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Lab-Scale Experiment for Assessing the Effect of Resuspension and Oxygenation on Sediment Oxygen Demand

Lab-Scale Experiment for Assessing the Effect of Resuspension and Oxygenation on Sediment Oxygen Demand

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in Biological Engineering

by

Grace Richardson University of Arkansas Bachelor of Science in Biological Engineering, 2008

August 2014 University of Arkansas

The thesis is approved for recommendation to the Graduate Council.

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Abstract

This study examined the effects of resuspending lake sediment for different time periods in a lab-scale tank under both oxic and anoxic conditions on sediment oxygen demand (SOD) and other related sediment properties. This lab-scale study was conducted as a first step to determine if a proposed method for reducing SOD by treating sediments in lakes and reservoirs is feasible. SOD is a critical process responsible for the formation of anoxic hypolimnia in lakes and reservoirs. A reduction in SOD may delay or eliminate the onset of anoxia in the hypolimnia, preventing adverse ecosystem effects and improving water quality and ecosystem function. The proposed treatment method for lakes and reservoirs would resuspend sediment into the water column and mix the suspension under aerated conditions such that near saturated DO conditions are maintained during mixing. The sediment is resuspended such that it is fully exposed to dissolved oxygen, thereby allowing oxygen-mediated chemical and biological reactions to proceed more rapidly without being rate-limited by oxygen availability as occurs within intact sediment. By maximizing oxygen uptake rates of sediment processes over a period of time, the oxygen consuming processes responsible for SOD may be partially quenched, thereby reducing SOD once the treated sediment has resettled. Current methods for oxygenation of sediments rely on oxygenation of overlying water such that oxygen diffuses into sediment. This process is typically conducted over a period of years. The method proposed may have economic benefits when compared to hypolimnetic water oxygenation treatments if the increased operating cost of rapid treatment is offset by a shorter overall required treatment time. The first step for testing the rapid method was done in a laboratory to determine if SOD is reduced due to the treatment and if oxygenation of sediments results in a greater decrease in SOD than resuspension without oxygenation. Bottom sediments were collected from a local eutrophic

reservoir, split into two samples, and then placed into 284 liter aquarium tanks. Sediment samples were resuspended for 3 hours, allowed to settle, resuspended for an additional 24 hours, allowed to settle, then resuspended for an additional 120 hours before being allowed to settle again. SOD, organic matter content, and sediment and water chemistry parameters were measured before and after each treatment period. Initial SOD in the lab experiments was greater than that measured in the lake. SOD was reduced an average of 32% over the course of the experiment, with no significant benefit from oxygenation during resuspension. Organic matter and other sediment quality parameters remained unchanged throughout the treatment. The concentration of all water quality parameters with the exception of Mn increased in the water column over the course of the treatment. Overall, the rapid oxygenation treatment method explored in this study did not appear to be an economically feasible alternative to existing longterm treatment methods of hypolimnetic oxygenation, but resuspension of sediments without oxygenation may have potential as a reservoir sediment remediation technique. However, due to the high degree of experimental error in this data, further studies must be conducted to determine if SOD reduction can be repeated under more closely controlled conditions. Further studies are also required to investigate the economic feasibility, potential benefits, and potential negative impacts of the resuspension treatment method.

Acknowledgements

I am so very thankful for Dr. Scott Osborn for giving me the opportunity to pursue this degree, and for graciously putting up with me and answering my constant questions over all these years. He has been my role model and mentor both in engineering and life. I would also like to thank my committee members: Drs. Julian Fairey, Brian Haggard, and Thad Scott for their valuable advice concerning the direction of my research. Additionally, without the help of Cameron Beyers, Thanh Thanh Dai, Shelby Owens, and Matthew McVey, I would most certainly have hurt myself or gotten myself stuck in mud or heavy machinery, and for them I am grateful.

My sincere thanks go to Keith Trost and others at the Arkansas Water Resource Center and UA Altheimer laboratories for processing a seemingly endless stream of samples. Most certainly, I would like to thank all of my friends and family including Mr. Mahmoud Sharara, Mr. Gurdeep Singh, Jeremy and Elizabeth Burns, Marcus Ward, and Elizabeth Richardson for providing me with continuous support and encouragement. And of course, I'd like to thank Ms. Linda Pate for having all the answers.

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Introduction

1.1 Background

1.1.1 Dissolved Oxygen

Low dissolved oxygen levels (hypoxia) in lakes, reservoirs, estuaries, and seas are a problem worldwide (UNEP, 2007). This hypoxia contributes to fish kills (Burton et al., 1908), the presence of metals (e.g. iron and manganese) and other reduced compounds (e.g. sulfides) in the water column (Sartoris and Boehmke, 1987), the release of nutrients (phosphorus (P) and nitrogen (N) compounds) from the sediment to the water column (Larsen et al., 1981; Cowan and Boynton, 1996), inefficiencies in hydropower production, and increased drinking water treatment costs (Monahan-Pendergast et al. 2008).

1.1.2 Eutrophication and Dissolved Oxygen

The USEPA, in its 2010 National Lakes Assessment (USEPA, 2010), estimates that over 40% of all lakes in the US are eutrophic or hypereutrophic because of a high concentration of nutrients. Just as with low dissolved oxygen levels, eutrophication can lead to nuisance and toxic algae, excessive plant growth, murky water, odors, fish kills, and increased drinking water treatment costs. Eutrophication is commonly measured using Carlson's (1977) trophic state index, which uses Secchi disk depth, total phosphorus, and chlorophyll-a concentrations to define the level of eutrophication on a scale of 1 to 100. A lake is considered oligotrophic when this value is below 40, mesotrophic when it is between 41-50, eutrophic when it is between 51-60, and hypereutrophic when it is greater than 60 (OWRB, 2002). The trophic index for some lakes in the Arkansas/Oklahoma region range from eutrophic to hypereutrophic (e.g. Lake Wister (Hession and Storm, 2000) and Lake Tenkiller, both in eastern Oklahoma (Nolen et al., 1989))

and from mesotrophic to eutrophic (e.g. Beaver Lake in Northwest Arkansas (Haggard et al., 1999)).

Eutrophication is particularly problematic in lakes when deeper waters of the hypolimnion become anoxic, releasing P stored in sediment into the overlying water. Therefore, for reservoirs containing P stored in sediments, preventing the formation of anoxic water in the hypolimnion can prevent the release of P into the overlying water and remove a source of P contributing to eutrophication (Cowan and Boynton, 1996).

1.1.3 Sediment Oxygen Demand

A critical factor responsible for the development of an anoxic hypolimnion is sediment oxygen demand (SOD). SOD is defined by Lee et al. (2000) as the rate of oxygen consumption exerted by the bottom sediment on the overlying water due to both the respiration of the benthic biological communities and the biochemical degradation of organic matter. This demand reflects decomposition of settling and deposited particulate organic matter that is formed mostly through the increase in primary productivity from eutrophication (Matthews and Effler, 2006). As organic matter accumulates over time, the SOD is increased due to benthic microorganisms consuming the excess organic matter and using oxygen for respiration (Meyers and Ishiwatari, 1993). SOD can cause the formation of anoxic hypolimnia in lakes and reservoirs when the rate of oxygen removed from the hypolimnion, which includes both SOD and oxygen consumption within the water column, exceeds the rate that oxygen is added to the water from the atmosphere and photosynthesis of the surface layers (Mackenthun and Stefan, 1995). There are several factors that influence the magnitude of SOD including temperature, oxygen concentration in the overlying water, flow velocity of the overlying water, the presence of reduced substances (e.g. NH_3 , Fe^{2+} , Mn^{2+} , etc.), sediment depth and aerobic layer thickness, organic matter content, and

the presence of macro-invertebrates (Charbonnet, 2003). SOD in shallow reservoirs can account for 60-95% of the total hypolimnetic oxygen demand (Beutel, 2003; Bouldin, 1968; Veenstra and Nolen, 1991). Typical SOD values range from 200 mg/m²-d in sandy lake bottoms to 1000 mg/m²-d in very organic sediments at 20°C (Thomann and Mueller, 1987). Reducing SOD would reduce the rate of oxygen removal from the hypolimnion and could reduce the likelihood of anoxic conditions.

1.1.4 Sediment Oxygen Demand Processes and Mechanisms

A reduction in SOD may not only delay or eliminate the onset of anoxia in the hypolimnion, but also increase the depth of the aerobic layer of sediment when overlying water is oxic, therefore increasing the thickness of the buffer layer between reductive dissolution in the anaerobic sediment and the sediment-water interface. The depth of the aerobic layer of the sediment just below the sediment-water interface is controlled by how deeply oxygen can diffuse into the sediment before it is consumed by oxygen demanding processes. Two independent factors control aerobic depth in the sediment: the rate of diffusivity of oxygen through the sediment, and the rate of oxygen consumption within the sediment.

Sediment having a higher diffusivity will allow oxygen to diffuse farther into the sediment when all other factors are constant. Sediment with a greater porosity (more open spaces filled by water) will possess a greater diffusivity than more densely packed sediment because oxygen diffusivity through water is greater than through solid sediment (Huettel and Webster, 2001). Therefore, increasing sediment porosity may result in a deeper oxic layer. However, as oxygen reaches deeper into the sediment, previously anaerobic processes will convert to aerobic processes and potentially increase oxygen demand within the previously anaerobic layers (Precht et al., 2004).

As oxygen diffuses into the sediment, it is consumed by chemical reactions such as oxidizing reduced metals and biological reactions such as bacterial respiration. The consumption of oxygen in the top layers of the sediment removes oxygen available to diffuse into the deeper layers of the sediment. Therefore, the rate of oxygen consumption by the sediment also affects oxic depth. If oxygen consumption rate by the sediment is reduced, then more oxygen is available to diffuse deeper into the sediment increasing oxic depth. However, the overall effect of a deeper oxic layer may also result in an increase in sediment oxygen consumption rate as previously anaerobic processes (such as denitrification and Fe³⁺ and Mn⁴⁺ reduction) shift to aerobic processes (such as nitrification, oxidation of metals, and aeration-dependent microbial activity) that consume oxygen more quickly (Skopp et al., 1990).

Suspended sediment oxygen uptake rate (sedOUR) is the mass-based oxygen uptake rate of the sediment (mg O₂/g sediment-d) measured when the sediment is fully suspended in aerated, fully aerobic conditions. This uptake rate is the theoretical maximum at which chemical and biological processes consume oxygen within the sediment since all of the sediment is fully exposed to fully aerobic conditions and there is minimal diffusion limitation. This rate is not realized in most of settled sediment because as settled sediment depth increases (away from the sediment-water interface), oxygen concentration within the sediment decreases as it is consumed. As the oxygen content is reduced in sediments below critical levels, chemical reactions slow as sufficient oxygen is not present to oxidize metals, and biological reactions slow as bacterial populations shift from faster aerobic respiration to slower anaerobic respiration. Oxygen uptake rate is affected by the concentration of substrates such as reduced metals (chemical) as well as organic matter and the concentration of dissolved oxygen in the sediment (biological). Oxygen uptake rate will be at a maximum in conditions where oxygen availability is not limited.

Since the concentration of oxygen within the sediment affects the oxygen uptake rate, then oxygen concentration in the sediment can affect SOD. Therefore, potential mechanisms for reducing SOD under oxic hypolimnetic conditions include temporarily increasing the oxygen availability within the sediment to maximize rates of chemical and biological processes in order to reduce the availability of reductive substrates. Once sediment is treated and allowed to resettle, this may reduce oxygen uptake rate and potentially SOD. This can be accomplished by increasing the diffusivity of oxygen into the sediment or by resuspending the sediment under oxygen-rich conditions to expose the entire layer to oxygen, removing diffusion as a rate-limiting step in the oxygenation of the sediment. However, increasing oxygen availability to sediments through resuspension and reducing diffusion limitations will initially increase the oxygen uptake rate and increase SOD by exposing a larger surface area of potentially oxygen-consuming sediments to oxygen-rich conditions (Sweerts, et al., 1989; Charbonnet, 2003; Brand et al., 2008).

1.1.5 Treatment Methods for Reducing SOD and Associated Problems in Sediments

A common treatment for reducing SOD and controlling P release is sediment dredging, i.e. physically removing a volume of sediment from a lake. However, this has several disadvantages including high costs, temporary P release from sediment, increased phytoplankton productivity, noise, lake drawdown, temporary reduction in benthic fish food organisms, the potential for toxic material release to the overlying water, and the potential for environmental degradation at the dredged material disposal site (Peterson, 1982).

Another method for treatment in lakes is hypolimnetic oxygenation which has been shown to improve water quality in lakes and reservoirs. This technique injects dissolved oxygen into the anoxic hypolimnion to create aerobic conditions in the water column above the sediment

without resuspending the sediment. Target oxygen content in the hypolimnion is typically around 4 mg/L, which will create aerobic conditions. Maintaining aerobic conditions in the hypolimnion would potentially reduce SOD by increasing the concentration of oxygen in the overlying water thereby increasing the rate of diffusion of oxygen into the sediments, quenching oxygen demand and reducing the concentration of bioavailable phosphorus by oxidation to the non-soluble form. Capital and operating costs of oxygenation systems increase substantially as target DO increases, so these systems are typically designed to provide the minimum DO required to maintain aerobic conditions. In lakes, hypolimnetic oxygenation is usually implemented seasonally over a period of one or more years.

The phenomenon of reducing oxygen demand through oxygenation is used frequently in wastewater processes. Oxygen demand is reduced through satisfying biological oxygen demand (due to microbes consuming organic matter), satisfying the chemical oxygen demand (from reduced compounds such as iron and sulfides), and satisfying the oxygen demand for nitrification (Metcalf, 2003). Gachter (1987) reported that while hypolimnetic oxygenation may help to decrease SOD and increase binding of the excess nutrients, it is not enough to permanently increase P sedimentation or change the trophic state of the reservoir. This indicates that once eutrophic reservoirs suffer an accumulation of nutrients and organic matter in bottom sediments, long term water quality may be compromised because of the sediments alone. So even if the total annual input of nutrients into a reservoir is reduced, legacy nutrients will remain in the bottom sediments of reservoirs and lakes and continue to contribute to water quality issues and potential reductions in recreation and tourism (Welch and Cooke, 1995; Gachter et al., 1998).

However, previous research also shows that, with increased hypolimnetic dissolved oxygen content, there are significant reductions in concentrations of nutrients in the water

column (Beutel and Horne, 1999). Gachter and Wehrli (1998) showed in Lake Baldegg, Switzerland, that after one year of injecting 6 tons/day of oxygen in November through May and 3-4 tons/day of oxygen in May through November, the DO levels remained above 3 mg/L, and the total phosphorus (TP) content dropped from 40 to 18 tons, though it was hard to discern between internal oxygenation and external controls. James et al. (1986) and Mauldin et al. (1988) investigated the effects of a dissolved oxygen injection system on Richard B. Russell Lake in Georgia, installed in 1985 in an attempt to remediate the hypolimnetic accumulation of ammonia, soluble reactive phosphorus (SRP), dissolved iron, and manganese. They found that, after one year of operation, in which the DO was maintained above 5 mg/L in most of the water column, hypolimnetic mass of ammonia, SRP, iron, and manganese in the reservoir's main basin dropped 50-85%. In Amisk Lake, in Alberta, Prepas et al. (1997) and Prepas and Burke (1997) found that, during three years of injecting oxygen at a rate ranging between 0.5 and 1.1 tons/day, the sediment phosphorus release dropped from 7.7 to 3.0 mg/m^2 -d and the volume-weighted mean hypolimnetic TP decreased from 123 to 56 µg-P/L. Additionally, they found that the volume-weighted mean hypolimnetic ammonia decreased from 120 to 50 µg-N/L with no concurrent increase in nitrate content over the same time period, possibly due to the inhibition of NH_4^+ release from the sediments or oxidation of NH_4^+ to NH_3 and subsequent loss to the atmosphere. Beutel and Horne (1999) found that, after the installation and operation of a downflow bubble contact system (DBCS) or Speece Cone, the deep-water ortho-P levels dropped from 200 µg-P/L prior to treatment to less than 50 µg-P/L after oxygenation. Additionally, deep-water ammonia dropped from 1000-1700 µg-N/L to less than 200 µg-N/L after oxygenation.

Research has also shown that with increased turbulence in the water above the sediment, oxygen can penetrate deeper into the sediment. Sweerts, et al. (1989) showed that the oxygen

content above the sediment-water interface (SWI) in an intact sediment core decreased by 70% and the oxygen penetration depth decreased from 4.2 mm to 2.0 mm, when the overlying water was not stirred for 24 h. Brand et al. (2008) studied the oxygen flux into sediments in Lake Alpnach in Switzerland, and found that when the vertical velocity fluctuations above the SWI increased from 0.6 to 1.1 mm/s, the oxygen flux into the sediment increased from 0.3 to 13.9 mmol/m²-d. This phenomenon is often explained by the convection mass transfer coefficient (h_m). This coefficient heavily influences the SOD, and is calculated from various properties, including density, viscosity, thermal conductivity, specific heat, temperature, and velocity (Charbonnet, 2003). As water velocity increases, the convection coefficient increases, resulting in an increased rate of diffusion of oxygen into the sediment.

And while there are methods available for treating the symptoms of eutrophication, such as reducing external P loading (Gachter and Muller, 2003; Carvalho et al., 1995) and the addition of chemicals including aluminum and Phoslock (a commercial bentonite product coated with lanthanum) (Egemose, 2010) or iron chloride (Wisniewski, 1999), little if anything has been done to find a remediation method to permanently reduce SOD and the likelihood of hypolimnetic anoxia. Additionally, little has been done to study the effect of rapid resuspension and oxygenation of sediments as a remediation method.

1.1.6 Proposed Rapid Method of Resuspension and Oxygenation of Sediments

The proposed remediation method described in this study includes intentionally resuspending the top layers (approximately 5 cm depth) of settled sediment at the bottom of a lake or reservoir. During the resuspension process, sufficient dissolved oxygen is added to the water/sediment mixture in the hypolimnion to overcome the increase in oxygen uptake rate caused by the additional surface area of resuspended anaerobic sediment being exposed to

oxygen and beginning to consume oxygen. Oxygenation would be conducted so aerobic DO conditions of near 4 mg/L would be maintained for the duration of the resuspension.

