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Characterizing Current and Geologic Phosphorus in Utah Lake Sediment Using
Field Samples, Laboratory Methods, and Statistical Analysis:
Implications for Water Quality Issues

Hani Yousef Ahmad Abu Hmeidan

A thesis submitted to the faculty of
Brigham Young University
in partial fulfillment of the requirements for the degree of

Master of Science

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ABSTRACT

Characterizing Current and Geologic Phosphorus in Utah Lake Sediment Using Field Samples, Laboratory Methods, and Statistical Analysis: Implications for Water Quality Issues

Hani Yousef Ahmad Abu Hmeidan
Department of Civil and Environmental Engineering, BYU
Master of Science

Phosphorus is an essential nutrient for aquatic life forms and plays a major role in the algae blooms that occur in lakes and reservoirs. It is considered a primary limiting nutrient of phytoplankton growth in streams, lakes, and reservoirs. Excess amounts of phosphorous may cause excess growth and biomass of algae. If phosphorus is available in excess, often from sewage and industrial discharges, the high levels in a lake or reservoir can lead to eutrophication.

Utah Lake is a shallow, basin-bottom lake in a semi-arid climate with sediments that are thousands of feet thick. Starting 165 years ago, humans have been discharging wastewater into Utah Lake, which in our day has raised serious questions on how the state can mitigate the negative effects of the external nutrient loading. Even though Utah Lake receives a significant amount of anthropogenic phosphorous, there are high levels of phosphorous in geologic deposits in the area, providing a long-term natural source. This study intends to provide data on the current distribution of phosphorous in lake sediments, potential for that phosphorous to be released into the water column affecting phytoplankton growth, and how historic lake sediment phosphorous levels compare to the levels in current sediments.

Sediments play an important role in the overall metabolism of shallow lakes. They supply the water column with phosphorus and must be considered as they serve as a sink and source. More than 50 branches of surface flow discharge into Utah Lake, 15 of which are major. Based on previous data, a positive retention of phosphorus from these branches occurs in the lake, of which the sediment plays a role. Phosphorus release from sediment occurs under very complicated processes under many different conditions. Some main influential factors include the iron and calcium content, redox potential, microbial processes, turbidity, sediment resuspension, temperature, and pH.

In this study, I analyzed 85 sediment samples sampled across Utah Lake for total phosphorus. I created Geospatial maps to show the phosphorous distribution. The data showed an average phosphorus level of 666 ppm and varied in distribution throughout the lake, though the majority of the lake had levels in the 600 to 800 ppm range. There were a few samples, which had lower total phosphorus levels, in the 200 to 300 ppm range. Based on the map, I found that these lower values were in locations representing potential springs. I hypothesize that this underground water source leached some of the phosphorous from the sediments in these areas. I found that total phosphorus concentrations in current lake sediment are quite similar to phosphorus levels in historic lake sediments levels. I also performed laboratory experiments to characterize sediment-water interactions and estimate the amount of phosphorus that could be released from lake sediments to the water column.

Keywords: total phosphorus, sediment

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My sincere thanks also go to my friends for all the fun and support. Last but not least, I would like to thank my wonderful parents for providing their continuous support and love over my whole lifespan and who without them none of this would have been possible.

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1 INTRODUCTION

Understanding phosphorous (P) availability in lakes around the world has grown rapidly in significance because of its effects on water quality. In addition to other factors such as nitrogen, light, wind, and temperature, phosphorus can be, and generally is, the limiting nutrient that causes eutrophication in lakes and water streams (Correll, 1999). Phosphorus supports the growth of floating organisms called phytoplankton which are predominantly algae and cyanobacteria (Zhu, Wan, & Zhao, 2010).

The Environmental Protection Agency (EPA) National Eutrophication Survey Program reported Utah Lake to be the most eutrophic lake in Utah which means that the lake has an excessive amount of nutrients that causes a concern about its water quality (UtahDepartmentofEnvironmentalQuality, 1973). The Utah State Department of Environmental Quality (DEQ) is currently considering implementing regulations to reduce the amount of nutrients coming into the Lake from wastewater treatment plants. Large amounts of phosphorus coming from human-related and natural sources are being delivered to and stored in the lake each year (UtahDepartmentofEnvironmentalQuality). Regulations would limit phosphorous inflows to reduce the concentration of phosphorus in the water column that is available to support phytoplankton growth.

However, because the lake is shallow, the sediments are also an important source of phosphorus to the water column. The sediments must be considered as they serve as both a sink and a source of phosphorus. Even with the regulations and the reduction of phosphorus coming from wastewater treatment plants, Utah Lake sediments already contain huge amounts of phosphorus that can maintain detrimental levels of phosphorous in the water column for many years.

The objective of this report is to provide an analysis on phosphorous levels in the sediments of Utah Lake, providing data for total phosphorus, and how that phosphorus is stored in the sediments. This study also provides phosphorous spatial distributions maps and how current lake sediment levels compare with historic lake sediments.

2 BACKGROUND AND LITERATURE REVIEW

This section of the report provides a Literature review about phosphorous, and a background about Utah Lake.

2.1 Phosphorus Cycle

Phosphorous is an important nutrient that occurs in the environment. It is a primary limiting nutrient of phytoplankton growth in streams, lakes, and reservoirs. Excess amounts of phosphorous may cause excess growth and biomass of algae (Carpenter, 2005). If phosphorus is available in excess, often from human sources like sewage, industrial discharges, and fertilizers and pesticides, the high levels in a lake or a reservoir can lead to eutrophication (Carpenter, 2005).

Also, Geologic sources can also provide high levels of phosphorous in some regions. Figure 1 shows the phosphorus cycle and how natural and human activities move phosphorus in the environment.

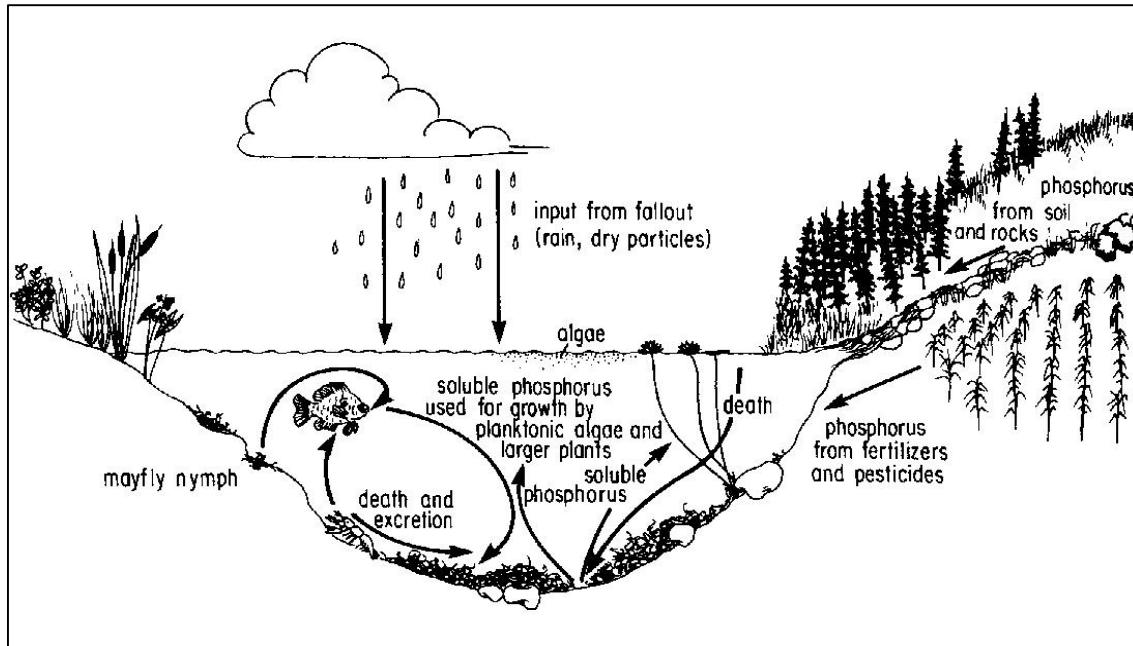


Figure 1: Phosphorus Cycle

2.2 Phosphorus in Water

Phosphorous is found in fresh water in two main phases, particulate phase, or dissolved phase. The sum of phosphorous in these two phases is called total phosphorous (TP).

Differentiation between soluble and dissolved phosphorous can be determined by whether they pass through a $0.45 \mu\text{m}$ membrane filter (Murphy & Riley, 1962). Dissolved phosphorous is divided into two components: soluble reactive phosphorous (SRP) that is also called orthophosphates and soluble unreactive or soluble organic phosphorous (Carlson, 1982). SRP-levels contribute to phytoplankton growth, as it is available to the organisms, unreactive phosphorous is not available to the organisms. Phosphorus naturally changes form, so scientists tend to measure total phosphorus rather than any single form to indicate the amount of Phosphorous available for algae growth.

Total phosphorus effects in fresh water have been studied widely and for most temperate reservoirs, phosphorous is accepted as the limiting nutrient that causes eutrophication in lakes and water streams. This theory has been tested by D.W. Schindler in 1974. The objective of the experiment was to test the effect of phosphorus on water quality. The methodology was to use a large lake, possessing two similar basins separated by a shallow neck, and to divide the lake into two equal sides. The sides were separated using a sea curtain of vinyl that was connected to bedrock in the narrow part of the lake. Starting in May 1973, systematic additions of Nitrogen and Carbon were applied to both basins. However, phosphorus was only added to the northeast basin of the lake. This was done to create a control environment to be compared to the test environment that had phosphorus added to it. The results showed that a layer of blue-green algae covered the northeastern basin, and none was found in the southwestern one. Figure 2 shows a photograph of the algae from September 4th 1973 (Schindler, 1974).



Figure 2: Effects of Phosphorus Addition to Lakes (Schindler, 1974)

2.3 Phosphorus in Sediments

Deposited sediments can act as either a source or a sink for phosphorous depending on the amount of dissolved oxygen and phosphorus in the water (Mayer & Gloss, 1980). Vollenweider established a model to demonstrate phosphorus retention, which is a mass flux computed as a difference between input phosphorus and output phosphorus of the lake on annual basis. In equilibrium, phosphorus retention is identical to net sedimentation which means that a part of phosphorus is retained within the sediment of the lake. The established model displays the retention percentage depending on the hydraulic retention time relating in-lake phosphorus (P_{lake}) to inlet concentrations (P_{in}) and hydraulic residence time (tw):

$$P_{lake} = P_{in}/(1+tw^{0.5}) \text{ (Vollenweider, 1969).}$$

In the case of Utah Lake, which is considered to have a high trophic levels, phosphorus release from sediments may exceed phosphorus sedimentation for periods of months, weeks, or years (Boström, Andersen, Fleischer, & Jansson, 1988), which means that in eutrophic lakes steady state is not achieved. Thus, the Vollenweider model cannot describe phosphorus retention in these kinds of lakes. For these transient conditions, Søndergaard et al. indicated that phosphorus retention is the net difference between “the downward flux caused mainly by sedimentation of particles continuously entering the lake or produced in the water column (algae, detritus etc.), and the upwards flux or gross release of phosphorus driven by the decomposition of organic matter and the phosphorus gradients and transport mechanisms established in the sediment” (Søndergaard, Jensen, & Jeppesen, 2003). If upward flux exceeds downward flux, reduction of phosphorous in lake inflows may have little to no effect on water quality conditions as phosphorous levels in the water column may be controlled by sediment interactions.

When phosphorus enters into sediments, it can become either permanently deposited or deposited in forms that can be released by various mechanisms described in the next section.

Figure 3 shows a schematic presentation of phosphorus pathways when entering a lake, and some phosphorus compounds found in the sediment (Søndergaard et al., 2003).

