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# Characterizing the Fate and Mobility of Phosphorus in Utah Lake Sediments

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# Characterizing the Fate and Mobility of Phosphorus in Utah Lake Sediments

Matthew Chambers Randall

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 the Fate and Mobility of Phosphorus in Utah Lake Sediments

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An increasing number of lakes worldwide are impacted by eutrophication and harmful algal blooms due to nutrient inputs. Utah Lake is a unique eutrophic freshwater lake that is naturally shallow, turbid, and alkaline with high dissolved oxygen levels. Recently, the Utah Division of Water Quality has proposed a new limitation of phosphorus (P) loading to Utah Lake from wastewater treatment plants in an effort to mitigate eutrophication. However, reducing external P loads may not lead to immediate improvements in water quality due to the legacy pool of nutrients in lake sediments. The purpose of this study was to characterize the fate and mobility of P in Utah Lake sediments to better understand P cycling in this unique system. We analyzed P speciation, mineralogy, and binding capacity in lake sediment samples collected from 15 locations across Utah Lake. P concentrations in sediment ranged from 306 to 1894 ppm, with highest concentrations in Provo Bay near the major metropolitan area. Sequential leach tests indicate that ~25-50% of P is associated with Ca ( $\text{CaCO}_3$  /  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH},\text{F},\text{Cl})_2 \approx \text{P}$ ) and 40-60% is associated with Fe ( $\text{Fe}(\text{OOH}) \approx \text{P}$ ). Ca-associated P was confirmed by SEM images, which showed the highest P concentrations correlating with Ca (carbonate minerals/apatite). The Ca-associated P fraction is likely immobile, but the Fe-bound P is potentially bioavailable under changing redox conditions. Batch sorption results indicate that lake sediments have a high capacity to absorb and remove P from the water column, with an average uptake of 70-96% removal over the range of 1-10 mg/L P. Mineral precipitation and sorption to bottom sediments is an efficient removal mechanism of P in Utah Lake, but a significant portion of P may be temporarily available for resuspension and cycling in surface waters. Mitigating lake eutrophication is a complex problem that goes beyond decreasing external nutrient loads to the water body and requires a better understanding in-lake P cycling.

Keywords: Eutrophication, Internal P loading, P speciation, P sorption, P mineralogy, Utah Lake, Lake sediments

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## 1. Introduction

In recent decades, an increasing number of lakes are impacted by eutrophication due to nutrient inputs from rising populations and urbanization. Excessive nutrients such as phosphorus (P) in a body of water may lead to eutrophication and transform a clear blue lake into a green algal swamp (Golterman and Oude 1991). These algal lakes occasionally contain harmful algae known as cyanobacteria, which can produce toxic microcystins and other neurotoxins (Christoffersen and Kaas 2000). However, it is not always obvious how much P is necessary to cause eutrophication and often lake sediments contain a substantial internal storage of nutrients that can outweigh inflows to the lake (Ogdahl et al. 2014). P concentrations and release have become a focus of study because it is widely regarded as a limiting nutrient in phytoplankton growth (Correll 1990).

External reductions of P loads does not always lead to the immediate improvement of water quality (Scheffer et al. 1993). Attempts have been made to severely limit the amount of P from entering lakes at great cost with little to no effect on the eutrophic state (Kapanen 2012). Although some measures can be taken to prevent nutrient pollution, it can be expensive and difficult to prevent entirely. The only escape for P from lake bioavailability is through the sediments, which act as a net P sink (Nowlin et al. 2005). Once P is bound into minerals it is typically unavailable for biological uptake (Lai and Lam 2008). However, sediments can function as a temporary source of P, depending on the physiochemical properties of the sediments and overlying water (Richardson 1985). The delayed response of lake improvement typically takes 10-15 years (Jeppesen et al. 2005), which time lag is often attributed to the continual release of P from bottom sediments (Carvalho et al. 1995)

P release in shallow lakes is a significant temporary source of nutrients due to the re-suspension of loosely bound nutrients in sediments (Nowlin et al. 2005), which become available after mixing (i.e. wind and boat propellers) (Søndergaard et al. 2013). Internal P release from lake sediments to overlying waters is strongly influenced by dissolved oxygen levels, pH, temperature, microbial activity, and pore water phosphorus concentrations (Moore et al. 1998). Shallow lakes are especially prone to P release given high surface area to volume ratio, making sediment-water interactions a particularly key role in P exchange (Søndergaard et al. 2001). As external loads are reduced, nutrient legacy pools from internal lake sediments and watershed soils can release P and drive eutrophication (Sharpley et al. 2013).

