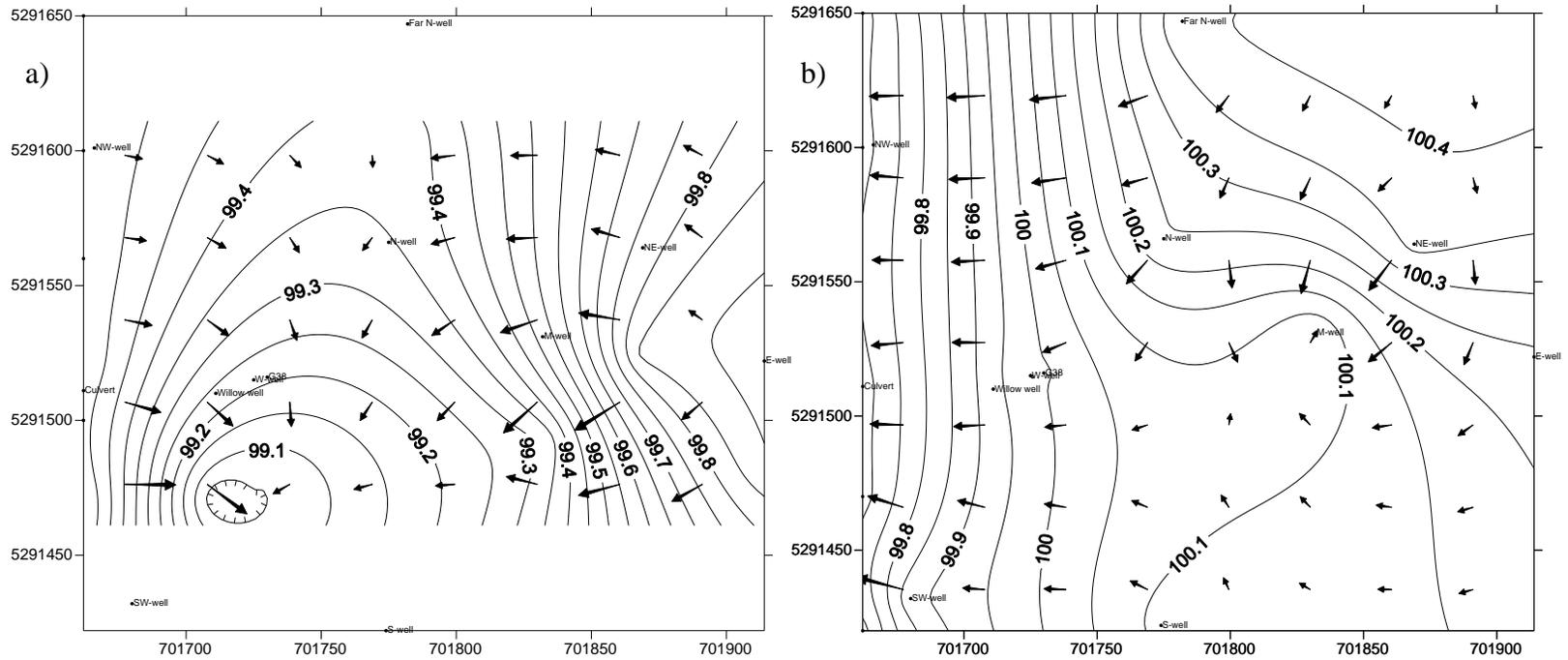


Figure 4. Groundwater flow in the study area east of JD66 (a) under baseflow conditions (10-2-08) and (b) after heavy rainfall (10-15-08), when 4.52 cm of rain fell October 11-13, 2008 in the watershed. JD66 runs along the left side of each figure. North is to the top of each figure. Water levels are in meters relative to the top of the culvert at the ditch crossing (= 100 m), not actual elevations. Coordinates of axes are UTM 14 NAD 1983.

Table 4. Reactive P and total P concentrations from (a) the four ditch sample sites along JD66, and (b) groundwater samples during the 2008 sampling season. ND = no detection, with a detection limit of 0.01 mg/L PO₄-P. A hyphen (-) indicates the well was not sampled on that date.

a)

Date	Reactive Phosphorus				Total Phosphorus			
	PT	UX	MC	HZ	PT	UX	MC	HZ
5/1/08	ND	ND	ND	ND	ND	ND	ND	0.01
5/29/08	ND	ND	ND	ND	ND	ND	ND	ND
7/14/08	ND	ND	0.01	0.01	ND	ND	0.01	ND
8/6/08	ND	0.01	0.01	0.01	0.03	0.01	0.02	0.01
10/3/08	ND	ND	ND	ND	0.07	0.02	0.02	0.03

b)

Date	Reactive Phosphorus					
	G38	E	Far N	M	Willow	NE
5/1/08	ND	-	-	-	-	-
5/29/08	-	ND	-	-	-	-
7/14/08	0.01	0.01	0.01	-	-	-
8/6/08	0.01	0.01	0.01	-	-	-
10/3/08	0.01	ND	0.01	0.01	0.01	0.01

Date	Total Phosphorus					
	G38	E	Far N	M	Willow	NE
5/1/08	ND	-	-	-	-	-
5/29/08	-	ND	-	-	-	-
7/14/08	0.53	0.15	0.07	-	-	-
8/6/08	0.12	0.05	0.31	-	-	-
10/3/08	0.08	0.33	0.67	1.14	0.08	1.38

Soil Properties and PSI

The mean BD increased with depth from 1.14 g/cm³ at the 10-18 cm depth to 1.46 g/cm³ at the 25-33 cm depth (Table 5). The grain size analysis showed little change with depth. Sand sized particles comprised 55% of the soil and silt 37%. Gravel and clay sized particles contributed the remaining 3.5% and 4.7%, respectively. Mean SOM decreased from 4.7% to 2.3% with depth, although with a large standard deviation. This likely explains the BD change with depth. Mean soil pH increased very slightly with depth from 7.82 to 7.90. The mean Fe_{ox} values showed no change with depth and

averaged 0.78 mg/g, while mean Al_{ox} values decreased from 0.94 to 0.73 mg/g with depth. Mean values of Ca_{ex} were much higher than either Fe_{ox} or Al_{ox} values and decreased from 3.29 mg/g to 3.10 mg/g with depth. Similar to SOM, Ca_{ex} , and Al_{ox} , mean total P values decreased from 437 $\mu\text{g/g}$ at 10-18 cm depth to 346 $\mu\text{g/g}$ at 25-33 cm depth.