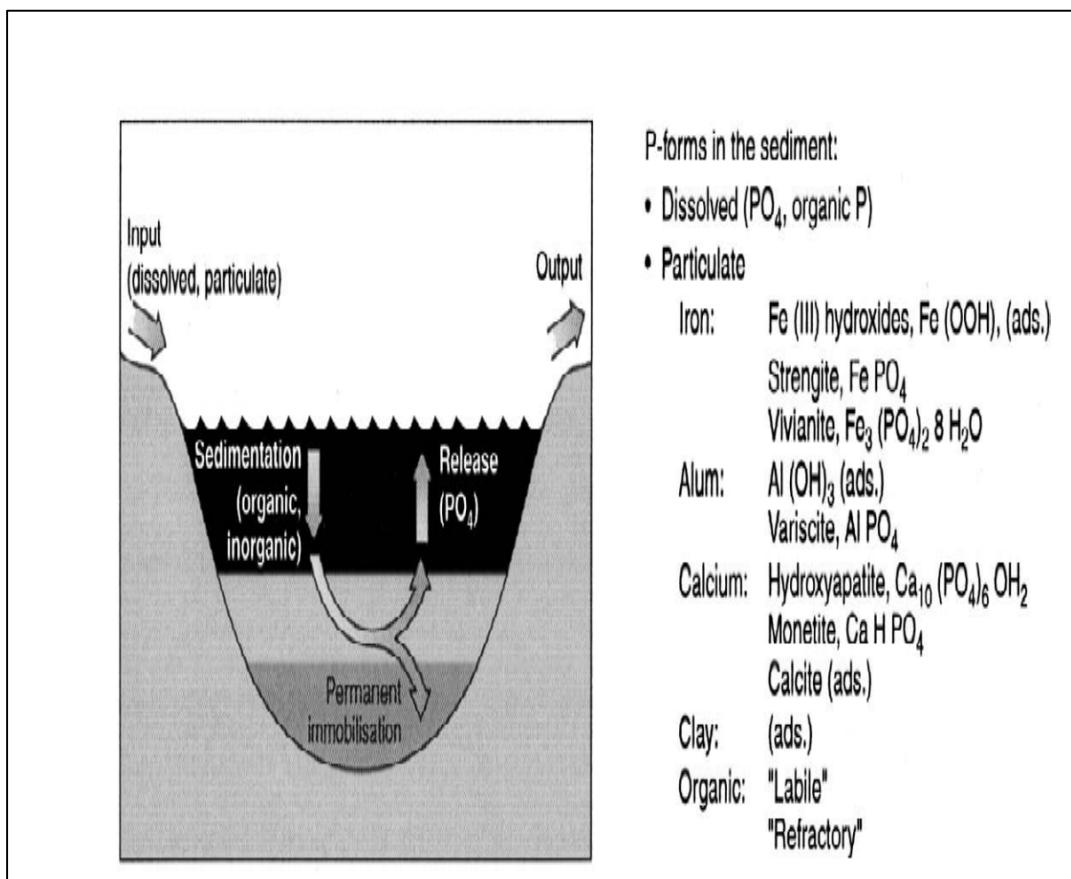


Figure 3: Schematic Presentation of Phosphorus Pathways when Entering a Lake, and Some Phosphorus Compounds Found in the Sediment (Søndergaard, 2003)

2.1 Phosphorus Release from Sediments

Sediment-water interaction related to phosphorous retention or release is a complicated process and is governed by different considerations depending on the type of lake and chemical compositions as no two lakes are the same (Søndergaard et al., 2003). Different parameters may influence these interactions including dry weight, organic content, and content of iron (Fe), aluminum, manganese, calcium, clay and other elements with the capacity to bind and release phosphorus, and many other parameters using different mechanisms (Søndergaard, Windolf, & Jeppesen, 1996). This section talks about some of these mechanisms such as: Fe:P ratio, reduction-oxidation (Redox) conditions, resuspension, turbidity, biological activity, microbial processes, temperature, and pH (Søndergaard et al., 2003).

2.1.1 Iron and Redox

Different studies have been performed that link retained phosphorus with iron levels in the sediment. In 1936, Einsele and Mortimer performed studies that showed that oxygenated sediments retain phosphorus by fixation to Fe(III) whereas reduced sediments release phosphorus by reduction and dissolution of Fe(II) (Einsele, 1936). More studies were conducted to confirm this conclusion and it was recently re-verified by Petticrew et al in 2001 who concluded that phosphorus release rates are strongly correlated to iron bound phosphorus with a variability coefficient of $r^2 = 0.88$ (Petticrew & Arocena, 2001).

Hydroxides and ferric oxides presented in sediments are highly effective in binding with phosphorus under oxidizing conditions (Søndergaard et al., 2003). Jensen et al. showed that the retention capacity is proportional to the Fe:P ratio as long as the ratio exceeds 15 by weight (Henning S Jensen, Kristensen, Jeppesen, & Skytthe, 1992). However, Caraco et al. proposed

that phosphorus release is present as long as the Fe:P ratio is less than 10 and can be regulated as long as the ratio exceeds that (Caraco, Cole, & Likens, 1993). An experiment performed by Jensen et al. to analyze phosphorus adsorption properties from 12 different lakes concluded that aerobic sediments' ability to buffer phosphates concentration is related to the Fe:P ratio whereas the total iron is connected to the maximum adsorption capacity (Henning S Jensen et al., 1992).

Classically, sediment-water interactions were explained by redox conditions in the surface sediments (Søndergaard et al., 2003). According to Boström and Pettersson, sediments are grouped into three groups according to their redox sensitivity. Type I are redox-insensitive and do not release phosphorus into water, while types II and III are redox-sensitive and redox-insensitive respectively but both release phosphorus into the water column. These categories are related to iron and other metal contents.

The redox conditions regulate phosphorus release into pore water for the fraction of phosphorous bond to the iron minerals. When dissolved oxygen is relatively available, iron is oxidized and precipitates as solid minerals. These minerals adsorb phosphorus and sediment retention increases. However, under anoxic conditions, iron is reduced, the iron minerals dissolve, and loses their binding capacity as the iron is solubilized and enters the water column. When Fe(III) is reduced into Fe(II), the iron mineral solubility is significantly higher, up to two orders of magnitude, resulting in iron dissolution. As the iron dissolves, phosphorus is released into water (Lijklema, 1977; Mortimer, 1941). In shallow lakes, as in the case of Utah Lake, the whole water column is usually oxic, thus, an oxic surface layer of the sediment is created with a high capability to bind with phosphorus (Søndergaard et al., 2003). Under well-mixed conditions in spring and autumn, this layer moderately prevents phosphorus release(Penn et al., 2000).

However, when this layer is chemically reduced at the onset of anoxia, phosphorus can be freed and high phosphorus release rates are observed (Søndergaard et al., 2003) particularly in sediments where the ratio of Fe (II) to phosphorus is greater than 2 (Gunnars, Blomqvist, Johansson, & Andersson, 2002). This work shows that the oxidized layer may control seasonal phosphorus release, but does not affect long term sediment-water exchange (Søndergaard et al., 2003) especially in shallow lakes where even minor disturbances may enhance the anoxic events (Søndergaard et al., 2003) or in lakes, such as Utah Lake, where other processes, such as boating and carp, regularly disturb the sediment, exposing anoxic sediments to the water column. The effect of the presence or absence of oxygen on phosphorus release was studied by D. Demare and V. Ruban who concluded that phosphorus release was avoided as long as the concentration of dissolved oxygen stayed above 0.5 mg/L (Ruban & Demare, 1998).

2.1.2 Microbial Processes and Organic Material

Phosphorus cycling may be affected by bacteria, which effectively catalyze the release processes and also create reducing conditions through decomposition of organic matter. Many of the chemical processes that affect phosphorous equilibrium are relatively slow, but are catalyzed by biological activity significantly increasing the rate at which these processes occur. For example, iron reduction or oxidation can be relatively slow in most chemical environments on the order of months to years or longer; biological processes can significantly increase the rate of this process to time scales on the order of hours to days (Grantham, Dove, & Dichristina, 1997).

Besides iron dissolution, biological processes can directly affect phosphorus levels. It is often difficult to distinguish whether oxygen depletion is the result or the cause of phosphorus release as microbial processes consume oxygen and produce phosphorus (Hupfer &

Lewandowski, 2008). Bacteria affect phosphorus cycling by releasing phosphorus during mineralization of organic matter. High amounts of freshly produced organic material are delivered to sediments in shallow lakes. When the electron acceptor supply, including oxygen and/or nitrate, is sufficient in an oxidizing environment, the high organic input leads to potential increase in mineralization rate, which increases phosphorus release. The electron acceptor supply under oxidizing conditions is often supplied, as both elements, oxygen and nitrate, are found a few millimeters beneath the sediment surface. Once oxygen and nitrate are exhausted as electron acceptors, then iron or sulfates are used. The environment where the iron is the electron acceptor results in the dissolution of iron from the sediments and release of the bound phosphorous.

2.1.3 Resuspension, Turbidity, and Biological Activity

The effects of resuspension of sediments on phosphorous release have been widely studied and many experiments have been conducted. Resuspension is a mechanical process that permits settled sediments to be redistributed in the water column. Koski-Vähälä and Hartikainen explained that phosphorus release to the water column is affected by different biological and physico-chemical factors, and resuspension is one of these factors that affect the internal P loading by “mechanically mediating the phosphorus exchange between suspended material and the water column” (Koski-Vähälä & Hartikainen, 2001). For resuspension, the majority of the phosphorous released is not from the iron-bound fraction, but more often from the salt-extractable, or sorbed fraction.

Different factors causing resuspension have been analyzed for their effect on phosphorus release. Waves (Lijklema, Aalderink, Blom, & Van Duin, 1994), wind (Kristensen, Søndergaard, & Jeppesen, 1992; Sondergaard, Kristensen, & Jeppesen, 1992), ice cover, and climate change

due to global warming (Niemistö & Horppila, 2007), are some of these factors. An experiment conducted by Fan, and Zhang estimated that internal phosphorus loading encouraged by resuspension to be 8 to 10 times greater than release from undistributed sediments (Fan, Zhang, & Qu, 2001). Another experiment by Søndergaard found that phosphorus release from resuspended sediments is about 20 to 30 times greater than undistributed sediments (Søndergaard et al., 1992).

Utah Lake is known of having large amounts of carp fish. Carp and other benthivorous fish have a great effect on resuspension of sediments and concentrations of nutrients in the water column. The digestive activities of these fish release phosphorus into the water and their activities increase the resuspension of sediments as they search for food. Thus, a dense population of these kinds of fish can lead to higher phosphorous content in the water column and algal growth in lakes.

In addition to resuspension and biological activity, turbidity plays a big role on internal phosphorus loading in Lakes by influencing nutrient levels, turbid lakes have higher total phosphorus during summer while in clear water lakes total phosphorus remains constant almost all season. Jeppesen indicates that the turbid state, as in Utah Lake's case, is characterized by having a lack of submerged macrophytes, low piscivorous: planktivorous fish ratio, low zooplankton: phytoplankton ratio, high algal biomass and usually high resuspension by wind disturbance of sediments and fish (Jeppesen, Jensen, Søndergaard, & Lauridsen, 1999). This high resuspension can release phosphorous into the water column.

2.1.4 Temperature

Temperature also affects phosphorus release from sediments, as higher temperatures can increase microbial activity creating anoxic or anaerobic conditions and catalyzing mineral dissolution (Gächter & Meyer, 1993; Holdren & Armstrong, 1980; Henning Skovgaard Jensen & Andersen, 1992). When microbial activity is increased, organisms use more oxygen leading to anoxic conditions, thus, a high release of Iron bounded phosphorus occurs (Mortimer, 1941). A study done in West Lake in China (Zhang et al., 2016) described the effect of temperature on phosphorus release in sediments. In this study, temperature and light were varied. The study concluded that released amount of phosphorus varied with the increase of temperature as shown in figure 4.

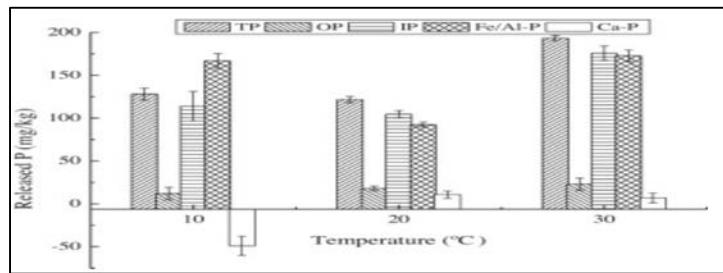


Figure 4: Effects of Temperature on Sediment Phosphorus Release (Zhang, 2016)

A laboratory experiment was also performed in 2008 by Hupfer; who concluded that the temperature controls the balance between both the phosphorus decomposition and phosphorus-Iron binding. At 15C, phosphorus was trapped at the sediment surface, while at 20 C, it was released. This can be explained by two impacts; the higher the temperature, the more mineralization rate of organic material, and the higher mineralization rate, the more oxygen is

needed, leading the reduction of the penetration depth of oxygen into the sediment (Hupfer & Lewandowski, 2008).

2.1.5 pH

Eutrophic lakes are commonly characterized of having high pH values and when intensive resuspension is linked tightly to high pH, internal phosphorus loading may increase. In addition, studies showed that high Si enrichment combined with elevated pH had a positive synergistic effect, resulting in the most extensive mobilization of phosphorus. Thus, a self-fueled increase in internal loading of phosphorus is expected to happen from this synergism (Koski-Vähälä & Hartikainen, 2001). A study performed in 2003 on Taihu Lake in China showed that there is a relationship between the rate of phosphorus release and pH. The rate of phosphorus release decreased as pH increased from 2 to 6, but increased as pH increased from 8 to 12. This actually means that phosphorus released can be increased in either acid or base environments, while in neutral environments it is not. The study also suggested that in heavily eutrophic sediments, inorganic phosphorus mainly consisted of NaOH-P, and a high pH promoted its release, and this might be the case of Utah Lake (Jin, Wang, Pang, & Wu, 2006). Thus, a competition between the hydroxyl ions and the bound phosphorus ions leads to an increase in pH which frees phosphorus from its binding with ferric complexes (Andersen, 1974; Kim, Choi, & Stenstrom, 2003).