The theory of how this proposed treatment would reduce SOD is that the upper layers of intact sediment will be resuspended and broken into smaller particles with increased exposure to oxygen. Because a large amount of the sediment will have been exposed to oxygen, those constituents buried in the sediment that readily accept oxygen (e.g. reduced metals) will be oxidized, and aerobic bacterial respiration will be supported, reducing organic matter, and thereby quenching some of the long-term demand for oxygen. Once the sediment is allowed to re-settle, the SOD may be reduced, thereby slowing the onset of anoxic conditions in the overlying water (hypolimnion). With a reduced overall oxygen consumption rate in the sediment, the aerobic layer of sediment could be deeper (Bryant et al, 2010), which will possibly allow for a larger buffer layer against the anoxic release of nutrients to the aerobic water column (Hupfer and Lewandowski, 2008). While nutrients can still be released from the sediments, a deeper buffer layer could reduce the rates at which they are released. Additionally, as more nutrient-binding iron and manganese are oxidized, nutrients would be bound and settled into the sediment (Austin et al., 2009).

This proposed method of treatment is not feasible in a full scale lake or reservoir with currently used equipment for oxygenating water in the hypolimnion. Current equipment is designed to add DO without resuspending sediment and uses either diffuser hoses that inject oxygen bubbles along the lake bottom that dissolve into the water as they rise through the water column, or water injection at a low velocity to avoid sediment disruption. Current equipment is also not designed to add sufficiently high concentrations of DO in water to be able to overcome the substantial increase in oxygen demand caused by resuspended sediment.

The proposed rapid technique is made feasible because of a new technology,

Supersaturated Dissolved Oxygen injection (SDOX[®]), manufactured by BlueInGreen, LLC (Fayetteville, AR). The SDOX outputs a high velocity stream of water (around 12 m/s) that is supersaturated with dissolved oxygen (around 350 mg/L). The principle of operation of the SDOX is to inject this supersaturated water stream into a larger body of water such that the high concentration of DO in the stream is diluted within the larger body of water such that the overall DO is below saturated conditions to prevent the DO from bubbling out of solution. For example, if a hypolimnion was to be treated to reach a target DO of 4 mg/L, the mass dilution factor would be 1 part SDOX water per approximately 88 parts anoxic lake water, assuming no oxygen demand in the water. For the proposed rapid treatment method, the dilution factor would be less than 88 parts anoxic lake water because of the high oxygen uptake. Because of the concentrated solution, the SDOX may be capable of adding sufficient DO to maintain aerobic conditions during resuspension of sediment. An injection apparatus could be designed to use the high velocity stream to be directed at lake sediments in such a manner as to cause a controlled depth of sediment over a controlled area of the lake floor to resuspend under oxygenated conditions. Treatment would consist of resuspending and oxygenating the sediments for a period of time, then allowing the sediment to resettle. The goal of the treatment would be to reduce long term SOD in the water body and to reduce the likelihood of creating an anoxic hypolimnion and associated problems.

This resuspension phenomenon was observed at an SDOX installation at Lake Thunderbird in Norman, OK in 2010 when a large volume of sediment was inadvertently resuspended for several days during start-up of the installation. Data from that treatment year indicated that low to negative oxidation-reduction potentials responsible for the solubilization of

metals and sediment-bound phosphorus in the water column were reduced from historical averages (OWRB, 2011). Because the inadvertent resuspension of sediment led to reduced release of sediment-bound phosphorus to the water column, further investigation of intentional sediment resuspension is warranted.

1.1.7 Estimating Treatment Time Requirement for Proposed Rapid Treatment Method

The goal of the proposed rapid resuspension method would be to cause the same amount of treatment and benefits as several years of typical hypolimnetic water oxygenation in much less time. If the proposed rapid treatment is to be a competitive alternative to hypolimnetic oxygenation, it cannot cost substantially more than hypolimnetic oxygenation. As previously discussed, hypolimnetic oxygenation of water relies on the diffusion of oxygen into the sediment for treatment and is typically conducted over a period of one or more years. If successful, the rapid suspension method could be effective in a much shorter period of time. However, the rapid method would use oxygen at a much greater rate than hypolimnetic oxygenation because of the added demand of the resuspended sediment. The rapid method would also use more power. If the rapid method is to be economically feasible, then the overall cost of treatment must be near that of hypolimnetic oxygenation. Since the power and oxygen costs for the rapid method would be greater than hypolimnetic oxygenation, then the treatment time must be considerably less. When designing experiments to test the rapid method, it is necessary to estimate the treatment time required using the rapid method to have at least approximately the same overall costs as hypolimnetic oxygenation. It is not only important to experimentally determine if the rapid method reduces SOD, but also if the time required for treatment is sufficiently short to allow it to be economically feasible. Some rough calculations were conducted to estimate the required treatment time to help create an experimental design.

To completely offset the SOD using hypolimnetic water oxygenation in a 647,497 m^2 lake with an average depth of 3 m and an average SOD of 540 mg/m²-d (upper value of average range found by Haggard et al. (2012) in Lake Wister, Poteau, OK), 479 kg/day of oxygen would be required (full calculations found in Appendix 1). Based on a typical oxygen injection system operating for five years, this would require a total project cost of \$1.2 million, including capital equipment installation and operation (Scott Osborn, personal communication). If the resuspension and oxygenation method proposed is effective, then cost estimates are as follows.

The same 647,497 m² lake would be treated by resuspending the top 5 cm of sediment having a volumetric oxygen uptake rate of 0.166 g O_2/m^3 -s (average found by Charbonnet et al. (2006) in the Arroyo Colorado River) and adding sufficient dissolved oxygen to meet the increased demand. To meet this demand, a portable oxygen injection type system that is capable of resuspending the sediment 9,300 m² at a time would be used. The leasing and operating costs of such a system are \$100,500/mo (Scott Osborn, personal communication). To reach the same total project cost of the five year system of \$1.2 million, the leasable system would need to provide equivalent reduction of SOD by operating for 5.2 days in each treated 9,300 m² section. Therefore, if the resuspension and oxygenation treatment can provide significant treatment in approximately 5 days, it may be a feasible alternative to hypolimnetic aeration.

1.1.8 Developing Tests to Determine Feasibility of Rapid Treatment Method

During normal *in-situ* conditions, most of the sediment is anaerobic. Even if the water above the sediments is fully saturated with DO, typically only the top few millimeters of sediment are aerobic because the oxygen demand within the sediment consumes oxygen faster than it can diffuse into the sediment from the overlying water. As previously discussed, fully oxygenating all of the sediment enables the facultative aerobes within the previously anaerobic

sediment to shift to aerobic respiration, which will allow more digestion of organic matter, shifting the solid-state carbon to CO_2 gas. Additionally, more oxidative material within the sediment (e.g. iron and manganese compounds) will be exposed to oxygen, causing it to shift to an oxidized state, which reduces the solubility of metals (Fe and Mn) in the overlying water. Iron and manganese in the oxidized state are able to bind with nutrients as they precipitate to the bottom of the water body (Christensen, 1998; Dunne et al., 2011; Sondergaard et al., 1992).

However, because the processes involved with SOD are very complicated, there are several indicators that the rapid method may not only be ineffective but may be harmful to the ecosystem of a reservoir if tested *in-situ*. For instance, suddenly exposing a large amount of sediment that had previously been anaerobic to oxygen can cause a very large increase in oxygen uptake rate in the water column (Bryant et al., 2010), which, without knowing whether or not the rapid method is effective, could be harmful to an ecosystem to which the method is applied. Further, testing the method in an open system would not only be very expensive but would result in many uncontrollable factors which may affect experimental results and lead to inconclusive treatment results. Because of these concerns, this study focuses on a lab-scale experiment to help answer one of the key questions regarding the effectiveness of the proposed rapid treatment method, that is, whether or not resuspension and oxygenation of sediments from a eutrophic reservoir could reduce SOD in an economically feasible time period. If the lab-scale tests are successful, then the continuation of development of the method on a larger and more expensive scale could be supported. The function of the SDOX in the rapid method would be to add sufficient oxygen to maintain aerobic conditions and provide the mixing energy to resuspend sediments. Converting the SDOX into a system to conduct this treatment in a reservoir would require a great deal of engineering design that will not be considered in this study. Providing

sufficient dissolved oxygen and mixing of sediments on a lab-scale can be done using simpler equipment than the SDOX. Therefore, the SDOX will not be used in this study.

1.2 Objectives

The objective of this study is to examine the effectiveness of full resuspension of sediment combined with oxygenation for reducing the SOD in lake sediments. Further, the study will examine the effects of the duration of resuspension and oxygenation on SOD. The study will also compare the effects of resuspension with and without oxygenation on SOD in an attempt to determine if any effective SOD reduction is because of resuspension alone. Resuspension alone may change the porosity and diffusivity of oxygen through the sediment, which could provide a separate effect on SOD than resuspension with oxygenation. The effect of resuspension and oxygenation treatments on the nutrients, metals, and some minerals in both the sediment and water column will also be measured.

Experimental Methods and Equipment

2.1 Preliminary Work

Before designing the experimental plan to test the hypotheses, the following tasks were conducted:

- Assembling the testing equipment and establishing initial experimental and analytical procedures.
- Conducting a set of preliminary experiments to establish procedures for conducting the final experiment, including lab analyses, sediment collection, and treatment procedures.
 Conducting these preliminary experiments ensured all procedures and equipment worked

properly and that desired dissolved oxygen conditions could be maintained during the treatments before the final experiments were conducted.

- Four sites were explored for potential sediment collection. Lake Wister in Oklahoma was selected as the best testing location, based on accessibility and representativeness of a eutrophic system.
- 2.2 Sediment Site

Lake Wister, located in Le Flore County, Oklahoma, is a reservoir that was constructed in the 1940s. It was built primarily for flood-control, but is also used for supplying drinking water for approximately 40,000 area residents (OWRB, 2003). Lake Wister was chosen for sediment collection because of its relative proximity to the laboratory in which experiments were conducted and its status as a hypereutrophic reservoir with excessive levels of primary productivity and nutrients (OWRB, 2003; ODWC, 2009). It was also listed as impaired by pH, turbidity, chlorophyll-a, color, low dissolved oxygen, and phosphorus by the Oklahoma Department of Environmental Quality (ODEQ, 2008). In a previous study, Haggard et al. (2012) measured values of SOD in Lake Wister ranging from 388-643 mg/m²-d in the same site from which the present study's sediment was collected. They also measured average soluble reactive P release rates from sediment ranging from between <0 to 3.3 mg/m²-d across several sites in Lake Wister, indicating periods of anoxic sediment conditions were present and that sediment was an appropriate candidate for treatment.

2.3 Sediment Collection

Sediment samples were collected from locations in the upper cove (Figure 1) using an intact sediment corer as well as a sediment dredge. Samples were collected once for each replication of the present experiment, on August 28th and September 19th, 2013. For each trial of

the experiment, 34 liters of sediment and 227 liters of water were collected from Lake Wister. Sediment samples were collected using a Wildco Ekman Sampler from the bottom of Lake Wister (5.5 m deep) and stored in sealed buckets while being transported to the lab. Water samples were collected by taking the top water layer above sediment collected with a Wildco Ekman Sampler as well as by taking grab samples near the water surface (5.5 m and <1 m, respectively). Water was stored in sealed buckets as well as in barrels for transport back to the lab. Water samples collected from the bottom of the lake were anoxic. These samples were stored in air-tight sampling bottles (0.5 L) to take to the water quality lab. Samples collected at the water surface were aerobic having dissolved oxygen levels between 5 and 8 mg/L and remained aerated during transport.



Figure 1. Lake Wister, OK, sediment sampling site in upper cove (image obtained from Google Maps); the GPS coordinates of the site were 34°56′45.83″N, -94°43′12.48″W.

2.4 Sediment Resuspension Treatment Equipment

Sediment treatment experiments were conducted in the University of Arkansas' Biological and Agricultural Engineering laboratory. The sediment resuspension and oxygenation treatments were conducted in two 284 L aquarium tanks, each with dimensions 1.20 m L x 0.46 m W x 0.50 m H. The aquarium is made from a painted metal frame holding glass panes on the bottom and four sides. The glass panes are also sealed with silicone caulk. The top is open to the atmosphere. Two tanks are located side-by-side so an oxygenated treatment and nonoxygenated treatment can be conducted simultaneously. The oxygenated test is resuspending the sediment while adding sufficient DO to the system to maintain aerobic conditions near saturation. The non-oxygenated test is a duplicate of the oxygenated with the exception that no oxygen is added to the system and anaerobic conditions are maintained. This allows analysis of the effect of oxygen addition separate from the effect of resuspension of the sediment. A photograph of the tanks as set up in the laboratory is shown in Figure 2.



Figure 2. Photograph of the two treatment tanks. Pumps and fans can be seen in the foreground. Aeration pumps (blue) can be seen resting on top of the tank on the right.

To prevent atmospheric oxygen from entering the non-oxygenated tank, both tanks are sealed from the atmosphere with lids. Each tank is covered with a two-piece lid. One piece is permanently mounted and made from plywood, and is built to include a mixing pump inlet and outlet, two aeration tube inlets, a port for allowing handheld DO probe measurements, and a port for pressure relief. The second piece is removable to allow sample collection and is made of plexiglass. Each lid piece is sealed to the top frame of each tank to prevent gas exchange with the atmosphere using petroleum jelly as a sealant and 2.3 kg weights at each corner.

Tanks were tested for oxygen leakage by filling with water with no oxygen demand, removing DO from the water using sodium sulfite and then monitoring the change in DO using YSI 6290v2 datasondes. There was only an increase of DO in the tanks when the pumps were operating. When pumps were not operating, there was no DO increase. Therefore, the conclusion was made that the DO increase was from oxygen in the air headspace dissolving into the water. This problem was addressed by adding a nitrogen purge to remove air from the headspace prior to testing.

Nitrogen gas cylinders are installed next to the tanks. Nitrogen gas is bled into the gas headspace of the non-oxygenated tank at a flow rate of 1 lpm to purge oxygen initially sealed in the gas headspace of the tank and to prevent buildup of any leaking oxygen into the headspace from the outside air. This purging process prevents oxygen in the headspace from dissolving into the water allowing for a more accurate accounting of oxygen in the sealed tank required for determining the oxygen uptake rate. The nitrogen purge line has a digital flow meter and needle valve to monitor and control the flow rate of nitrogen into the non-oxygenated tank's headspace. Tanks were retested for leaks, and DO remained low when the nitrogen was bled into the headspace.

Each tank is wrapped in black plastic to prevent light from reaching the water, potentially causing oxygen production through photosynthetic reactions. Sediment is mixed using pumps that recirculate the water and sediment in the tanks. These pumps added heat to the system during the process of recirculation because of friction in the pump housing. To remove some of this heat addition, each tank has fans directed at the tops and sides of the outsides of the tanks. The room was also cooled to 18°C during the experiments to help maintain the temperature in

the tanks below 29°C, the typical maximum water temperature found on the floor of Lake Wister during the summer when oxygen deficits are most problematic.

The difference between the oxygenated and non-oxygenated tanks is that the oxygenated tank is set up such that at all times during mixing of the sediment, the entire tank will be maintained in aerobic conditions. The non-oxygenated tank was maintained at anaerobic conditions for the duration of mixing. All other aspects of the tanks are identical. Therefore, each tank can be either oxygenated or non-oxygenated. Since there was one replication of this test, for the second test, the tanks were switched such that each glass tank was used once as an oxygenated tank and once as a non-oxygenated tank.

2.5 Loading Sediment into Laboratory Tanks

Before adding the sediment to the tanks, the tanks were filled with water to a point approximately one inch above the ports for the inlet and outlet of the pumps, which were approximately 1.3 cm above the bottom of the tank, then, using tap water, the pumps were primed completely to prevent disturbances in the sediment later in the experiment. Next, the sediment was weighed and split evenly between the two 284 L tanks, such that there was approximately 4 cm of sediment in each tank. The sediment was carefully raked across the bottom to ensure even distribution throughout the tank. To estimate the surface area of the sediment for SOD calculations by assuming it was equal to the floor area of the tank, it was necessary that the sediment be evenly distributed and flat. The procedure to collect sediment from Lake Wister, store it, transport it to the lab, and place it in the tanks changed it from its *insitu* state. This disturbance was minimized as best as possible.

Once the sediment was added to the tanks, the water from Lake Wister was added (carefully poured against the side of the tank to minimize disturbance of the sediment) until the tanks were approximately half full. A datasonde set to log DO and temperature every ten minutes was placed in each of the tanks, hanging from a hook to prevent the sensor from being covered in sediment. Four air diffuser bars (aerators) were installed directly above the sediment surface (but not yet activated) in the treatment tank such that dissolved air would be distributed evenly throughout the tank. These aerators were attached to two air pumps made for aquarium aeration resting beside the tank. The capability of the two air pumps to maintain aerobic conditions during treatment was confirmed during in a preliminary test. The sediment was allowed to settle for 24 h in undisturbed, unaerated water to allow anaerobic conditions to be established in the bottom layers of the sediment. Then, more of the source water, which had been aerated to saturation DO conditions, was added to each tank until filled within 4 cm of the top of the tank with aerated water, and allowed to settle for 24 h without installing the plexiglass lids. The water was added in stages so that the beginning of the experiment would commence with the sediment in conditions similar to those *in-situ*, i.e. having a dissolved oxygen content above the sediment of at least 2 mg/L. The staging of water addition was also done in order to minimize the consumption of the oxygen in the water from any sediment that had yet to settle. After this period, if the DO was at least 2 mg/L in the tank, samples of the sediment and water were collected, and the treatment began.

2.6 Analytical Methods

2.6.1 Sediment Oxygen Demand

Two methods were used for measuring SOD in this experiment: using intact sediment cores in conjunction with membrane inlet mass spectrometry (MIMS), and directly measuring

the change in dissolved oxygen concentration over time for a known volume of water and surface area of the bottom of the 284 L aquarium tanks.

2.6.1.1 SOD Using Cores

Sediment cores were used in conjunction with MIMS in the Scott Biogeochemistry Lab at the University of Arkansas to measure sediment oxygen demand (SOD) from within the lake and within the 284 L aquarium tanks. Intact lake sediment cores were collected from Lake Wister using a hammer corer, then plugging the ends with rubber stoppers and sealing with electrical tape. These cores were made of acrylic plastic with a floor surface area of 28.3 cm² and a height of 70 cm. Smaller in-tank sediment cores were collected from the middle of each tank before the beginning of each treatment by inserting the core into the sediment manually, then sliding a metal plate under the bottom of the core in order to remove an intact cross section of the sediment. The cores used for the tank sediment were made of acrylic plastic with a floor surface area of 17.3 cm² and a height of 21 cm. A plastic cap was then pushed onto the top of the core in order to create a vacuum while another plastic cap was fixed onto the bottom of the core and sealed with electrical tape. The vacuum prevented any sediment from falling out of the core while the bottom cap was being installed.