The mean PSI at the shallow depth was 23.5 and increased slightly with depth to 25.8. These values had a high standard deviation, meaning that the PSI has a large variability in the soils that were sampled. Grouping the samples by soil series shows that the mean PSI is lowest in the Hedman Loam (soil series no. 1117, $n = 10$) at 13.1 and in the Hedman-Fram complex (1142, $n = 2$) at 11.5, while the PSI is highest in the Rosewood-Venlo complex (1278, $n = 2$) at 82.2 (Table 6). The PSI was lowest in sample 11S at -1.9, which was the only negative PSI, and highest in sample 15D at 131.3 (Appendix D). A negative PSI indicates that during extraction none of the added P was adsorbed by the soil, and some P already present in the soil was released.

Factors Relating to Phosphorus Retention

The Spearman Rank correlation analyses indicated many significant correlations between soil properties, with similar results between each depth interval (Table 7). Soil organic matter had a significant negative correlation to BD and pH, and a positive correlation to Ca_{ex} and total P at both depths. There were no significant correlations with Fe_{ox} at either depth interval. A negative correlation exists between pH and Al_{ox} at both depths. In the shallow samples, Al_{ox} is also correlated to SOM and total P. Total P is correlated with most parameters at the shallow depth, with SOM and Ca_{ex} showing the

Table 5. Mean values of soil properties at each depth interval. The row labeled “Both” includes all samples. Standard deviations are shown below the mean values at each depth interval in italics. Appendix D provides the complete analysis results for each sample.

Depth cm	BD g/cm ³	pH	SOM %	Fe_{ox} mg/g	Ca_{ex} mg/g	Al_{ox} mg/g	Total P µg/g	PSI X/log C	Gravel %	Sand %	Silt %	Clay %
10-18^a	1.14	7.82	4.7	0.77	3.29	0.94	437	23.5	3.0	54.7	38.1	4.2
	<i>0.26</i>	<i>0.31</i>	<i>4.3</i>	<i>0.41</i>	<i>1.01</i>	<i>0.53</i>	<i>189</i>	<i>17.6</i>	<i>7.3</i>	<i>16.5</i>	<i>14.7</i>	<i>4.4</i>
25-33^a	1.46	7.90	2.3	0.78	3.10	0.73	346	25.8	3.9	55.2	35.7	5.1
	<i>0.25</i>	<i>0.27</i>	<i>2.2</i>	<i>0.63</i>	<i>1.76</i>	<i>0.45</i>	<i>145</i>	<i>27.5</i>	<i>8.3</i>	<i>18.1</i>	<i>15.7</i>	<i>4.2</i>
Both^b	1.30	7.86	3.5	0.78	3.20	0.84	391	24.7	3.5	54.9	36.9	4.7
	<i>0.30</i>	<i>0.29</i>	<i>3.6</i>	<i>0.53</i>	<i>1.43</i>	<i>0.50</i>	<i>173</i>	<i>23.0</i>	<i>7.8</i>	<i>17.2</i>	<i>15.1</i>	<i>4.3</i>

^an = 40 for this depth interval, ^bn = 80 (all samples)

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Table 6. Mean values of soil properties for samples in each NRCS soil series. Descriptions of each soil series can be found in Table 2 and locations in Figure 3.

Soil Series	Samples (n = 80)	BD g/cm ³	pH	SOM %	Fe_{ox} mg/g	Ca_{ex} mg/g	Al_{ox} mg/g	Total P µg/g	PSI X/log C	Gravel %	Sand %	Silt %	Clay %
15	8	1.21	7.37	3.3	0.55	2.16	1.23	356	20.6	1.7	74.5	20.8	3.0
296	4	1.13	7.71	6.0	0.63	3.46	1.11	591	29.5	3.0	45.4	44.5	7.1
439	24	1.21	7.87	3.8	0.99	3.80	0.78	441	31.2	4.4	48.9	42.2	4.5
547	12	1.30	7.89	3.6	0.50	3.23	0.57	367	23.2	0.3	58.0	36.6	5.1
704	2	1.37	7.94	4.4	0.87	5.07	1.91	697	43.6	3.0	47.5	47.0	2.5
712	16	1.44	7.90	2.2	0.68	2.79	0.92	312	16.2	1.7	61.8	32.6	4.0
1117	10	1.42	8.06	4.5	1.00	2.79	0.66	356	13.1	9.8	46.7	38.7	4.8
1142	2	1.44	8.27	0.3	0.68	2.72	0.46	301	11.5	5.8	38.1	47.3	8.8
1278	2	1.24	7.92	3.1	0.65	3.27	0.45	276	82.2	0.4	61.4	29.4	8.8

highest correlation. For the deep samples, total P correlates negatively with BD and positively with SOM, Ca_{ex}, and silt. The PSI correlated positively with SOM, Ca_{ex}, total P, and silt in the shallow samples, and negatively with sand. For the deep samples, PSI correlated positively with SOM, Ca_{ex}, silt, and clay, and negatively with sand.

Table 7. Spearman Rank correlation coefficients at each depth interval. Coefficients in **bold** are significant at the 95% confidence level. Coefficients with asterisk (*) are significant at the 99% confidence level.

10-18 cm	pH	SOM	Fe _{ox}	Ca _{ex}	Al _{ox}	Total P	PSI	Gravel	Sand	Silt	Clay
BD	0.39	-0.53*	-0.08	-0.44*	-0.13	-0.45*	-0.40	-0.38	0.44*	-0.36	-0.10
pH		-0.57*	-0.20	-0.31	-0.46*	-0.42*	-0.06	-0.23	0.05	0.02	0.18
SOM			0.29	0.81*	0.57*	0.88*	0.63*	-0.15	-0.35	0.37	-0.05
Fe _{ox}				0.26	0.02	0.31	0.05	0.02	-0.20	0.21	0.01
Ca _{ex}					0.32	0.83*	0.78*	-0.11	-0.58*	0.61*	0.13
Al _{ox}						0.51*	0.18	-0.24	0.14	-0.06	-0.40
Total P							0.68*	-0.13	-0.44*	0.54*	-0.04
PSI								-0.31	-0.56*	0.58*	0.37
Gravel									-0.40*	-0.05	-0.33
Sand										-0.86*	-0.41*
Silt											0.35
25-33 cm	pH	SOM	Fe _{ox}	Ca _{ex}	Al _{ox}	Total P	PSI	Gravel	Sand	Silt	Clay
BD	0.50*	-0.67*	-0.04	-0.35	-0.30	-0.53*	-0.34	-0.02	0.00	-0.21	0.12
pH		-0.58*	0.06	-0.10	-0.57*	-0.30	-0.06	0.00	-0.16	-0.01	-0.09
SOM			0.04	0.67*	0.37	0.77*	0.48*	-0.03	-0.37	0.52*	0.05
Fe _{ox}				0.14	-0.17	0.16	0.18	0.05	-0.26	0.14	0.20
Ca _{ex}					-0.06	0.64*	0.77*	-0.30	-0.64*	0.76*	0.43*
Al _{ox}						0.39	-0.17	-0.08	0.31	-0.15	-0.31
Total P							0.30	-0.01	-0.40	0.56*	0.03
PSI								-0.27	-0.51*	0.60*	0.44*
Gravel									-0.37	-0.05	-0.39
Sand										-0.84*	-0.58*
Silt											0.40