2.1.6 Ca

Phosphorous releases are also affected by Calcium concentrations. The same study done on Taihu Lake showed that sediments with Fe-P/Ca-P (iron bound phosphorus & Calcium bound

phosphorus) ratios less than 0.5 released more phosphorous to the water under acidic conditions, while sediments of higher ratios released more phosphorous under alkaline conditions. This happens because calcium compounds associated with phosphorus become more at lower pH (Huang, Wang, Wang, Wang, & Jin, 2005).

2.2 Utah Lake

This section provides information about Utah Lake, including basic information, hydrologic statistics, historical data, and past studies about the external loading of phosphorus into the Lake.

2.2.1 Basic Information

Utah Lake (displayed in figure 5) is considered the third largest freshwater lake west of the Mississippi with a surface area of about 95,000 acres (384.5 km^2). It is a shallow lake with an average depth of 9 ft. (2.74 m). The lake lies in Utah valley surrounded by the major cities of Provo, Orem, and Saratoga Springs. Its water is considered saline, which means that it contains a significant amount of dissolved salts. Other than springs, groundwater, and precipitation, the Lake has fifteen key tributaries going into the Lake including the Provo River, and the Spanish Fork River, which together account for approximately 60% of the inflow. The Jordan River is the only surface outlet and is located on the north end of the Lake.

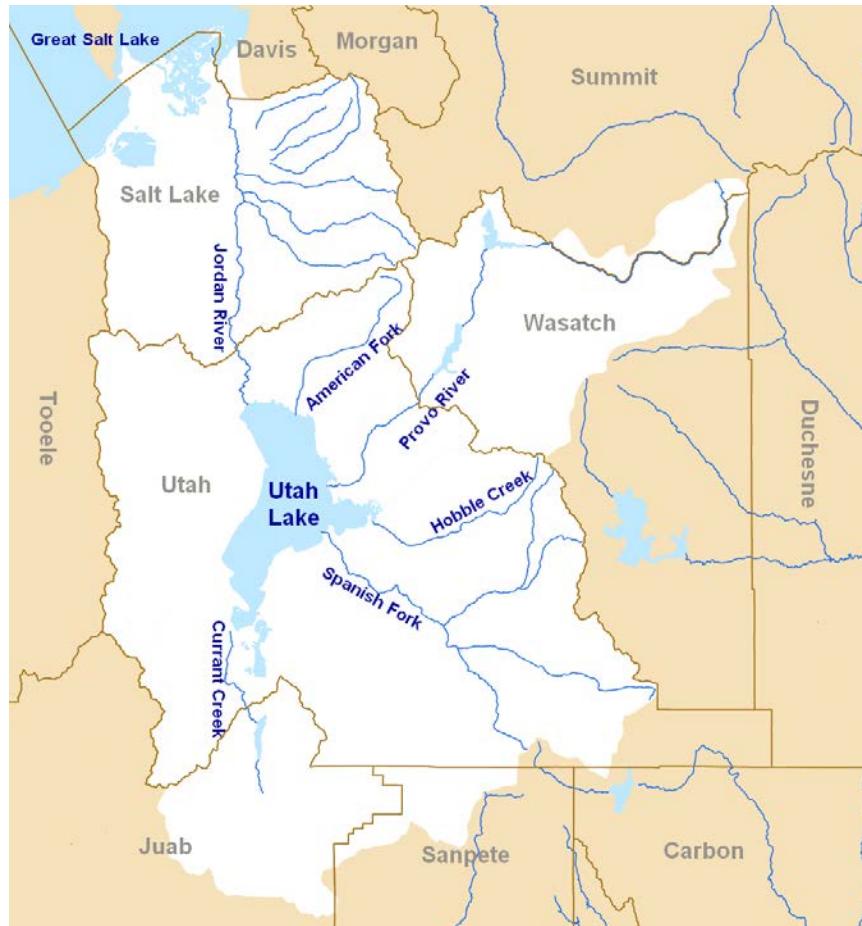


Figure 5: Jordan River Basin (UtahGISportal)

Evaporation plays a big role in the hydrology of Utah Lake, as almost 41% of its water is lost due to that each year. There is an average annual precipitation in the area of about 19.8 in. (50.3 cm). This high evaporation rate coupled with the very shallow nature of Utah Lake causes the high dissolved solids in the lake water. Table 1 shows the hydrologic statistics of Utah Lake including total inflows and outflows.

**Table 1: Hydrologic Statistics of Utah Lake [(+) Inflow, (-) Outflow]
(Utah State Division of Water Quality Report, 2007)**

Tributary	Average Flow (acre-ft./year)	Tributary	Average Flow (acre-ft./year)
Provo River	+ 151,000	Provo River	+ 151,000
Spanish Fork River	+ 99,700	Spanish Fork River	+ 99,700
Benjamin Slough	+ 36,700	Benjamin Slough	+ 36,700
Other Inflows	+ 134,200	Other Inflows	+ 134,200
Total Inflow	+ 829,300	Total Inflow	+ 829,300
Evaporation	- 349,800	Evaporation	- 349,800
Jordan River	- 428,200	Jordan River	- 428,200

Utah Lake is a popular lake because of its proximity to the metropolitan areas of Provo and Orem. Approximately 150,000-200,000 people visit Utah Lake each year for its nice picnic areas, fishing, and water activities. It also supports agriculture uses as it is used to irrigate about 50,000 acres (202.3 km²) yearly.

2.2.2 External Loading of Phosphorus into Utah Lake

Different studies have been done on external loading of phosphorous into Utah Lake. The different inflows provided from streams, springs, drains, wastewater plants, and other sources carry phosphorus into the lake. The State of Utah DEQ Division of Water Quality conducted a study to calculate total phosphorus loads for each inflow to the lake. Evaporation and

precipitation were ignored in these calculations assuming that they have negligible phosphorus amounts.

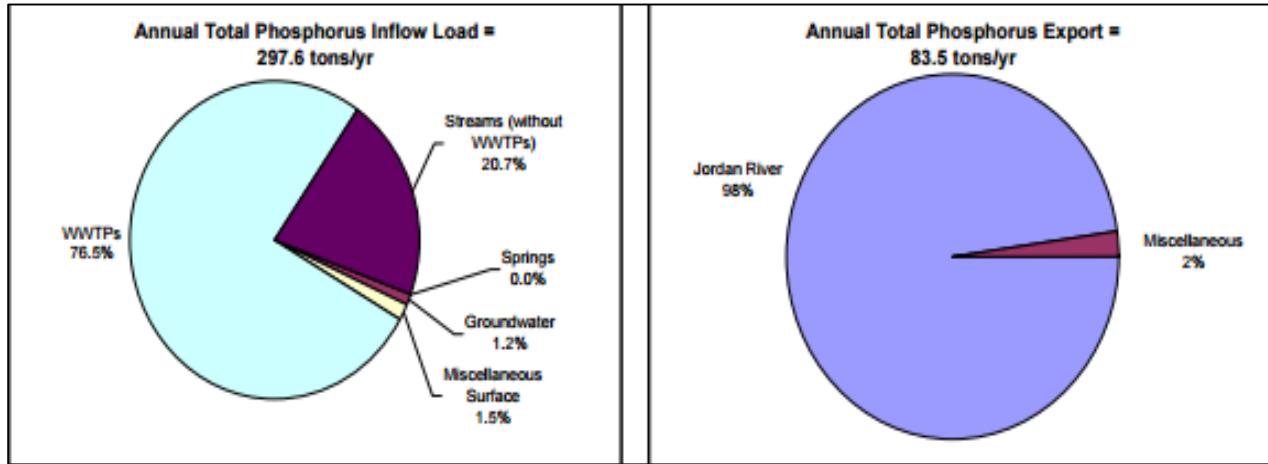


Figure 6: Total Phosphorous Load Summary (The State of Utah Division of Water Quality, 2007)

Figure 6 shows the total phosphorous coming into and out of Utah Lake. The total phosphorous coming into the lake constitutes of different natural and human-related causes. The amount from human-related sources, especially WWTPs provide about 77% of the total phosphorous intake. According to Figure 6, the total Phosphorous intake is approximately 297.6 tons/yr. from the different sources measured in the study, while the only phosphorous outflow is the Jordan River with a total phosphorus of approximately 83.5 tons/yr. These amounts mean that the difference of total phosphorus amounts between the inflows and the outflow is being retained within the lake, which proves that the sediments of the lake act as a sink for phosphorous. Later we will show that current lake sediments total phosphorus levels are similar to geologic lake sediments exposed on the west shoreline.

3 METHODOLOGY

This section provides an overview of the methods I used for field sampling and laboratory analysis. This includes general considerations for sampling in a lake. One objective for this project includes sediment sampling across Utah Lake to determine total phosphorus content in sediments. This section provides the sampling outline, procedure, and lab experiments I used for this study.

3.1 Methodological Consideration

When performing sediment sampling in a specific lake, essential methodological considerations must be established in order to ascertain a successful attempt. There are many factors that need to be considered when defining the number of samples that should be used in the study and their locations. According to MacKnight and Mudroch, these factors are: purpose of sampling, study objectives, historical data and other available information, bottom dynamics at the sampling area, size of the sampling area, and available funds vs. estimated (real) cost of the project (Mudroch & MacKnight, 1994).

Several reviews and articles have been published in order to describe different ways in determining the number of samples and their locations. The most basic approach is to leverage the ease of access in selecting sampling stations. This approach can be further improved by previous team knowledge of the site that can impact the judgment in the selection process. That

knowledge can be also used to gather background information and topographical details of the site. However, that knowledge is not always available, and is sometimes not enough to determine sites of importance. There are many approaches that can make better judgments in this regard. If the team applies statistical modeling of the site, they can uncover trends in data and recognize locations with high concentrations of target substances. A more systematic approach is to divide the area into a grid of blocks or triangles and identify sites within units or intersections. An important consideration that a team should take into account is the relationship of the distance of the source of contamination and its corresponding impact. For this purpose, the team must apply Sediment sampling near the point source. Finally, the sampling process will depend on the scope of the project and its target distribution.

3.2 Objectives

The main purpose of this study is to determine sediment-water interactions, and to determine potential sediment phosphorus release, which can impact algae growth and eutrophication in Utah Lake. This report serves as an important step towards the final purpose by providing an analysis on the condition of the sediments of Utah Lake, providing data for total phosphorus, how that phosphorus is stored in the sediments, and what portion of this stored phosphorus might be released. This study provides maps that present phosphorous spatial distributions and how current lake sediment phosphorous levels compare with phosphorous levels in historic lake sediments.

3.3 Sampling Outline

The outline of this work is as follows:

1. Develop a sampling plan
2. Obtain sediment samples with a hand corer
3. Store the samples in a cooler until arrival to the environmental lab
 - Total phosphorus
 - Phosphorus Fractionation
4. Test the sample in the lab for sediment phosphorus release
5. Analyze the data

3.4 Sampling

We took many factors into consideration during the design and implementation of the sampling process. I wanted to representatively sample the parts of the lake that are expected to have high phosphorous amounts and also have enough data to infer the spatial distributions and patterns. I selected a combination of the traditional approach of choosing by ease of access and random sampling near the point sources. We also chose points near the center of the lake to provide more complete data and be able to generate spatial maps using a more general and even distribution of samples in the lake. However, we did not select points separated by a particular distance; but rather, we chose to sample additional points to represent the locations of the inflows from wastewater treatment plant locations and the river outlet locations.

We took samples using a pontoon boat and a WildCo 2424 Series Hand Corer. Figure 7 shows the device used during sampling. After we completed the sampling from the lake, we took some shore samples. Most of the samples were analyzed for phosphorous, but a few samples were taken to the laboratory to test the sediment phosphorus release with distilled water and river water.



Figure 7: WildCo 2424 Series Hand Corer

We stored the gathered samples in Ziploc bags, labeled with number, location, and date information, and kept them in a cooler. We took the samples to the BYU environmental lab to be analyzed for total phosphorus and phosphorous fractionation analysis. I performed the sampling over a three-month period from June to August, and took a total of 36 samples. Also, I used data taken during a previous year to gain a better understanding of what is happening in Utah Lake (Merrell, 2015).