In each case, cores were moved to the lab, and the water column above the sediment in the cores was aerated overnight. The following day(s), a small sample of water was collected from each core and preserved with ZnCl periodically until 4-6 samples had been collected. Each time a sample was taken from a core, the water was replaced with water collected from the epilimnion of Lake Wister.

Oxygen to argon (O₂:Ar) ratios were analyzed in each sample using MIMS, and each was converted to O₂ concentration using equation 1 (adapted from Grantz et al., 2012). Sample O₂ concentration ($[O_2]_{sample}$) was defined as

$$[O_2]_{sample} = (O_2: Ar_{sample} \times [Ar]_{exp}) \left(\frac{[O_2]: [Ar]_{exp}}{O_2: Ar_{standard}}\right)$$
(1)

where O_2 : Ar_{sample} is the measured sample signal and O_2 : $Ar_{standard}$ is the measured signal for well-mixed deionized water open to the atmosphere at the same temperature as the samples. The terms $[Ar]_{exp}$ and $[O_2]$: $[Ar]_{exp}$ were the theoretical saturated concentration and ratio, respectively, calculated for each sample temperature using gas solubility tables (Weiss, 1970; Grantz et al., 2012). The SOD in each core was then found using equation 2 as follows:

$$SOD = \frac{\Delta O_2 \times V}{SA} \tag{2}$$

where SOD is sediment oxygen demand in mg/m²-d, ΔO_2 is the slope of the line comprised of the oxygen concentrations over time, i.e. the change in oxygen content over time in mg/L-d, V is the volume of the water column above the sediment in the core in L, and SA is the cross sectional area of the core in m². There was no replication of any data point when measuring SOD in the cores in order to minimize the amount of sediment being removed from the tanks over the course of the experiment. There was also no replication of the intact sediment cores collected *in-situ*.

2.6.1.2 SOD Using Tanks

The SOD was determined in each of the 284 L aquarium tanks at the beginning of each experiment, and between each treatment by calculating the slope of the line representing oxygen concentration from the tanks (mg O_2/L water-d) then multiplying the slope by the volume of

water in each tank then dividing by the tank floor surface area (assumed to be constant throughout the experiment) (mg O_2/m^2 -d). The oxygen concentration was measured with a YSI 6920v2 datasonde in each tank every ten minutes over the course of each experiment. From the DO versus time data, the final SOD value for each treatment time was determined once the system reached steady-state, as indicated when the DO versus time graph became linear. In all cases, at least ten points (90 minutes) along the curve of DO versus time were considered in the calculation of the slope of the line. This slope was then converted to a sediment oxygen demand in mg/m²-d as previously described.

2.6.2 Suspended Sediment Oxygen Uptake Rate

Suspended sediment oxygen uptake rate (sedOUR) is the mass-based oxygen uptake rate of the sediment (g O_2/g sediment-s). Measuring the sedOUR gives an estimate of the maximum oxygen uptake rate that would be possible for the sediment fully suspended in aerated conditions. The sedOUR is measured by collecting a small (1-2 g) sample of sediment and placing it in a 330 mL BOD bottle, then filling the rest with reagent grade water that contained DO near saturation. Reagent grade water was used in order to be able to disregard the oxygen uptake rate from the water in the BOD bottle. The oxygen concentration is measured in the bottle every few minutes while it is being stirred. A plot of DO versus time was created and the slope of the line (mg O_2/L -min) calculated. Once the rate of change of oxygen concentration over time is calculated, the sediment is filtered from the water and dried overnight at 105° in order to determine the dry mass of sediment contributing to the oxygen uptake rate.

2.6.3 Sediment Organic Matter Content

Sediment organic matter content was measured to determine if oxygenation of the resuspended sediment would result in a decrease in organic matter due to consumption by

microbes in the sediment. The loss on ignition method is used to estimate the percent organic matter content in a sediment sample. The standard operating procedure (USGS Wisconsin Mercury Research Laboratory, (USGS, 1989)) was used for this study. In short, samples are dried overnight (approximately 8 hr) in an oven at 105°C, weighed, then transferred to a furnace and heated for two hours at 550°C and reweighed. The percent difference is the approximate organic matter content in percent. Results are reported as LOI%.

2.6.4 Sediment Components

Sediment composition parameters were analyzed to determine if sediment treatments changed the composition by shifting components to or from the water column to or from the sediment. Sediment components, including total phosphorus, potassium, calcium, manganese, sulfur, sodium, iron, manganese, zinc, copper, boron, aluminum, nitrate- plus nitrite-nitrogen, ammonia nitrogen, and percent loss on ignition were analyzed by the Altheimer Agriculture Diagnostic Laboratory at the University of Arkansas, Fayetteville. All analyses were done after the sample had dried at 50°C until sufficiently dry to grind and sieve through a 2mm (10 mesh) sieve. Total elemental concentrations were measured. The total digest was done using EPA Method 3050B (USEPA, 1986), loss on ignition by muffle furnace, and inorganic N by KCl extraction and scalar autoanalyzer. Results were reported in mg/kg, with the exception of loss on ignition, which was reported in a percent.

2.6.5 Water Quality Parameters

Water quality parameters were analyzed to determine if there would be a change in the amounts of the constituents due to transfer to or from the sediment as well as to examine whether or not nitrification was occurring, which could be detected by examining nitrate- plus nitritenitrogen and ammonia nitrogen. Chemical analysis of water samples was performed by the
Arkansas Water Resources Center (AWRC) in Fayetteville, AR using standard methods. Dissolved aluminum, dissolved iron, dissolved manganese, dissolved orthophosphate, dissolved nitrate, and ammonia were analyzed using EPA methods 200.7, 365.2, 300.0, and 351.2 (USEPA, 1983; USEPA, 1993). COD, TOC, TN, and TP were measured using APHA methods 5220D, 5310C, and 4500-P (APHA, 1997). Results were reported in mg/L. More information on the AWRC can be found at their website: <u>www.uark.edu/depts/awrc</u>.

2.7 Test Procedures

Once the sediment was placed in the tanks and distributed as described in section 2.3.3, the tanks were prepared for resuspension tests by first filling to within 4 cm of the top with water. A YSI 556MPS handheld DO sensor was placed in the tanks and logged DO every hour for a full working day. This manual DO measurement was done as a method for monitoring the SOD regularly during the experiment and as a check against the datasonde and core measurements. The tanks were then allowed to sit uncovered overnight to allow some reaeration of water to occur so that the reduction of DO of the water caused by the consumption of oxygen could be measured and SOD determined. Without some reaeration, the DO of the water was too low to measure the oxygen consumption rate. After reaeration, the lids were re-installed and sealed so that oxygen consumption caused DO to decrease. The DO of the water was measured to determine SOD of the sediment in the tanks prior to the resuspension treatment.

After SOD measurements were conducted in the tanks, short core samples were taken from each tank for analysis, and water and sediment samples were also collected for measurement of water and sediment quality parameters. After collecting these samples, treatment proceeded, the lids were sealed onto the tank, and the headspace of the non-oxygenated tank was purged with N_2 gas for 20 minutes at 3 lpm, then at 1 lpm for the remainder of the

experiment. After nitrogen purging, the pumps that resuspended sediment on both the oxygenated and non-oxygenated tanks were turned on, the aerators in the oxygenated tank were turned on, cooling fans were turned on, and the temperature in the room was lowered to 18°C.

The resuspension process continued for 3 hours then the pumps, fans, aeration, and nitrogen were turned off. The sediment in the tanks was allowed to settle with the tank lids removed to allow some reaeration to determine the SOD in the tanks. After approximately 12 hours, the lids were resealed onto the tanks, and the DO was measured over the course of the day to calculate SOD as previously described. After performing this procedure the first time, it was evident that simply removing the lid overnight was not sufficient to aerate the non-oxygenated tank to a sufficient DO to allow proper measurement of SOD, so for subsequent tests, bubble bars were inserted into the both of the tanks above the sediment to aerate the water column during sediment settling. Care was taken to minimize resuspension of the sediment. Additionally, simply measuring DO over the course of one day to determine SOD was not sufficient, so, after aeration, DO was measured over the course of two days instead of one. Once the SOD had been measured, short sediment core samples, water samples, and sediment samples were collected, and then treatment proceeded to the next duration test. The resuspension tests were repeated for 24 hr and 5 day durations. The entire test was repeated once, using sediment collected from the same location in Lake Wister on a different date, and using the same operating procedures as just described. The first test was completed in August 2013, and the replication was conducted between September and October 2013.

2.8 Data Analysis Procedures

The data was analyzed for statistical significance using JMP software. Each of the 28 measured parameters (SOD in the tanks, SOD in the cores, sedOUR, sediment components, and

water components) was analyzed using a generalized linear model (GLM) with a cross of the treatment (added oxygen versus no added oxygen) and time (modeled as a continuous variable between 0-147 hours of treatment) variables. The GLM was run assuming a normal distribution and using an identity link function. The estimation method used by JMP was maximum likelihood. The chi squared statistic was observed for all model parameter estimates (intercept and slopes for treatment effect, time effect, and treatment*time effect). Additionally, p-values were generated for each model parameter estimate as well as for the model as a whole. These were used to determine statistical significance of each of the effects of treatment (resuspension with oxygen versus resuspension without oxygen, and time). An alpha value of 0.05 was used to determine statistical significance. A p-value <0.05 indicated that the slope of the line representing the model parameter in the GLM was significantly different from zero.

The p-value for the treatment effect was used to determine if there was a significant difference between resuspension with added oxygen and resuspension without added oxygen. The p-value for treatment*time was used to determine if, over the course of the treatment from 0-147 hours, there was a significant difference between the slopes of the regression lines for resuspension with added oxygen and resuspension without added oxygen. The p-value for time was used to determine if there was a significant difference in the measured parameter over time during the course of the treatment, from 0-147 hours. When the treatment effect was ignored and the values for the oxygenated and non-oxygenated tanks combined, the p-value for the whole model was used to determine if there was a significant change in the parameter over the course of the treatment, from 0-147 hours. In the cases in which the resuspension with oxygen and resuspension without oxygen data were combined, the p-value for the whole model was the same as the p-value for the time effect, because only the time effect was being examined.

For each of the measured parameter analyses of resuspension with oxygenation versus resuspension without oxygenation, there were 16 observations and 3 degrees of freedom, with the exception of SOD as measured in the tanks, in which there were 14 observations and 3 degrees of freedom because of lost data for one of the reps at two times. For the analyses in which the resuspension with oxygen and resuspension without oxygen data were combined, there were 16 observations (4 times x 4 steps) and 1 degree of freedom, again with the exception of SOD as measured in the tanks, which had 14 observations. Full GLM analysis result details are found in Appendix 4.

2.9 Hypothesis

There are three hypotheses being tested in this lab-scale study:

1. H_a: Oxygenation of resuspended sediment in the tank will have an effect on SOD.

H_o: Oxygenation of resuspended sediment in the tank will not have an effect on SOD.

If the null hypothesis fails to be rejected, then all data for oxygenated and non-oxygenated resuspended sediment will be combined for testing of the second hypothesis. If the null hypothesis is rejected, the oxygenated resuspended data set will be examined separately from the non-oxygenated resuspended data set for the second hypothesis.

2. H_a: Resuspension of sediment in the tank will reduce SOD.

H_o: Resuspension of sediment in the tank will not reduce SOD.

 H_a: Oxygenation of resuspended sediment in the tank will have an effect on the concentrations of metals (Fe, Mn), minerals (e.g. Ca, Na, K), organic matter, and nutrients (P, N) in the sediment and water column. H_o: Oxygenation of resuspended sediment in the tank will not have an effect on the concentrations of metals (Fe, Mn), minerals (e.g. Ca, Na, K), organic matter, and nutrients (P, N) in the sediment and water column.

Results and Data Analysis

3.1 Sediment Oxygen Demand (SOD) and Suspended Sediment Oxygen Uptake Rate (sedOUR)

3.1.1 Results of Measurements

The dissolved oxygen data from the datasondes installed in the tanks is shown in Figures 3 and 4. It can be seen in Figures 3 and 4 that during the resuspension treatment, the oxygenated tank remained at saturation DO conditions (between 9 and 10 mg/L), and the non-oxygenated tank remained at anoxic conditions (below 2 mg/L). The point of aeration in order to measure SOD from DO concentrations above 2 mg/L can be seen as a spike in DO before each asterisk in the figures. Two illustrative portions of these graphs used to calculate SOD in the tanks are shown in Figure 5 and 6. The remainder of the graphs used to calculate SOD in the tanks at each point can be found in Appendix 2 along with the raw data used to calculate SOD in the cores. Additionally, the graphs of the dissolved oxygen content over time in the BOD bottles used to measure sedOUR can be found in Appendix 3. Two representative graphs of this data are shown in Figures 7 and 8.



Figure 3. Dissolved oxygen content over the course of the first replication of the experiment. Test durations are labeled at the peak values of oxygenated tests. * indicates segment used for SOD determination.



Figure 4. Dissolved oxygen content over the course of the second replication of the experiment. Test durations are labeled at the peak values of oxygenated tests. * indicates segment used for SOD determination.



Figure 5. Dissolved oxygen data from the tank after the 24 hr treatment in the first replication of the test in the non-oxygenated tank. Data was taken on 9/8/2013.



Figure 6. Dissolved oxygen data from the tank after the 24 hr treatment in the second replication of the test in the oxygenated tank. Data was taken on 9/30/2013.



Figure 7. Dissolved oxygen data in a BOD bottle used to calculate sedOUR before any treatment had occurred in the tank for the oxygenated tank in the first replication of the test.



Figure 8. Dissolved oxygen data in a BOD bottle used to calculate sedOUR after the 3 hr treatment in the tank for the oxygenated tank in the second replication of the test.

The results for the measurement of SOD after resuspension only (non-oxygenated) and resuspension with oxygenation (oxygenated) using the core and tank methods are reported in Tables 1 and 2, respectively. All values were measured at room temperature, and the values for

both SOD and sedOUR have all been corrected to 20°C by multiplying the raw percent DO saturation data by the saturation DO at 20°C (9.02 mg/L) and dividing by 100 before using the DO values to calculate SOD and sedOUR, since there was some fluctuation in the temperature of the laboratory during the experiment. The values for SOD in the tanks measured during the second replication of the test at the three hour duration point are missing due to a laboratory error.

	In-lake core						
	SOD						
	(mg/m^2-d)	In-tank	short cores	s SOD (mg	$/m^2$ -d)		
		Cumulative Duration of Treatment (hrs)					
Test (replication)		0	3	27	147		
Oxygenated (1)	661	731	794	546	679		
Non-oxygenated (1)	001	717	933	530	958		
Oxygenated (2)	156	1376	817	578	641		
Non-oxygenated (2)	430	1059	729	723	661		

Table 1. Sediment oxygen demand as measured in cores.

SOD as measured in tanks (mg/m^2-d) Cumulative Duration of Treatment (hrs) 0 Test (replication) 27 147 3 1241 Oxygenated (1) 606 610 146 Non-oxygenated (1) 1680 714 1740 221 Oxygenated (2) 1097 1159 748 Non-oxygenated (2) 871 1750 676

Table 2. Sediment oxygen demand as measured in tanks.

Values for sedOUR measured after each resuspension treatment are shown in Table 3. For direct comparison and to determine the percentage of maximum theoretical oxygen consumption (sedOUR) the SOD comprises, the values of SOD as measured in the tanks are converted to the same units as sedOUR by using the mass of sediment in each tank and are shown in Table 4.

	sedOUR (mg O ₂ /g sediment-d)						
	Cumulative Duration of Treatment (hrs)						
Test (replication)	0	3	27	147			
Oxygenated (1)	8.5	9.9	13.7	5.4			
Non-oxygenated (1)	9.4	10.0	12.7	7.4			
Oxygenated (2)	14.9	17.1	16.1	16.2			
Non-oxygenated (2)	14.1	17.9	16.0	17.8			

Table 3. Suspended sediment oxygen uptake rate as measured in BOD bottles with sediment collected from each tank (mg O_2/g sediment-d).

Table 4. SOD values from tanks converted to oxygen uptake rate per mass of sediment (mg O_2/g sediment-d).

	Oxygen uptake rate in the tank (mg O_2/g sediment-d)							
	Cumulative Duration of Treatment (hrs)							
Test (replication)	0	3	27	147				
Oxygenated (1)	0.035	0.017	0.017	0.004				
Non-oxygenated (1)	0.047	0.020	0.048	0.006				
Oxygenated (2)	0.031		0.032	0.021				
Non-oxygenated (2)	0.049		0.019	0.024				

3.1.2 Statistical Analysis

The GLM analysis produced parameter estimates for the linear model and p-values for comparative statistics for SOD from the tanks, SOD from the cores, and sedOUR. Model parameter estimates are listed in Table 5, and p-values are listed in Table 6. Full GLM analysis results for all measured parameters are found in Appendix 4.

Table 5. Model parameter estimates from GLM analysis of SOD tanks and SOD cores (mg/m^2 -d), and sedOUR ($mg O_2/g$ sediment-d).

	SOD Tanks	SOD Cores	sedOUR
Intercept	1183.9	814.4	13.4
TRT	146.1	9.4	0.23
Time	-4.7	-0.8	-0.009
TRT*Time	-1.0	0.7	0.006

	SOD Tanks	SOD Cores	sedOUR	
Whole Model	0.0425*	0.6626	0.9090	
TRT	0.1598	0.8511	0.8119	
Time	0.0097*	0.3472	0.5571	
TRT*Time	0.5268	0.4003	0.7007	

Table 6. p-values for GLM analysis of SOD tanks, SOD cores, and sedOUR. Significant p-values are denoted with an asterisk.

For SOD as measured in the tanks, there was no statistically significant difference between resuspension with oxygen and resuspension without oxygen. For SOD as measured in the cores, there was no statistically significant difference between resuspension with oxygen and resuspension without oxygen. For sedOUR, there was no statistically significant difference between resuspension with oxygen and resuspension without oxygen. Based on these tests, it is concluded that for hypothesis one the null hypothesis is not rejected and there is no difference between oxygenated sediments and non-oxygenated sediments. Because we failed to reject the null of the first hypothesis and there is then no statistical difference between the oxygenated and non-oxygenated sediment, and therefore all data points were combined for analysis for hypothesis 2 examining SOD over time.