A correlation analysis was also performed on mean soil property values for each of the nine soil series sampled (Table 8). For this analysis, Fe_{ox} was positively correlated to gravel. There was a strong negative correlation between Al_{ox} and clay. Total P was positively correlated to SOM. The strong negative correlation between sand and silt is a result of the grain size analysis procedure (Appendix B). The PSI was only correlated to Ca_{ex} in this analysis.

Table 8. Spearman Rank correlation coefficients using the mean values for each of the nine soil series sampled (n = 9). 95% significance in **bold**, 99% marked by asterisk (*).

Soil Series	pH	SOM	Fe _{ox}	Ca _{ex}	Al _{ox}	Total P	PSI	Gravel	Sand	Silt	Clay
BD	0.75	-0.52	0.32	-0.42	-0.22	-0.42	-0.58	0.25	-0.05	0.12	-0.10
pH		-0.25	0.58	-0.03	-0.45	-0.30	-0.30	0.52	-0.50	0.47	0.25
SOM			0.20	0.60	0.45	0.82	0.30	0.25	-0.35	0.25	-0.20
Fe _{ox}				0.28	0.00	0.13	-0.12	0.87*	-0.43	0.47	-0.18
Ca _{ex}					0.20	0.68	0.78	0.07	-0.27	0.40	-0.07
Al _{ox}						0.67	0.13	0.07	0.15	0.07	-0.83*
Total P							0.33	0.18	-0.30	0.45	-0.48
PSI								-0.42	0.23	-0.12	-0.02
Gravel									-0.70	0.67	-0.08
Sand										-0.90*	-0.38
Silt											0.10

Phosphorus Fractionation

The sequential extraction calculated the bioavailable P, mineral bound P, and Ca-bound P pools. Organic P was then calculated using these values and the total P value. There was little change with depth between the P pools. Mean organic P comprised 80% of the total P in the shallow samples with Ca-bound P making up nearly all of the rest (Figure 5). Mean bioavailable P and mineral bound P each comprised less than 1% of the total P at both depths. Separating the data by soil series showed similar results.

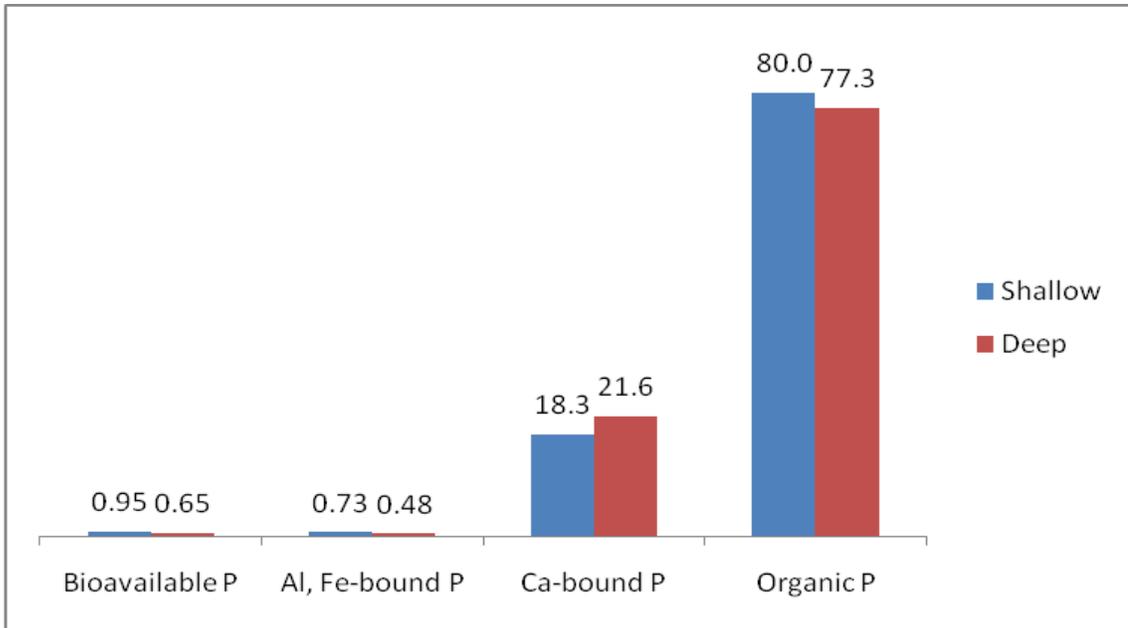


Figure 5. Phosphorus fractionation results showing the percentages of soil P in each pool for shallow (left) and deep (right) samples. Results for each sample are in Appendix E.

CHAPTER V

DISCUSSION

Phosphorus Transport in Surface and Groundwater

Under baseflow conditions, water level data showed a water table depression near the ditch, indicating water from the ditch is flowing into the surrounding soils and sediments. Analysis of ditch water indicated very low concentrations of reactive and total P. Therefore, under baseflow conditions little P is being added to the sediments surrounding the ditch. Even if the soil is releasing P to porewater, it will not be transported to the ditch and is likely to be readsorbed by the surrounding soil. However, soils and sediments within the water table depression are likely to be more saturated in P. After a period of heavy rainfall, the water table rose and the depression disappeared, indicating groundwater flow into the ditch. Consequently, if P is released by the soil under this flow pattern, it could enter the ditch and be transported downstream.

Groundwater samples show very little reactive P, but higher concentrations of total P. This shows that any reactive P in the sediment is quickly immobilized and stored. Even after periods of heavy rainfall, when groundwater is flowing into the ditch, little P will be transported into the ditch, because P is bound to sediment, not in solution. Concentrations of P in the ditch leaving the study area are very low, indicating that the sediment and soils within the watershed are able to store P and little is transported downstream.