Figure 8 shows the distribution and location of sample points used in this study. The figure indicates the points sampled in 2015 (blue squares) the points sampled in 2016 (red circles) and land samples (black triangles) which represent geologic lake sediments. Phosphorus fractionation analysis was done on the 36 samples collected in 2016.

I found that the sediments sampled in 2015 have an average total phosphorus of 719 ppm. Sediments sampled in 2016 have an average total phosphorus of 604 ppm. Land samples have an average total phosphorus of 786. My analysis showed that these sample groups, while having

different averages, are not significantly different. One of the differences appears to be if samples were taken in areas of groundwater or spring discharge to Utah Lake. As noted, these areas have significantly lower phosphorous that I attribute to leaching or flushing by the groundwater.

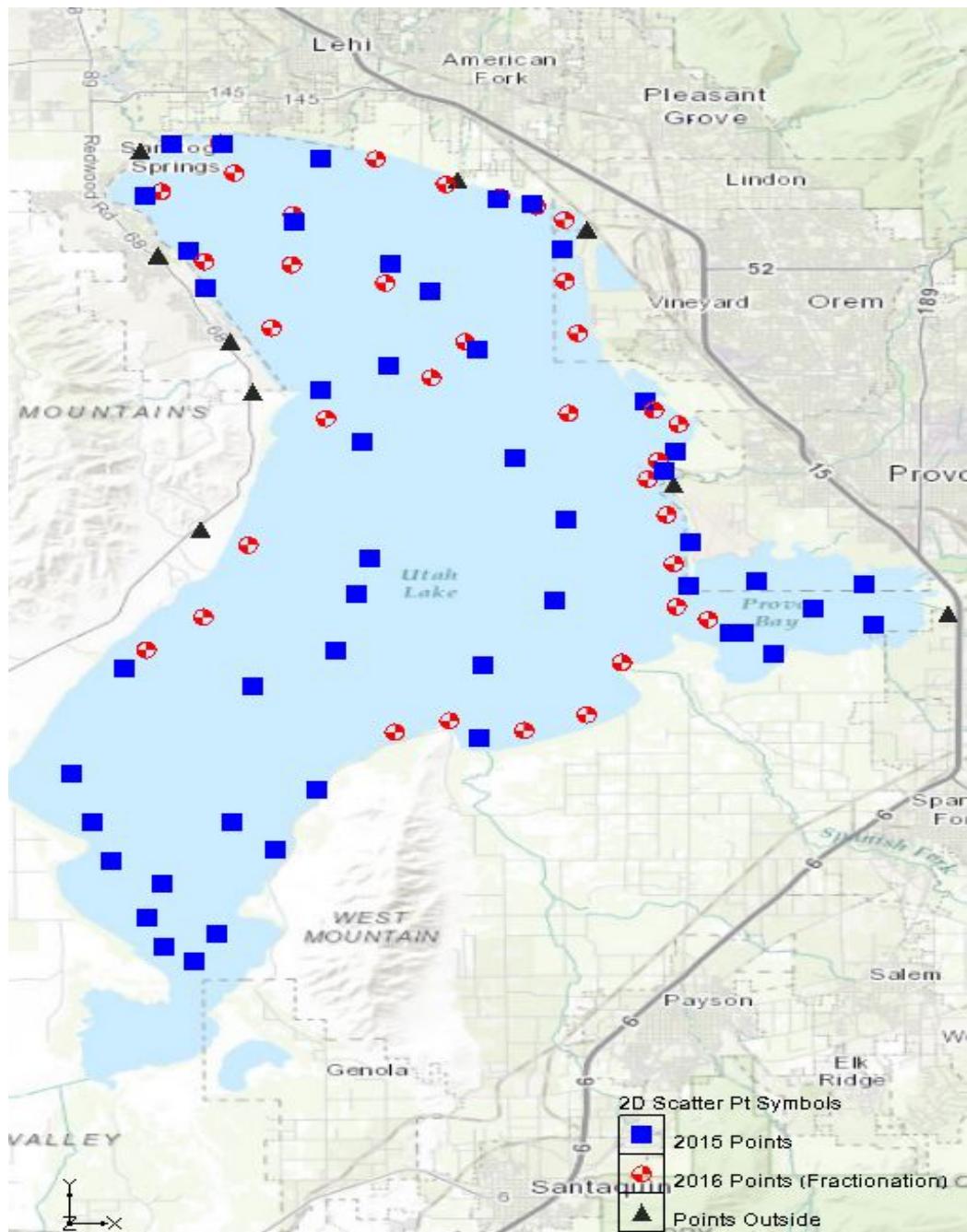


Figure 8: Distribution of Samples

The instruments the BYU Environmental Lab used to calculate total phosphorus were an Ethos OZ Microwave digestion system and Thermo Scientific iCAP™ 7400 ICP-OES Analyzer. They used microwave digestion to dissolve heavy metals in sediments (See Appendix B for Microwave procedure). Then they used the ICP-OES Analyzer, which is an effective metal detection machine with a phosphorus detection limit of 1.55 µg/L, to analyze the phosphorus content in each sample (See Appendix B for Machine specifications).

3.5 Lab Experiments of Phosphorus Release

I conducted two lab experiments to calculate phosphorus release from sediments to the water column. I used a shore sediment sample and a lake sediment sample that were stored in Ziploc bags in a refrigerator for about a week until the lab was ready for the experiment. For these two experiments I placed these samples into eight transparent beakers (2.5 X 4.5 in, diameter X height).

I used distilled water for the first experiment. I placed 2 inches of each sample into four beakers, two with lake sediments and two with shore sediments. I then added distilled water to fill up the beaker. I added some table salt to reduce the aggressive nature of distilled water in solubilizing phosphorus. I shook two of the four bottles, one lake and one shore, manually once every 3 days for a period of 1 month. The other two beakers, one lake and one shore, were not disturbed during this period. At the end of the month, I took samples of the water to the BYU Environmental Lab to measure amount of phosphorus in the water column. .

For the second experiment, I used Provo River water instead of distilled water. Again I used two beakers with lake sediments and two with shore sediments, one of each shaken and one not disturbed. I re-did the experiment to more closely match actual conditions in Utah Lake. And gain a better understanding of sediment-water interactions. I followed the same approach as the first experiment; two beakers were shaken manually every day for a period of 2 weeks. I measured the phosphorus concentration in Provo River water before and after the experiment to calculate the amount of phosphorus transferred from sediments to water. Figure 9 shows a picture of the beakers filled with sediments used in the experiments.



Figure 9: Beakers Used for the Two Experiments

3.6 Geostatistical Analysis of Phosphorus Distribution

I used the total phosphorus data obtained for all the samples collected over a two-year period (2015 & 2016) to create a spatial model that shows phosphorus distribution patterns around the lake. I used a program developed by Aquaveo named Groundwater Modeling System (GMS), which contains different geostatistical tools useful in interpolation and autocorrelation amongst measured points, to create these models.

I created two different coverages representing the boundaries of models and data sets used in the models. I interpolated the data sets containing phosphorus concentrations using Shepard's Method. Two models were:

1. Points inside the lake – only lake sediment samples.
2. Points inside and outside the lake – lake sediment and shore sediment samples.

I generated two maps, the coverage or map for the first model was clipped to shore line representing the boundary of the lake as shown in figure 10, while the coverage used for the second model included a portion of the land surrounding the lake as shown in figure 11. These maps show the distribution of phosphorous in Utah Lake sediments and how that distribution relates to distributions in the shore sediments.



Figure 10: Coverage Used in the First Three Models

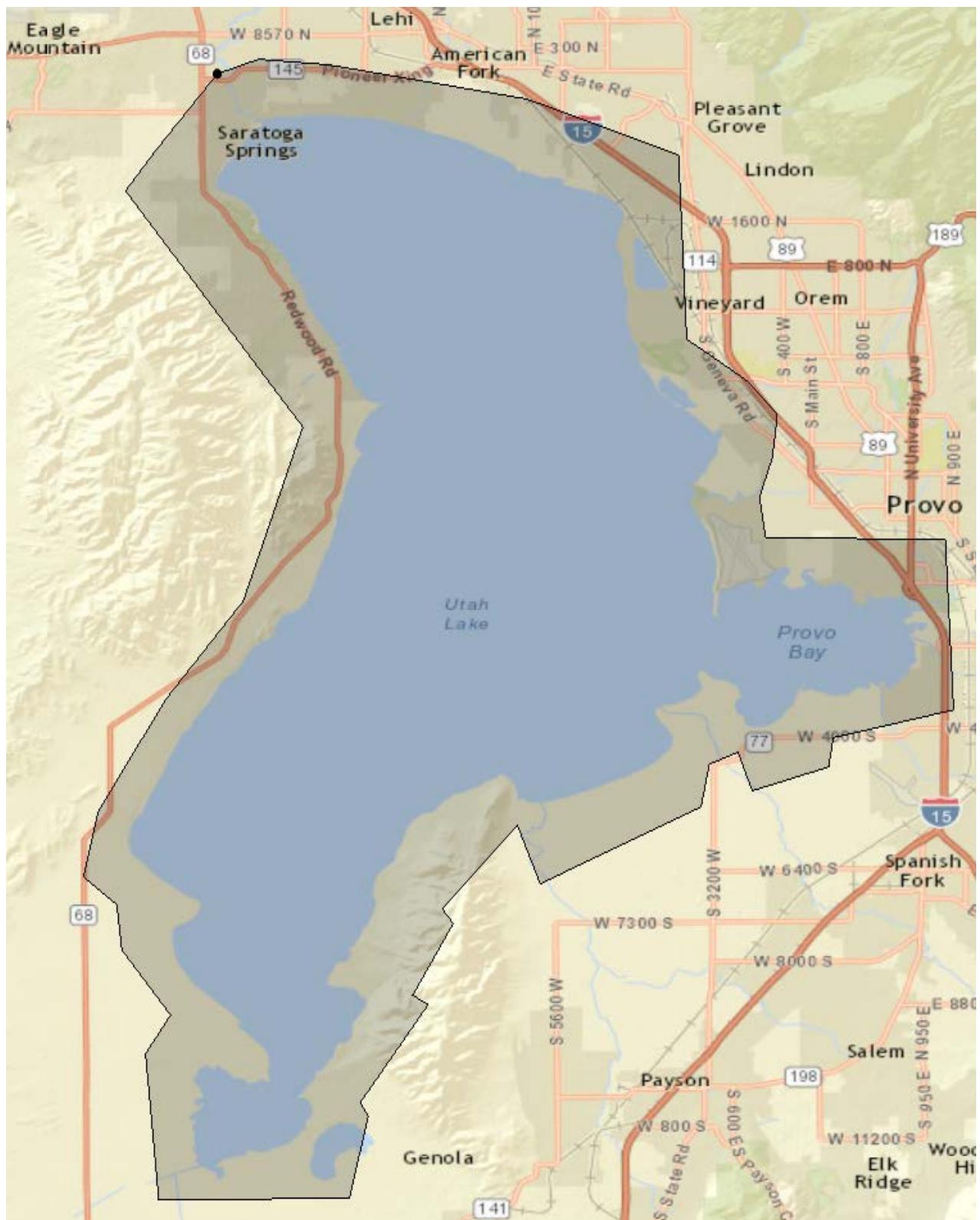


Figure 11: Coverage Used in the Fourth Model

3.7 Phosphorus Fractionation

Fractionation is a process of separation in which a quantity of a mixture is divided into different fractions. The concentration of different phosphorous fractions in lake sediments depend on many different factors such as, its concentrations in the lake water, the transport of soluble phosphate between solid components, adsorption-desorption mechanisms, the chemisorption ability of the sediments, and biological uptake (Andersen, 1974; Koski-Vähälä & Hartikainen, 2001; Sondergaard et al., 1992). Thus, the biogeochemical cycling of P fractions and, thus, their concentrations in sediments can vary greatly (Kapanen, 2008).

Phosphorus fractionation has been an important subject of study because of its significance in the development of lake ecosystems (Kapanen, 2008). Many approaches have been developed to extract the different fractions or pools from sediment into a solution for measurement.

The BYU environmental laboratory used the fractionation procedure described by Moore & Coale (2000) to analyze the sediment samples. Refer to appendix (B) for procedure and steps. The equipment used for the measurement of phosphorus pools are the Microwave and iCAP™ 7400 ICP-OES Analyzer. Refer to Appendix (B) for description and procedure.

The fractionation scheme allowed the measurement of sediment phosphorus content in five different pools (Casbeer, 2009), which are:

- Fr. W: water soluble phosphorus
- Fr.KCl: loosely sorbed phosphorus
- Fr.NaOH: Al- and Fe-bound phosphorus
- Fr.HCl: Ca-bound (apatite) phosphorus

- Fr.PFD: residual phosphorus, probably mostly organic

Raw ICP concentrations in mg/L for the extractant liquid were determined using a iCAP™ 7400 ICP OES Analyzer. The fractionation concentrations were determined for 36 samples out of the 85 samples used in this study.