The GLM analysis results for each measured parameter once the treatment effect was removed are shown in Tables 7 and 8 with corresponding plots shown in Figures 9-11.

Table 7. Model parameter estimates from GLM analysis of SOD tanks, SOD cores, and sedOUR $(mg/m^2-d \text{ and } mg O_2/g \text{ sediment-d}, \text{ respectively})$ without a treatment effect (combined oxygenated and non-oxygenated data).

	SOD Tanks	SOD Cores	sedOUR
Intercept	1183.9	814.4	13.3506
Time	-4.7	-0.8	-9.3E-3

		SOD Tanks	SOD Cores	sedOUR
Whole Model		0.0155*	0.3581	0.5596
Time		0.0155*	0.3581	0.5596
SOD Tanks (mg/m ² -d)	2000 1500- 1000- 500- 0	: · · · : : 0 50	100 Fime (hr)	:

Table 8. p-values for GLM analysis of SOD tanks, SOD cores, and sedOUR without a treatment effect (combined oxygenated and non-oxygenated data). Significant p-values are denoted with an asterisk.

Figure 9. Regression plot of GLM analysis of SOD as measured using datasonde data in the tanks (mg/m²-d) without a treatment effect (combined oxygenated and non-oxygenated data) (p-value: 0.0155, $R^2 = 0.34$).



Figure 10. Regression plot of GLM analysis of SOD as measured in short cores collected from the tanks (mg/m²-d) without a treatment effect (combined oxygenated and non-oxygenated data) (p-value: 0.3581, $R^2 = 0.05$)



Figure 11. Regression plot of GLM analysis of sedOUR (mg O2/g sediment-d) without a treatment effect (combined oxygenated and non-oxygenated data) (p-value: 0.5596, R2 = 0.02).

For SOD of the tanks, the whole model is significant and has a negative slope (Figure 10). For SOD of the cores, the whole model is not significant. To examine the difference in the SOD data between measurement methods, all of the SOD data was then combined, and a GLM analysis run with the measurement method as an effect. The slope of the resulting model was significant and there was no significant difference between the results from the core and tank

methods. Thus, the two methods were combined and analyzed together with only the effect of time on SOD. The plot of the resulting model is shown in Figure 12.



Figure 12. Regression plot of GLM analysis of all SOD data combined (mg/m²-d). The slope of this model is significantly different from zero (p-value: 0.017, $R^2 = 0.17$), and is negative (slope estimate: -2.62 mg/m²-d/hr), thus, for hypothesis two, we reject the null hypothesis. The resuspension of sediment does reduce the SOD for sediments treated in a laboratory.

The model for sedOUR shows no significance for any parameter estimate or treatment effect through the whole model, even when the treatment effect is disregarded. This suggests that resuspension either with or without oxygen does not affect the capability of the sediment to uptake oxygen in a fully aerobic state.

3.2 Sediment Components

3.2.1 Results of Measurements

The results for the measurement of sediment components are shown in Tables 9 and 10.

	Component							
	Cumulative							
	Treatment						NO ₃ -N	
Test (rep)	Time	Al	Iron	Mn	TP	NH ₄ -N	$+NO_2-N$	LOI%
Oxygenated (1)	0	13145	29691	1379	615	59	0.68	6.8
Oxygenated (1)	3	12522	30101	1330	654	50	$< 0.21^{+}$	7.3
Oxygenated (1)	27	12944	29301	1349	622	74	$< 0.21^{+}$	7.0
Oxygenated (1)	147	14911	29716	1304	620	44	0.28	7.0
Non-oxygenated (1)	0	12926	30044	1373	655	60	0.33	6.2
Non-oxygenated (1)	3	12907	28837	1339	636	56	$< 0.21^{+}$	6.9
Non-oxygenated (1)	27	13099	30256	1420	673	60	$< 0.21^{+}$	6.8
Non-oxygenated (1)	147	14107	28739	1312	592	51	$< 0.21^{+}$	6.6
Oxygenated (2)	0	21013	38269	1737	847	94	$< 0.21^{+}$	8.9
Oxygenated (2)	3	20793	37751	1715	858	107	$< 0.21^{+}$	9.3
Oxygenated (2)	27	21334	38713	1761	847	93	$< 0.21^{+}$	9.4
Oxygenated (2)	147	20852	40694	1782	926	65	7.69	7.6
Non-oxygenated (2)	0	21543	38762	1786	837	104	$< 0.21^{+}$	8.6
Non-oxygenated (2)	3	20798	38432	1726	847	102	$< 0.21^{+}$	9.3
Non-oxygenated (2)	27	21164	37431	1671	840	93	$< 0.21^{+}$	7.7
Non-oxygenated (2)	147	21026	41357	1853	907	79	5.34	7.4

Table 9. Sediment components measured in the tanks during the course of the experiment. All values are reported in mg/kg, with the exception of LOI%, which is reported as a percent. All values are measured as total elemental concentrations.

⁺Values measured are below method detection limit.

	Component								
	Cumulative								
	Treatment								
Test (rep)	Time	В	Zn	Cu	Κ	Ca	Mg	S	Na
Oxygenated (1)	0	12	69	17	1532	912	1516	371	62
Oxygenated (1)	3	12	72	18	1511	988	1502	364	63
Oxygenated (1)	27	11	67	17	1463	892	1457	339	61
Oxygenated (1)	147	12	69	20	1476	973	1465	359	63
Non-oxygenated (1)	0	12	69	17	1509	923	1498	374	64
Non-oxygenated (1)	3	12	69	17	1528	928	1503	365	63
Non-oxygenated (1)	27	12	71	18	1478	951	1526	377	62
Non-oxygenated (1)	147	11	67	20	1398	913	1419	356	60
Oxygenated (2)	0	15	95	21	2059	1196	2086	464	92
Oxygenated (2)	3	15	93	21	2034	1239	2027	446	86
Oxygenated (2)	27	15	95	21	2086	1237	2109	447	88
Oxygenated (2)	147	14	105	25	2224	1513	2160	530	101
Non-oxygenated (2)	0	15	94	21	2062	1158	2099	460	85
Non-oxygenated (2)	3	14	92	23	1975	1370	2056	449	83
Non-oxygenated (2)	27	15	95	23	2102	1259	2091	453	88
Non-oxygenated (2)	147	14	103	22	2148	1318	2122	527	91

Table 10. Sediment quality parameters measured in the tanks during the course of the experiment. All values are reported in mg/kg. All values are measured as total elemental concentrations.

3.2.2 Statistical Analysis

GLM analysis of the sediment components produced parameter estimates and p-values for each statistical model parameter. These are listed in Tables 11 and 12. Full GLM analysis results for all parameters are found in Appendix 4.

	Al	Iron	Mn	TP	NH ₄ -N
Intercept	16963.4	33897.3	1548.0	743.0	80.5
TRT	3.5	-23.6	7.7	-0.1	1.4
Time	5.2	8.1	0.1	0.1	-0.1
TRT*Time	-1.6	-0.7	0.1	-0.1	0.04
	NO ₃ -N +NO ₂ -N	LOI%	В	Zn	Cu
Intercept	+	7.9	13.3	81.3	19.3
TRT	+	-0.2	-0.04	-0.3	0.1
Time	+	-0.005	-0.004	0.03	0.02
TRT*Time	+	6E-4	-7E-4	-5E-3	-0.008
	К	Ca	Mg	S	Na
Intercept	1776.0	1081.8	1788.6	407.4	74.5
TRT	-11.6	-8.1	-0.5	2.6	-1.3
Time	0.2	0.7	0.03	0.2	0.03
TRT*Time	-0.2	-0.5	-0.2	-0.03	-0.02

Table 11. Model parameter estimates for GLM analysis of sediment quality parameters measured in the tanks (mg/kg).

+Because values were reported as below detection limit, GLM analysis was not possible.

	Al	Iron	Mn	TP	NH ₄ -N
Whole Model	0.9903	0.9823	0.9972	0.9914	0.3966
TRT	0.9971	0.9841	0.8814	0.9966	0.7673
Time	0.7491	0.6817	0.9100	0.8012	0.0996
TRT*Time	0.9200	0.9721	0.9084	0.8409	0.6419
	NO ₃ -N +NO ₂ -N	LOI%	В	Zn	Cu
Whole Model	+	0.4801	0.9062	0.9601	0.3238
TRT	+	0.3388	0.9016	0.9217	0.8287
Time	+	0.2021	0.4665	0.5940	0.0940
TRT*Time	+	0.8871	0.9112	0.9367	0.3854
	К	Ca	Mg	S	Na
Whole Model	0.9926	0.7683	0.9992	0.8194	0.9285
TRT	0.8792	0.8609	0.9948	0.8600	0.7182
Time	0.8497	0.4026	0.9842	0.3469	0.6255
TRT*Time	0.8514	0.5143	0.8865	0.9149	0.7625

Table 12. p-values for GLM analysis of sediment quality parameters measured in the tanks. Significant p-values are denoted with an asterisk.

+Because values were reported as below detection limit, GLM analysis was not possible.

There was no significant difference between resuspension with oxygen and resuspension without oxygen treatments for any sediment component. All data for each treatment was combined and the GLM analysis conducted again without the treatment effect. The results are that there are no significant differences in sediment component concentration over time due to resuspension. Based on these results, the null hypothesis was not rejected, and there is no effect on the sediment component concentrations over time due to resuspension of the sediment.

3.3 Water Quality Parameters

3.3.1 Results of Measurements

The results for the measurement of water quality parameters are shown in Tables 13 and 14.

Table 13. Water quality parameters as measured at each point during the treatment. All values are reported in mg/L. Al, Fe, Mn, OrthoPO₄, and NO₃-N+NO₂-N were measured in the dissolved form.

Test (rep)	Time	Al	Iron	Mn	OrthoPO ₄	TP	COD
Oxygenated (1)	0	0.01	0.05	1.13	0.01	0.16	19.76
Oxygenated (1)	3	0.01	0.03	1.58	0.01	0.21	37.23
Oxygenated (1)	27	0.02	0.10	1.60	0.01	0.27	61.01
Oxygenated (1)	147	0.02	0.16	0.13	0.02	0.64	168.23
Non-oxygenated (1)	0	0.01	0.06	1.25	0.01	0.20	23.49
Non-oxygenated (1)	3	0.02	0.03	1.64	0.01	0.31	31.83
Non-oxygenated (1)	27	0.01	0.06	0.92	0.01	0.25	41.53
Non-oxygenated (1)	147	0.02	0.09	1.59	0.02	0.44	36.16
Oxygenated (2)	0	0.01	0.01	1.17	0.01	0.12	16.23
Oxygenated (2)	3	0.01	0.01	1.61	0.01	0.23	33.83
Oxygenated (2)	27	0.01	0^+	1.39	0.01	0.16	28.17
Oxygenated (2)	147	0.01	0.07	0.57	0.02	0.22	36.35
Non-oxygenated (2)	0	0.01	0^+	1.08	0.01	0.12	16.50
Non-oxygenated (2)	3	0.01	0^+	2.05	0.01	0.25	44.04
Non-oxygenated (2)	27	0.02	0^+	1.78	0.01	0.16	52.07
Non-oxygenated (2)	147	0.02	0.01	1.59	0.03	0.23	62.15

⁺Values measured are below method detection limit.

Table 14. Water quality parameters as measured at each point during the treatment. All values are reported in mg/L.

Test (rep)	Time	NH ₄ -N	NO ₃ -N+NO ₂ -N	TN	TOC
Oxygenated (1)	0	0.39	0.07	0.54	2.34
Oxygenated (1)	3	0.54	0.08	1.39	2.77
Oxygenated (1)	27	0.5	0.22	1.79	2.51
Oxygenated (1)	147	0.2	0.76	3.05	3.89
Non-oxygenated (1)	0	0.41	0.07	0.56	2.77
Non-oxygenated (1)	3	0.57	0.06	1.25	2.97
Non-oxygenated (1)	27	0.68	0.07	1.61	2.83
Non-oxygenated (1)	147	0.1	0.78	1.67	2.56
Oxygenated (2)	0	0.4	0.03	0.77	2.33
Oxygenated (2)	3	1.61	0.05	1.23	8.98
Oxygenated (2)	27	0.69	0.36	1.34	9
Oxygenated (2)	147	0.71	0.73	1.54	2.17
Non-oxygenated (2)	0	0.34	0.03	0.66	2.38
Non-oxygenated (2)	3	0.06	0^+	1.22	7.62
Non-oxygenated (2)	27	1.38	0.11	1.73	13.92
Non-oxygenated (2)	147	0.58	0.66	2.02	2.33

⁺Values measured are below method detection limit.

3.3.2 Statistical Analysis

GLM analysis of the oxygenated and non-oxygenated values for the water quality parameters produced parameter estimates and p-values for each statistical model parameter. These are listed in Tables 15 and 16. Full GLM analysis results for all parameters are found in Appendix 4.

	Al	Iron	Mn	OrthoPO ₄	TP
Intercept	0.01	0.03	1.5	0.01	0.2
TRT	2.0E-4	-0.01	0.2	9.4E-4	-2.0E-3
Time	3.0e-5	4.0E-4	-3.3E-3	6.5e-5	1.3E-3
TRT*Time	3.0e-6	-2.0E-4	4.1E-3	1.3e-5	-4.5E-4
			NO ₃ -N		
	COD	NH ₄ -N	$+NO_2-N$	TN	TOC
Intercept	30.2	0.6	0.05	1.1	5.0
TRT	-5.8	-0.06	-0.03	-0.06	0.2
Time	0.3	-1.3E-3	5.0E-3	7.2E-3	-0.01
TRT*Time	-0.2	4.5E-4	6.8e-5	-1.5E-3	-3.2E-3

Table 15. Model parameter estimates from GLM analysis of water quality parameters (mg/L).

Table 16. p-values from GLM analysis of water quality parameters. Significant p-values are denoted with an asterisk.

	Al	Iron	Mn	OrthoPO ₄	TP
Whole Model	0.0178*	0.0302*	0.0027*	< 0.0001*	0.0452*
TRT	0.6956	0.1462	0.0343*	0.1233	0.9350
Time	0.0016*	0.0133*	0.0171*	< 0.0001*	0.0065*
TRT*Time	0.7220	0.1488	0.0040*	0.1782	0.3022
			NO ₃ -N		
	COD	NH ₄ -N	$+NO_2-N$	TN	TOC
Whole Model	0.0227*	0.7879	< 0.0001*	0.0069*	0.8201
TRT	0.3729	0.5535	0.0317*	0.5803	0.7961
Time	0.0078*	0.4217	< 0.0001*	0.0006*	0.3689
TRT*Time	0.0811	0.7815	0.7709	0.3994	0.8159

There was a significant difference between resuspension with oxygen and resuspension without oxygen for Mn and $NO_3-N + NO_2-N$. There was no significant difference between

resuspension with oxygen and resuspension without oxygen for any other water quality parameter. The oxygenated and non-oxygenated data were then combined for all parameters except Mn and $NO_3-N + NO_2-N$. The results were that slope of the model and time effects were significantly different from zero except for in the cases of NH₄-N and TOC. These two showed no significant change over time, and thus, for these two parameters, the null hypothesis is not rejected, and resuspension has no effect on concentration over the treatment period. For all other parameters, there is a significant effect due to resuspension, and for Mn and $NO_3-N + NO_2-N$, a significant effect due to resuspension and oxygenation, therefore the null hypothesis is rejected for these parameters. Additionally, for all parameters for which there was a significant effect, the slope of concentration versus time is positive, indicating that the concentrations of these parameters increased over the duration of the test. The model plots for Mn and $NO_3-N + NO_2-N$ are shown in Figure 13. For Mn, over the course of the treatment, there was a negative change in concentration for the tank with added oxygen, and a positive change in concentration for the tank without added oxygen. For $NO_3-N + NO_2-N$, there was a positive change in concentration for both resuspension with oxygen and resuspension without oxygen, though the values were significantly different between treatments.



Figure 13. Mn and NO₃-N+NO₂-N GLM regression model plots (mg/L). Oxygenated data is shown in blue, and non-oxygenated is shown in red.

3.4 Difference Between Replications

The data for many measured parameters appeared to differ between replications of the tests, so all data was analyzed to determine if this difference was significant. The data was analyzed in JMP using a GLM as in previous analyses, but adding the replication as a treatment effect. For parameters in which there was no difference between resuspension with oxygen and resuspension without oxygen, all data was combined for this analysis. Even though there was no significant difference between SOD between methods, each method was examined both separately and combined because of the amount of error present in the measurement methods.

The two water quality parameters (Mn and NO₃-N+NO₂-N) for which there was a significant difference between resuspension with oxygen and resuspension without oxygen were separated into resuspension with oxygen and resuspension without oxygen before analysis of the effect of the replication. Table 17 shows the resulting p-values for this analysis. For those parameters in which there was no significant difference between replications, the combined replication data was further analyzed to determine if there was a significant time effect between times of treatment (0 versus 3 hr, 3 hr versus 27 hr, and 27 hr versus 147 hr of treatment) to determine where if any change was occurring during the course of the experiment. This can be seen in Table 17.

		p-value			
Parameter	Rep Effect	Time Effect	0-3 hr	3-27 hr	27-147 hr
SOD Combined	0.3477	0.0170*	0.0292*	0.7815	0.1457
SOD Cores	0.0032*	-	-	-	-
SOD Tanks	0.9270	0.0155*	0.0537	0.2317	0.0887
sedOUR	< 0.0001*	-	-	-	-
		SEDIMENT			
Al	< 0.0001*	-	-	-	-
Fe	< 0.0001*	-	-	-	-
Mn	< 0.0001*	-	-	-	-
TP	< 0.0001*	-	-	-	-
NH_4-N	0.0002*	-	-	-	-
NO ₃ -N+NO ₂ -N	-	-	-	-	-
В	< 0.0001*	-	-	-	-
Zn	< 0.0001*	-	-	-	-
Cu	0.0106*	-	-	-	-
K	< 0.0001*	-	-	-	-
Ca	0.0040*	-	-	-	-
Mg	< 0.0001*	-	-	-	-
S	< 0.0001*	-	-	-	-
Na	< 0.0001*	-	-	-	-
LOI%	0.0015*	-	-	-	-
		WATER			
Al	1.0000	0.0017*	0.0462*	0.1950	0.0005*
Fe	0.1065	0.0257*	0.4640	0.2480	0.0695
Mn (Oxygenated)	0.8568	0.0065*	0.0626	0.5856	0.0028*
Mn (Non-oxygenated)	0.3381	0.3459	-	-	-
OrthoPO4	0.2694	< 0.0001*	0.3434	0.3434	0.0002*
TP	0.3559	0.0083*	0.0480*	0.2685	0.3335
COD	0.8831	0.0155*	0.4897	0.7240	0.2552
NH_4-N	0.9459	0.4277	-	-	-
NO ₃ -N+NO ₂ -N (Oxygenated)	0.9540	0.0007*	0.7997	0.0146*	0.0012*
NO ₃ -N+NO ₂ -N (Non- oxygenated)	0.8529	0.0005*	0.7476	0.2989	0.0003*
TN	0.6783	0.0008*	0.0459	0.2389	0.1336
TOC	0.8839	0.3707	-	-	-

Table 17. p-values for the analysis of the difference between replications of the experiments. In cases where there was not a significant difference between replications, the time effect between each treatment time was examined and the p-value reported. Significant p-values are denoted with an asterisk.