Soil Properties and PSI

The relatively high BD and low SOM indicate that the study area contains mineral soils. Organic soils contain $\geq 20\%$ organic carbon (Soil Survey Division Staff, 1993). High Ca_{ex} concentrations and relatively low Fe_{ox} and Al_{ox} concentrations show that the soils are predominately Ca-rich. This is because the soils are formed on the carbonate-rich Red Lake Falls Till. All but one sample has a pH greater than 7.0, with the average pH nearly 8.0; therefore, the soils are slightly basic. Under basic conditions, P adsorption and precipitation will be controlled by reactions with calcium and magnesium (Mitsch and Gosselink, 2000). The higher total P in the shallow samples is most likely a result of agricultural application. Similar total P values were found in active- and abandoned-intensive dairy sites that were proposed for wetland construction (Pant et al., 2002). Natural wetland sites in Minnesota had total P concentrations that were significantly higher, most likely due to organic matter accumulation (Bruland and Richardson, 2006).

Overall, the PSI values were relatively low compared to other studies, suggesting a fairly high degree of P saturation in the soils. Bruland and Richardson (2006) calculated mean PSI in wetlands to be above 85 for each depth interval. Since the wetlands in their study contained high total P, but also high PSI, this may indicate that wetlands can potentially store a large amount of P. They also indicated PSI decreased with depth, which was not found in this study. The slight increase in PSI with depth at this study site is most likely a result of lower total P values, but a similar amount of P sorption sites.

Factors Relating to Phosphorus Retention

Soil properties that correlated strongest with PSI at both depth intervals were SOM, Ca_{ex} , and silt. Bruland and Richardson (2006) also showed a correlation of PSI to SOM and Ca_{ex} . Other studies found Al_{ox} or Fe_{ox} to correlate with PSI; however, these studies were done on acidic soils (Richardson, 1985; Khalid et al., 1977; Hogan et al., 2004). The Ca_{ex} correlated with the silt fraction as well, which explains why PSI is correlated to silt. When separated by soil series, PSI correlated highest to Ca_{ex} . The dominant soil component that controls P sorption seems to be Ca_{ex} . Precipitation of dicalcium phosphate (CaHPO_4) or octacalcium phosphate ($\text{Ca}_8(\text{HPO}_4)_2(\text{OH})_2$) will remove SRP from solution (von Wandruszka, 2006). Since calcium is not redox sensitive, Ca-bound P will not be released under anaerobic conditions. Negatively charged SOM can bind with Ca and Mg cations, which in turn bind with and immobilize P (Brady and Weil, 1999). This can explain the high correlation between Ca_{ex} and SOM.

Phosphorus Fractionation

Since the study site contains mineral soils it is surprising that organic P comprises about 80% of total soil P. The high organic P is probably a result of decaying material that is tilled into the soil. Bruland and Richardson (2006) found wetlands soils contained 60-80% organic P, but the soils also had greater than 40% SOM. In the soils at this study site, greater than 99% of the total P is in the organic P and Ca-bound P pools. Anaerobic conditions caused by wetland construction will not affect these P pools. Therefore, soils at the site are unlikely to release P to solution. Since wetlands accumulate organic matter, after restoration the organic P pool will continue to be stored and may increase.

Predicted Changes After Wetland Restoration

Soil flooding during wetland restoration will change the redox conditions of the soil. Research has shown that changes in P retention are likely to be seen within the first month after wetland construction. However, at this site nearly all P in the soil is Ca-bound or organic P, and redox changes will not affect these P pools. Even if all the P in the bioavailable and mineral bound P pools were released, solution P would not noticeably increase and it is likely the P in solution would be quickly reabsorbed. While the PSI of the soils is relatively low, the soils are still capable of adsorbing more P. Therefore, if inputs of P are reduced, the soils will be able to adsorb, in the long-term, any P in solution. Consequently, restoring wetlands at this site will apparently not cause a significant P release and should not influence water quality downstream.

Continued monitoring of this site is imperative in verifying the predictions of this research. Since the soil will quickly respond to redox changes, it is important to collect water P data within the first month after wetland construction. This is likely when the largest amount of P, if any, will be released. Monitoring should continue at least until redox conditions have stabilized. If wetland restoration does not cause a significant release of P, sites with similar soils can be selected for wetland construction throughout the Red River Valley; however, new sites should also be monitored for effectiveness. If wetland construction is a viable method to reduce P loading downstream, water quality throughout the Red River Valley and Lake Winnipeg can be improved.

CHAPTER VI

CONCLUSIONS

Most P in the water samples collected was particulate P, not SRP. Concentrations of P leaving the watershed in the ditch were very low; therefore, most P was stored in the soils and sediments in the watershed. Soils at the site show a moderate degree of P saturation, but still have the ability to store more P. The soil properties that correlated highest with PSI were SOM and Ca_{ex} . Nearly 80% of the total soil P is organic P, with the remaining amount predominantly Ca-bound P. Therefore, adsorption and precipitation of P with Ca, along with organic matter accumulation, are the dominant P storage mechanisms at this site. Wetland restoration will affect redox conditions by creating anaerobic conditions at the site; however, P is not significantly bound to any redox sensitive compounds. Wetlands decrease water velocity and allow particulate P to settle out of the water column. In addition, wetlands accumulate organic matter, thereby storing P. Therefore, wetland restoration will apparently not cause a significant P release to solution and can be a long-term P sink. Restoring wetlands at sites with similar soils can improve water quality throughout the Red River Valley and Lake Winnipeg.

APPENDICES

Appendix B Soil Analysis Procedures

Procedure for Grain Size Analysis

1. Weigh out approximately 60 g of oven-dried sample into aluminum tray, recording weight.
2. Make a 4% Calgon solution by mixing 400 g Calgon to 10 L deionized water.
3. Add soil sample to plastic bucket, then add 125 mL of 4% Calgon solution.
4. Mix thoroughly and let sit over night.
5. Next day, stir soil/Calgon solution and pour into 1 L settling tube.
6. Wash out plastic bucket with deionized water into tube and add deionized water until settling tube is at 1 L mark.
7. Make a blank settling tube by adding 125 mL of 4% Calgon and filling to 1 L mark with deionized water.
8. Mix settling tube end-over-end until no sediment remains on bottom of settling tube.
9. Let settling tube sit for 2.5 hours.
10. After 2.5 hours take hydrometer reading. Measure blank sample first. Subtract blank value from sample readings. This is the clay fraction in grams.
11. Rinse settling tube solution through a No. 230 sieve with tap water until waste water turns clear.
12. Transfer remaining sediment into aluminum tray and place in oven at 105°C overnight.
13. Next day, stack sieves using No. 10, 18, 120, and 230.
14. Place sediment into top sieve, cover, and shake for 10 minutes.
15. Weigh sediment in No. 10 sieve = gravel fraction. Sediment in remaining sieves is sand fraction. Sediment that goes through No. 230 sieve is smaller than sand and does not need to be weighed.
16. Calculate percentages of each fraction using fraction weights and total weight. The missing weight is considered the silt fraction.