4 RESULTS

I collected samples in five sampling trips, over a three-month period in the summer, specifically, June to August 2016. I stored the samples collected in a big refrigerator in the lab and took them to the BYU environmental laboratory to measure total Phosphorus in each sample. The total number of samples analyzed for total phosphorus was 36. However, the total number of samples used for the study is 85 samples which included samples collected in 2015.

4.1 Sampling Results

Samples were collected near the top of the sediment layer from 0 to 4 inches in depth. Total phosphorus concentration varied throughout the lake ranging from 280 to 1710 ppm, with an average value of 666 ppm. The 10 on-shore samples collected in 2015 from geologic sediments had an average total phosphorus of 786 ppm. Figure 12 shows the results and sampling points. Some of the points were excluded from the map in order to have a clear view of the points without overlap.

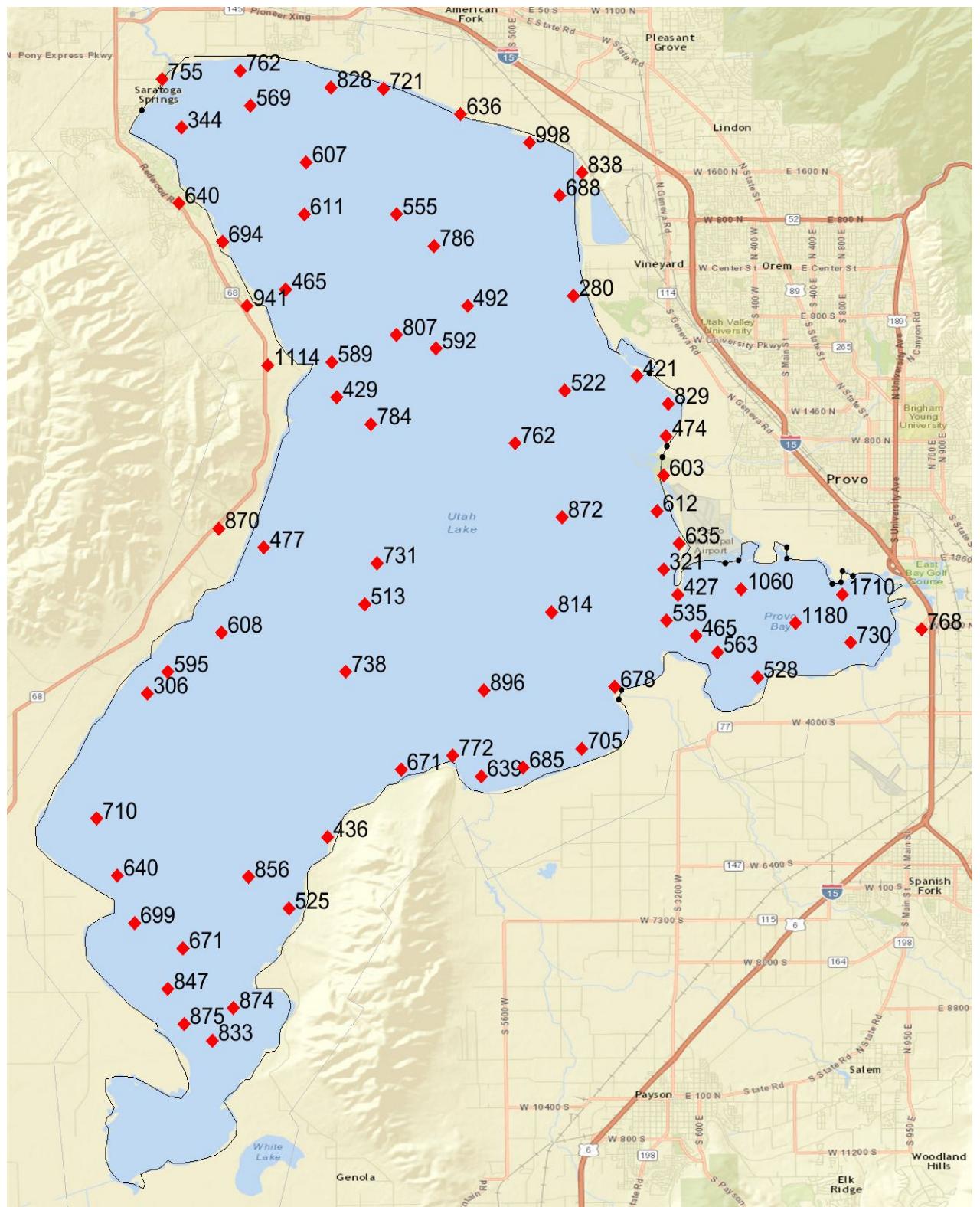


Figure 12: Sampling Distribution and Results

For analysis, I divided the lake horizontally into 4 sections as shown in Figure 13. I calculated statistics for each section including means and standard deviations and show the results in Table 2.

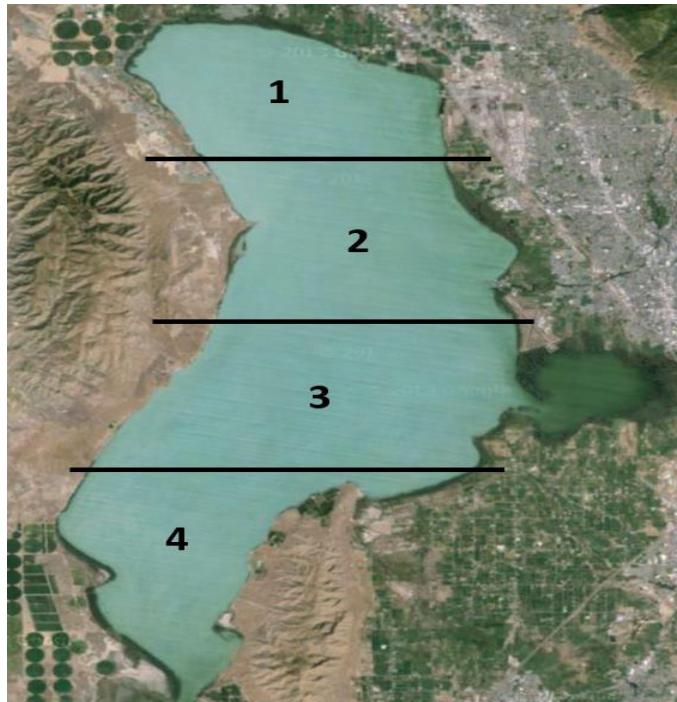


Figure 13: The Division of Utah Lake

Table 2: Statistics for Each Section of Utah Lake

Section	Number of Samples	Average (ppm)	Standard Deviation
1 (Upper)	24	660	150
2 (Middle Upper)	20	631	174
3 (Middle Lower)	24	668	304
4 (Lower)	17	714	118
Land Samples	10	786	151

Averages for sections and Land Samples were compared with each other using a bar plot (Figure 14) and a box plot (Figure 15).

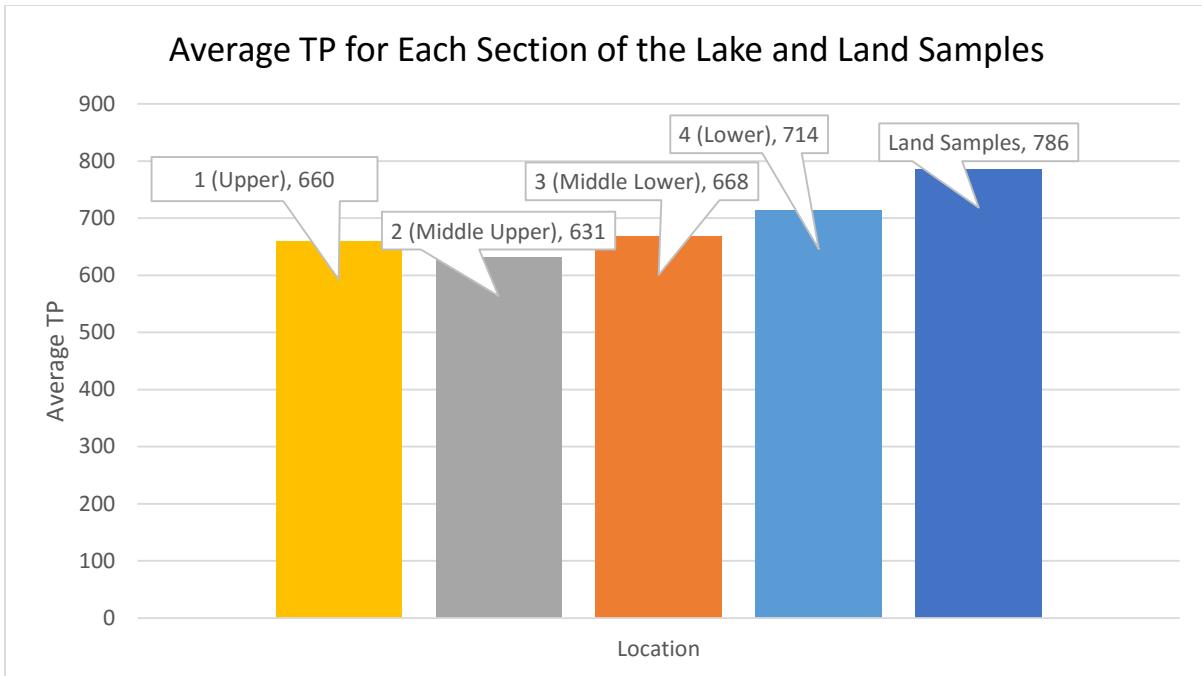


Figure 14: Average Total Phosphorus for Each Section of the Lake, and Land Samples

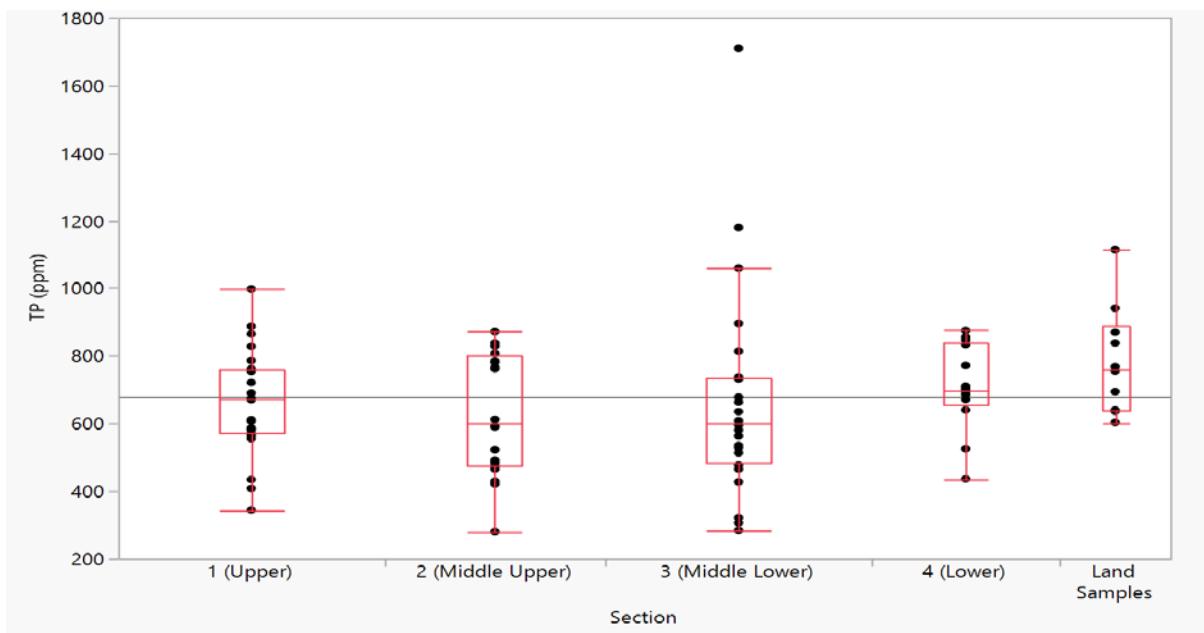


Figure 15: Box Plot Comparing Lake Sections and Land Samples

The results show that Area 4 and the geologic sediment samples (on-shore) have the highest average with the remaining three sections having similar averages. The middle section shows the highest standard deviation and later maps will show it contains some hot spots, most likely due to agricultural impacts, and low spots, most likely due to spring water inflows. The box plots in Figure 15 show that the distribution of results is quite similar for all 5 regions. I explain the differences through physical processes such as on-shore corrals and in-lake springs. The shallow end of the lake (Area 4) has the highest total phosphorous content and these values closely match those of the on-shore or geologic sediments.

