

Technical Report No: ND13-02

**Tile drain water: identification of sources
and quality improvement by a constructed wetland**

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June 2012

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Tile drain water: identification of sources and quality improvement by a constructed wetland

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The work upon which this report is based was supported in part by federal funds provided by the United States of Department of Interior in the form of ND WRI Graduate Research Fellowship for the graduate student through the North Dakota Water Resources Research Institute.

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Project Period: March 1, 2012 – February 28, 2013

Project Number: 2012ND254B

**North Dakota Water Resources Research Institute
Director, G. Padmanabhan
North Dakota State University**

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Abstract

North Dakota clay soils have naturally high concentrations of sulfate. With the increasing popularity of putting tile drains into agricultural fields, this sulfate can be released into the environment. Sulfate, when chemically reduced to sulfide, can displace phosphate bound to soil particles. Phosphate, often a limiting reagent for growth in aquatic systems, can cause eutrophic conditions. Treatment wetlands can be built to intercept and mitigate elevated sulfate concentrations and the subsequent release of phosphate through the use of anaerobic and aerobic environments with the help of soil, organic matter, and wetland plants. These treatment wetlands can also provide additional revenue to landowners through the growth of willow trees that can be used as biofuel for the production of heat and or electricity via biogasification.

Acknowledgements

We are very thankful to the North Dakota Water Resources Research Institute, North Dakota State Water Commission, ND USDA-CIG, ND INBRE, EPA-319, NDSU Department of Biological Sciences, NDSU Environmental and Conservation Sciences Graduate Program.

We would like to personally thank Dr. Wei Lin for his treatment wetland design. We would also like to thank Mr. Michael Ell of the North Dakota Department of Health and Mr. Steve Fischer formerly of the NRCS. Also, a special thanks to Dr. La Toya Kissoon, Dr. Sharmila Sunwar, Dr. Dimuthu Wijeyaratne, Carrie Werkmeister, Alex Yellick, and Aida Asgary for their help and support through this research.

Introduction

Public view of wetlands has changed drastically over the past half century. Prior to the mid-1970's wetlands were treated as unusable portions of land within many countries and often drained for agricultural or urban purposes (Mitsch & Gosselink, 2007). This all changed with the passing of the Clean Water Act of 1972, under Section 404 there is to be 'No Net Loss' of wetlands within the United States (National Research Council, 2001). The phrase 'No Net Loss' implies that any wetland changed from its natural function will be replaced via mitigation processes by either restoring or constructing a wetland of 'equal value' elsewhere at the landowners expense (National Research Council, 2001). The United States has yet to achieve this goal, but losses from agricultural practices and nonagricultural between 1986 and 1997 dropped 90% and 63% respectively (National Research Council, 2001). Since the advent of the Clean Water Act there have been efforts to help educate the public, including farmers, through outreach programs to show the effectiveness and importance of natural, restored, and constructed wetlands (Lemke et al., 2010).

The implementation and research of treatment wetlands is a relatively new idea (Mitsch & Gosselink, 2007). The field stems from two different approaches, which are: German scientists researching the use of macrophytes for wastewater improvements and United States scientists studying the role of wetlands in their natural setting and the part they play in water and nutrient cycling, in the 1950's and 1970's respectively (Mitsch & Gosselink, 2007). The merger of these two ideas spawned the field of 'treatment wetlands' (Mitsch & Gosselink, 2007) and has since diversified into nutrient removal, agricultural runoff mitigation, hydrological buffers, ecological restoration, graywater treatment, and many other applications (Budd et al., 2011; Erler et al., 2011; Mitsch & Day, 2006). Both natural and constructed wetlands can be considered 'treatment wetlands' (Vought & Lacoursiere, 2002; Higgins, 2003).

The Midwestern United States has become heavily modified through ditching and tile draining; Minnesota alone has 21,000 miles of ditches and channelized streams; the United States, as of 1990, has drained a total of 43,500,000 ha of land (Sands, 2010; Mitsch & Gosselink, 2007). It is estimated that approximately 98% of North America's prairie and forests have been turned into crop land (Blann et al., 2009). Tile draining a field also increases the amount of nutrients lost from the soil as well as pesticides (Lennartz et al., 2010; Kladvko et al., 1999). Of these nutrients sulfate can be released and cause water quality issues related with its chemical reduction.

Sulfates are common in North Dakota soils as salt compounds, mostly as sodium sulfate (Na_2SO_4), calcium sulfate (CaSO_4), and magnesium sulfate (MgSO_4) (Franzen, 2007). Compounds such as sodium sulfate easily dissociate in water and release sulfate into the environment. With draining activities such as ditching and tile draining sulfate becomes a much larger concern (Ekholm & Lehtoranta, 2012). The dilemma at hand is how to cope with increasing need for higher crop production, which tiling a field can improve, and mitigating downstream eutrophication via the chemical reduction of sulfate to hydrogen sulfide and the subsequent release of phosphate into the watershed. A role which constructed treatment wetlands can fill within the Midwestern United States.