Procedure for Organic Matter Determination by Loss-on-Ignition

(Obtained from Agvise Laboratories, Northwood, ND)

1. Preheat a drying oven to 150° ± 5°C.
2. Check the balance calibration following SOP NUT.01.01 – “Use of Maintenance of Laboratory Balances”

Appendix B
Soil Analysis Procedures

3. Use porcelain crucibles in metal organic matter trays. Record the tray letter and the crucible number for each sample on an organic matter lab sheet. Record two check soils at the beginning of each run.
4. Using a 5 g soil scoop, scoop the soil and check samples into the crucibles, according to SOP NUT.06.02 – “Soil Scoop Procedure”.
5. Place each tray of crucibles in the drying oven for a minimum of 2 hours.
6. Turn on the computer, printer and balance. Remove each tray from the drying oven after at least 2 hours and weigh at once, using the appropriate computer program.
7. Place the tray in a muffle furnace that has been pre-heated to $360^{\circ} \pm 30^{\circ}\text{C}$.
8. Set up the computer for the second weighing and start removing the trays from the muffle furnace after 4 hours and weigh the crucibles.
9. Write the results from the printout into the proper spaces on the lab sheet, checking to see that the check sample results are correct.
10. Empty the crucibles, being sure to remove all soil.
11. The percent organic matter is reported as percent loss on ignition.

Procedure for Total Phosphorus by Acid Digestion

(Obtained from Agvise Laboratories, Northwood, ND)

1. Transfer 0.25 g of sample to digestion tube. Run a reagent blank through the process.
2. Check the volume of the repipettes and/or adjustable pipettes that will be used to add the HNO_3 , DI H_2O , H_2O_2 , and HCl using a graduated cylinder. Use multiple dispenses to measure a minimum of 5 mL with the graduated cylinder. Record the volumes dispensed on the appropriate lab sheet.
3. In a fumehood, add 2 mL of DI H_2O and 2 mL of HNO_3 . Mix the slurry, and place a funnel in the digestion tube. Heat the sample to 95°C on the block heater and reflux 10 and 15 minutes without boiling. Allow the sample to cool, then add 1 mL of HNO_3 , replace the funnel, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3 , repeat the addition 1 mL of HNO_3 until no brown fumes are given off by the sample. Replace the glass funnel in the digestion tubes and heat at 95°C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times.
4. After step #3 has been completed and the sample has cooled, add 1 mL of water and 2 mL of H_2O_2 . Replace the glass funnel in the digestion tube and return to the block heater to start the Peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and allow the vessel to cool.

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Soil Analysis Procedures

5. Continue to add H₂O₂ in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.
6. Place glass funnel in the digestion tube and heat at 95°C without boiling for two hours. Maintain a covering of solution over the bottom of the vessel at all times.
7. Using an adjustable pipette, add 2 mL HCl to the sample digest and place glass funnel into the digestion tube. Place the sample on the heating block and reflux at 95°C for 15 minutes.
8. Bring sample digests to volume of 25 mL with DI H₂O and mix thoroughly.
9. Make sure the sample has settled out prior to analysis, filter using a Whatman No. 41 or similar filter paper if necessary.

Appendix C
Water Sampling Data

Table 10. Water Sampling Chemical Data

Date (2008)	Sample	Cond. μS/cm	Temp. °C	pH	Turbidity NTU	ORP mV	Reactive P mg/L P	Total P mg/L P
5/1	PT	451	7.6	8.79	3.14	181	ND	ND
	UX	535	8.2	7.90		214	ND	ND
	MC	519	7.7	8.37	5.20	186	ND	ND
	HZ	567	6.8	8.16	5.70	356	ND	0.01
	G-38	785	4.5	7.21		187	ND	ND
5/29	PT	425	16.2	8.74	7.12	217	ND	ND
	UX	497	17.3	7.62	1.11	224	ND	ND
	MC	501	16.5	8.07	5.95	222	ND	ND
	HZ	522	15.4	8.25	3.19	235	ND	ND
	W-well	725	11.2	7.85		243	-	ND
	E-well	676	9.60	7.77		238	ND	ND
7/14	PT	352	24.8		2.75	129	ND	ND
	UX	414	23.6		1.17	178	ND	ND
	MC	401	24.3		2.20	211	0.01	0.01
	HZ	421	24.5		1.44	113	0.01	ND
	E-well	515	17.5			180	0.01	0.15
	Far N-Well	569	17.4			190	0.01	0.07
	G-38	710	15.3			214	0.01	0.53
8/6	PT	344	27.9	8.41	3.55	113	ND	0.03
	UX	375	24.8	7.23	1.38	141	0.01	0.01
	MC	384	23.8	7.76	2.52	209	0.01	ND
	HZ	434	22.2	7.77	4.98	106	0.01	0.01
	E-well	661	19.4	7.18		187	0.01	0.05
	Far N-Well	579	16.6	6.90		163	0.01	0.31
	G-38	707	14.1	7.25		177	0.01	0.12
10/3	PT	365	19.4	8.57	21.75	126	ND	0.07
	UX	352	15.4	8.10	2.75		ND	0.02
	MC	394	16.4	8.42	6.95	107	ND	0.02
	HZ	418	10.1	8.10	3.28	127	ND	0.03
	E-well	628	18.8	6.37		124	ND	0.33
	M-well	562	15.9	7.62			0.01	1.14
	Far N-Well	650	16.3	7.10			0.01	0.67
	Willow	824	16.8	7.11		147	0.01	0.08
	NE-well	519	18.1	7.40		137	0.01	1.38
	G-38	762	15.3	7.31		160	0.01	0.08

Appendix C
Water Sampling Data

Table 11. Water Level Data. Water levels are in meters relative to the top of the culvert at Middle Crossing (MC), which is arbitrarily set to 100 m. Dates are in 2008. Locations of wells can be found in Figure 2.