These results infer that while the current phosphorous balance indicates that Utah Lake is acting as a phosphorous sink for anthropogenic phosphorous, these processes are not significant compared to the historic geologic processes. Based on the different areas, it even appears that spring inflow and other hydrologic processes have reduced sediment phosphorus in the areas of the Lake near the outfall and near underwater springs.

4.2 Lab Experiments Results

Table 3 shows the results of the two lab experiments I conducted to estimate phosphorus release in sediment-water interactions. As noted, I performed the first experiment using distilled water with the addition of some table salt to reduce the aggressiveness of distilled water in solubilizing phosphorus. I performed the second experiment using Provo river water to more closely match actual conditions and gain a better understanding of sediment phosphorus release in Utah Lake. Two of the four beakers used for each experiment contained lake sediments and the other two contained shore sediments. I had the BYU environmental laboratory measure phosphorus concentration in each beaker in the lab and all results are in mg/L for the water

above the sediments. These samples were filtered on a 0.45 μm filter to only measure the dissolved content. The initial concentration of phosphorus in Provo River water was 0.05 mg/L before being added to the beakers. Figures 16 and 17 show plots representing the two experiments.

Table 3: Lab Experiments Results

Experiment	Land Soil No shake (mg/l)	Land Soil Shake (mg/L)	Lake Sediment No Shake (mg/L)	Lake Sediment Shake (mg/L)
1	7.70	14.0	3.00	19.0
2	2.60	5.10	0.24	0.24

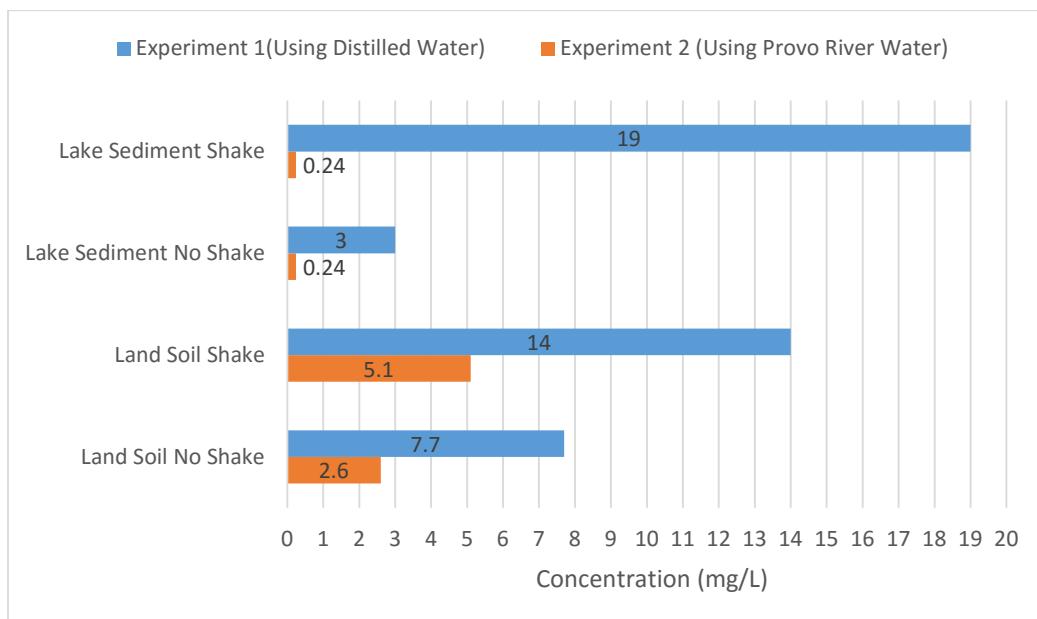


Figure 16: Lab Experiments Results

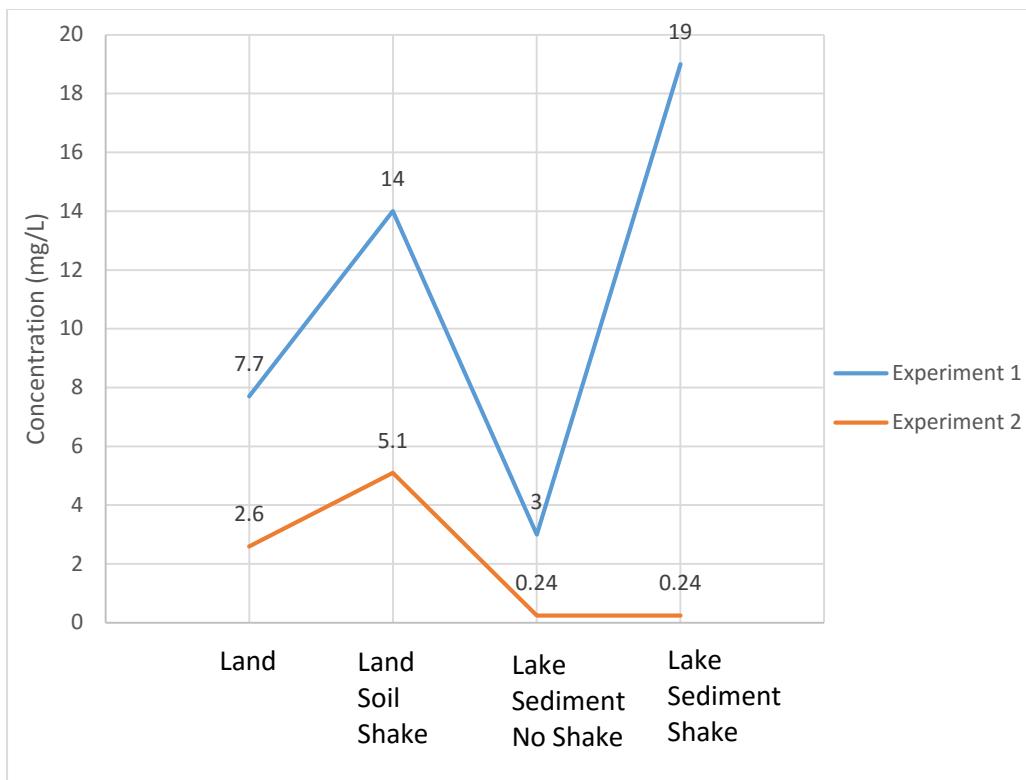


Figure 17: Lab Experiments Results

These plots show that there is a significant difference in comparing phosphorus concentration using the different waters. The experiment attempts to provide some insight into several potential processes. The sediments should be in anaerobic or reducing conditions after three weeks, though I did not measure these conditions. The two different water types, distilled or Provo River water, show the potential for releases when the sediments are in contact with water already high in dissolved solids (Provo River water) or relatively clean (distilled water) such as precipitation or run-off.

I used sediments from both in-lake and on-shore to determine the potential for phosphorous release from these locations and to help to characterize the difference or similarity among the geological and current sediments.

The distilled water releases significantly more phosphorous from the sediment than the Provo river water. For the on-shore sediments, the distilled water extracts about 3 times as much phosphorous as the Provo River water, for the lake sediments this ranges from 10 times for the non-disturbed samples to 100 times for the shaken samples. Distilled water would extract both the interstitial water and the ion-sorbed phosphorous. As distilled water has no dissolved solids, it would also be much more aggressive in dissolving iron minerals under the anoxic conditions, resulting in the higher phosphorous releases.

Shaking the beakers simulates mechanical resuspension of the sediments. Without resuspension, even if phosphorus is released into the pore water of the sample, it must diffuse through the sediment to the sediment surface in the beaker to be released. Shaking eliminates the need for the diffusion. For the lake sediments with Provo River water, there was essentially no difference between the shaken and non-disturbed samples and the released amounts were low. I attribute this to two things. The first is a smaller release because the Provo River water is less aggressive and does not dissolve as much of the iron minerals, this results in a lower concentration gradient limited diffusion. It would be interesting to run this experiment over a longer time to determine if the low release using Provo River water is partially due to slower kinetics and that more phosphorous might be released over time.

While earlier analysis showed little difference in the total phosphorous between the current lake and geologic sediments, this experiment shows that the geologic sediments are much more prone to release phosphorous under these conditions. This could mean that these on-shore sediments might be a significant phosphorous source.

4.3 Statistical Analysis of Results

I used JMP Pro 12 to do statistical analysis of the results. Basic statistics comparing the four lake sections of the lake and the land samples are presented as box plots in Figure 15. I used a Chi-square test to compare the results from the different sections. Specifically, I tested the null hypothesis that all sections have the same average level of phosphorus to determine if statistically the sections, including the historic sediments on the ancient shore were different from each other. I obtained a p-value of 0.1210 which means that at a confidence level of 0.05, we fail to reject the null hypothesis and prove that there is no difference in averages between the different sections or stated another way, we are 95% confident that they are the same. In addition to a Chi-square analysis, I performed a nonparametric Tukey analysis to compare all individual means between all pairs from each section for statistical significance. Figure 20 shows the statistical analysis done including the different p-values for each pair. While these calculated p-values between pairs show that we cannot reject the null hypothesis of equal means, or stated another way, the differences are not statistically significant, the collected historic samples, or land samples are only 10 samples. It is also essential to state that the collection of samples was random indicating that population inferences can be drawn and conclusions represent the whole population of sediment samples throughout Utah Lake.

Connecting Letters Report	
Level	Mean
Land Samples	A 785.90000
4 (Lower)	A 713.64706
3 (Middle Lower)	A 684.91304
1 (Upper)	A 659.87500
2 (Middle Upper)	A 631.30000

Levels not connected by same letter are significantly different.

Ordered Differences Report						
Level	- Level	Difference	Std Err Dif	Lower CL	Upper CL	p-Value
Land Samples	2 (Middle Upper)	154.6000	78.68559	-64.495	373.6948	0.2917
Land Samples	1 (Upper)	126.0250	76.46865	-86.897	338.9469	0.4712
Land Samples	3 (Middle Lower)	100.9870	76.95603	-113.292	315.2659	0.6843
4 (Lower)	2 (Middle Upper)	82.3471	67.02101	-104.268	268.9626	0.7347
Land Samples	4 (Lower)	72.2529	80.96681	-153.194	297.6996	0.8990
4 (Lower)	1 (Upper)	53.7721	64.40379	-125.556	233.1001	0.9190
3 (Middle Lower)	2 (Middle Upper)	53.6130	62.11620	-119.345	226.5715	0.9095
4 (Lower)	3 (Middle Lower)	28.7340	64.98172	-152.203	209.6713	0.9919
1 (Upper)	2 (Middle Upper)	28.5750	61.51136	-142.699	199.8493	0.9903
3 (Middle Lower)	1 (Upper)	25.0380	59.28284	-140.031	190.1072	0.9932

Figure 18: Statistical Analysis Results

4.4 Geostatistical Analysis Results

I created two geostatistical models to provide a spatial interpretation of total phosphorus distributions. I show the results of these models in Figures 18 and 19. Figure 18 shows the model that includes all the points inside the lake. Figure 19 shows the model, which includes the points inside and outside the lake. If you compare these two models it appears that the phosphorous distribution inside the lake extends to historic geologic sediments indicating that recent processes have had a minimal impact on lake sediment phosphorous levels.