For SOD in the cores and sedOUR there is a significant difference between replications of the test. For SOD combined (cores and tanks) there is no significant difference between replications. For SOD in the tanks, there is no significant difference between replications. For all sediment composition parameters there is a significant difference between replications. For no water quality parameter is there a significant difference between replications of the test.

For SOD combined there was a significant effect due to time between 0 and 3 hr of treatment and no significant effect due to time for any other time step. For SOD in the tanks, there was an overall significant effect due to time, but there is no significant effect due to time for any individual time step. For all water quality parameters with the exception of Mn (non-oxygenated), NH₄-N, and TOC, there was an overall significant effect due to time, with varying results for significance due to the effect of time at different time steps. These can be seen in Table 17.

For the parameters in which there was a significant difference between the two replications, the overall time effect was examined for significance, and, if there was a significant effect due to time, the time effect between each treatment time was examined during each replication individually. These results are shown in Tables 18 (first replication) and 19 (second replication).

	p-value			
Parameter	Time Effect	0-3 hr	3-27 hr	27-147 hr
SOD Combined	-	-	-	-
SOD Cores	0.1336	-	-	-
SOD Tanks	-	-	-	-
sedOUR	0.0063*	0.3084	0.0187*	0.0014*
	SEDIMENT			
Al	0.0181*	0.3846	0.4037	0.0107*
Fe	0.7710	-	-	-
Mn	0.1088	-	-	-
TP	0.4489	-	-	-
NH_4-N	0.1427	-	-	-
NO ₃ -N+NO ₂ -N	-	-	-	-
В	0.9753	-	-	-
Zn	0.6390	-	-	-
Cu	0.0268*	0.3027	0.9319	0.0174*
Κ	0.1191	-	-	-
Ca	0.6833	-	-	-
Mg	0.2586	-	-	-
S	0.6809	-	-	-
Na	0.5317	-	-	-
LOI%	0.3454	-	-	-
	WATER			
Al	-	-	-	-
Fe	-	-	-	-
Mn (Oxygenated)	-	-	-	-
Mn (Non-oxygenated)	-	-	-	-
OrthoPO4	-	-	-	-
TP	-	-	-	-
COD	-	-	-	-
NH4-N	-	-	-	-
NO ₃ -N+NO ₂ -N (Oxygenated)	-	-	-	-
NO ₃ -N+NO ₂ -N (Non-oxygenated)	-	-	-	-
TN	-	-	-	-
TOC	-	-	-	-

Table 18. p-values for the analysis of the overall time effect and the time effect between each treatment for the first replication of the experiment. Significant p-values are denoted with an asterisk.

	p-value				
Parameter	Time Effect	0-3 hr	3-27 hr	27-147 hr	
SOD Combined	-	-	-	-	
SOD Cores	0.0299*	0.0249*	0.3889	0.9965	
SOD Tanks	-	-	-	-	
sedOUR	0.0439*	0.0126*	0.1049	0.2528	
	SEDIMENT				
Al	0.1866	-	-	-	
Fe	0.0196*	0.5125	0.9752	0.0075*	
Mn	0.2229	-	-	-	
TP	0.0030*	0.3026	0.3682	0.0012*	
NH_4-N	0.0237*	0.4712	0.1454	0.0285*	
NO ₃ -N+NO ₂ -N	-	-	-	-	
В	0.0026*	0.0432*	0.0234*	0.0008*	
Zn	0.0007*	0.1335	0.0523	0.0005*	
Cu	0.5102	-	-	-	
Κ	0.0262*	0.1799	0.0604	0.0560	
Ca	0.1694	-	-	-	
Mg	0.0267*	0.0521	0.0349*	0.0926	
S	< 0.0001*	0.0080*	0.4456	< 0.0001*	
Na	0.2114	-	-	-	
LOI%	0.1577	-	-	-	
	WATER				
Al	-	-	-	-	
Fe	-	-	-	-	
Mn (Oxygenated)	-	-	-	-	
Mn (Non-oxygenated)	-	-	-	-	
OrthoPO4	-	-	-	-	
TP	-	-	-	-	
COD	-	-	-	-	
NH4-N	-	-	-	-	
NO ₃ -N+NO ₂ -N (Oxygenated)	-	-	-	-	
NO ₃ -N+NO ₂ -N (Non-oxygenated)	-	-	-	-	
TN	-	-	-	-	
TOC	-	-	-	-	

Table 19. p-values for the analysis of the overall time effect and the time effect between each treatment for the second replication of the experiment. Significant p-values are denoted with an asterisk.

SOD as measured in the cores showed no significant change over time when examining the first replication, and did show a significant change over time when examining the second replication. During the second replication in which the time effect was significant, the only time period in which the change was significant was between the initial value and after 3 hours of treatment. SedOUR showed a significant effect due to time in both the first and second replication of the test. When examining the time steps for significance, in the first replication there was a significant effect due to time between 3 and 27 hr of treatment and between 27 and 147 hr of treatment. In the second replication there was a significant effect due to time between the initial value and after 3 hours of treatment.

Of the sediment component parameters, only Al and Cu showed a significant effect due to time in the first replication of the test, while no others showed a significant effect due to time in the first replication. Both Al and Cu, when examining the time steps separately, showed a significant effect due to time only between 27 and 147 hr of treatment. All of the sediment quality parameters with the exception of Al, Mn, Cu, Ca, Na, and LOI% showed a significant effect due to time in the second replication of the test, with varying results in significance between time steps, the results of which are shown in Table 19.

Discussion

- 4.1 Sediment Oxygen Demand and Suspended Sediment Oxygen Uptake Rate
- 4.1.1 Comparison to Literature Values

A range of SOD and sedOUR values from the literature is shown in Table 20.

Constituent	Range (mg/m^2-d)	Source
SOD	456-661	Present study (Intact Cores)
SOD	146-1750	Present study (Tank Method)
SOD	539-1376	Present study (Short Core Method)
SOD	200-1000	Thomann and Mueller (1987)
SOD	561-2230	Charbonnet et al. (2006) (River)
SOD	388-643	Haggard et al. (2012) (Lake Wister)
Constituent	Range (mg/g-d)	Source
sedOUR	5.4-17.85	Present study
sedOUR	3.2-15.56	Charbonnet et al. (2006) (River)

Table 20. SOD and sedOUR values found in the literature.

Thomann and Mueller (1987) report that typical SOD values range from 200 mg/m²-d in sandy lake bottoms to 1000 mg/m²-day in very organic sediments at 20°C. Charbonnet et al. (2006) reported SOD values at 20°C in the Arroyo Colorado River near Weslaco, TX using a chamber-based measurement method. Haggard et al. (2012) measured values of SOD using the intact core method in Lake Wister in the same site from which the present study's sediment was collected. The average of the in-lake core SOD measurements from this study is in the range of values found previously by Haggard et al. (2012). Nearly all of the SOD values measured after resuspension using both the tank and core method are greater than SOD values measured by Haggard et al. (2012), and nearly a third of the SOD measurements exceed the range identified by Thomann and Mueller (1987). This indicates that transferring sediment to the laboratory and placing it in the tanks increased SOD value.

The sedOUR values in this study are comparable to the range found by Charbonnet et al. (2006) who measured between sedOUR at 6 sites in the Arroyo Colorado River. This indicates that the maximum oxygen uptake rate of the sediment under fully exposed and oxygenated

conditions was similar even though this work used lake sediment and Charbonnet et al. (2006) used stream sediment. No other values for sedOUR were found in the literature.

4.1.2 Discussion of Results

Nearly all of the SOD values measured after resuspension using both the tank and core method are greater than SOD values measured by Haggard et al. (2012), and nearly a third of the SOD measurements exceed the range identified by Thomann and Mueller (1987). However, the SOD measured from the intact cores taken directly from the lake is within the range measured by Haggard et al. (2012), while still being two to three times lower than that measured in the sediment using either measurement method before operation of the treatment. It is possible that, though the values for calculating SOD were corrected to 20°C, the fact that the operating temperature in the tanks was close to 29°C during the treatment could have increased respiration rates of any biological activity contributing to SOD (Seiki et al., 1994). It is also possible that the higher values are due to suspended sediment in the tanks and in the cores which would have effectively increased the surface area of sediment coming into contact with any oxygen available in the water, thus increasing the amount of constituents (both biological and chemical) contributing to the oxygen demand in both the cores and the tanks (Sweerts, et al., 1989; Charbonnet, 2003; Brand et al., 2008). A factor that is likely to be contributing to this difference between the values for SOD measured in the lake and those measured in the lab is the collection and preparation procedures increasing the SOD.

The collection and preparation of the sediment for the tests had a considerable effect on the sediment, likely because, when the sediment was resuspended during the collection and preparation, the oxygen uptake of the sediment would have been increased due to increased exposure of previously buried and anoxic sediments to oxygen and other constituents in the

water column, shifting the slower, anaerobic processes (e.g. fermentation, denitrification, etc.) to faster, aerobic processes (e.g. aeration-dependent microbial activity, oxidation of metals, etc.) that consume oxygen more quickly (Skopp et al., 1990). Because oxygen diffusion limitations contribute to the amount of oxygen being consumed by the sediment, the disturbance of the sediment during the collection process and the subsequent increase in porosity of the sediment may have increased the SOD between collection of the sediment and placing it in the tanks in the lab. Thus, the experiment as set up in the lab is not likely representative of a larger-scale experiment performed *in-situ*, because the larger-scale experiment would not include physical removal and replacement of the sediment with intermittent exposure to oxygen. The continual addition of oxygen into the water column in both tanks to measure SOD likely had an effect on the SOD, since there was potentially oxygen available to at least some of the sediment for most of the duration of the experiment.

While anaerobic conditions were maintained in the non-oxygenated tank during the treatment periods, there is no evidence to suggest that oxygenation had any effect on the SOD different than non-oxygenation. The fact that oxygenation of sediment during the resuspension treatment had no significant additional effect on the SOD suggests that the mechanism reducing SOD is not dissolved oxygen in the water. Also, the fact that sedOUR was not significantly changed suggests that this maximum potential sediment oxygen uptake rate is not a dominant driving force in SOD. If it were a dominant driving force in SOD, then the trend of the change in SOD over the course of the treatment would have more closely resembled that of sedOUR and would not have been reduced over time. This also suggests that the reduction in SOD during resuspension is not due to any reduction in oxygen uptake rate in the sediment.

When comparing the values for sedOUR to the values of SOD as measured in the tanks (Tables 3-4, pg. 36), when converted to the same units as sedOUR using the mass of sediment in each tank, the SOD as measured in the tanks ranges between 0.075 and 0.50% of the sedOUR. This suggests that, while there may be overestimation of SOD in the tanks and cores due to suspended sediment, the majority of the oxygen demand measured is due to settled sediment in the cores and the tanks. That is, if the SOD measured in the tanks and cores were actually measuring the SOD of the suspended sediment (resuspended and exposed to oxygen), then the value of the SOD would be closer to that of the sedOUR. Since it is not, and is in fact only a small percentage of the sedOUR, which is a measure of maximum oxygen uptake rate when the sediment is resuspended and exposed to oxygen, the SOD measured in the tanks and cores is likely not due to the suspended sediment.

If we assume that sedOUR is the maximum potential oxygen uptake rate of fully suspended and oxygenated sediment, then it is reasonable to assume that the measured SOD in the tanks is the oxygen uptake rate due solely to the aerobic layer of sediment. When the SOD as measured in the tanks is converted to the same units as sedOUR, it is then possible to estimate the percentage of the sediment in the tank that is consuming oxygen. Using this percentage to estimate the depth of the aerobic layer of sediment out of the 4 cm total of sediment shows an aerobic depth ranging from 0.03 to 0.20 mm of aerobic sediment depth. Because the aerobic depth is so small and the contours of the actual sediment in the tank so variable, this is another source of experimental error associated with the SOD measurement made in the tanks. The change in topography potentially changed the surface area of the sediment by a considerable amount, which may have accounted for the 32% measured change in SOD. Because of the assumption of a flat surface for the SOD calculations and the presence of this variability, the

32% change in SOD cannot be used for definitive conclusions of the effectiveness of the treatment on reducing SOD.

The fact that the oxygen uptake rate is not the driving force in any change in SOD leads to a potential whole-lake treatment by only resuspension. Since the results of this work indicate that the addition of excess oxygen may not be required in order to reduce SOD, treatment costs for whole-lake remediation could be significantly reduced from what was previously thought. Returning to the illustrative example from the introduction and Appendix 1, oxygen costs represented almost 60% of daily operating costs. If this is no longer a cost, then, even though only 32% of the SOD was removed after 5 days of treatment, without oxygen costs, there can be a longer treatment and still be a viable alternative to a more permanent equipment installation and operation. However, suddenly exposing a large amount of sediment that had previously been anaerobic to oxygen can cause a very large increase in oxygen uptake rate in the water column (Bryant et al., 2010), which, without knowing how effective the rapid treatment method is, could be harmful to an ecosystem to which the method is applied. More research is required before large-scale testing could be implemented.

Further, when analyzing the time effect at each stage of treatment, there was only a significant effect due to time for the combined SOD data after 3 hours of treatment and no further significant effect for further treatment (section 3.4). This suggests that it may be possible to achieve a considerable amount of SOD reduction after only 3 hours. The analysis of each time step indicated that, for SOD combined data, the slope of the line representing the change in SOD over time after the first 3 hours of treatment was 439 mg/m²-d/hr of treatment. While this slope is not conclusive because of the considerable amount of error contained within it (due to limited data points and inability to extrapolate further), it does show that there was a large and

significant reduction after only 3 hours of treatment. If this SOD reduction is sufficient to prevent the formation of an anoxic hypolimnion, then this method may still have potential viability as a treatment option if the previously mentioned disadvantages can be overcome. This was demonstrated for the second replication of the experiment when observing the change in SOD measured in the cores and when observing the sedOUR, in both of which there was a significant reduction in value after 3 hours of treatment, but in no other point. For sedOUR, however, in the first replication of the experiment, the results showed a significant time effect between 3 and 27 hours of treatment and between 27 and 147 hours of treatment. Thus, further study is still required to determine with any confidence the feasibility of implementing this method on a larger system.

4.1.3 Limitations of SOD Measurement Methods

Because of the experimental design and the novelty of the two main SOD measurement methods, there were some limitations to the corresponding results and conclusions for the SOD and sedOUR measurements. Primarily, there were a few differences in the results between the two methods for measuring SOD. The SOD measured using the core method showed no significance in change over time, while the tank method did show a significant time effect. The SOD measured using the cores and the tanks both showed no significant difference between oxygenated and non-oxygenated resuspended sediment. Both methods appeared to have large measurement error indicated by the variability of the measurements. It is unclear whether the difference in results from the two methods is primarily because of the error in the tank method or in the core method for measuring SOD, or if neither of them is appropriate for measuring SOD changes as a result of oxygenation and resuspension. Another potential source of error when measuring SOD is the variability in topography of the sediment in the tanks as the sediment was

resuspended and settled into a non-flat surface. The inconsistency of the surface topography would cause a change in the surface area of the sediment from the assumed condition of a flat sediment surface. If the actual sediment surface area is greater than the assumed flat surface area, this will result in an actual SOD that is less than that which is calculated. The error in surface area measurement may be large enough to account for any decrease in SOD measured using the datasonde data in the tanks compared to the cores. The total surface area of the tanks was much greater than the cores, so this type of error is more likely to occur in the tank data. Because of the assumption of a flat surface area (i.e. the same surface area as the tank bottom), any change in surface area of the sediment due to the resuspension and settling changing the topography results in a range of actual surface area that is changing over the course of the experiment. Overall, when examining the R^2 values of all of the SOD GLM plots, they are very low (0.05, 0.34, and 0.17 for SOD in the tanks, SOD in the cores, and SOD combined respectively). Thus there was low correlation between time and SOD based on this statistical analysis, reducing confidence in the results.

The difference between the two measurement methods is likely due to experimental error in both methods. The confidence interval in the GLM analysis of SOD as measured in the cores overlaps 43% of the confidence interval for that of SOD as measured in the tanks (See Appendix 4 for confidence interval calculations). Because for the cores, with each time of collection, water had to be replaced, there is error in the measurement, since the DO of the water, which was replacing collected water, was not controlled to the exact conditions of the remaining water in the core and thus conditions were changing over the course of the measurement.

A further limitation is in the potential administration of this method of treatment in-situ in a lake. Because resuspending sediment in a lake can potentially have many negative effects on
an ecosystem, especially if done without oxygenation, without further study and understanding of what is actually occurring for all sediment components and water quality parameters, it is unadvisable to implement the method on a lake system. Further study is required before this treatment may be used on a large scale.

4.2 Sediment Components

4.2.1 Comparison to Literature Values

A range of values for certain sediment components from literature are presented in Table

21.

Table 21. Range of values for sediment components found in the literature.

Constituent	Range (%)	Source
OM Content	6.2-9.4	Present study
OM Content	13-36	Wisniewski (1999) (Lakes)
OM Content	29 (avg)	den Heyer and Kalff (1998) (Lakes)
OM Content	21.27-40.84	Malecki and White (2004) (Lakes)
	Range (mg/L)	
TP	615-926	Present study
TP	330-4230	Kopacek et al. (2005)

Organic matter content (LOI%) values in this work are low as compared to what was expected from the literature. Wisniewski (1999) measured values for organic matter content in two hypereutrophic lakes in Poland, and den Heyer and Kalff (1998) measured organic matter content in the bottom of 9 Quebec lakes. Malecki and White (2004) measured organic matter contents in three lakes in northern Florida. Kopacek et al. (2005) reported average TP values in sediment samples from 43 European and North American lakes. Values found in Lake Wister in this study are in the lower half of this range.