A variety of methods are typically used to investigate P characteristics and potential release in lake sediments. Sequential extractions are used to determine the P binding forms in aquatic sediments (Boström et al. 1988, Hupfer et al. 2009, Istvánovics et al. 1989), while batch sorption is used to quantify P sorption characteristics of bottom sediments (Olila 1993). Identification of lake sediment mineralogy and P-bearing minerals using x-ray diffraction (XRD) (Ippolito et al. 2003) and scanning electron microscope imaging (Hupfer et al. 2009) also help to determine the risk of internal P release by identifying elemental associations of P mineralogy in lake sediments. X-ray fluorescence (XRF) has also been used to quantify elemental compositions of lake sediments, including P (Lopez et al. 1996).

Utah Lake, located in northern Utah, USA, (Fig. 1) is a large but shallow lake with a high surface area to volume ratio, with potential for P release from the sediment-water interface. With the growing population and development of the Utah Lake watershed, greater discharge of domestic, agricultural and industrial wastewater, and nutrient loading to Utah Lake has led to water quality degradation (PSOMAS 2007). The Utah Department of Water Quality has recently

adopted a new TP loading policy to control effluent discharge of local wastewater treatment plants (WWTP) in order to prevent further P pollution loading (Hood 2016). Yet, the role of P retention and release and the cycles involved in Utah Lake sediments is largely unknown. The purpose of this study is to identify the sinks and subsequent mobility of P in Utah Lake. Specific objectives are to (1) characterize P concentrations in sediment; (2) characterize sediment mineralogy, with emphasis on P-bearing mineral phases; (3) evaluate P fractions in sediment via sequential extraction experiments; (4) evaluate P binding capacity via batch sorption experiments; and (5) evaluate P dynamics in surface water via mixing experiments of effluent water and lake water.

## 2. Materials and Methods

### *2.1 Site Description*

Utah Lake (Fig. 1) is the largest natural freshwater lake in the state of Utah and one of the largest west of the Mississippi River (PSOMAS 2007). It is located on the eastern edge of the Basin and Range Province juxtaposed to the Wasatch Range and about 48 kilometers southeast of the Great Salt Lake. The lake stretches 38 kilometers long from N-S with a maximum width of 21 kilometers E-W, covering a surface area of approximately 375 square kilometers. Utah Lake is shallow, with an average depth of 2.7-3 meters and a maximum depth of 5.5 meters during normal lake levels (PSOMAS 2007). This shallow depth allows for wind to easily re-suspend bottom sediments, generating turbid water. This turbid water influences the photosynthetic activity by limiting the amount of light that penetrates the water column. Light limiting turbidity has been speculated to slow, and even limit, the amount of algae present in lakes (Havens et al. 2003). However, while turbidity may be a major issue on Utah Lake, no major studies have been done to investigate the links between turbidity and algal growth.

Snowmelt from the Wasatch Range and Uinta mountains is the largest source of water for Utah Lake as well as the many natural springs due to faults from the horst and graben features of the Basin and Range Province (Hooton 1989). Primary inputs to the lake come from the Spanish Fork, Provo, and American Fork Rivers, with minor inflows from Hobble Creek and Currant Creek, with the only output being the Jordan River, which flows north to the Great Salt Lake. Average inflow into the lake is about 888.1 million cubic meters/year with an average outflow of 426.6 million cubic meters/year, with 468.6 million cubic meters/year lost to evaporation (Hooton 1989). Sedimentation rates average about 1.4mm/yr.—mostly from calcium carbonate precipitation, silicate minerals, and iron hydroxides (Macharia 2012).