These systems when combined would make a treatment wetland capable of chemically reducing sulfate, binding subsequent released phosphate to soil, and produce higher quality water than what entered the treatment wetland. But the truth of the matter is that most landowners, when mitigating wetland loss, aren't looking to, or even know about, these processes. So there needs to be another way of grabbing their attention. By utilizing a treatment wetland as a potential source of income for the landowner by producing biofuel, landowners may be more likely to adopt such systems. A potential plant family that could be used for biofuel applications is that of *Salicaceae* or more commonly known as the willow family. There is a dire need around the globe to find sources of bioenergy that are not agricultural crops, due to higher populations, more and more crop production is needed for food

(Schmidt-Walter & Lamersdorf, 2012). The European Union projects that to meet their goal of 20% renewable energy by the year 2020, which is a goal they can meet but at a high cost, approximately 85% of their current agricultural crops would have to be allocated towards biofuel production (Schmidt-Walter & Lamersdorf, 2012). This is why non-agricultural crops, such as switch grass and possibly willow trees, are being researched heavily for their biofuel applications and possibilities (Schmidt-Walter & Lamersdorf, 2012).

By allocating land that would normally be kept out of production for crops, due to soil type or position within the landscape, towards possible biofuel opportunities, we may be able to move towards a much more sustainable energy economy. Willow trees would be ideal for a treatment wetland system because they are adapted to many hydrologic settings, from dry sandy soils all the way up to inundated wetlands (Savage & Cavender-Bares, 2012). This means that willow trees could serve as a model species for biofuel production and potentially grown in almost all hydrologic settings throughout an agricultural environment. Land that would normally be drained for crop production could be allocated towards the production of willow trees for biofuel applications, instead of a traditional food crop, as well as increase the farmers income. A willow harvest is commonly referred to as a 'coppice' or the act of cutting the trees as 'coppicing' (Schmidt-Walter & Lamersdorf, 2012).

Another ideal quality of *Salix sp.* is that they undergo vegetative reproduction or the ability to grow new shoots when cut (Radtke et al., 2011). This amazing ability allows willow trees to overcompensate, after being coppiced, by producing more stems than then previous year, effectively increasing potential crop yield over time (Radtke et al., 2011). Willow trees could serve as a biofuel for farmers by being burned for heat or electricity via biogasification, which is primarily researched using corn stover (Zheng et al., 2009). Biogasification works by digesting plant material through anaerobic processes, and as a byproduct, produces biogas (Zheng et al., 2009). This gas can then be burned to

generate steam, which can be used to generate electricity via electrical turbines. For a landowner to have a biogasification facility on-site is highly unlikely. However, rural communities could band together to produce biomass for such facilities and be paid for their efforts. If all else fails, the biomass can be used to generate heat for the farmer and their family and mitigate their standard heating costs over colder months.

Study area

This project is primarily done at a treatment wetland at the Discovery Farm near Embden, ND (Figure 1). The treatment wetland is approximately 0.034ha in area. The treatment wetland intercepts tile drain water from the nearby agricultural field via a basin that collects water at the tile drain outlet. The study site was excavated and built with a clay liner to reduce ground seepage, a large basin and PVC pipes to direct flow to the treatment wetland, a parent soil and organic matter (decomposed bovine manure) mixture to facilitate the growth of anaerobic bacteria within the first treatment cell, cinder blocks to create walls to increase residence time, and wetland plants (*Typha latifolia* and *Salix viminalis* QC83) to increase the aerobic environment within the second treatment cell via radial oxygen loss (ROL) via their root systems (Figure 2).



Figure 1. Treatment wetland, Discovery Farm near Embden, ND. Treatment wetland targets removal of sulfate and phosphate from agriculture tile-drainage. Red square approximate treatment wetland area.

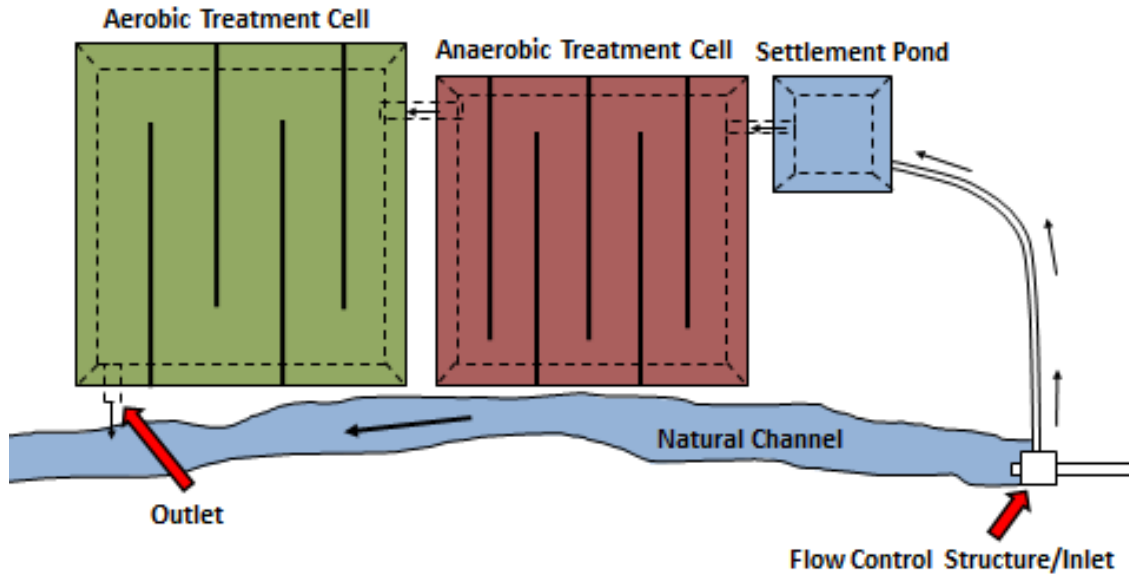


Figure 2. Treatment wetland layout including the Inlet, Settlement Pond, Anaerobic Treatment Cell, Aerobic Treatment Cell, and the Outlet.