4.2.2 Discussion of Results

No sediment components were significantly changed in concentration over the course of the treatment. Since total concentrations of elements were measured, any component would need to transfer to the water column to be reduced in concentration or transfer from the water column to the sediment to increase in concentration. The fact that none of the components significantly changed in concentration suggests that either the resuspension has no effect on parameters from the water column settling into the sediment or that any parameters in the water column that might be changed and settled into the sediment by the treatment are changed by slower processes than would have been seen over the course of the five day treatment period. Further studies could examine either a prolonged treatment or a longer-term effect of the rapid treatment on nutrients and other constituents settling into the sediment. The lack of a change in any constituents in the sediment, however, may also be explained by the relative mass of the constituents in the water to the sediment. Even if some particles had settled from the water column to the sediment, there was so much smaller mass of each of the constituents in the water column than in the sediment that a change in component concentrations in the sediment would not have been measurable. For instance, if all of the iron from the initial water sample in the first repetition of the test (0.05 mg/L * 250 L of water = 12.5 mg) had settled into the sediment, which initially contained 585506.5 mg of iron (29691 mg/kg *19.72 kg of sediment = 585506.5 mg), there would have only been an increase of 0.002%. This level of change in the sediment is not easily detectable.

It was expected that the organic matter content would be affected by the treatment as increasing oxygen uptake rate (sedOUR and SOD) would convert organic matter to CO₂ gas and exit the system (Gnaiger, 1983). However, since neither organic matter content nor sedOUR changed significantly over the course of the treatment from 0-147 hours, there is no evidence to suggest that this was the case. This is possibly due to the fact that the organic matter content was relatively low in the sediments used in the present study (Table 9) and any possible change that would occur in the organic matter content and possibly subsequently the sedOUR may only occur when there is sufficient organic matter to be digested through biological processes occurring in the sediment, and these processes may be limited by organic matter content or bacterial populations. Or, it could be that sedOUR is rate limited by other substrates and thus sufficient digestion of the available organic matter resulting in a measurable change did not occur due to a limiting of other necessary components of aerobic digestion. Further, the time allowed for the experiment may not have been sufficient to allow measurable changes in organic matter concentration.

The organic matter may have been so low in this study due to the method by which the sediment was collected. Sediment was collected to approximately 3 to 4 inches deep. If the majority of the more reactive organic matter was only present in the top layers of sediment, collecting so deep may have diluted the organic matter content by mixing any organic matter-rich sediment with inorganic matter-rich sediment. Meyers and Ishiwatari (1993) showed that microbial reworking diminishes the total amount of organic matter in during settling and sedimentation of organic compounds settling into lacustrine sediments.

When examining the time effect after each treatment period for the sediment quality data, there were more significant changes in the parameters during the second replication than the first

replication of the experiment. The concentrations of those sediment components also happened to be much higher in the sediment used for the second replication of the test than the sediment used for the first replication. The fact that there was a higher concentration of each constituent during the second replication could have led to the greater changes in the constituents present in the sediment over time. A possible reason for the difference in concentrations between replications could be the sampling method. Because it was difficult to capture the same depth of sediment with any accuracy between grabs, it is possible that there may have been less dilution of parameters in the second replication if a shallower sample of sediment had been collected. There may have been a difference in the sediment between collection locations or times, as well. Håkansan (1977) showed that, due to wind, water depth, and other factors, the sediment distribution changes the properties in any one small area of sediment over time.

4.3 Water Quality Parameters

4.3.1 Comparison to Literature Values

A range of values for certain water quality parameters found in the literature are presented in Table 22.

Constituent	Range (mg/L)	Source
$NO_3-N + NO_2-N$	0.03-0.78	Present Study
$NO_3-N + NO_2-N$	0.05-1.2	Haggard et al. (2006) (Lake Wister)
NH ₄ -N	0.06-1.61	Present Study
NH ₄ -N	$4.2 \times 10^{-5} - 0.00012$	Prepas and Burke (1997) (Amisk Lake)
TP	0.12-0.64	Present Study
TP	0.007-0.261	USACE (2002) (Lake Wister)
TP	$5.6 \times 10^{-5} - 0.000123$	Prepas and Burke (1997) (Amisk Lake)
Ortho-P	0.01-0.03	Present Study
Ortho-P	0.01-15.43	McCowan et al. (2002) (Lake Wister)
Fe	0.01-0.16	Present Study
Fe	0.05-0.20	USACE (2002) (Typical Lakes)
Fe	0.5-26.3	USACE (2002) (Lake Wister)
Fe	0.003-0.012	Xue et al. (1997) (Lake Sempach)
Fe	0.014-0.028	Xue et al. (1997) (Lake Griefen)
Mn	0.13-2.05	Present Study
Mn	0.01-0.85	USACE (2002) (Typical Lakes)
Mn	0.09-7.5	USACE (2002) (Lake Wister)
Mn	$5.5 \times 10^{-5} - 0.0016$	Xue et al. (1997) (Lake Sempach)
Mn	$5.5 \times 10^{-5} - 0.014$	Xue et al. (1997) (Lake Griefen)

Table 22. Ranges of values of certain water quality parameters found in the literature.

Haggard et al. (2003) found NO₃-N+NO₂-N values in the Beaver Lake Basin, NW Arkansas in a range close to the range found in Lake Wister in this study and using the same methods as in this study. NH₄-N and TP was measured by Prepas and Burke (1997) in the hypolimnion of Amisk Lake, Alberta before treatment by injection of dissolved oxygen and after daily injection of oxygen over a three year period. Values in Table 22 for both NH₄-N and TP for Prepas and Burke's (1997) study show a reduction in concentration after oxygenation, that is, in oxic conditions relative to previously anoxic conditions (1.0 mg/L to 4.6 mg/L average DO before and after the addition of daily dissolved oxygen addition). For both NH₄-N and TP, the values measured in the present study are several orders of magnitude greater. TP was measured by the US Army Corps of Engineer in the surface waters of Lake Wister, (USACE, 2002). The average of the range of values found in the water column of Lake Wister in this study falls within those found in other studies. Orthophosphate has been found in surface runoff from Lake Wister (McCowan et al., 2002). The Ortho-P found in this study falls in the lower end of the range found by McCowan et al.

The US Army Corps of Engineers measured Fe and Mn in the surface waters of Lake Wister (USACE, 2002). The values of found in this study for iron concentration in the water column of Lake Wister are on the lower end of the typical range, and are much lower than those previously found in Lake Wister. Xue et al. (1997) measured both dissolved Fe and dissolved Mn in Lake Sempach, a eutrophic but artificially oxygenated lake and in Lake Greifen, a lake with a seasonally anoxic hypolimnion. The values of Fe and Mn concentrations found in this study are much higher than those found in the hypolimnia of both the oxic Lake Sempach and the anoxic Lake Greifen. The values found in Lake Wister in this study for Mn concentration in the water column of Lake Wister are in the lower range of average, but are slightly higher than those previously found in Lake Wister. Whether the Mn samples measured by the USACE (2002) were in total or dissolved form was not indicated in the cited article, so comparison to values in this study may not be appropriate.

4.3.2 Discussion of Results

Water samples for each treatment were collected immediately before the beginning of each subsequent treatment, which was between 24 and 48 hours after the water columns were aerated in order to measure SOD for each treatment. Because the water was thus aerobic for at least a short period of time for both tanks, this may have had an effect on the constituents found in the water column. Additionally, chemical reactions in both aerobic and anaerobic water/sediment systems are complex and dynamic, and with the amount of time and resources

allotted for this experiment, it was not possible to completely capture all of the effects of water chemistry dynamics of the treatments. For both Mn and NO₃-N + NO₂-N in the water, there was a significant difference between the oxygenated and non-oxygenated tanks. For NO₃-N + NO₂-N, the concentration in the water for both the oxygenated and non-oxygenated tanks increased significantly over time. This is not surprising since there was oxygen in both tanks at some points over the course of the entire experiment. This suggests that there was nitrification occurring in both tanks, though one might have expected a decrease in NH₄-N if nitrification was occurring. The fact that it did not could mean that the NH₄-N from the sediment was moving into the water to replace any that was lost. The concentration changes of NO₃-N + NO₂-N could also have been affected by denitrification that may have occurred in the anaerobic portions of the settled sediment.

For dissolved Mn, the non-oxygenated tank did not experience a significant change in concentration over the course of the treatment, but the oxygenated tank did experience a significant decrease. This could potentially have been because the oxygenated tank provided conditions to oxidize Mn converting it from a soluble form to an insoluble form thereby reducing the concentration of dissolved Mn in the water (Zaw and Chiswell, 1999). However, another oxidizible metal, dissolved Fe, increased in concentration over the course of the treatment suggesting that either there is some experimental variability that may not have captured what process is actually occurring over the course of the treatment time, or that dissolved Fe was added to the water from the sediment. The concentrations of dissolved Fe measured during these experiments were very low near the detection limit, so the measurement error is likely relatively high between readings. Since Fe has a lower oxidation-reduction potential than Mn, and thus

tends to react more slowly than Mn, any decrease in concentration of dissolved Fe may require a longer treatment time period than Mn.

The increase in concentrations of the water quality parameters could be due to a release of the constituents from the sediment. The lack of a corresponding decrease in concentration in the sediment quality parameters because of interaction with the water column is likely due to the large difference in quantity of the parameters in the sediment and in the water. Since the total mass of sediment constituents in the tanks was much greater than that contained in the water column, a very large change in the concentration of the parameter in the water column may have had very little effect on concentration in the sediment. Further, because for several constituents (including Al and Ortho-P), the concentrations were very low and near the detection limits of the lab measurement methods, there is a high measurement error and thus conclusions cannot be readily drawn as values change over time.

When examining the time effect after each treatment period for the water quality data, there were varying effects for different components. For example, for Mn in the oxygenated tank, there appeared to only be an effect due to the treatment after 5 days of treatment, but the TP changed significantly only after 3 hours of treatment. Coming back to the discussion of Mn and Fe, both oxidizible metals, since Mn only showed an effect after 5 days of treatment, and Fe reacts slower, it is not surprising that no effect was seen for Fe in the amount of time allowed for treatment. TP may have changed significantly with so little treatment time due to P being released from the sediment with mixing. Further, when examining the difference between the oxygenated and non-oxygenated tanks for a change in NO₃-N+NO₂-N to indicate whether nitrification was occurring, nitrification was observed sooner in the oxygenated tank (as evidenced by a significant time effect after 27 hours of treatment and 147 hours of treatment in

the oxygenated tank but only a significant time effect after 147 hours in the non-oxygenated tank). This suggests that there was possibly oxygen leaking into the non-oxygenated tank during the longer treatment period or that oxygen added to the water column to measure SOD between treatments did not significantly contribute to nitrification until after a period of time passed.

Summary and Conclusions

5.1 Sediment Oxygen Demand and Suspended Sediment Oxygen Uptake Rate

For SOD as measured in both tanks and cores, there was no significant difference between the sediment resuspended with oxygen and the sediment resuspended without oxygen. For the tank data, there was a significant change in the SOD over time when the effect of added oxygen was removed. For the core data, there was no significant change in SOD over time when the effect of added oxygen was removed. When the tank and core data were combined and analyzed over the duration of the treatment period, there was no significant difference between the core data and the tank data. When this was then combined and all SOD data analyzed together, there was a significant change in the SOD with time. This change was in the negative direction, and the SOD was reduced at a rate of approximately 2.62 mg/m²-d/hr of treatment. For the average initial value for all SOD measurements (1206 mg/m²-d), this corresponds to a total decrease of 32% in the SOD over 147 cumulative hours of treatment, though the variability and error in measurements may account for any significant decrease in SOD, and thus there is very little confidence in the 32% reduction estimate.

For sedOUR, there was no significant difference between the sediment resuspended with oxygen and the sediment resuspended without oxygen. Additionally, when the oxygenated and

non-oxygenated data were combined, there was no significant change in the sedOUR over time for 147 hours of treatment.

In the illustrative example mentioned previously, for the remediation method of the present study to be economically feasible as compared to a typical hypolimnetic oxygen injection system, the rapid resuspension system would need to fully meet the SOD after operating for 5.2 days. Based on the results of the present study, 5 days of treatment will reduce the SOD by 32%, and thus may not be economically feasible as a full-lake remediation technique when operated in the manner presented in this study. Though if this 32% can be achieved after only 3 hours, as the time step analysis suggests, this treatment method may be economically feasible treatment if the 32% reduction in SOD is sufficient to prevent the formation of an anoxic hypolimnion. Further, results suggest that oxygenation of resuspended sediments adds no additional effect to the treatment method, and so the oxygen costs may be removed from the calculations for the duration of treatment necessary for economic feasibility. Since oxygen costs are approximately 60% of the total costs of the treatment operation, removing these would potentially render the treatment economically feasible. While there is still considerable error in these measurements, and they should not be used directly without further research, they do indicate that there is a possibility of a shorter treatment time required than previously thought, which, if found to be true, could further reduce the treatment costs associated with this treatment method. It would be necessary to determine if the amount of treatment possible with this proposed method is sufficient to prevent the formation of an anoxic hypolimnion before *in-situ* treatment was used.

Even though, based on the methods described in the present study, the rapid resuspension method as executed in the study is likely not economically feasible, the results do suggest that resuspension of lake sediments decreases SOD. Further optimization of the method could result

in more economic feasibility, especially if it is determined that a 32% reduction in SOD after 3 hours of treatment is sufficient to prevent or reduce the duration of the formation of an anoxic hypolimnion.

5.2 Sediment Components

There was no significant change in concentration for any of the measured sediment components over time. This suggests that either the resuspension treatment is not sufficient to cause any change in the concentration of the sediment components or that the period of time of either treatment or monitoring is not sufficient to observe any possible effects due to the treatment.

5.3 Water Quality Parameters

For Al, Iron, Mn, OrthoPO₄, TP, COD, NO₃-N+NO₂-N, and TN, there was a significant change in the concentration in the positive direction over time for the treatment. For NH₄-N and TOC, there was not a significant change in concentration over time. However, for some constituents – Al, Iron, and Ortho-P – the measured concentrations were close to non-detectable and thus too susceptible to large measurement errors to reach any definitive conclusions. For Mn and NO₃-N+NO₂-N, there was a significant difference between the tank with added oxygen and the tank without added oxygen. For Mn, over the course of the treatment, there was a negative change in concentration for the tank with added oxygen. For NO₃-N+NO₂-N, there was a positive change in concentration over time for both with and without added oxygen. The effects on NO₃-N+NO₂-N were significantly different between the oxygenated and non-oxygenated tanks. Nitrification likely occurred in both tanks as evidenced by the increase in NO₃-N+NO₂-N

Suggestions for Future Work

Because there was such an increase in SOD between in-lake conditions and laboratory conditions, it would be useful to design an experiment to test the rapid resuspension method on sediments that have not previously been considerably disturbed, including not having been exposed to oxygen. This could potentially be accomplished by collecting intact sediment cores from a lake and reproducing the resuspension and oxygenation within the cores themselves. This would eliminate many of the problems associated with disturbing the sediment to prepare it for the experimental methods in this study, as well as would allow more controlled measurement of SOD in conjunction with the MIMS.

In order to reduce error, it would also be useful to measure the OUR of the water collected in the lake and during each test when collecting water and sediment for the measurement of sedOUR, SOD in the tanks, and SOD in the cores. This would allow a more complete accounting for all of the oxygen uptake in both the tanks and cores.

Additionally, because the initial organic matter in the sediments used in this study was low when compared to the organic matter content of other eutrophic lakes, it would be interesting to determine if the method presented in this study would have a greater effect on organic matter content when applied to lakes with considerably more organic matter content in the sediment to begin with.

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Appendix 1: Oxygen Injection Cost Calculations

Constant Parameters:

Lake surface area: 160 acres = 647,497 m² Average water depth: 2 m Average sediment depth: 2" = 0.051 m Volume: 1.295E9 L SOD: 540 mg/m²-d Hypolimnetic water column oxygen uptake rate: 0.1 mg/L-d sedOUR: 0.166 g O_2/m^3 -s Cost of oxygen (LOX): \$0.33/kg

1.1 Installing a Permanent Hypolimnetic oxygenation system

For a permanent hypolimnetic oxygenation system, it is assumed that there is minimal disturbance to the sediment due to the injection of oxygen. To meet the sediment oxygen demand of a 160 acre (647,497 m²) lake, 349 kg O₂/day would be required (647,497 m² * 540 mg O₂/m²-d). The hypolimnetic water column oxygen uptake rate requires an additional 129 kg O₂/day (0.1 mg/L-d * 1.295E9 L). The total oxygen required, then, is 479 kg O₂/day.

An oxygen injection system capable of delivering this has a capital cost of \$750,000 (BlueInGreen, LLC, personal communication) and a daily operating cost of \$81. 479 kg O₂/day would cost \$158.2/day at a cost of \$0.33/kg.

For five years of operation, this results in a total installation and operating cost of \$1.19 million.

1.2 Leasing a portable oxygen injection system

Rental costs for a portable oxygen injection system are \$33,000/mo with an oxygen generator and an additional \$67,500/mo for electricity costs (numbers provided by BlueInGreen, LLC, personal communication), for a total of \$100,500/mo.

To meet the oxygen uptake rate of 0.166 g O_2/m^3 -s, in a sediment volume of 33022 m³, requires 473,189 kg O_2/day . Since the portable oxygen injection system can put out 6899 kg O_2/day , the area that can be covered at a time is 1.46% (6899/473,189) of 160 acres, which is 2.33 acres.

The treatment time to equal the \$1.19 million permanent installation can be found using the following equation.

Number of days required = $\frac{\$1.19m}{\$100,500/mo} * \frac{30 \text{ days}}{mo} * \frac{2.33 \text{ acres}}{160 \text{ acres}}$

This results in a required treatment time of 5.17 days.

Assuming the costs of the oxygen generator (including rental and electricity) are 60% of the entire rental costs for a portable oxygen injection system that is still capable of operating as a sediment resuspension device without adding oxygen, the monthly rental costs total \$40,200. Replacing the previous monthly rental cost with this value in the previous equation results in a required treatment time of 12.9 days.