The lake is also popular among the locals for boating and other recreational use, making it a great income source for the Utah state government (PSOMAS 2007). By 2050, reports suggest that the population surrounding Utah Valley will double in size, increasing the need for municipal water and other amenities (Development 2013). Cleaning up Utah Lake has long been a debated issue and is becoming of increasing interest to local residents. Utah Lake is commonly thought of as a polluted lake because of the amount of agricultural runoff and wastewater effluent entering the lake (PSOMAS 2007). Currently, seven local WWTPs discharge into Utah Lake with variable nutrient loads. It has also recently received a lot of media attention because of noticeable algal blooms that occur during the late summer or early fall, which have been reported to kill local livestock and in one instance, a family dog (Quality 2015). Cyanobacteria, the toxic algae of interest, can be found at times throughout the lake.

## *2.2 Sediment Sampling*

To evaluate temporal and spatial variability in P sediment chemistry, we collected sediment samples from Utah Lake over the course of one year. A total of 26 major sediment

samples were collected from 15 sites across Utah Lake during four months: October 2015 (“A”), May 2016 (“B”), August 2016 (“C”), and November 2016 (“D”). Not all sample sites were visited each sampling trip. Sample sites 1, 11-13, and 15 are located in Provo Bay and sample sites 2-10 and 14 are located in the main body of the lake (Fig. 1). Lake sediment consisting of the top 5 cm was collected using a stainless-steel Ekman dredge and stored in 3.8 liter Ziploc bags on ice until transported back to the laboratory (within 12h). Sediments were stored at 4°C until subsamples were taken for physiochemical analyses and sorption experiments. An additional 55 minor sediment samples were collected between August 2015 and September 2015 to provide a higher resolution map of the spatial variability of TP in sediments throughout Utah Lake. These additional 55 sediment samples are characterized as minor because they were only analyzed for TP by total digestion.

### *2.3 Sediment Analysis*

Sediment samples were analyzed by a variety of instruments to determine their physiochemical properties. Lake sediments were dried at 60°C until a constant weight to determine water content (~3 days). Dried samples were then crushed with a mortar and pestle and stored at room temperature in air-tight Ziploc bags until physiochemical analysis. Organic matter and carbonates were determined using mass loss on ignition (LOI) difference before and after combustion in a Lindberg Blue M muffle furnace at 1000°C for 4h (Oliver Heiri 2001).

Dried sediment samples were analyzed for major and trace element oxide concentrations using x-ray fluorescence (XRF). All 26 dried sediment samples were then powdered using a tungsten carbide TS 250 Siebtechnik Shatter Box and dried at 105°C overnight. Samples were then compacted into pressed pellets and melted into glass discs using a Katanax K1 Prime electric furnace for analysis on a Rigaku ZSX Primus II XRF spectrometer of major element

oxide weight percentages ( $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{MnO}$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ , and  $\text{P}_2\text{O}_5$ ) and trace elements (Ba, Ce, Cl, Cr, Cu, F, Ga, La, Nb, Nd, Ni, Pb, Rb, S, Sc, Sm, Sr, Th, U, V, Y, Zn, and Zr). Weight percent P was then calculated to mg P/kg dried sediment or parts per million.

Mineralogy of lake sediment was determined using x-ray diffraction (XRD). 23 of the 26 dried powdered samples prepared for XRF analysis were also used in XRD analysis using a Rigaku MiniFlex 600 XRD. XRD patterns were analyzed using Rietvelt PDXL2 with 2-theta mineral intensity peaks obtained from the American Mineralogist Crystal Structure Database.

Scanning electron microscope (SEM) images were taken to provide an in-situ and non-invasive view of elemental correlations within sediment grains. Four samples were used in Environmental SEM imaging (4-B, 1-A, 2-A, and 3-A). 2-A and 3-A were selected because they were taken from the center of the lake and are representative of regular lake conditions. 4-B and 1-A were selected because of their proximities to wastewater effluent and high concentrations of TP in sediments. To prepare samples for the ESEM, 5 mg of dried crushed lake sediment was suspended in 50mL of Milli-Q water and shaken until 8  $\mu\text{L}$  of water was extracted and dropped onto a silicon chip, that was carbon taped onto a stainless-steel stud. The stud was then placed in a flow hood until the Milli-Q water evaporated leaving behind a shallow film of dried sediment grains. The stud and silicon chip were then carbon coated for analysis on the ESEM and Helios microscopes. Scanning images and dot maps were created using energy dispersive spectroscopy capabilities of the ESEM and FEI Helios Nanolab 600 DualBeam FIB/SEM (Helios) microscopes analyzing for Ca, P, Fe, Mn, Al, K, and O