Aims, hypotheses and objectives of the project

The overall aims of this project are to demonstrate the effectiveness of the treatment wetland to chemically reduce the concentration of sulfate within the agricultural tile drain water and to show the efficacy of using a hybrid species of willow (*Salix viminalis* QC83) for biofuel applications.

It was expected that,

1. There is a detectable concentration of sulfate within the agricultural tile drain water.
2. The willow tree *Salix viminalis* QC83 would grow within the North Dakota climate.

The objectives of this study were,

1. To assess the ability of the treatment wetland to reduce the chemical concentration of sulfate and phosphate.
2. To assess the biofuel applications of *S. viminalis* QC83 via biogasification.

Methodology

Treatment wetland sampling

At each sampling point three samples will take at random locations using VWR 150ml plastic bottles. There are five sampling locations: Inlet (IN), anaerobic treatment cell (AN), aerobic treatment cell (AE), outlet (OUT), and the Maple River (RV). To collect each sample, submerge bottle underwater while capped, unscrew cap while underwater to minimize the samples exposure to air, and screw the cap back on while underwater. Samples are then put on ice until they can be processed for [sulfate] (turbidimetric), [phosphate] (colorimetric), total S & total P (ICP-OES).

Well sampling

Remove the cap from the pipe and insert siphon tube (6mm I.D., 11mm O.D.). Wells should be siphoned dry; samples will consist of redraw into the well to account for actual groundwater and not stagnant water. Three samples of 150ml into VWR plastic bottles will be taken at each well. Samples are then put on ice until they can be processed for [sulfate] (turbidimetric), [phosphate] (colorimetric), total S & total P (ICP-OES).

Greenhouse wetland sampling

Model wetlands placed within plastic containers and filled with soil from the anaerobic treatment cell. Model wetlands are then saturated with water from tanks filled with one of two different concentrations of sulfate; 'High' 250 mg/l, and 'Low' 80 mg/l.

One sample per model wetland should be taken twice per week. Place a 20ml scintillation vial under the outlet. Wait until vials are at least 90% full before removing them from underneath the outlet. At least 11ml of sample is needed for analysis. Samples should then be prepared using sulfate – turbidimetric and phosphate – colorimetric methods for their respective analysis.

Sulfate analysis - Turbidimetric

Samples are initially collected using a Kimble 20ml Scintillation Vials. Samples are then filtered through a PALL Supor-405 0.45 μ m 25mm filter using a PALL 25mm Easy Pressure Syringe Filter Holder and a BD 60ml Slip Tip Syringe. Filtered samples are then placed within a VWR 60ml plastic bottle. Samples are then fixed via 2 drops of ACS grade concentrated nitric acid per bottle. One milliliter of filtered sample is then placed within a 15ml centrifuge tube and 2ml of buffer solution are then added to the same tube. The buffer solution is produced by: dissolving 1.5g of $MgCl_2 \cdot 6H_2O$, 0.25g $CH_3COONa \cdot 3H_2O$, 50mg KNO_3 , and add 1ml of CH_3COOH (99%) in 25ml distilled water made up to 50ml. Invert the centrifuge tube several times to mix. Then add a small scoop of BaCl crystals (20 to 30 mesh) to each centrifuge one sample at a time (add to one, analyze, then add to the next). Once crystals have been added mix individual centrifuge tube for one minute and pour 2ml of contents into a cuvette for analysis. The cuvette is then placed within a Helios λ Spectrophotometer, set at $\lambda 420nm$, and against a blank of deionized water. An initial reading is then taken and every 30 seconds another reading will be taken up to 5 minutes. After 5 minutes use the highest reading and disregard the others.

Phosphate analysis - Colorimetric

Samples are initially collected using a Kimble 20ml Scintillation Vials. Samples are then filtered through a PALL Supor-405 0.45 μ m 25mm filter using a PALL 25mm Easy Pressure Syringe Filter Holder and a BD 60ml Slip Tip Syringe. Filtered samples are then placed within a VWR 60ml plastic bottle. Samples are then fixed via 2 drops of ACS grade concentrated nitric acid per bottle. Ten milliliters of sample are then added to a 15ml centrifuge tube and 1ml of mixed reagents is added to the same centrifuge tube. There are four reagents to produce to make the 'mixed reagents' they are called: a, b, c, and d. The reagents are produced by: a, dilute 38.9ml of H_2SO_4 within 250ml; b, dissolve 1.5g ammonium molybdate $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 50ml dH_2O ; c, dissolve 2.7g ascorbic acid in 50ml dH_2O ; and d, dissolve 0.34g potassium antimonyl tartrate in 250ml dH_2O . Once made, the reagents are mixed

within the following ratio for a:b:c:d, 10:4:4:2. Once the mixed reagents have been added to the 10ml of sample, allow the sample to sit for a minimum of 30 minutes but analyzed before 12 hours. Place 2ml of sample into a cuvette and ran on a Helios λ Spectrophotometer, set at λ 885nm, and against a blank of deionized water.