Appendix 2: Dissolved Oxygen Data Used for Calculating SOD in Tanks and Cores

Tanks:



Test 1: Time 0 hr, Oxygenated, 8/30/2013

Test 1: Time 0 hr, Non-oxygenated, 8/30/2013





Test 1: Time 3 hr, Oxygenated, 9/4/2013







Test 1: Time 24 hr, Oxygenated, 9/8/2013

Test 1: Time 24 hr, Non-oxygenated, 9/8/2013





Test 1: Time 140 hr, Oxygenated, 9/17/2013

Test 1: Time 140 hr, Non-oxygenated, 9/15/2013-9/17/2013



Test 2: Time 0 hr, Oxygenated, 9/23/2013



Test 2: Time 0 hr, Non-oxygenated, 9/23/2013



Test 2: Time 24 hr, Oxygenated, 9/30/2013



Test 2: Time 24 hr, Non-oxygenated, 9/30/2013



Test 2: Time 140 hr, Oxygenated, 10/7/2013



Test 2: Time 140 hr, Non-oxygenated, 10/8/2013



Cores:

Test 1: 0 hr, Oxygenated, 9/3-9/4/2013



Test 1: 0 hr, Non-oxygenated, 9/3-9/4/2013



Test 1: 3 hr, Oxygenated, 9/6/2013



Test 1: 3 hr, Non-oxygenated, 9/6/2013



Test 1: 24 hr, Oxygenated, 9/10/2013



Test 1: 24 hr, Non-oxygenated, 9/10/2013



Test 1: 140 hr, Oxygenated, 9/18/2013



Test 1: 140 hr, Non-oxygenated, 9/18/2013



Test 2: 0 hr, Oxygenated, 9/25/2013



Test 2: 0 hr, Non-oxygenated, 9/25/2013



Test 2: 3 hr, Oxygenated, 9/27/2013



Test 2: 3 hr, Non-oxygenated, 9/27/2013



Test 2: 24 hr, Oxygenated, 10/3/2013



Test 2: 24 hr, Non-oxygenated, 10/3/2013





Test 2: 140 hr, Oxygenated, 10/9/2013

Test 2: 140 hr, Non-oxygenated, 10/9/2013



Appendix 3: Graphs of Dissolved Oxygen Content over Time used to Measure Sediment Oxygen Uptake Rate

For each sedOUR point, three replicate measurements were performed. These are shown below.



Test 1: Time 0 hr, Oxygenated


Test 1: Time 0 hr, Non-oxygenated



Test 1: Time 3 hr, Oxygenated



Test 1: Time 3 hr, Non-oxygenated







Test 1: Time 24 hr, Non-oxygenated



20.00

0.00

Test 1: Time 140 hr, Oxygenated

60.00

80.00

40.00



Test 1: Time 140 hr, Non-oxygenated







Test 2: Time 0 hr, Non-oxygenated



Test 2: Time 3 hr, Oxygenated







y = -0.0089x + 7.2768

 $R^2 = 0.9581$

20.00

30.00

Time (min)

7 6.9

6.8

6.7

0.00

10.00

40.00

50.00

60.00



Test 2: Time 24 hr, Non-oxygenated







Test 2: Time 140 hr, Oxygenated



Test 2: Time 140 hr, Non-oxygenated

0 0.00

60.00

80.00

 $R^2 = 0.9661$

40.00 Time (min)

20.00

Appendix 4: GLM Analysis of Sediment and Water Quality Parameters

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: SOD Cores (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16 Regression Plot



Whole Model	Test			
Model	-LogLikelihood	L-R	DF	Prob>ChiSq
	-	ChiSquare		_
Difference	0.79287498	1.5857	3	0.6626
Full	107.43547			
Reduced	108.228345			

Effect Tests			
Source	DF	L-R	Prob>ChiSq
		ChiSquare	
TRT	1	0.0352449	0.8511
Time	1	0.8835018	0.3472
TRT*Time	1	0.7074843	0.4003

Parameter Estimates

Term	Estimate	Estimate Std Error		L-R Prob>ChiS		Upper
			ChiSquare	q	CL	CL
Intercept	814.39	61.88	39.52	<.0001*	685.44	943.33
TRT[CTRL]	9.37	49.87	0.035	0.85	-94.55	113.29
Time	-0.79	0.83	0.88	0.35	-2.51	0.94
TRT[CTRL]*(Time-	0.709	0.83	0.71	0.40	-1.02	2.43

44.25)

Generalized Linear Model Fit

Overdispersion parameter estimated by Maximum Likelihood

Response: SOD Cores (data combined)

Distribution: Normal

Link: Identity

Estimation Method: Maximum Likelihood

Observations (or Sum Wgts) = 16





Whole Model Test

Model	-LogLikelihood	L-R	DF	Prob>ChiSq
		ChiSquare		
Difference	0.4222718	0.8445	1	0.3581
Full	107.806074			
Reduced	108.228345			

Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare		-		
Time	1	0.8445436	0.35	81		
Parameter Esti	mates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	814.39	63.33	38.85	<.0001*	682.42	946.35
Time	-0.79	0.85	0.84	0.36	-2.55	0.98

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: SOD Tanks (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 14 Regression Plot



Model	-LogLikelihood		L-R	DF	Prob>ChiSq
		C	hiSquare		
Difference	4.08	925303	8.1785	3	0.0425*
Full	102.	851619			
Reduced	106.	940872			
Effect Tests					
Source	DF	L-R	Prob>ChiSq		
		ChiSquare	-		
TRT	1	1.9764449	0.1598		
Time	1	6.692244	0.0097*		
TRT*Time	1	0.400477	0.5268		
Parameter Estim	ates				
Term		Estimate S	Std Error	L-R	Prob>ChiS

Term	Estimate	Estimate Std Error L-R Prob>ChiS		Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1183.85	128.82	27.31	<.0001*	913.03	1454.68
TRT[CTRL]	146.12	100.29	1.98	0.16	-64.73	356.98
Time	-4.72	1.61	6.69	0.0097*	-8.11	-1.33
TRT[CTRL]*(Time-	-1.03	1.61	0.40	0.53	-4.42	2.36
50.1429)						

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: SOD Tanks (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 14



Time

-4.72

1.75

Whole Model	Test					
Model	-LogLi	kelihood	L-R	DF	Prob>ChiS	q
			ChiSquare			
Difference	2.9	2683081	5.8537	1	0.0155	*
Full	104	.014041				
Reduced	106	5.940872				
Effect Tests						
Source	DF	L-R	Prob>ChiS	Sq		
		ChiSquare				
Time	1	5.8536616	0.0155	5*		
Parameter Est	imates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1183.85	139.97	25.34	<.0001*	889.57	1478.13

5.85

0.0155*

-8.41

-1.04

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: SOD (tank method versus core method) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 30 Regression Plot



Whole Model Test

-LogL	ikelihood	L-R ChiSquare	DF	Prob>ChiSq
3.	90840326	7.8168	2	0.0201*
21	7.263128			
22	21.171531			
DF	L-R ChiSquare	Prob>ChiSq		
1	6.3685079	0.0116*		
1	2.1164724	0.1457		
	-LogI 3. 21 22 DF 1 1	-LogLikelihood 3.90840326 217.263128 221.171531 DF L-R ChiSquare 1 6.3685079 1 2.1164724	-LogLikelihood L-R ChiSquare 3.90840326 217.263128 221.171531 DF L-R ChiSquare 1 6.3685079 0.0116* 1 2.1164724 0.1457	-LogLikelihood L-R DF ChiSquare 3.90840326 7.8168 2 217.263128 221.171531 Prob>ChiSq ChiSquare 1 6.3685079 0.0116* 1 2.1164724 0.1457

Parameter Estimates

Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	990.1	78.06	55.51	<.0001*	832.07	1148.14
Time	-2.7	1.01	6.37	0.0116*	-4.73	-0.65
ID[Cores]	-91.7	61.93	2.12	0.15	-217.07	33.66

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: SOD (all SOD data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 30



Time

-2.62

1.04

Whole Model	Test					
Model	-LogLi	kelihood	L-R	DF	Prob>ChiS	q
			ChiSquare			
Difference	2.8	5016709	5.7003	1	0.0170	*
Full	218	3.321364				
Reduced	221	.171531				
Effect Tests						
Source	DF	L-R	Prob>Chis	Sq		
		ChiSquare				
Time	1	5.7003342	0.0170	0*		
Parameter Est	imates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	980.62	80.59	53.42	<.0001*	817.46	1143.78

5.70

0.0170*

-4.73

-0.50

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: sedOUR (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16 Regression Plot



Whole Model Test

Model	-LogLikelihood	L-R ChiSquare	DF	Prob>ChiSq
Difference	0.27216631	0.5443	3	0.9090
Full	44.0586541			
Reduced	44.3308204			

Effect Tests

Source	DF	L-R ChiSquare	Prob>ChiSq
TRT	1	0.0566498	0.8119
Time	1	0.34475	0.5571
TRT*Time	1	0.1477721	0.7007

Parameter Estimates

Term	Estimate	Std Error	L-R	Prob>ChiSq	Lower	Upper
			ChiSquare		CL	CL
Intercept	13.35	1.18	35.20	<.0001*	10.90	15.81
TRT[CTRL]	0.23	0.95	0.057	0.81	-1.75	2.21
Time	-0.0093	0.016	0.34	0.56	-0.04	0.02
TRT[CTRL]*(Time-44.25)	0.0061	0.016	0.15	0.70	-0.027	0.04

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: sedOUR (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16

Regression Plot



Whole Model Test

	Juci I cs	ι				
Model	-Lo	gLikelihood	L-R ChiSquare	DF	Prob>ChiSq	
Difference	e 0.17	021526	0.3404	1	0.5596	
Full	44.1	606051				
Reduced	44.3	308204				
Effect Tes	sts					
Source	DF	L-R	Prob>ChiS	q		
		ChiSquare				
Time	1	0.3404305	0.5596			
Parameter	Estimat	tes				
Term	Feti	mate Std Erro	or L-R ChiSau	are Pr	ob>ChiSa Lower (٦٢

Term	Estimate	Std Error	L-R ChiSquare	Prob>ChiSq	Lower CL	Upper CL
Intercept	13.35	1.19	35.01	<.0001*	10.88	15.82
Time	-9.30E-3	0.02	0.34	0.56	-0.04	0.024

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Al in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model Test

Model	-LogLikelihood	L-R	DF	Prob>ChiSq
		ChiSquare		
Difference	0.05617894	0.1124	3	0.9903
Full	154.993588			
Reduced	155.049767			
Effect Tests				

Source	DF	L-R	Prob>ChiSq
		ChiSquare	
TRT	1	0.0000129	0.9971
Time	1	0.1023262	0.7491
TRT*Time	1	0.0100831	0.9200

Parameter Estimates

Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	16963.4	1209.1	41.41	<.0001*	14444.0	19482.8
TRT[CTRL]	3.5	974.4	1.29E-5	0.99	-2027.0	2034.0
Time	5.2	16.2	0.10	0.75	-28.5	38.9
TRT[CTRL]*(Time-	-1.6	16.2	0.01008	0.92	-35.3	32.1
44.25)						

Generalized Linear Model Fit

Overdispersion parameter estimated by Maximum Likelihood

Response: Al in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



	Whole	Model	Test
--	-------	-------	------

					D 1 0110	
Model	-LogLi	kelihood	L-R	DF	Prob>ChiS	q
			ChiSquare			
Difference	0.0	5113093	0.1023	1	0.749)1
Full	154	1.998636				
Reduced	155	5.049767				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare		1		
Time	1	0.1022619	0.74	91		
Parameter Est	imates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
		200 2000	ChiSquare	q	CL	CL
Intercept	16963.4	1209.5	41.398	<.0001*	14443.2	19483.6
Time	5.2	16.2	0.102	0.75	-28.5	38.9

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: B in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



124

Upper

14.17

0.008

0.012

0.70

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: B in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16

Regression Plot



Whole Model Test	
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Model	-LogLi	kelihood	L-	R DF	Prob	>ChiSq
	-		ChiSqua	re		-
Difference	0.2	6465392	0.529	03 1		0.4669
Full	28.	2670503				
Reduced	28.	5317042				
Effect Tests						
Source	DF	L-F	R Prob>	ChiSq		
		ChiSquare	e			
Time	1	0.5293078	8 0).4669		
Parameter Es	timates					
Term	Estimate	Std	L-R	Prob>Chi	Lower	Upper
		Error	ChiSquare	Sq	CL	CL
Intercept	13.26	0.44	64.96	<.0001*	12.34	14.17
Time	-0.0043	0.0059	0.53	0.47	-0.017	0.0079

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Ca in Sediment (oxygenation versus non-oxygenation) **Distribution:** Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



CL

88.46

2.25

1.10

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Ca in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model	Test					
Model	-LogLi	kelihood	L-R	DF	Prob>ChiS	q
			ChiSquare			
Difference	0.34	4064314	0.6813	1	0.409	91
Full	106	5.491487				
Reduced	10	6.83213				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare				
Time	1	0.6812863	0.40	91		
Parameter Est	timates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1081.81	58.34	49.81	<.0001*	960.25	1203.37
Time	0.65	0.78	0.68	0.41	-0.98	2.28

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Cu in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model Test Model I og ikelihood

D ...

	1030			
Model	-LogLikelihood	L-R	DF	Prob>ChiSq
		ChiSquare		
Difference	1.73833839	3.4767	3	0.3238
Full	34.4113014			
Reduced	36.1496398			

Effect Tests

ъ

DF	L-R	Prob>ChiSq
	ChiSquare	
1	0.0467934	0.8287
1	2.804216	0.0940
1	0.7534695	0.3854
	DF 1 1 1	DF L-R ChiSquare 1 0.0467934 1 2.804216 1 0.7534695

Parameter Estimates						
Term	Estimate	Std Error	L-R I	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	19.33	0.65	64.74	<.0001*	17.99	20.68
TRT[CTRL]	0.11	0.52	0.047	0.83	-0.97	1.20
Time	0.015	0.0086	2.80	0.09	-0.0029	0.03
TRT[CTRL]*(Time-	-0.0076	0.0086	0.75	0.39	-0.026	0.01
44.25)						

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Cu in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model	Test				
Model	-Log	Likelihood	L-R	DF	Prob>ChiSq
	_		ChiSquare		_
Difference	1	.33928172	2.6786	1	0.1017
Full	3	4.8103581			
Reduced	3	6.1496398			
Effect Tests					
Source	DF	L-R	Prob>ChiSq		
		ChiSquare	-		
Time	1	2.6785634	0.1017		
Parameter Est	imates				
Term	Estima	te Std Error	L-R Pro	ob>ChiS	Lower U
			ChiSquara	a	СІ

Term	Estimate S	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	19.33	0.66	63.95	<.0001*	17.95	20.71
Time	0.02	0.009	2.68	0.10	-0.003	0.034

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Iron in Sediment (oxygenation versus non-oxygenation) **Distribution:** Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



CL

49.2

40.4

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Iron in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



		Chisquare	
Time	1	0.1681701	0.6817

Parameter E	stimates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	33897.3	1473.2	56.5	<.0001*	30827.5	36967.0
Time	8.1	19.7	0.2	0.7	-33.0	49.2

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: K in Sediment (oxygenation versus non-oxygenation) **Distribution:** Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Upper

146.9 2.9

2.46

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: K in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model	Test					
Model	-LogLi	kelihood	L-R	DF	Prob>ChiSc	1
			ChiSquare			
Difference	0.0	1789529	0.0358	1	0.8499)
Full	114	.218382				
Reduced	114	.236278				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare				
Time	1	0.0357906	0.84	99		
Parameter Est	timates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1775.97	94.55	50.20	<.0001*	1578.9	1972.9
Time	0.24	1.27	0.04	0.8499	-2.4	2.9
Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Organic matter in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Upper

CL

8.52

0.27

0.003

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Organic matter in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16

Regression Plot

Time

-0.005



Whole Model	Test				
Model	-LogLi	kelihood	L-R	DF	Prob>ChiSq
			ChiSquare		
Difference	0.7	6961641	1.5392	1	0.2147
Full	22.	4340198			
Reduced	23.	2036362			
Effect Tests					
Source	DF	L-R	Prob>ChiS	q	
		ChiSquare		1	
Time	1	1.5392328	0.214	7	
Parameter Esti	mates				
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower
			ChiSquare	q	CL
Intercept	7.90	0.31	60.17	<.0001*	7.27

0.004

1.54

0.2147

-0.01

Upper CL 8.54

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Mg in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Upper

1986.4

158.8

2.7

2.5

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Mg in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model	Test					
Model	-LogLil	celihood	L-R	DF	Prob>ChiSe	q
			ChiSquare			
Difference	0.00	0019702	0.0004	1	0.984	2
Full	114	.284639				
Reduced	114	.284836				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare				
Time	1	0.000394	0.98	42		
Parameter Est	imates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1788.6	94.9	50.3	<.0001*	1590.8	1986.5
Time	0.03	1.3	0.0004	0.9842	-2.6	2.7

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Mn in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Upper

1681.2

115.03

CL

1.9

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Mn in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16





Whole Mode	l Test					
Model	-LogLi	ikelihood	L-	R DF	Prot	>ChiSq
	•		ChiSqua	re		-
Difference	0.0	0638088	0.012	28 1		0.9101
Full	1	07.97266				
Reduced	10	7.979041				
Effect Tests						
Source	DF	L-F	R Prob>	ChiSq		
		ChiSquare	e			
Time	1	0.012761	8 ().9101		
Parameter Es	stimates					
Term	Estimate	Std	L-R	Prob>Chi	Lower	Upper
		Error	ChiSquare	Sq	CL	CL
Intercept	1548.0	64.0	58.02	<.0001*	1414.7	1681.4
Time	0.1	0.9	0.01	0.9101	-1.7	1.9

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Na in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



44.25)

Lower

CL

65.57

-8.45

-0.09

-0.14

Upper

83.44

5.95

0.15

0.10

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Na in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16

Regression Plot

Time



0.03

Whole Model	Test					
Model	-LogLil	kelihood	L-R	DF	Prob>ChiSq	
			ChiSquare			
Difference	0.	1174759	0.2350	1	0.6279	
Full	6	4.83847				
Reduced	64.9	9559459				
Effect Tests						
Source	DF	L-R	Prob>Chis	Sq		
		ChiSquare				
Time	1	0.2349518	0.627	79		
Parameter Est	imates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	74.51	4.32	47.61	<.0001*	65.51	83.50