Sediment samples from May 2016 (10 total samples) were used in a sequential extraction procedure to investigate phosphorus fractionation in bottom sediments. These samples were



chosen because they represented 10 of the total 15 sites across the lake. TP in sediments also varied little with respect to the timeframe of this study. Sediment P species were determined using a sequential extraction scheme for calcite rich lakes from Hupfer et al. (2009), modified after Psenner et al. (1984). After each fraction the supernatant was filtered through a 0.45- $\mu$ m nylon filter, acidified with 2.4% v/v  $\text{HNO}_3$ , and stored at 4°C until analyzed for TP using a Thermo Scientific Inductively Coupled Plasma Optical Emission Spectroscope (ICP-OES) iCAP 7000 Series. The instrument was calibrated using Inorganic Ventures IV-ICPMS-71A and 71B aqueous solutions for trace elements (Al, As, B, Ba, Ca, Fe, K, Li, Mg, Mn, Mo, Na, P, Pb, Sb, Se, Si, Sr, Tl, V, Zn) and the IV-Stock-50 for major cations (Ca, Fe, K, Mg, and Na). A standard of a known concentration of IV-ICPMS-71A and 71B was run every 10 samples to check for instrument drift. New standards were mixed before each run to ensure concentrations had not changed in standards after a couple months in between sampling events. An independent standard (High Purity Standards 68-A) was analyzed with a subset of samples to verify accuracy of measurements.

Steps included: 1 M  $\text{NH}_4\text{Cl}$  (deoxygenated  $-\text{N}_2$  purged) shaken for 0.5h, 0.11 M BD (bicarbonate/dithionite-buffered to a pH 7 using  $\text{NaHCO}_3$ ) shaken for 1 h, 1M NaOH shaken for 16 h, 0.5 HCl shaken for 16 h, and 1 M boiling HCl for 0.25 h after a 550°C ignition for 2 h. Extractant supernatant was also analyzed for Fe and Ca to ensure release of Fe-P during the BD step and Ca-associated P during the HCl step of the sequential extraction procedure.

Total digestion and analysis of TP using ICP-OES and XRF was also done to each sample to check the efficiency and to verify total concentrations from the extraction procedure. A total of 81 samples (26 major and 55 minor sampling locations) were collected across the lake during the time frame of the study (August 2015-November 2016) for total digestion analysis of

TP in sediments. Samples analyzed for TP in sediments by total digestion were determined using US EPA method 3052, where 8mL HNO<sub>3</sub> and 2 mL H<sub>2</sub>O<sub>2</sub> is added to 0.1g of sample, then digested in an ETHOS UP Microwave Digestion System at 180°C for 15min before analysis for TP by ICP-OES.

To quantify the binding capacity of P in lake sediments, batch sorption experiments were conducted on sediment samples from May 2016 and November 2016 (14 total samples). These samples were chosen to represent spatially variability of lake sediment and to provide a temporal variability in the results received from experimentation. Two separate batch sorption experiments were performed to observe phosphorus sorption under low and high TP concentrations. The high TP experiment was performed by adding NaH<sub>2</sub>PO<sub>4</sub>•H<sub>2</sub>O to background lake water solution in the amounts of 0, 0.7, 2.1, 6.9, and 20.7 mg/L TP. The low TP experiment was done at lower concentrations by adding NaH<sub>2</sub>PO<sub>4</sub>•H<sub>2</sub>O to background lake water solution in the amounts of 0, 0.07, 0.21, 0.7, 2.1, and 6.9 mg/L TP. Prior to experimentation, lake water was collected and stored to use as a background for batch sorption experimentation. One gram of wet lake sediment was weighed into a 50mL polyurethane centrifuge tube to which were added 50 mL of background lake water solution containing the different P concentrations. Samples were allowed to equilibrate for 24h on an end-to-end shaker, after which they were centrifuged at 5000 rpm for 15m. The supernatant was collected using a syringe and immediately filtered through a 0.45-μm nylon filter, acidified with 2.4% v/v HNO<sub>3</sub>, and stored at 4°C until analyzed for TP on ICP-OES. TP sorbed by lake sediment was calculated as the difference between the initial and equilibrium concentrations.