Reduction/Oxidation - Redox

Redox readings were taken with a SympHony multi-meter and a redox probe in the field. The probe should first be calibrated within the lab before heading to the field. Calibrate the probe using a two point calibration via solutions at pH 4 and 7. Second wet probe with deionized water when in the field and clear the ground of excess debris. Insert probe into the soil/parent material and take the redox reading in mV.

Inductively Coupled Plasma Optical Emission Spectrometry – ICP-OES

Water samples (5ml minimum) were analyzed for element concentrations of the following elements: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, SiO₂, Sn, Sr, Ti, Tl, V, Zn, Zr via Spectro Genesis ICP-OES with the SmartAnalyzer program.

Biomass

A native willow species, *Salix exigua*, was harvested to achieve a length:mass ratio. All trees were cut 12 cm from the base of the main stem to account for differences in the topography and to normalize the cutting procedure. Tree length was a composite measurement of main stem length and all branches. To determine mass the trees were dried at 48.8°C in a walk-in drier for a week. The dried trees were then measured with a hand scale for mass. This relationship can then be related to an energy:mass ratio. The dried trees are to be processed at the University of Minnesota Morris campus at their biogasification plant. Through the gasification process we will be able to relate how much energy is produced per unit mass. This is to be completed in the spring of 2013.

Results and Discussion

Treatment wetland

The treatment wetland showed sulfate concentrations averaging 56.59 ± 19.0 mg/l amongst all sample locations throughout the 2012 field season. The four sample locations are the **Inlet**, **Anaerobic** treatment cell, **Aerobic** treatment cell, and the **Outlet** (Figure 3). The averages for these sample locations for the entire field season were; 79.5 ± 54.9 , 50.2 ± 4.12 , 48.0 ± 7.90 and 48.8 ± 9.14 mg/l respectively. It should be noted that the first sample date, May 22nd, only shows data for the Inlet. May 22nd was the first record of water flowing from the tile drain into the settlement pond. Also, sample dates June 20th and June 22nd show sulfate concentrations which are possibly artificially elevated due to the lack of water (Figure 3). By the end of June, 2012 there was no water entering the system due to lack of water throughout the system and sulfate may have become more concentrated as water began to evaporate from the treatment wetland. This can explain why the standard deviation for the Inlet is so high (79.5 ± 54.9 mg/l).

Over the course of the 2012 field season it can be seen that the sulfate concentrations entering the system do not significantly decrease by the time they exit. However, on May 31st, sulfate concentrations began to significantly decrease from 49.63 mg/l at the Inlet to 39.75 mg/l at the outlet (Figure 3). Although this is a minor decrease it is a statistically significant decrease. Based on these results alone, it is hard to determine whether the treatment wetland is efficiently lowering the concentration of sulfate from the tile-drainage. When we look at the concentrations of phosphate over time within the treatment wetland (Figure 4) we see another interesting relationship.

Phosphate, as discussed earlier, is displaced from soil particles as a consequence of the chemical reduction of sulfate to sulfide. So, if we examine the increase of phosphate concentrations from the Inlet to the Anaerobic treatment cell (Figure 4) the data suggests that sulfate chemical reduction is taking place. For example, on May 31st, 2012, 16.3 ± 0.00 $\mu\text{g/l}$ phosphate was found at the Inlet and

440±17.1 µg/l phosphate was measured within the Anaerobic treatment cell. This shows that, even though we don't see a large significant difference between sulfate concentrations entering and exiting the treatment wetland, sulfate chemical reduction is taking place (Figure 3). The data suggests this due to the increase in phosphate concentrations (Figure 4). On May 31st, 2012 the phosphate concentrations measured at the Outlet, 215±2.55 µg/l, is a significant drop from the Anaerobic treatment cell, 440±17.1 µg/l, but is a significant relative to what entered the system at the Inlet, 16.3±0.00 µg/l (Figure 4). This shows that the treatment wetland was reducing the levels of phosphate released through the chemical reduction of sulfate by approximately 50%. However, the entire system is netting an increase in phosphate concentrations by over tenfold (Figure 4). By June 15th, 2012 these amounts changed to 66% reduction of phosphate and a net increase in phosphate concentrations of under twofold (Figure 4). This trend suggests that if there were a longer wet period during the summer of 2012 that the system may have eventually net a reduction in total phosphate.