Well	Ground Level	8/28	9/5	9/11	9/18	9/25	10/2	10/15	10/31
E-well	101.37	99.80	99.90	99.90	100.00	99.95	99.89	100.24	100.30
NE-well	100.91		99.89	100.00	99.95	99.91	99.87	100.37	100.36
Far N-well	100.44	98.79	99.40	99.84	99.59	99.45	99.38	100.39	100.36
N-well	100.48				99.45	99.32	99.26	100.24	100.19
M-well	100.15		99.20	99.52	99.43	99.38	99.30	100.05	100.13
W-well	99.92	98.49	98.90		99.23	99.13	99.06	100.00	99.97
Willow well	99.95		98.95	99.43	99.17	98.80	99.01	99.93	99.89
G38		98.20		99.34	99.23	99.14	99.05	99.99	99.98
Culvert	100.00	99.36	99.47	99.50	99.41	99.43	99.55	99.68	99.55
NW-well	100.02							100.07	99.84
SW-well	99.95							99.89	99.75
S-well	100.24							100.12	99.90

Appendix D Soil Sampling Data

Table 12. Results of Soil Analyses

Sample	BD g/cm ³	pH	SOM %	Fe _{ox} mg/g	Ca _{ex} mg/g	Al _{ox} mg/g	Total P μg/g	PSI	Gravel %	Sand %	Silt %	Clay %
1S	1.05	7.74	3.8	0.77	3.75	0.71	406	20.7	44.5	36.1	18.5	0.8
1D	1.50	7.96	1.1	0.57	2.52	0.44	261	11.2	30.8	35.9	30.8	2.5
2S	0.92	7.83	2.4	0.78	3.33	0.45	330	37.1	6.8	37.6	50.6	5.0
2D	1.32	8.04	0.3	0.52	2.86	0.22	187	98.1	12.0	55.1	24.5	8.3
3S	0.88	7.35	5.6	2.46	3.53	1.52	427	11.6	0.3	58.9	39.1	1.7
3D	1.10	7.60	3.2	2.47	2.53	1.50	313	16.4	2.3	59.9	36.9	0.8
4S	1.21	7.78	0.8	0.22	0.86	0.62	266	2.3	4.7	81.0	12.7	1.7
4D	1.35	7.66	0.8	0.17	0.82	0.59	196	2.6	2.0	85.9	10.5	1.7
5S	1.03	7.65	3.9	0.44	3.92	1.27	485	29.5	3.8	36.6	53.8	5.8
5D	1.85	7.92	6.1	0.27	3.41	0.43	252	35.9	6.8	33.0	54.3	5.8
6S	1.20	7.88	1.2	0.80	3.22	0.48	310	8.8	6.5	47.5	44.3	1.7
6D	1.49	7.70	5.8	0.50	3.56	1.51	532	13.8	3.0	55.8	37.8	3.3
7S	1.53	8.32	0.1	0.93	1.89	0.29	191	8.2	1.2	84.7	10.8	3.3
7D	1.61	8.15	0.2	2.61	1.05	0.18	182	10.8	0.5	90.5	7.3	1.7
8S	1.45	8.07	0.8	0.78	2.70	0.24	168	13.5	0.2	70.2	21.3	8.3
8D	1.58	7.74	0.8	1.11	3.19	0.21	207	41.6	0.2	39.1	53.2	7.5
9S	1.10	7.88	2.5	0.49	3.14	0.74	266	6.4	1.3	75.0	18.7	5.0
9D	1.51	7.97	0.5	0.31	2.53	0.51	130	3.7	0.2	78.0	18.5	3.3
10S	0.50	8.13	9.9	0.79	4.05	0.93	693	68.1	0.0	50.9	45.6	3.5
10D	1.51	7.88	2.4	0.40	3.33	0.85	406	18.9	0.0	76.0	21.5	2.5
11S	1.12	7.29	3.1	0.70	2.12	1.80	331	-1.9	3.0	77.8	18.3	0.8
11D	1.26	7.51	2.2	0.66	2.09	1.73	329	3.1	3.2	78.2	17.0	1.7
12S	1.33	7.90	5.4	0.53	3.36	2.31	420	15.9	0.0	70.0	26.7	3.3
12D	1.33	8.00	1.0	0.24	3.12	1.07	295	9.1	0.0	63.3	34.2	2.5
13S	1.06	7.92	2.1	0.33	3.25	0.81	405	11.2	1.3	41.9	53.4	3.3
13D	1.57	7.70	1.1	0.29	2.40	0.83	328	4.8	2.5	56.3	37.0	4.2
14S	1.36	7.78	3.4	0.68	3.18	0.97	353	19.4	0.7	63.7	29.0	6.7
14D	1.62	7.93	0.7	0.80	3.09	0.63	229	18.2	0.0	84.0	12.7	3.3
15S	1.20	7.88	4.1	0.72	3.53	0.64	327	33.1	0.3	54.3	31.2	14.2
15D	1.28	7.95	2.1	0.58	3.01	0.26	225	131.3	0.5	68.6	27.6	3.3
16S	0.93	7.74	2.6	0.64	2.94	0.45	231	9.8	0.3	72.4	25.6	1.7
16D	1.32	7.77	2.8	0.40	2.56	0.71	396	6.2	0.3	86.0	11.1	2.5
17S	1.26	7.76	8.7	0.85	4.11	0.77	553	33.3	0.2	41.2	53.7	5.0
17D	1.33	7.79	2.3	0.73	3.73	0.34	330	30.2	0.2	38.2	53.3	8.3
18S	1.05	7.46	10.6	0.40	4.59	0.82	716	62.7	0.5	42.8	52.5	4.2
18D	1.00	7.78	5.7	0.29	4.04	0.62	511	57.8	0.3	37.7	57.0	5.0
19S	0.86	7.42	11.0	0.42	4.45	1.34	690	63.3	0.2	53.3	44.0	2.5
19D	0.93	7.51	5.8	0.37	3.87	1.12	444	88.0	0.0	49.9	46.7	3.3
20S	1.45	6.67	1.5	1.00	1.51	0.99	313	2.7	0.2	83.7	13.7	2.5
20D	1.51	7.15	1.1	0.89	1.57	1.61	279	4.7	0.2	86.1	3.7	10.0