## 2.4 Mixing Experiments

Mixing experiments between various percentages of wastewater and lake water were conducted to observe changes in P speciation. Two mixing experiments were conducted in the laboratory by mixing varying amounts of wastewater effluent water with Utah Lake water and measuring total dissolved phosphorus (TDP) and soluble reactive phosphorus (SRP) concentrations over time. Mixtures included effluent to lake water ratios of 1:9, 3:7, 5:5, 7:3, and 9:1 in 50mL polyurethane centrifuge tubes. After mixing, samples were allowed to equilibrate for 24h on an end-to-end shaker, after which TDP samples were filtered through a 0.45- $\mu$ m nylon filter and analyzed for TDP (acidified to 2.4% v/v HNO<sub>3</sub>) by ICP-OES. SRP was measured using the molybdenum blue method (Murphy and Riley 1962). Sample solutions were mixed with ammonium molybdate, antimonyl potassium tartarate and ascorbic acid in an acidic medium and the absorbance of the phosphomolybdenum blue complex was measured colorimetrically at 882 nm after 10 min using a Thermo Scientific Genesys 20 Visible Spectrophotometer.

## 3. Results

### 3.1 Major elemental compositions of lake sediments

Major element composition of lake sediments determined by XRF shows that Utah Lake sediment is primarily composed of Si, Ca, and organic/carbonate material (LOI) with trace amounts of other elements (Table 1). Silica (SiO<sub>2</sub>) ranged from 20.5 – 71.0 wt.%, averaging 39.0 wt.%; Calcium (CaO) ranged from 8.3 – 36.9 wt.%, averaging 25.6 wt.%; and organic/carbonate material (LOI) ranged from 8.2 – 36.4 wt.%, averaging 24.7 wt.%. Phosphorus was measured at a range of 0.14 – 0.43 wt.% with an average of 0.22 wt.%. Weight percent TP was then calculated to concentration TP through the conversion 1 wt.% TP = 10,000 ppm TP.

Concentrations of TP in sediment measured by XRF varied spatially throughout the lake with a maximum of 1894 ppm TP and a minimum of 615 ppm (Table 2 & Fig. 2).

Total digestion of sediments shows variable TP concentrations in bottom sediments across Utah Lake (Fig. 3 & Table 2, see also Supplementary Data Table S6). Concentrations of TP in sediment varied spatially throughout the lake with a maximum of 1710 ppm TP and a minimum of 306 ppm. Areas near WWTP effluent (i.e. Lindon Marina (sample site 4) and the east side of Provo Bay, sites 1, 12, and 11) had higher concentrations of TP in sediment, reaching a maximum of 1710 ppm TP. However, the main body of the lake fluctuated between 1165 and 306 ppm. Differences in concentration between total digestion and sequential extraction might arise from the differences in number of acids and bases used in digestion, time allotted for equilibration to be reached, and solution:sediment ratios used in extraction (Fig. 4 & Supplemental Fig. S1).

### *3.2 Mineralogy of lake sediments*

X-ray diffraction results showed that lake sediments were primarily composed of carbonates (calcite, aragonite, and dolomite) and quartz (Table 3). Calcite ( $\text{CaCO}_3$ ) dominated mass percentages averaging 39.7%, with a minimum of 8.9% and maximum of 69.9%. Aragonite and dolomite only averaged 4.4% and 2.0%, respectively. Quartz ( $\text{SiO}_2$ ) was found at an average mass percentage of 25.3% with a minimum of 7.4% and a maximum of 49.7%. Feldspars (plagioclase (albite ( $\text{NaAlSi}_3\text{O}_8$ ) and anorthite ( $\text{CaAl}_2\text{Si}_2\text{O}_8$ )) and k-feldspar (microcline –  $\text{KAlSi}_3\text{O}_8$ )) averaged a mass percentage of 15.4%, with a maximum of 32.9% and a minimum of 5.2%. Clay particles (kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ), montmorillonite ( $((\text{Na,Ca})_{0.33}(\text{Al,Mg})_2(\text{Si}_4\text{O}_{10})(\text{OH})_2 \cdot n\text{H}_2\text{O})$ ), and muscovite ( $\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$ ) averaged a mass percentage of 9.8% with a maximum of 19.7% and a minimum of 1.0%. Other mineral