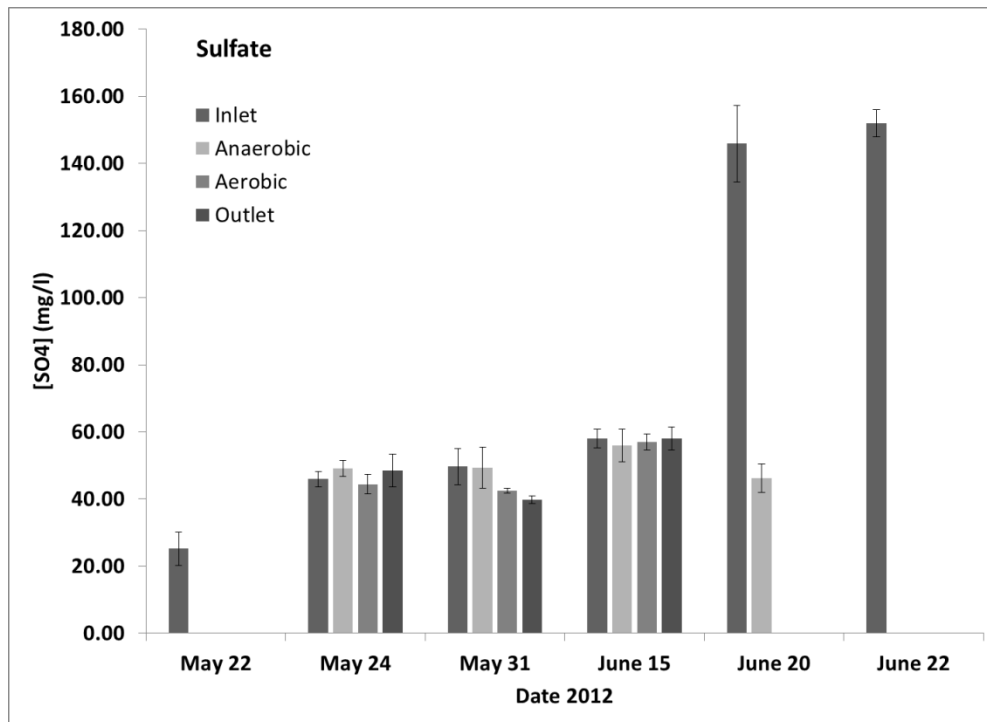


Figure 3. Sulfate concentrations over time within the treatment wetland. Samples are taken at the Inlet, Anaerobic treatment cell, Aerobic treatment cell, and Outlet. n=3 per sample location per date.

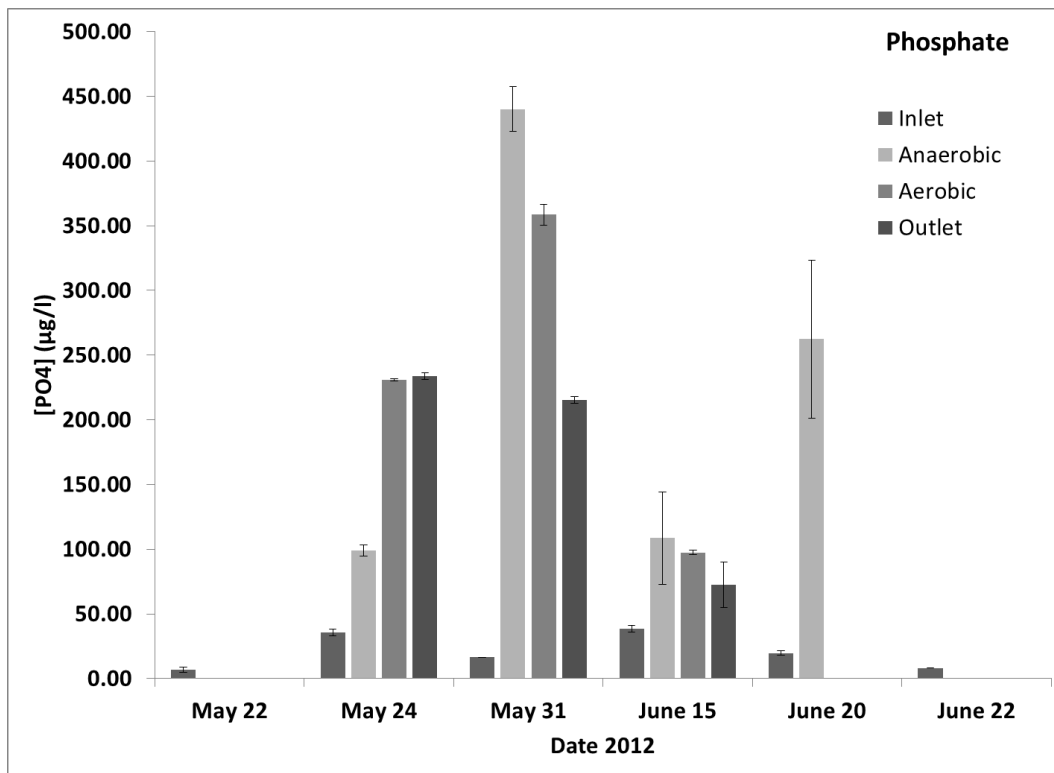


Figure 4. Phosphate concentrations over time within the treatment wetland. Samples are taken at the Inlet, Anaerobic treatment cell, Aerobic treatment cell, and Outlet. n=3 per sample location per date.