Appendix D Soil Sampling Data

Table 12. Continued

Sample	BD g/cm ³	pH	SOM %	Fe _{ox} mg/g	Ca _{ex} mg/g	Al _{ox} mg/g	Total P µg/g	PSI	Gravel %	Sand %	Silt %	Clay %
21S	1.49	8.13	5.4	0.78	3.50	0.98	470	27.3	0.2	64.5	32.0	3.3
21D	1.39	7.86	3.2	0.38	2.97	0.74	352	14.1	0.2	69.7	28.5	1.7
22S	1.11	7.97	3.6	0.91	3.38	1.28	486	21.9	0.7	42.0	54.0	3.3
22D	1.25	8.03	4.2	0.81	3.35	0.48	783	19.8	0.3	43.5	51.2	5.0
23S	1.34	7.79	3.0	0.39	2.30	2.37	413	14.7	0.0	68.2	31.0	0.8
23D	1.74	7.96	0.1	0.13	0.95	1.18	224	3.5	0.8	83.3	14.2	1.7
24S	1.26	7.84	5.5	0.77	2.97	0.85	462	26.6	0.3	67.2	29.2	3.3
24D	1.69	8.14	0.7	0.40	2.87	0.52	203	16.2	0.0	51.2	31.3	17.5
25S	1.52	8.19	0.8	0.36	2.77	0.38	324	9.5	0.0	56.3	41.2	2.5
25D	1.60	8.37	0.1	0.31	2.56	0.15	286	15.4	0.0	49.5	47.2	3.3
26S	0.66	7.92	3.4	1.01	3.50	0.62	563	29.9	0.2	43.7	52.6	3.5
26D	0.95	7.57	10.2	1.81	4.37	0.80	665	40.5	0.3	35.5	55.8	8.3
27S	1.54	8.01	1.5	0.35	3.26	1.26	278	25.4	0.2	69.8	28.3	1.7
27D	1.54	7.84	1.1	0.58	3.17	0.91	265	41.7	0.2	58.3	34.8	6.7
28S	1.16	7.71	5.9	0.99	3.75	1.19	499	29.1	0.2	47.5	46.5	5.8
28D	1.53	7.98	1.7	0.85	3.30	0.36	328	24.1	0.0	49.2	40.8	10.0
29S	1.17	7.88	1.6	0.82	3.18	0.47	444	15.9	0.5	54.5	42.5	2.5
29D	1.18	7.78	4.0	1.39	3.60	0.64	449	29.7	2.3	29.8	59.5	8.3
30S	0.61	7.61	10.6	0.64	4.03	0.82	977	47.7	0.8	45.2	49.8	4.1
30D	1.52	7.60	4.7	0.47	3.82	0.71	638	32.0	10.0	37.7	47.3	5.0
31S	1.37	8.26	0.1	0.32	1.07	0.79	163	2.3	6.0	83.5	8.8	1.7
31D	1.96	8.32	0.2	0.40	1.51	0.77	206	6.8	3.5	57.8	37.0	1.7
32S	0.76	7.42	18.4	1.16	3.22	1.37	573	17.9	13.2	44.1	40.9	1.8
32D	1.15	7.76	2.5	0.63	2.47	1.04	400	10.1	5.5	54.8	38.0	1.7
33S	0.72	7.85	6.5	0.69	3.35	1.62	444	20.1	0.8	59.0	38.5	1.7
33D	1.66	7.77	2.1	0.73	2.62	1.27	306	18.3	0.3	39.8	42.3	17.5
34S	1.50	8.06	2.2	0.97	3.24	0.55	397	10.9	5.3	46.7	44.7	3.3
34D	1.66	8.19	0.9	0.73	3.04	0.33	314	9.0	4.8	50.2	42.5	2.5
35S	1.04	8.23	0.4	0.76	2.90	0.35	254	15.0	5.2	29.2	53.2	12.5
35D	1.84	8.30	0.2	0.60	2.54	0.58	348	8.1	6.5	47.0	41.5	5.0
36S	1.24	8.28	0.7	0.43	2.79	0.12	127	31.8	0.2	18.5	56.3	25.0
36D	2.01	8.59	0.2	2.30	2.54	0.22	267	6.1	42.5	35.3	18.0	4.2
37S	1.08	7.63	17.2	1.79	4.42	0.86	677	19.2	6.7	37.2	53.7	2.5
37D	1.44	8.11	2.9	1.31	3.63	0.57	438	16.4	10.7	39.2	46.8	3.3
38S	1.32	7.67	5.9	1.24	3.95	0.68	532	15.7	0.5	40.7	55.5	3.3
38D	1.66	8.02	0.9	1.96	3.42	0.15	298	20.9	0.2	34.7	50.2	15.0
39S	1.06	7.96	6.6	0.66	3.93	0.84	712	59.0	2.0	42.5	53.8	1.7
39D	1.26	8.01	3.2	0.54	12.70	0.73	440	51.1	1.5	37.0	58.2	3.3
40S	1.28	7.81	5.7	0.95	6.75	1.97	805	45.0	3.0	46.5	48.0	2.5
40D	1.47	8.06	3.0	0.80	3.39	1.85	588	42.3	3.0	48.5	46.0	2.5