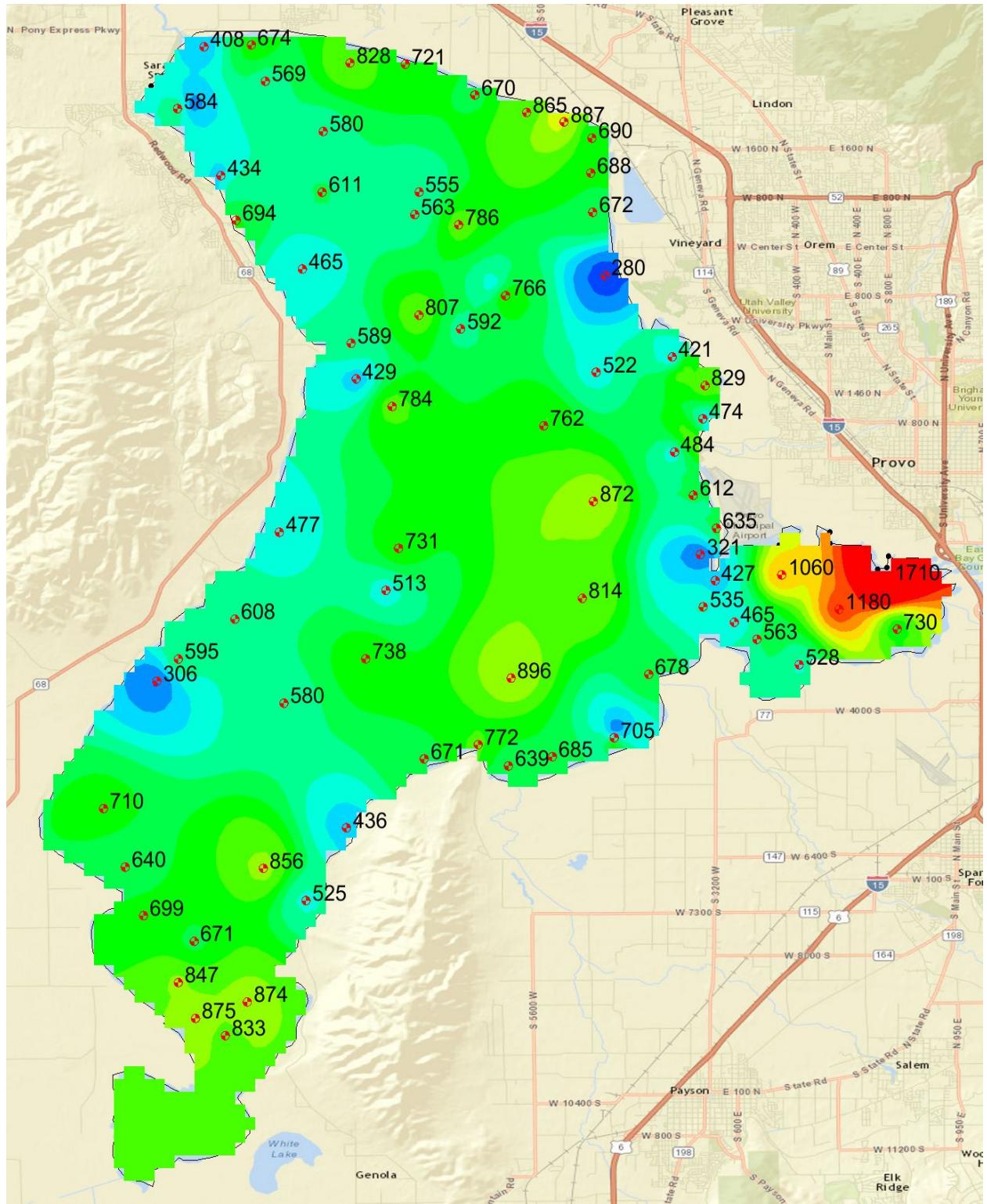


Figure 19: Geostatistical Model including the Points inside the Lake

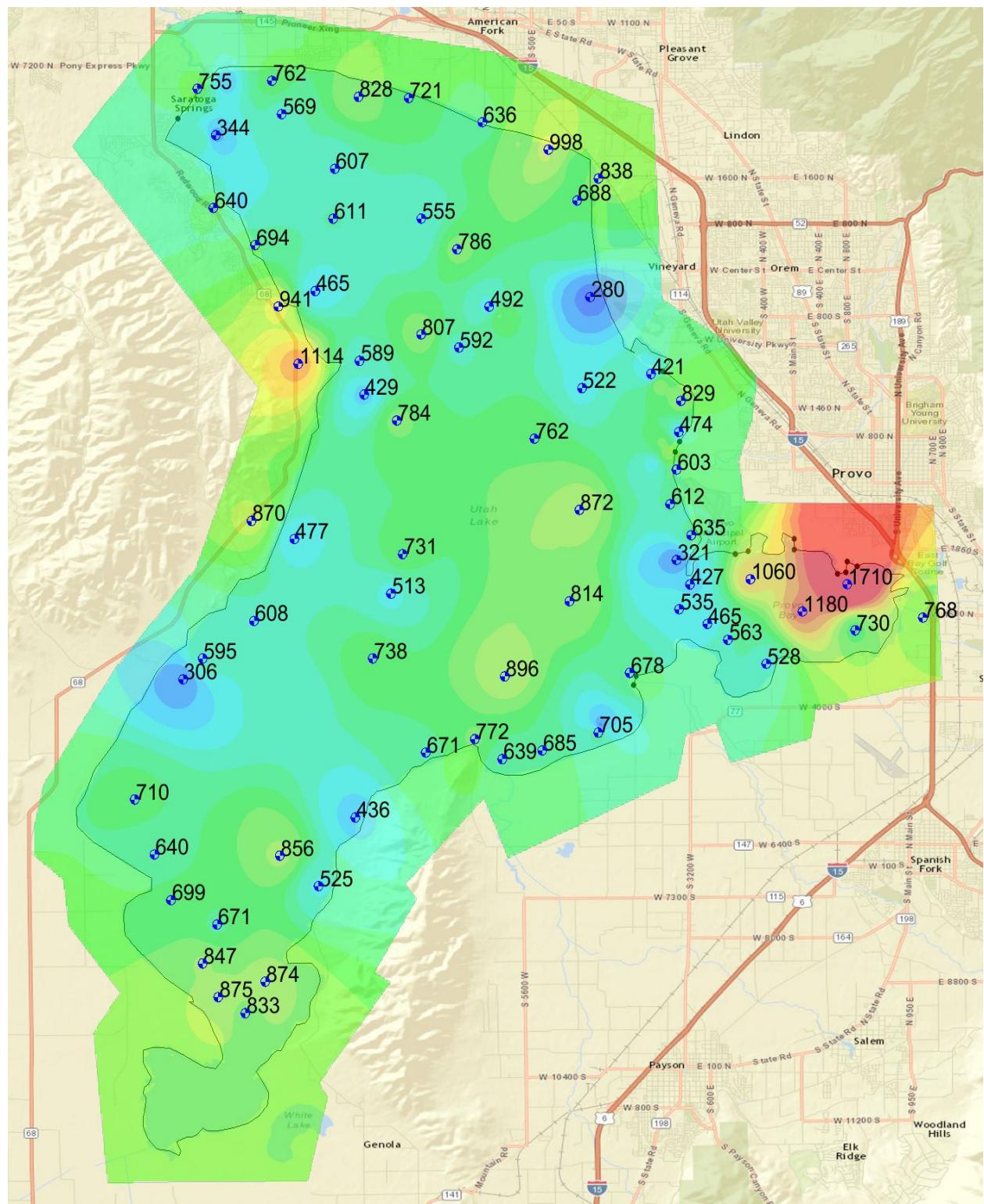


Figure 20: Geostatistical Model including the Points Inside and Outside the Lake

The previous 2 figures show that the average concentration throughout the lake is in the 600 to 800 ppm range. There are also some low concentration spots around the shoreline of the lake representing potential spring places.

Both maps show sediments with very high phosphorous concentrations on the north shore of Provo Bay. I believe that prior to recent restoration efforts in Provo Bay, a livestock corral existed near this location. If this is correct, these very high phosphorous levels could be the result of long-term, up to 100 years, of manure and other inflows to the local sediments.

Around the shoreline there are several samples with apparent low phosphorous levels, such as the location with 280 ppm near the Vineyard area in Orem. The sample was taken in this location because of the inflow from the Orem Wastewater treatment plant. However, my understanding is that this area has a number of underwater seeps and springs. These seeps and springs were evident this fall (2016) when these sediments were exposed because of low water levels. I believe that these seeps and springs have dissolved or washed out the phosphorous in this area. Other samples around Utah Lake with similar low phosphorous levels also seem to be associated with areas known to have underwater seeps and springs.

4.5 Fractionation Results

The fractionation procedure used was described by Moore & Coale (2000). Due to some misunderstandings between “me” and the lab providing the concentration measurements for sediments, water content and dry weight were measured before performing the required steps for fractionation.

The right way to perform the fractionation is by following the steps described by Moore & Coale (2000), then determining the water content and dry weight. Thus, the results of the

required calculations needed to convert the concentrations in (mg/L) to actual amounts in (mg/Kg) were not as expected. Refer to Appendix (B) for raw ICP concentrations. These are not reported in the main body of the report because of the miss-application of the correct methods.

5 CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE WORK

5.1 Discussion of Results

The sampling results show that Utah Lake sediments have huge amounts of total phosphorus content with an average concentration of 666 ppm inside the borders of the lake. The concentrations in historic geologic sediments sampled from the west shore are similar, but higher, with an average concentration of 786 ppm. The availability of these massive amounts of phosphorus in sediments increases the availability of these sediments to act as a source of phosphorus into the water column. The simple lab experiments performed in this study showed that phosphorus release from sediments is present under both conditions of shaking and not shaking. The second experiment using Provo River water to determine sediment-water interactions represents a more accurate situation than the first experiment using distilled water. As indicated previously, the testing times for both experiments varied and manual shaking was used to count for any possible movements in the bottom of Utah Lake. The varied testing times and inconsistent manual shaking may be the reason behind not having an increase in phosphorus release when shaking occurred in the second experiment.

From the statistical analysis and the spatial maps, we showed that historic sediments and current sediments have very similar total phosphorus levels. Thus, the current phosphorous load coming into the lake does not seem to significantly impact sediment phosphorus levels and may not necessarily affect levels in the water column.

5.2 Conclusions

My research attempted to characterize the large concentrations of total phosphorus trapped into Utah Lake's sediments. While I did not perform any in-depth experiments, literatures reviews, fractionation results, and a simple water bottle test showed that the phosphorous in the sediments could significantly affect phosphorous levels in the water column. Utah Lake physical properties (shallow, carp content, thick sediments, and wind) can cause more mixing in the top layer of sediments which increases the chances of releasing relatively high amounts of phosphorus into the water column as the simple lab experiments proved. These high sediment phosphorus levels combined with Utah Lake physical processes increase the probability that the phosphorus in the sediment significantly impacts lake water quality.

Phosphorus release from sediment occurs under very complicated processes under many different conditions. Thus, more knowledge is needed to develop a conclusion for the phosphorus release mechanism in Utah Lake and the relative impacts of historic high phosphorus levels in Lake Sediments versus the current high inflow loadings

5.3 Future Work

Several recommendations for future work are listed below and are followed by brief explanations of each.

1. Sediment water interactions
2. Repeating Fractionation Procedure to obtain better, more accurate data

5.3.1 Sediment Water Interactions

The lab experiments I performed in this study showed that sediments act as a source of phosphorus for the water column. As mentioned previously, the mechanism of shaking and the times of testing varied between the two experiments and within each experiment itself. Thus, it is recommended to redo these simple lab experiments but in a broader fashion keeping in mind the following suggestions: 1. Use samples from each lake section in addition to land samples and a sample from a potential spring place. 2. Use a shaker table to insure consistent shaking. 3. Use the same time frame for all experiments. It would also be interesting to resample the few areas that had lower phosphorus levels and determine if the hypothesis, that spring, or ground water inflow, causes this is correct.

5.3.2 Fractionation of Phosphorus

Fractionation of phosphorus must be repeated using the right way described in Moore & Coale (2000). Refer to Appendix (B) for procedure. Unfortunately, while these tests were performed as part of this work, as noted above because of problems with laboratory procedures the data are not usable.

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APPENDIX A. SAMPLE COORDINATE LOCATIONS AND TOTAL PHOSPHORUS

Sample #	TP (ppm)	Location	
		Latitude	Longitude
1	484	40.23582	-111.74336
2	827	40.26077	-111.74147
3	672	40.30755	-111.76908
4	887	40.33456	-111.77805
5	670	40.34261	-111.80601
6	612	40.22292	-111.73746
7	321	40.2052	-111.73527
8	535	40.18957	-111.73437
9	465	40.18493	-111.72459
10	678	40.16939	-111.75146
11	284	40.15282	-111.76155
12	705	40.150407	-111.762317
13	685	40.14475	-111.78171
14	772	40.14837	-111.80501
15	671	40.14411	-111.82196
16	781	40.24248	-111.74016
17	829	40.25572	-111.73376
18	280	40.28865	-111.76512
19	690	40.3297	-111.76929
20	754	40.33814	-111.78929
21	721	40.35181	-111.82788
22	674	40.35758	-111.8762
23	569	40.3467	-111.87183
24	580	40.3317	-111.85366
25	563	40.30685	-111.82493
26	492	40.2856	-111.80004
27	522	40.25972	-111.76796
28	595	40.173946	-111.899122
29	608	40.185845	-111.881385
30	477	40.21186	-111.867434
31	429	40.257714	-111.843293
32	465	40.290551	-111.860211

33	587	40.314537	-111.881088
34	344	40.340015	-111.894573

Sample #	TP (ppm)	Location	
		Latitude	Longitude
35	611	40.313525	-111.854005
36	592	40.272634	-111.810505
37	421	40.26426425092004	-111.74407973885536
38	688	40.31928806315549	-111.7696438729763
39	896	40.33523144859279	-111.77808176726103
40	677	40.34296739759075	-111.80575512349606
41	555	40.31357522507134	-111.82355426251888
42	589	40.2683834002936	-111.84491269290447
43	513	40.19449255252672	-111.83400381356478
44	639	40.14198487994471	-111.79551847279072
45	473	40.16862169116434	-111.75221353769302
46	663	40.18018792680401	-111.7139196395874
47	635	40.21306697842025	-111.73019427806139
48	1060	40.199104817443605	-111.70973338186741
49	1710	40.1974940176514	-111.67623460292816
50	730	40.18289520023535	-111.67345315217972
51	528	40.17225785529139	-111.70421976596117
52	563	40.179860300712186	-111.71751379966736
53	1180	40.188832944004815	-111.69172637164593
54	427	40.19743153116509	-111.73059090971947
55	448	40.235706516769916	-111.74185048788786
56	474	40.24581978918884	-111.73448044806719
57	689	40.343832400999474	-111.80529814213514
58	828	40.35218262713112	-111.8452375754714
59	762	40.357303046035	-111.87517873942852
60	408	40.35699416097002	-111.8910600990057
61	584	40.338537946354606	-111.8994128331542
62	434	40.31847872907794	-111.88577245920897
63	607	40.32935407983717	-111.85344748198986
64	998	40.33544152770277	-111.77958715707064
65	865	40.33740044558262	-111.78980134427547
66	719	40.14427950911718	-111.82161334902048
67	436	40.12346226090437	-111.84633191674948
68	525	40.10171464096386	-111.85905866324903

APPENDIX B.