Ground wells

In addition to the treatment wetland sampling, ground wells were placed on site in three locations (Figure 5). The ground wells will help determine the nutrient loading on site by capturing groundwater within the North and South fields before they enter the treatment wetland. Based on data found in the summer of 2012, the North field, specifically near Well 2 at 448 ± 35.3 mg/l compared to 19.3 ± 1.56 mg/l (Well 1) and 224 ± 10.5 mg/l (Well 3), contributes the largest amount of sulfate to the tile drain system (Figure 6). It is interesting that these concentrations are so high relative to the concentrations of sulfate reported at the inlet of the treatment wetland. Further investigation could tease out whether this is due to dilution or if there is a loss of sulfate within the drain tile itself.

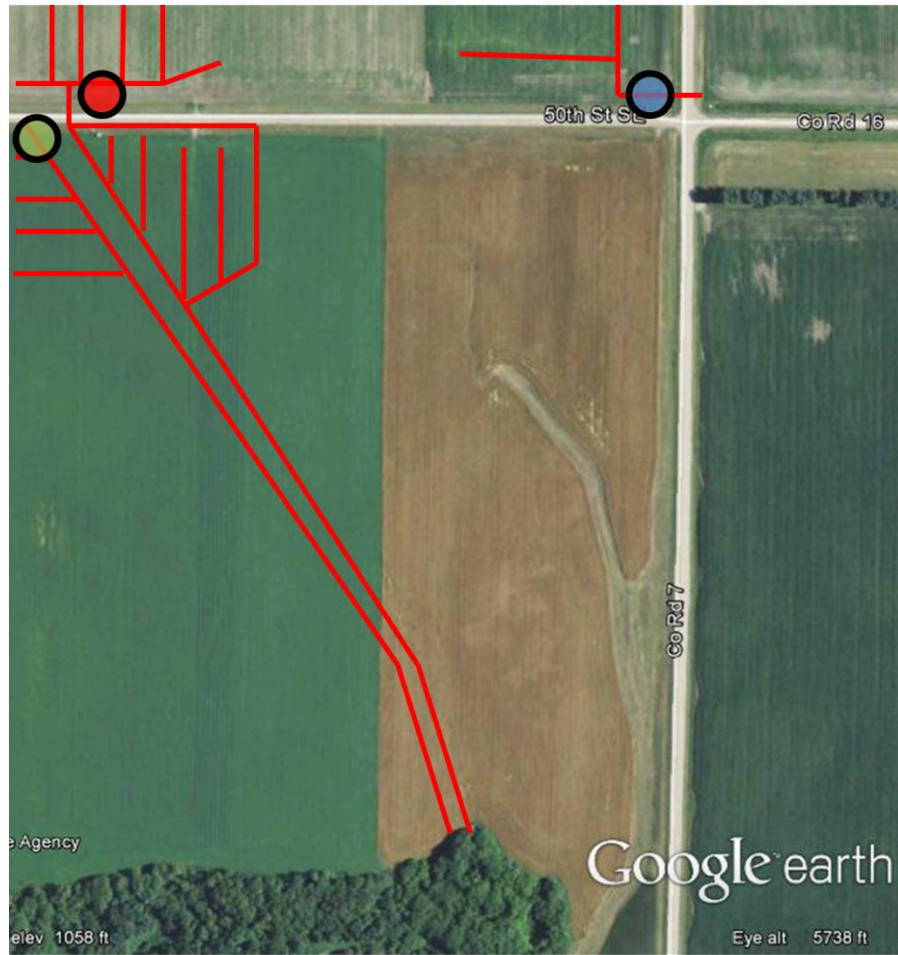


Figure 5. Discovery Farm near Embden, ND. Circles represent ground wells on site. From right to left: Well 1 (blue), Well 2 (red), Well 3 (green). Wells 1 and 2 are located in the North field and Well 3 is located within the South field. Red lines signify approximate underground tile drain pattern.