Appendix E
Phosphorus Fractionation Data

Table 13. Results of Phosphorus Fractionation

Sample	Total P μg/g	P _{bioavailable} μg/g	P _{Al,Fe} μg/g	P _{Ca} μg/g	P _{organic} μg/g	P _{bioavailable} %	P _{Al,Fe} %	P _{Ca} %	P _{organic} %
1S	406	6.3	4.9	102.5	292.3	1.56	1.20	25.25	71.99
1D	261	3.1	2.4	106.5	149.0	1.20	0.91	40.82	57.08
2S	330	3.4	2.0	58.1	266.5	1.04	0.62	17.59	80.74
2D	187	1.4	0.7	47.0	137.5	0.73	0.35	25.22	73.71
3S	427	6.6	8.5	26.0	385.9	1.54	1.99	6.09	90.38
3D	313	3.2	3.2	24.0	282.6	1.03	1.02	7.67	90.28
4S	266	4.9	2.4	47.5	211.3	1.84	0.89	17.85	79.42
4D	196	3.5	1.6	47.0	143.8	1.81	0.83	23.98	73.38
5S	485	3.3	2.4	81.5	397.8	0.69	0.49	16.80	82.02
5D	252	1.1	0.5	77.5	172.9	0.46	0.19	30.74	68.61
6S	310	1.8	2.6	89.5	216.1	0.57	0.84	28.88	69.70
6D	532	2.7	2.9	69.0	457.4	0.50	0.54	12.97	85.99
7S	191	1.3	1.3	46.0	142.4	0.66	0.68	24.09	74.57
7D	182	1.1	1.5	47.0	132.3	0.63	0.85	25.82	72.70
8S	168	1.1	0.7	95.0	71.1	0.68	0.44	56.57	42.31
8D	207	1.3	0.7	52.5	152.5	0.61	0.35	25.37	73.67
9S	266	2.9	1.6	45.5	215.9	1.10	0.61	17.11	81.17
9D	130	1.5	1.0	44.5	83.0	1.12	0.75	34.26	63.86
10S	693	6.2	3.7	85.5	597.6	0.89	0.54	12.33	86.24
10D	406	2.7	2.2	63.5	337.6	0.67	0.54	15.65	83.14
11S	331	6.4	5.4	32.0	286.7	1.93	1.63	9.69	86.76
11D	329	2.9	2.8	36.5	286.8	0.89	0.84	11.10	87.17
12S	420	2.7	2.5	47.8	367.0	0.63	0.60	11.38	87.39
12D	295	1.7	1.1	61.5	230.7	0.57	0.39	20.86	78.19
13S	405	2.6	2.2	75.5	324.6	0.64	0.54	18.65	80.16
13D	328	1.6	1.1	79.0	246.3	0.48	0.35	24.09	75.09
14S	353	3.6	2.0	77.1	270.4	1.01	0.58	21.83	76.59
14D	229	1.7	0.8	54.5	172.0	0.73	0.36	23.79	75.12
15S	327	2.6	1.5	80.5	242.3	0.80	0.47	24.62	74.11
15D	225	1.5	0.5	56.8	166.2	0.67	0.24	25.23	73.86
16S	231	2.6	2.0	55.5	170.9	1.13	0.85	24.04	73.98
16D	396	2.3	1.6	57.5	334.6	0.58	0.41	14.52	84.49
17S	553	3.7	3.8	91.0	454.6	0.66	0.68	16.46	82.20
17D	330	1.3	1.0	85.5	241.8	0.38	0.30	25.95	73.38
18S	716	4.7	3.5	81.5	626.3	0.66	0.49	11.38	87.47
18D	511	2.1	1.9	75.5	431.5	0.41	0.37	14.78	84.44
19S	690	5.1	3.4	57.5	623.9	0.74	0.50	8.34	90.42
19D	444	1.9	1.2	54.5	386.4	0.42	0.28	12.27	87.03
20S	313	6.9	7.5	28.8	269.8	2.22	2.40	9.19	86.20
20D	279	4.4	3.8	27.0	243.8	1.57	1.37	9.68	87.37

Appendix E
Phosphorus Fractionation Data

Table 13. Continued

Sample	Total P μg/g	P _{bioavailable} μg/g	P _{Al,Fe} μg/g	P _{Ca} μg/g	P _{organic} μg/g	P _{bioavailable} %	P _{Al,Fe} %	P _{Ca} %	P _{organic} %
21S	470	4.1	3.2	72.5	390.2	0.87	0.68	15.43	83.02
21D	352	2.6	2.5	95.0	251.9	0.74	0.72	26.98	71.57
22S	486	4.5	3.8	103.0	374.7	0.92	0.79	21.20	77.09
22D	783	3.0	4.5	120.0	655.5	0.39	0.57	15.32	83.72
23S	413	4.8	4.7	49.0	354.5	1.16	1.14	11.86	85.83
23D	224	1.3	1.5	51.5	169.7	0.56	0.69	22.99	75.76
24S	462	4.9	3.3	66.6	386.8	1.06	0.71	14.42	83.81
24D	203	1.1	0.8	47.5	153.6	0.57	0.38	23.41	75.65
25S	324	1.8	1.1	86.0	235.1	0.55	0.35	26.54	72.56
25D	286	0.9	0.6	89.1	195.4	0.33	0.20	31.14	68.33
26S	563	4.6	3.3	127.1	428.0	0.82	0.59	22.57	76.02
26D	665	6.6	7.0	89.5	561.9	0.99	1.05	13.46	84.49
27S	278	1.3	1.3	69.0	206.4	0.45	0.47	24.82	74.26
27D	265	1.0	0.2	67.5	196.2	0.39	0.09	25.47	74.05
28S	499	4.7	4.3	94.5	395.5	0.94	0.87	18.93	79.26
28D	328	1.6	1.0	84.5	240.9	0.48	0.30	25.78	73.45
29S	444	2.7	1.9	108.8	330.6	0.61	0.43	24.50	74.46
29D	449	3.2	2.6	76.5	366.6	0.72	0.58	17.04	81.66
30S	977	15.7	5.7	124.5	831.1	1.60	0.58	12.75	85.07
30D	638	4.0	2.3	112.9	518.8	0.62	0.36	17.70	81.32
31S	163	1.1	0.7	44.0	117.2	0.70	0.40	27.00	71.90
31D	206	1.1	0.7	46.5	157.7	0.56	0.32	22.57	76.55
32S	573	8.0	8.3	53.5	503.1	1.40	1.45	9.34	87.81
32D	400	2.4	2.3	84.5	310.8	0.60	0.57	21.13	77.70
33S	444	3.8	3.1	60.5	376.6	0.85	0.70	13.63	84.83
33D	306	1.5	0.7	59.5	244.3	0.48	0.24	19.45	79.83
34S	397	2.5	1.6	82.5	310.4	0.63	0.41	20.78	78.17
34D	314	1.3	1.1	89.0	222.6	0.40	0.35	28.35	70.90
35S	254	1.7	0.3	64.0	188.0	0.66	0.13	25.19	74.03
35D	348	1.0	1.1	82.0	263.9	0.30	0.30	23.56	75.84
36S	127	1.5	0.2	22.5	102.9	1.15	0.13	17.72	81.00
36D	267	1.0	0.6	81.6	183.8	0.39	0.21	30.55	68.85
37S	677	5.6	5.4	60.5	605.4	0.83	0.80	8.94	89.43
37D	438	1.6	1.1	75.0	360.3	0.36	0.26	17.13	82.26
38S	532	3.0	3.4	97.8	427.3	0.57	0.64	18.40	80.39
38D	298	1.3	0.7	87.5	208.6	0.42	0.22	29.35	70.01
39S	712	4.6	3.0	81.0	623.4	0.65	0.42	11.38	87.55
39D	440	2.4	1.1	64.7	371.7	0.55	0.26	14.71	84.48
40S	805	5.4	3.3	76.0	720.3	0.67	0.41	9.45	89.47
40D	588	3.4	1.6	74.1	508.9	0.59	0.28	12.59	86.54

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