phases present consisted of minor Goethite ( $\text{Fe}(\text{OOH})$ ), averaging 0.9% with a max of 2.5%; ankerite ( $\text{Ca}(\text{Fe,Mg,Mn})(\text{CO}_3)_2$ ), averaging 1.3% and a max of 3.4%; hornblende ( $\text{Ca}_2(\text{Mg,Fe,Al})_5(\text{Al,Si})_8\text{O}_{22}(\text{OH})_2$ ), averaging 0.6% with a maximum of 2.4%; And pyrite ( $\text{FeS}_2$ ), averaging 0.8% with a max of 3.2%. A representative graph of intensity versus 2-theta peaks is provided in Fig. 5 (also see Supplementary Table S4).

### *3.3 In-situ imagery of elemental correlations in lake sediments*

Images of lake sediment taken by ESEM and the Helios electron microscope show P disseminated throughout mineral grains associated with Fe and Ca (Fig. 6), with a few highly-concentrated P grains corresponding with Ca rich grains (Fig. 7). Backscattered electron microscope images overlain with energy dispersive spectroscopy dot maps show in-situ elemental correlations of Ca, P, and O within specific mineral grains. This represents most of the elemental assemblage of an apatite crystal ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH,F,Cl})_2$ ). Some, but not all, of the highly-concentrated grains also showed the hexagonal mineral structure of apatite. Other highly concentrated Ca, P, and O grains were more amorphous in shape are likely to be precipitated amorphous calcium apatite, as the formation of crystalline apatite in solution is intimately related to amorphous calcium apatite (Eanes et al. 1973) (Supplementary Fig. S2). Other mineral assemblages that were apparent in imaging were calcite ( $\text{CaCO}_3$ ), iron (oxy)hydroxides ( $\text{Fe}(\text{OOH})$ ), and pyrite ( $\text{FeS}_2$ ).

### *3.4 Sorption capacity of lake sediments*

Sorption experiments showed high P sorption capacity in Utah Lake sediments, with some spatial variability (Fig. 8). Sorption was particularly high in all samples at low P concentrations in water with some spatial variability. Two experiments were conducted; a high concentration experiment with a range of 0.7 mg/L to 20.7 mg/L TP and a low concentration

experiment with a range of 0.07 mg/L to 6.9 mg/L TP. In the high batch sorption experiment, 91-99% of initial TP concentrations of 0.7 mg/L were sorbed by lake sediments, with an average of 94.6%. At 2.1 mg/L initial TP concentrations, 62-97% was sorbed by sediments, with an average of 79.9%; 6.9 mg/L TP sorbed 32-86%, with an average of 55.7%; and 20.7 mg/L initial TP, sorbed 11-63%, with an average of only 31.0%. Sorption characteristics of sediments varied spatially across the lake and were mostly dependent on the physio-chemical properties of the sediment. Muddy or fine grained areas, such as Lindon Marina (sample site 4) or the center of the lake (sample site 3), tended to have higher sorption capacities than coarser grained areas (i.e. sample site 9) (Fig. 9A).

Low batch sorption experiments varied in sorption capacity across the lake, depending largely on the physio-chemical properties of the sediment. Results from low batch sorption experiments showed that 61-100% of initial TP concentrations of 0.07 mg/L were sorbed by lake sediments, with an average of 85.1%. At 0.21 mg/L initial TP concentrations, 86-100% was sorbed by sediments, with an average of 94.1%; 0.7 mg/L TP sorbed 88-97%, with an average of 90.8%; 2.06 mg/L TP sorbed 75-87%, with an average of 82.7%; and 6.9 mg/L TP sorbed 51-71%, with an average of 66.3% (Supplementary Table S5). As concentrations of initial TP in solution increased, percentage of uptake decreased. Low batch sorption isotherms vary spatially across the lake and, just like the high batch sorption results, have a higher sorption capacity in muddy or fine grained areas than in coarser grained areas (Fig. 9B). These results suggest that in 24 hours Utah Lake sediments sorb the majority of TP in the overlying water column at low initial concentrations (<1-2.1 mg/L TP), and have a differing maximum sorption capacities, depending on the physio-chemical properties of the sediment.