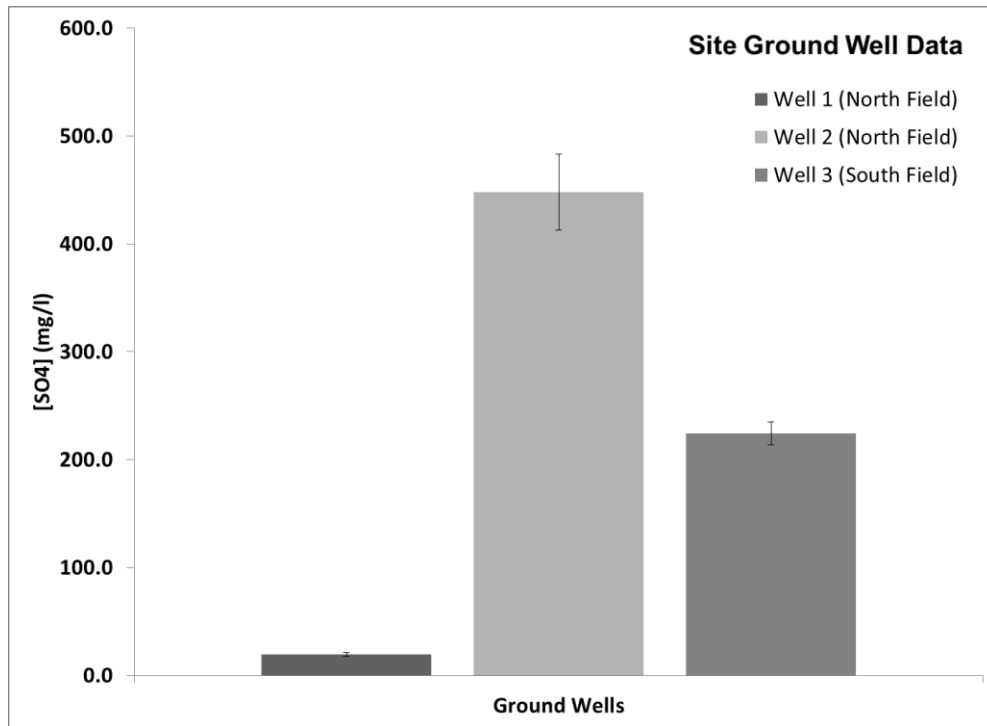


Figure 6. Sulfate concentrations from ground wells on site with treatment wetland. Wells located in North and South fields on site.

Greenhouse wetlands

To compensate for the lack of water during the summer of 2012 a greenhouse study involving model treatment wetlands was conducted. The main goal of this experiment was to assess the efficiency of the soil within the treatment wetland to remove sulfate and phosphate from tile-drainage. The experiment consisted of two treatments; a) high sulfate concentration, 250 mg/l and b) low sulfate concentration, 80 mg/l. These concentrations were chosen because 250 mg/l was slightly higher than any recorded natural sulfate concentration on site, and 80 mg/l because it was approximately the average sulfate concentration found entering the treatment wetland during the summer of 2012. The High treatment seems to be too much sulfate for the soil to efficiently handle. The percent removal quickly dropped from over 30% to under 10% by the second sample period (Figure 7). However, the efficiency of the model wetlands came back up to 30% by December 17th. It should be noted that some of the treatment wetlands became dry at this time and may have become aerobic for a period of time

thus increasing the efficiency of the model wetlands for a brief period of time. The Low treatment maintained a percent removal around 30% for 4 sampling periods and then fell around 20% by December 17th (Figure 7). After December 17th, 2012, when the model wetlands went dry, it took almost a month for the percent removal to bounce back above 20% (Figure 7).

The data seems to suggest that there is a threshold for the sulfate reducing potential of the wetland’s soil. At 80 mg/l sulfate the model wetlands removed sulfate at approximately 30% removal for 5 samplings, whereas the 250 mg/l sulfate solution only removed sulfate at 30% efficiency for one week before dropping down to under 10% (Figure 7). This data suggests that the treatment wetland at the Discovery Farm near Embden, ND should have at least a percent removal of approximately 30%. Continued sampling during the 2013 field season should illuminate whether or not this relationship holds true.

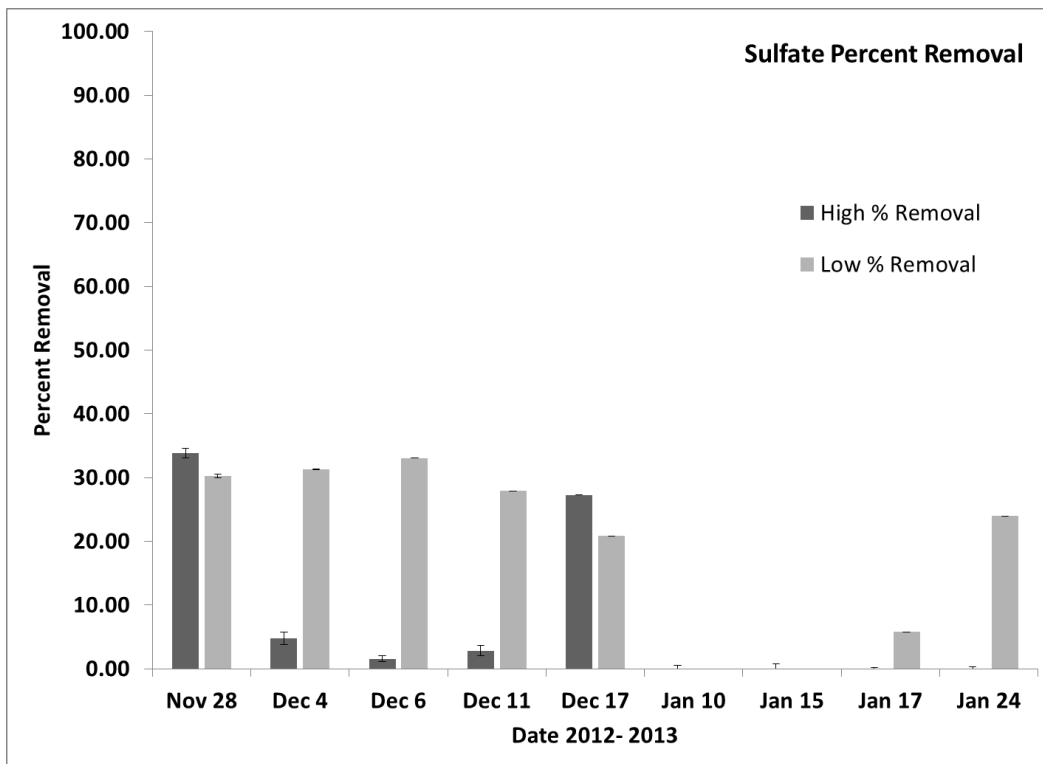


Figure 7. Greenhouse wetland sulfate percent removal over time. ‘High’ concentration at 250 mg/l and ‘Low’ concentration at 80 mg/l. n=3 per treatment per date.