B.1 Fractionation

This fractionation scheme is based on Moore & Coale (2000), roughly equivalent to van Eck (1982) as modified by Moore & Reddy (1994).

Weigh centrifuge tube (wt) prior to placement of sediment sample in tube. The weight of each successive fraction is needed to calculate the entrained liquid (containing soluble Phosphorus) from the prior extraction.

B.1.1 Water Soluble Phosphorus

1. Place ~10 g sediment sample into tube
2. Reweigh tube with sediment (wt+s), determine wet weight of sediment
3. Add 20 mL of DI water to tube 77
4. Centrifuge for 20 minutes at 7500 rpm
5. Filter through 0.45 micron membrane filter (use vacuum filter)
6. Save sample for analysis/measurement, refrigerate ASAP to avoid evaporation

B.1.2 Loosely Sorbed Phosphorus

1. Reweigh tube to determine how much water removed (wwat.sol)
2. Homogenize pellet left in tube with a spatula
3. Add 20 mL of deaerated 1 M KCl to tube

4. Shake for 2 h on reciprocating shaker
5. Centrifuge for 20 minutes at 7500 rpm
6. Filter immediately through 0.45 micron membrane filter
7. Save sample for analysis/measurement, refrigerate ASAP to avoid evaporation
8. Reweigh tube to determine weight after loosely sorbed P released (w_{loose})

B.1.3 Aluminum and Iron-bound Phosphorus

1. Add 20 mL 0.1 M NaOH to tube
2. Shake for 17 h on reciprocating shaker
3. Centrifuge at 7500 rpm for 20 minutes
4. Filter with 0.45 micron filter
5. Save sample for analysis/measurement, refrigerate ASAP to avoid evaporation
6. Reweigh tube to determine new weight (w_{Fe+Al})

B.1.4 Calcium-bound (Apatite) Phosphorus

1. Reweigh tube prior to Ca-bound P extraction
2. Add 20 mL of 0.5 M HCl
3. Shake for 24 h on reciprocating shaker
4. Centrifuge at 7500 rpm for 20 minutes
5. Filter through 0.45 micron filter
6. Save sample for analysis/measurement, refrigerate ASAP to avoid evaporation

B.1.5 Residual (Organic) Phosphorus

We used the persulfate digestion method with remaining sediment after step #4 (apatite-P). This can also be used with a new sediment aliquot for determination of total P.

Materials:

- Hot plate
- Glass scoop (to hold persulfate crystals)
- Sulfuric acid solution
- Potassium persulfate ($K_2S_2O_8$) solid
- 1 N (1 M) NaOH

Procedure:

1. Obtain 50 mL (or suitable portion) of thoroughly mixed sample
 - For sediments, mix ~60 mg sediment sample (record weight) to 50 mL H₂O 2.
2. Add 1 mL sulfuric acid solution
3. Add 0.5 g solid potassium persulfate
4. Boil gently on preheated hot plate for 30-40 min (or until 10 mL left)
5. Cool and dilute to 30 mL (with DI water)
6. Neutralize solution with NaOH
7. Dilute to 100 mL with distilled water

B.2 Water Content

1. Weigh ceramic bowl, record weight (mbowl)
2. Add sediment sample

3. Reweigh bowl with wet sediment, record weight (mwet.bowl)
4. Heat in oven at 105 °C for 24 h
5. Reweigh bowl, record weight (mdry.bowl)
6. Determine water content

B.3 Equipment

The equipment used for the measurement of phosphorus pools are described below.

B.3.1 Microwave

Additional PPE: gloves, goggles

Reagents:

Nitric Acid, 69.6% (concentrated)

Hydrogen peroxide, 50%

Batch size: 24: 22 samples, 1 standard, 1 blank

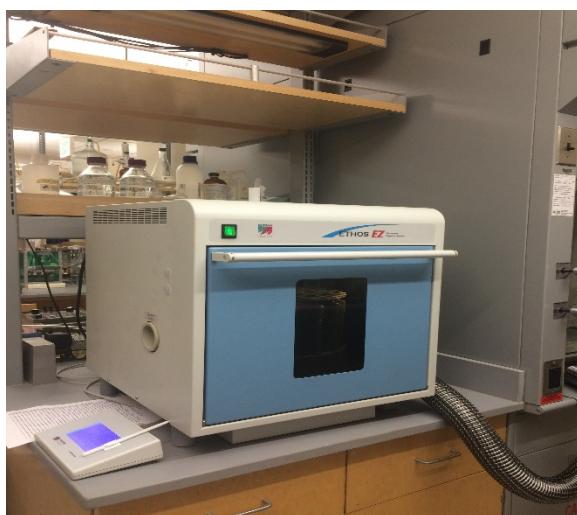


Figure 21: Microwave

Set up:

1. Weigh approximately 0.25 grams of prepared plant material (0.1 g for soils). Record the weight and tube number for each sample. Be sure to include a known check (alfalfa, tube 1) and a process blank (last tube). All sample must be either soil or plant, you cannot mix matrices!
2. Place in a white Teflon vessel.
3. Add 8 ml nitric acid (1 pump from the repipette).
4. Add 2 ml hydrogen peroxide (1 pump from the repipette, this is stored in the fridge).
5. Using the reshaping tool, recondition the white Teflon cap.
6. Place the Teflon cap on the vessel, and make sure that it fits well.
7. Place the vessel inside the safety shield (brown).
8. Tighten the safety valve (lid) by hand.

For the sample in tube 1 (reference vessel).

- Do not put the safety valve on yet.
 - Place the thermowell into the Teflon cap.
9. Once you have completed this for all samples, place the carousel lid in place above the samples.
 10. Take the samples to the microwave.

For the sample in tube 1 (reference vessel).

- Slide the ATC sensor through the safety valve (make sure the valve does not have a TFM foil in it).
- Slide the ATC sensor through a TFM foil.
- Place the sensor into the thermowell and tighten the safety valve.

Instrument:

1. Place the rotor inside the microwave; ensure that it locks into place.
2. Plug in the jumper of the ATC sensor.
3. Place exhaust tube in fume hood.
4. Turn on the microwave.
5. Login.
6. Go to program, select “epa.2”.
7. Press start.
8. Check on the temperature profile about every 20 minutes to make sure it is working properly.
9. The run should take about an hour. When the temperature is 40°C or lower, the samples can be removed from the microwave.

Sample transfer:

1. Collect the following items:
 - Purple centrifuge rack
 - Small distilled water wash bottle
 - 2 L 10% Nitric acid bath
 - Pressure release tool
 - Vessel removing tool
 - Washable marker
2. Retrieve the carousel containing samples from the microwave digester and place in the fume hood.
3. Number the centrifuge tubes using the washable marker.

4. Working inside the hood, use the pressure relief tool to release excess pressure from the safety valve.
5. Carefully remove the safety valve from the first brown safety shield.
6. Using the vessel-removing tool, pop the vessel out of the shield by lining up one of the two holes in the bottom of the shield with the two projections in the tool and pressing down.
7. Remove the white Teflon cap from the vessel.
8. Using distilled water; quantitatively transfer the contents of the vessel to the corresponding centrifuge tube.
9. Dilute the sample up to the 25 ml mark on the centrifuge tube, also mark the dilution on the sample sheet.
10. Cap the tube and invert it 3 times. Repeat with remaining samples.

Clean up:

1. Place white Teflon vessels and caps in a 10% Nitric acid bath (let sit for 30 minutes- 1 hour).
2. Rinse the white Teflon vessels and caps with distilled water.
3. Rinse safety shields and safety valves with distilled water be sure to tap additional water out of safety valves.
4. Return sample carousel to the microwave digester.

B.3.2 iCAP™ 7400 ICP-OES Analyzer



Figure 22: iCAP 7400 ICP-OES Analyzer

Refer to ThermoFisher website for full description.

<https://www.thermofisher.com/order/catalog/product/842320074081>

B.4 Raw ICP Concentrations

Sam ple	Mass (g)					Raw ICP Concentrations [mg/L]			
	Mass tube	Mass tube plus soil	Mass after water	Mass after loose	Mass after Al-Fe	Water -P	Loose -P	Al- Fe -P	Ca -P
1	11.564	21.371	25.047	24.68	25.077	0.57	0.14	4.78	0.35
2	11.457	22.132	28.035	28.903	30.574	0.62	0.28	11.85	1.49
3	11.685	20.096	25.383	26.399	27.492	0.46	0.23	6.43	1.21
4	11.496	20.932	26.53	27.463	29.047	1.09	0.58	10.85	0.66
5	11.501	21.128	27.162	28.307	28.84	0.22	0.1	5.43	0.52
6	11.475	21.449	24.858	24.867	25.165	0.6	0.23	3.7	0.93
7	11.643	21.456	25.095	25.155	25.356	0.91	0.31	7.48	0.9
8	11.568	21.729	25.705	25.829	26.611	0.92	0.37	5.12	1.55
9	11.533	22.538	26.959	27.234	27.065	0.65	0.31	17	1.15
10	11.307	21.453	26.339	26.761	28.501	0.95	0.37	16.1	2.74
11	11.468	22.776	26.549	26.579	26.863	0.29	0.08	2.27	0.18
12	11.672	22.559	27.835	28.426	28.581	0.53	0.19	11.7	1.16
13	11.505	23.044	28.247	28.7	28.301	0.51	0.17	6.69	1.02
14	11.563	20.994	26.498	27.368	27.561	0.72	0.22	9.69	1.34

15	11.51	21.479	27.615	28.408	29.377	0.35	0.16	4.08	1.03
	11.468	22.059	28.549	29.482	30.121	0.37	0.16	3.65	1.01
16	11.649	22.359	27.368	27.913	27.986	0.39	0.19	9.65	1.1
17	11.461	21.045	26.617	27.291	27.796	0.43	0.16	10.7	0.76
18	11.522	21.021	24.209	24.174	24.232	0.77	0.28	1.33	0.37
19	11.519	21.331	27.071	27.793	28.955	0.33	0.17	8.19	1.07
20	11.466	21.406	26.307	27	27.875	0.29	0.13	7.84	0.59
21	11.645	21.263	26.688	27.456	27.778	0.41	0.17	8.2	1.02
22	11.531	23.015	29.785	31.028	31.961	0.32	0.17	7.21	1.51
23	11.512	22.63	29.298	30.803	31.108	0.28	0.15	4.22	0.87
24	11.473	23.222	30.142	31.658	32.278	0.32	0.14	4.61	1.15
25	11.419	22.445	28.978	30.432	30.89	0.24	0.14	4.31	0.83
26	11.407	20.882	26.666	27.996	27.967	0.17	0.11	1.64	0.59
27	11.422	21.697	28.09	29.436	29.862	0.22	0.12	1.97	0.76
	11.684	21.401	27.476	28.911	29.24	0.21	0.12	1.9	0.62
28	11.557	21.796	28.022	29.102	30.501	0.38	0.21	7.45	1.47
29	11.492	21.925	28.321	28.803	30.382	0.35	0.15	5.6	1.37
30	11.316	22.976	28.446	29.073	29.484	0.17	0.09	2.75	0.55
31	11.45	21.705	26.189	26.598	26.48	0.39	0.19	3.42	1.34
32	11.735	20.865	26.306	27.391	27.559	0.17	0.11	2.22	0.43
33	11.498	22.923	29.136	30.32	31.802	0.44	0.2	9.63	1.64
34	11.507	21.863	29.447	30.041	29.993	0.14	0.11	1.65	0.48
35	11.464	21.578	27.676	28.851	29.818	0.35	0.16	6.07	1.12
36	11.5	21.156	26.929	28.138	28.619	0.3	0.16	4.99	0.89