### *3.5 Phosphorus speciation in lake sediments*

P speciation in lake sediments, determined through sequential extraction experiments, was found predominantly associated with Fe and Ca minerals. Results show >5% of the TP fraction was loosely sorbed to lake sediments or interstitial pore space ( $\text{NH}_4\text{Cl}$  step), ~40-60% of the TP fraction bound with redox sensitive Fe/Mn (oxy)-hydroxides (BD step), 2-6% bound to clay minerals (NaOH step), ~25-50% bound with calcium minerals (HCl step), and >8% as residual TP (Residual) (Fig. 4 and 10). Minimal amounts of Mn were found in Utah Lake sediments, suggesting most of the Fe/Mn fraction to be associated with primarily surface complexation sorption to Fe (oxy)-hydroxides ( $\text{Fe}(\text{OOH}) \approx \text{P}$ ). Releases of Fe and Ca were also measured in the sequential extractant solution and were found to correlate with the release of TP during their particular extractant phases, phases 2 (BD) and 4 (HCl) respectively (Fig. 11 & Supplementary Table S2). Overall, Fe-P ( $\text{Fe}(\text{OOH}) \approx \text{P}$ ) and Ca-associated P ( $\text{CaCO}_3/\text{Ca}_{10}(\text{PO}_4)_6(\text{OH},\text{F},\text{Cl})_2 \approx \text{P}$ ) were the dominant phosphorus forms in Utah Lake sediments, accounting for 85-91% of the TP in sediments (Fig. 10). For a list of all analysis run on sediment samples see table 4.

### *3.6 Mixing experiment between wastewater and lake water*

Mixing experiments between wastewater treatment (WWTP) effluent and lake water show changes in speciation of SRP. After starting concentrations of wastewater effluent (6.54 mg/L TDP & 3.87 mg/L SRP) and Utah Lake water (0.05 mg/L TDP & 0.02 mg/L SRP) mixing at 5 different ratios (9:1, 7:3, 5:5, 3:7, 1:9) and shaking for 24 h there were no significant deviations to TDP from the calculated mixing line (Fig. 12). However, when SRP was measured after 24 h of shaking, significant deviations from the calculated mixing line occurred at ratios of 9:1, 7:3, 5:5 and 3:7 wastewater to lake water (Fig. 12). This can be regarded as a change in

speciation, from SRP to dissolved organic P. After 24h of mixing WWTP and lake water, the amount of SRP decreased by a maximum of 1.2 mg/L (41%), likely converting to dissolved organic P. TDP represents the sum of SRP and dissolved organic P and remains unchanged after 24h of mixing. Therefore, the 1.2 mg/L decrease of SRP means an increase of 1.2 mg/L of dissolved organic P after 24h mixing at 70% WWTP effluent and 30% lake water. Raw data from the experiment are provided in the Supplementary data (Table S3).

## 4. Discussion

### *4.1 Spatial variability of phosphorus in Utah Lake Sediments*

Overall, Utah Lake sediments were higher in TP near zones of WWTP effluent than the rest of the lake. This can likely be attributed to the external source of TP being added due to the lack of major nutrient removal steps in current wastewater practices. The major WWTP inflows to Utah Lake are located near the east side of the lake; including the east side of Provo Bay (sample sites 1 and 11-15) and Lindon Marina (sample site 4). The east side of Provo Bay is dominated by wastewater treatment (WWTP) effluent inlets. Due to the shallow nature of Utah Lake, TP in the sediment is relatively uniform across the main body of the lake because of how well mixed the water is by the prevailing winds. High TP concentrations in sediment relative to main lake conditions, are typically seen near areas of WWTP effluent, suggesting strong correlations between sediment TP and surface water concentrations of TP. While flux rates are generally unknown, the sediment-water interface seems to be a major controlling factor of P cycling in Utah Lake. Sediment concentrations generally change by a factor of three across the lake (600-1900 ppm).