Biomass experiment

Based on the tree data collected we were able to develop a length:mass ratio (Figure 8). After processing over 300 trees (n=341) the length:mass ratio produced has an R^2 value of 0.7 (Figure 8). This ratio is a first step in the process of developing a length:energy ratio. To do this we need to first establish a energy:mass ratio with the help of the biogasification plant at the University of Minnesota Morris. The two ratios can then be combined into a length:energy ratio so that trees may be measured in the field and their approximate caloric value can be determined without sacrificing the tree. This relationship can help determine the best harvest cycle for a stand of willow trees.

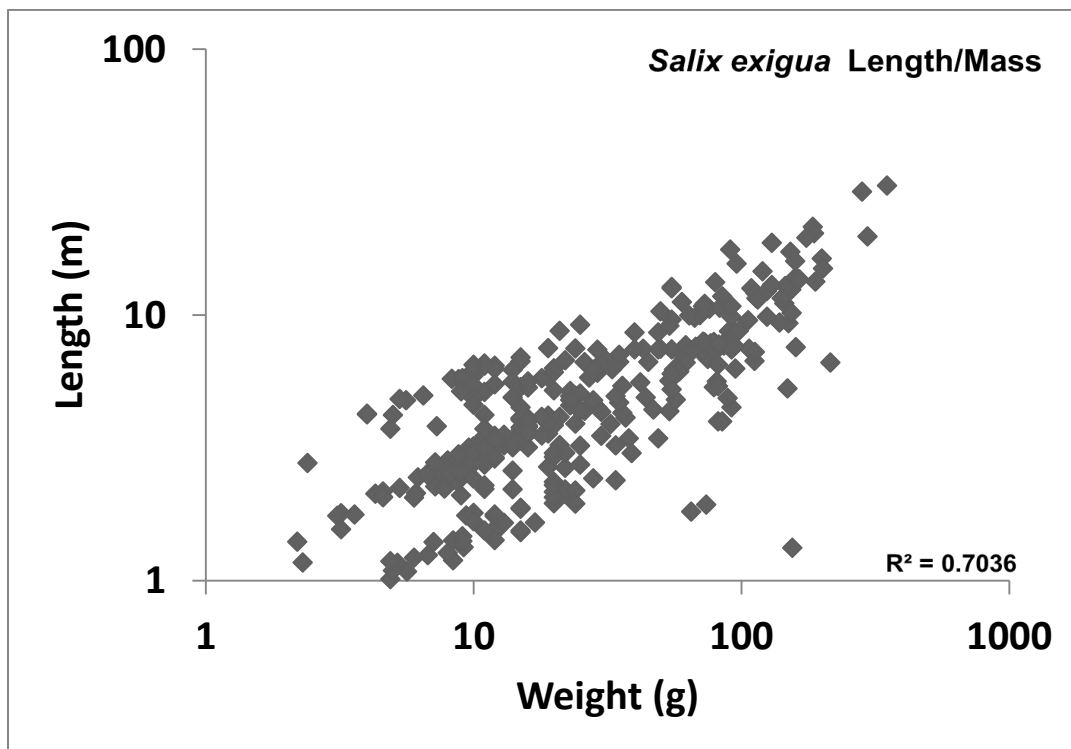


Figure 8. *Salix exigua* mass:length ratio. Length is composed of main stem length (m) and branch length (m). (n=341)

Conclusion

From the data we can see that the north field at the Discovery Farm near Embden, ND supplies the majority of the sulfate to the system. This is most likely due to the soil and a higher concentration of sulfate compounds. From the greenhouse experiments we can infer that the treatment wetland will have a percent sulfate removal of at least 30% or more. We say 'or more' because there is a possibility that the size of the greenhouse experiment limited the ability of the soil to remove sulfate from the water. Even though we had a limited sampling time at the treatment wetland in the summer of 2012 due to weather, we know that the wetland is working as intended. We can say this, not because of the sulfate concentrations recorded, because of the phosphate concentrations recorded. There would not be an increase of phosphate within the treatment cells if it was not being displaced by the chemical reduction of sulfate to sulfide. Finally, from initial data on the potential use of a *salix* species as a biofuel, we feel we have established a good length:mass ratio. This ratio will be related to a mass:calorie ratio so that in the future we will not have to sacrifice trees to know how much potential energy they will produce. This information will help us determine a proper harvest schedule for the trees. Continued sampling throughout the spring and summer of 2013 should shed more light onto the processes taking place within the treatment wetland.

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