0.06

0.23

0.6279

-0.09

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: NH4-N in Sediment (oxygenation versus non-oxygenation) **Distribution:** Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



CL

92.51

11.10

0.03

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: NH4-N in Sediment (data combined) **Distribution:** Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Time



-0.13

Whole Mode	l Test					
Model	-LogLi	kelihood	L-R	DF	Prob>ChiSq	
			ChiSquare			
Difference	1.3	3267632	2.6654	1	0.1026	
Full	69.	6768724				
Reduced	71.	0095488				
Effect Tests						
Source	DF	L-R	Prob>ChiS	q		
		ChiSquare		-		
Time	1	2.6653526	0.102	.6		
Parameter Es	timates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	80.45	5.84	40.85	<.0001*	68.28	92.63

0.08

2.67

0.1026

-0.30

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: S in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



			ChiSquare	q	CL	CL
Intercept	407.38	18.02	55.91	<.0001*	369.83	444.94
TRT[CTRL]	2.56	14.53	0.03	0.8600	-27.70	32.83
Time	0.23	0.24	0.88	0.3469	-0.27	0.73
TRT[CTRL]*(Time-	-0.03	0.24	0.01	0.9149	-0.53	0.489
44.25)						

Upper

Lower

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: S in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16





Whole Model 7	Гest					
Model	-LogLil	kelihood	L-R	DF	Prob>ChiSq	
			ChiSquare			
Difference	0.44	4129273	0.8826	1	0.3475	
Full	87.	7196734				
Reduced	88.	1609661				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare				
Time	1	0.8825855	0.34	75		
Parameter Estin	mates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	407.38	18.05	55.87	<.0001*	369.78	444.99
Time	0.23	0.24	0.88	0.3475	-0.27	0.73

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Zn in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Upper

90.35

6.94 0.15

0.12

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Zn in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16





Whole	Model	Test
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Model	-LogLi	kelihood	L-R	DF	Prob>ChiSq	
	U		ChiSquare			
Difference	0.1	4195197	0.2839	1	0.5942	
Full	64	1.911144				
Reduced	65.	0530959				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare		•		
Time	1	0.2839039	0.59	42		
Parameter Esti	mates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	81.32	4.34	50.14	<.0001*	72.28	90.36
Time	0.03	0.06	0.28	0.5942	-0.09	0.15

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TP in Sediment (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Upper

819.40

61.42

1.15

0.92

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TP in Sediment (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16





Whole	Model	Test
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Model	-LogLi	kelihood	L-R	DF	Prob>ChiSc	1
	U		ChiSquare			•
Difference	0.	0316279	0.0633	1	0.8014	1
Full	99	0.073983				
Reduced	99.	1056109				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare		-		
Time	1	0.0632558	0.80	14		
Parameter Est	imates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	743.03	36.70	52.51	<.0001*	666.57	819.49
Time	0.12	0.50	0.06	0.8014	-0.90	1.15

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Al in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



150

Upper

CL

0.01

0.001

0.00005

1.94E-5

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Al in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16

Regression Plot



Whole Model Test

-Log	Likelihood	L-R	DF	Prob>ChiSq
0		ChiSquare		1
4	.90758391	9.8152	1	0.0017*
-	77.310065			
-	72.402482			
DF	L-R	Prob>ChiSq		
	ChiSquare			
1	9.8151678	0.0017*		
	-Log 4 - - - DF 1	-LogLikelihood 4.90758391 -77.310065 -72.402482 DF L-R ChiSquare 1 9.8151678	-LogLikelihood L-R ChiSquare 4.90758391 9.8152 -77.310065 -72.402482 DF L-R Prob>ChiSq ChiSquare 1 9.8151678 0.0017*	-LogLikelihood L-R DF ChiSquare 4.90758391 9.8152 1 -77.310065 -72.402482 DF L-R Prob>ChiSq ChiSquare 1 9.8151678 0.0017*

Parameter Estimates

Term	Estimate	Std Error	L-R ChiSquare	Prob>ChiSq	Lower CL	Upper CL
Intercept	0.016	0.0006	55.30	<.0001*	0.01	0.01
Time	2.95E-5	8.00E-6	9.82	0.0017*	1.3E-5	4.6E-5

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Iron in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16 Regression Plot



Model	-LogLikelihood		L-R	DF	Prob>ChiSq
		С	hiSquare		
Difference	4.46	6462554	8.9293	3	0.0302*
Full	-31	.901462			
Reduced	-27	.436836			
Effect Tests					
Source	DF	L-R	Prob>ChiSq		
		ChiSquare	1		
TRT	1	2.1112746	0.1462		
Time	1	6.1328739	0.0133*		
TRT*Time	1	2.0842344	0.1488		
Parameter Estin	nates				
Term		Estimate S	td Error	L-R	Prob>ChiSq

Term	Estimate	Std Error	L-R	Prob>ChiSq	Lower	Upper
			ChiSquare		CL	CL
Intercept	0.03	0.01	5.24	0.0220*	0.004	0.05
TRT[CTRL]	-0.01	0.008	2.11	0.1462	-0.03	0.005
Time	0.0004	0.0001	6.13	0.0133*	8.9E-5	0.0007
TRT[CTRL]*(Time-	-0.0002	0.0001	2.08	0.1488	-0.0005	8.1E-5
44.25)						

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Iron in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16

Regression Plot



Whole Model	Test
Model	LogIik

Model	-LogLi	kelihood	L-R ChiSquare	DF I	Prob>ChiSq	
Difference		2.48858	4.9772	1	0.0257*	
Full	-29	9.925416				
Reduced	-27	7.436836				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare		-		
Time	1	4.97716	0.025	7*		
Parameter Es	timates					
Term	Estimate	Std Error	L-R	Prob>ChiSq	Lower CL	Upper CL
			ChiSquare	1		11
Intercept	0.03	0.01	4.23	0.0396*	0.0014	0.05
Time	0.0004	0.0002	4.98	0.0257*	5.15E-5	0.0007

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Mn in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16 Regression Plot



Model	-LogLikelihood		L-R	DF	Prob>ChiSq
	-	(ChiSquare		-
Difference	7.09	9293123	14.1859	3	0.0027*
Full	3.45	5292772			
Reduced	10.5	5458589			
Effect Tests					
Source	DF	L-R	Prob>Ch	iSq	
		ChiSquare		-	
TRT	1	4.4776093	0.03	43*	
Time	1	5.6810721	0.01	71*	
TRT*Time	1	8.2787403	0.00	40*	
Donomaton Estis	motos				
Parameter Estil	nates				

Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1.46	0.09	44.73	<.0001*	1.27	1.65
TRT[CTRL]	0.17	0.08	4.48	0.0343*	0.014	0.33
Time	-0.003	0.001	5.68	0.0171*	-0.006	-0.0007
TRT[CTRL]*(Time-	0.004	0.001	8.28	0.0040*	0.002	0.007
44.25)						

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: COD in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16 Regression Plot



Whole Model Test

Model	-LogLikelihood	L-R	DF	Prob>ChiSq
		ChiSquare		
Difference	4.77955455	9.5591	3	0.0227*
Full	74.6962176			
Reduced	79.4757721			

~

Effect Tests

DF	L-R	Prob>ChiSq
	ChiSquare	
1	0.7941095	0.3729
1	7.0857252	0.0078*
1	3.0424255	0.0811
	DF 1 1 1	DF L-R ChiSquare 1 0.7941095 1 7.0857252 1 3.0424255

Parameter Estimates Term

Term	Estimate	Std Error	L-K	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	30.15	8.0	10.17	0.0014*	13.49	46.81
TRT[CTRL]	-5.82	6.44	0.79	0.3729	-19.24	7.61
Time	0.32	0.11	7.09	0.0078*	0.10	0.54
TRT[CTRL]*(Time-	-0.20	0.117	3.04	0.0811	-0.42	0.037
44.25)						

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: COD in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16

Regression Plot



Model	-LogLikelihood	L-R	DF	Prob>ChiSq
	-	ChiSquare		_
Difference	2.92864793	5.8573	1	0.0155*
Full	76.5471242			
Reduced	79.4757721			

Effect Tests

Source	DF	L-R	Prob>ChiSq
		ChiSquare	
Time	1	5.8572959	0.0155*

Parameter Estimates

Term	Estimate	Std Error	L-R ChiSquare	Prob>ChiSq	Lower CL	Upper CL
Intercept	30.15	8.98	8.54	0.0035*	11.44	48.86
Time	0.32	0.12	5.86	0.0155*	0.069	0.57

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TOC in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16**Regression Plot**



Model	-LogLik	elihood	L-]	R I	DF	Prob>ChiSq	
	U		ChiSquar	e		1	
Difference	0.4ϵ	5104466	0.922	1	3	0.8201	
Full	41	692634	01722	-	e	0.0201	
Reduced	42.1	536786					
Reduced	12,1	550700					
Effect Tests							
Source	DF	L	-R Prob	>ChiSq			
		ChiSqua	ire	1			
TRT	1	0.06675	55	0.7961			
Time	1	0.80726	23	0.3689			
TRT*Time	1	0.05421	66	0.8159			
Doromotor Estin	notos						
	nates		0.15		ΙD	$\mathbf{D} = 1 \cdot \mathbf{C} 1 \cdot \mathbf{C}$	т
Term		Estimate	e Std Error		L-K	Prob>ChiS	Lower
				ChiS	quare	q	CL
Intercept		5.01	1.02	14.77	70844	0.0001*	2.89
TRT[CTRL]		0.21	0.82		0.07	0.7961	-1.50

2.89 7.13 -1.50 1.92 0.210.07 0./961 IKI[CIKL] 0.82-0.01 0.014 0.81 -0.04 0.02 Time 0.3689 TRT[CTRL]*(Time-0.014 0.05 0.03 -0.003 0.8159 -0.03 44.25)

Upper

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TOC in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Time



-0.01

0.014

Whole Model 7	Гest					
Model	-LogLil	kelihood	L-R	DF	Prob>ChiSq	
	-		ChiSquare		_	
Difference	0.4	0067125	0.8013	1	0.3707	
Full	41.	7530074				
Reduced	42.	1536786				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sq		
		ChiSquare				
Time	1	0.8013425	0.37	07		
Parameter Estin	nates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	5.01	1.02	14.70	0.0001*	2.88	7.13

0.80

0.3707

-0.04

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: NH₄-N in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16 **Regression Plot**



Model	-LogLik	elihood	L-F	R DF	Prob>ChiS	Sq
			ChiSquare	e		
Difference	0.52	767803	1.0554	4 3	0.787	79
Full	7.47	490052				
Reduced	8.00	257855				
Effect Tests						
Source	DF	L-l	R Prob	>ChiSq		
		ChiSquar	e			
TRT	1	0.351049	6	0.5535		
Time	1	0.645702	5	0.4217		
TRT*Time	1	0.076916	7	0.7815		
Parameter Estim	ates					
Term		Estimate	Std Error	L-F	R Prob>ChiS	Lower
				ChiSquar	e q	CL
Intercept		0.63	0.12	16.0	7 <.0001*	0.38

Intercept	0.63	0.12	16.07	<.0001*	0.38	0.88
TRT[CTRL]	-0.06	0.10	0.35	0.5535	-0.26	0.14
Time	-0.001	0.002	0.65	0.4217	-0.005	0.002
TRT[CTRL]*(Time-	0.0004	0.0022	0.08	0.7815	-0.003	0.004
44.25)						

Upper

CL

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: NH₄-N in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



-0.001

Time

0.002

Whole Model	Test					
Model	-LogLil	kelihood	L-R	DF	Prob>ChiSq	
	_		ChiSquare		_	
Difference	0.3	1452753	0.6291	1	0.4277	
Full	7.6	8805103				
Reduced	8.00	0257855				
Effect Tests						
Source	DF	L-R ChiSquare	Prob>ChiS	q		
Time	1	0.6290551	0.427	7		
Parameter Est	imates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	0.63	0.12	15.80	<.0001*	0.377	0.88

0.63

0.4277

-0.005

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: NO_3 -N+NO₂-N in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model	Test
Model	-LogLikelihood
Difference	26.3167048

Effect Tests

Full Reduced

Source	DF	L-R	Prob>ChiSq
		ChiSquare	-
TRT	1	4.6140457	0.0317*
Time	1	52.429627	<.0001*
TRT*Time	1	0.0847774	0.7709

-23.447145

2.86955963

Parameter Estimates

Term	Estimate	Std	L-R	Prob>ChiSq	Lower	Upper
		Error	ChiSquare		CL	CL
Intercept	0.05	0.02	6.08	0.0137*	0.01	0.08
TRT[CTRL]	-0.03	0.01	4.61	0.0317*	-0.06	-0.003
Time	0.005	0.0002	52.43	<.0001*	0.004	0.005
TRT[CTRL]*(Time- 44.25)	6.8E-5	0.0002	0.0847774	0.77	-0.0004	0.0006

DF

3

Prob>ChiSq

<.0001*

L-R

ChiSquare 52.6334

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TN in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16 **Regression Plot**



Whole	Model	Test
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Model	-LogLik	elihood	C1 . 0	L-R	D	F	Prob>ChiSq
Difference	6.06	7/133/		quare		3	0 0060*
Full	0.00 8.76	445665	14	.1340		5	0.0007
Reduced	14	4.83187					
Effect Tests							
Source	DF	L-	R	Prob>C	ChiSq		
		ChiSquar	re				
TRT	1	0.305752	23	0	.5803		
Time	1	11.6425	57	0.0	0006*		
TRT*Time	1	0.710231	7	0	.3994		
Parameter Estin	nates						
Term		Estimate	Std E	Error		L-R	Prob>ChiS
					ChiSc	quare	q
Τ		1 00		0 1 0	_		00011

Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1.08	0.13	26.77	<.0001*	0.81	1.35
TRT[CTRL]	-0.06	0.10	0.31	0.58	-0.28	0.16
Time	0.007	0.0017	11.64	0.0006*	0.0036	0.01
TRT[CTRL]*(Time-	-0.001	0.0017	0.71	0.40	-0.005	0.002
44.25)						

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TN in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Whole Model	Test					
Model	-LogLil	kelihood	L-R ChiSquare	DF	Prob>ChiSq	
D:ff	5.5	(500072		1	0.0000*	
Difference	5.50	0399872	11.1520	1	0.0008*	
Full	9.20	6587127				
Reduced	1	4.83187				
Effect Tests						
Source	DF	L-R	Prob>Chi	Sa		
		ChiSquare		~ 1		
Time	1	11.131997	0.000	8*		
Parameter Esti	mates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	1.08	0.134	25.96	<.0001*	0.80	1.36
Time	0.007	0.002	11.13	0.0008*	0.003	0.011

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: Ortho-PO₄ in Water (oxygenation versus non-oxygenation) **Distribution:** Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16**Regression Plot**



whole whole i cou

Model	-LogLik	kelihood	L-R	DF	Prob>ChiSq
	U	Cł	niSquare		
Difference	11.1	1917363	22.3835	3	<.0001*
Full	-74	.195473			
Reduced	-63	.003736			
Effect Tests					
Source	DF	L-R	Prob>Ch	iSq	
		ChiSquare		-	
TRT	1	2.3753933	0.12	233	
Time	1	21.237492	<.00	01*	
TRT*Time	1	1.8125891	0.1	782	
Parameter Estin	mates				

Term Estimate Std Error L-R Prob>ChiS Lower Upper ChiSquare CL CL q 0.0097 0.00073 39.92 <.0001* 0.0082 0.01 Intercept TRT[CTRL] 0.0009 0.00056 2.38 0.12 -2.8E-4 0.002 <.0001* Time 6.48E-5 21.24 4.5E-5 8.5E-5 9.73E-6 TRT[CTRL]*(Time-1.35E-5 9.73E-6 1.81 0.18 -6.7E-6 3.3E-5 44.25)

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: OrthoPO4 in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 16



Time

6.48E-5

1.1E-5

Whole Mode	l Test					
Model	-LogLi	kelihood	L-R	DF	Prob>ChiSq	l
			ChiSquare			
Difference	9.	2168437	18.4337	1	<.0001*	:
Full	-7	72.22058				
Reduced	-63	3.003736				
Effect Tests						
Source	DF	L-R	Prob>ChiS	Sq		
		ChiSquare				
Time	1	18.433687	<.000	1*		
Parameter Es	timates					
Term	Estimate	Std Error	L-R	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	0.0097	0.0008	36.34	<.0001*	0.008	0.01

18.43

<.0001*

4.18E-5

8.77E-5

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TP in Water (oxygenation versus non-oxygenation) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 14 **Regression Plot**



Whole Model	Test	
Model	-LogLikelihood	L-R
		ChiSquare
Difference	4.0189996	8.0380
Full	-12.156412	
Reduced	-8.1374119	

Effect Tests

Source	DF	L-R	Prob>ChiSq
		ChiSquare	
TRT	1	0.0066554	0.9350
Time	1	7.3970001	0.0065*
TRT*Time	1	1.0643405	0.3022

Parameter Estimates

Term	Estimate	Std Error	L-R	Prob>ChiSq	Lower	Upper
			ChiSquare		CL	CL
Intercept	0.18	0.034	16.0	<.0001*	0.11	0.25
TRT[CTRL]	-0.002	0.027	0.0067	0.94	-0.06	0.05
Time	0.001	0.0004	7.40	0.0065*	0.0004	0.002
TRT[CTRL]*(Time- 46.7143)	-0.0004	0.0004	1.06	0.30	-0.001	0.0004

DF

3

Prob>ChiSq

0.0452*

Generalized Linear Model Fit Overdispersion parameter estimated by Maximum Likelihood Response: TP in Water (data combined) Distribution: Normal Link: Identity Estimation Method: Maximum Likelihood Observations (or Sum Wgts) = 14



Time

0.001

4.4E-4

Whole Model	Test					
Model	Iodel -LogLikelihood		L-R	DF	Prob>ChiSq	
	-		ChiSquare		_	
Difference	3.4	8374519	6.9675	1	0.0083*	
Full	-11	.621157				
Reduced	-8.	1374119				
Effect Tests						
Source	DF	L-R ChiSquare	Prob>ChiSc	1		
Time	1	6.9674904	0.0083*	k		
Parameter Est	imates					
Term	Estimate	Std Error	L-R F	Prob>ChiS	Lower	Upper
			ChiSquare	q	CL	CL
Intercept	0.184	0.035	15.28	<.0001*	0.11	0.25

6.97

0.0083*

0.0004