#### *4.2 Phosphorus sinks in Utah Lake*

P in Utah Lake sediments is primarily found in two fractions: P associated with iron ( $\text{Fe}(\text{OOH}) \approx \text{P}$ ) and (2) P co-precipitated with carbonate minerals and apatite ( $\text{CaCO}_3/\text{Ca}_{10}(\text{PO}_4)_6(\text{OH},\text{F},\text{Cl})_2 \approx \text{P}$ ). Both of these sites act as potential sinks that will sequester P out of the water column and into the sediment (Boström et al. 1988, Istvánovics et al. 1989). Batch sorption results indicate that Utah Lake sediments have a high capacity to absorb P out of the water column at low TP surface water concentrations. This sorption characteristic of Utah Lake sediments has the potential to sequester bioavailable P out of the water column, limiting the amount of P available for phytoplankton use. Alongside batch sorption data, SEM imaging and sequential extraction results confirmed calcium's strong relationship with TP in sediments. The Ca-associated P bond is strong at or above neutral pH values (Gomez et al. 1999). Utah Lake is an alkaline lake with pH values of approximately 8.5 and is highly buffered by the large amount of bicarbonate, suggesting variation in pH very unlikely. The majority of Ca-associated P is a relatively permanent sink for P in Utah Lake with alkaline pH values. While SEM imagery did not show strong correlations with Fe-P (Fig. 6), likely due to release of surface sorbed P in sample preparation and small amounts of P that were mostly disseminated amongst grains, sequential extraction results show a significant amount of Fe-bound P fraction (Fig. 11).

#### *4.3 Phosphorus mobility in Utah Lake*

Utah Lake sediments can function as either a sink or temporary source for P, depending on the physiochemical properties of the sediments and overlying water. Batch sorption results show the majority of overlying TP concentrations in surface waters of 0.1 – 1 mg/L is ~95% absorbed by Utah Lake sediments. However, once trapped in the sediments a resuspension event, drops in pH, changes in dissolved oxygen levels, and increased microbial activity can re-suspend

this P back into the water column. P flux from sediments to overlying waters are typically related to dissolved oxygen levels, pH, temperature, microbial activity, and pore water phosphorus concentrations, which strongly influence P release (Moore et al. 1998). This potential internal source of P release could prove to be a large implication for lake restoration if external P loading inputs are terminated.

Ca-associated and Fe-bound P account for 85-91% of the TP in sediments; Ca-associated P is relatively immobile while pH values remain alkaline, but Fe-bound P is considered a redox-sensitive P fraction sorbed by surface complexation to goethite ( $\text{Fe}(\text{OOH})$ ) subject to mobilization depending on sediment redox potential, making it a highly labile fraction. This redox-sensitive relationship where P can be released under reducing or anoxic conditions often develops in sediments during summer time when both temperature and microbial activity are high. This creates an environment where Fe-bound P is subject to mobilization or dissolution under reducing conditions (Lai and Lam 2008).

However, simple anoxic conditions are not the only explanation for release of Fe-bound P. Sulfate-controlled P release is proposed to be another reason for P release, as sulfate reduction occurs in sediments it permanently immobilizes Fe. Consequently, less Fe is therefore available for P re-precipitation resulting in decoupling of the Fe and P cycles (Hupfer and Lewandowski 2008).

#### *4.4 Implications for lake restoration*

Wastewater is a major source of TP to Utah Lake but the reduction of external P loads may not result in immediate enhancement of water quality due to P release from lake sediments. P fluxes from sediment to the water column need to be quantified since it will have major implications on lake restoration. The nutrient-enriched sediments of Utah Lake are a potential P

source to the overlying water and high concentrations of the redox-sensitive/sulfate-controlled Fe-P fraction in lake sediments suggest that substantial P release would occur upon dissolution of ferric oxides (Fe-P) under strongly reducing conditions (Lai and Lam 2008) or during the reduction of sulfate in deeper sediments. Therefore, spending money to enhance wastewater treatment to remove a significant amount of nutrients might not result in the immediate enhancement of water quality (Scheffer et al. 1993) and could take 10-15 years before improvement is seen (Jeppesen et al. 2005). Apart from lowering external loads, proactive measures could be taken to reduce temporary P release from Utah Lake sediments, e.g. applying chemical amendments to bind mobile P, before any significant water quality improvements could be achieved (Lai and Lam 2008Lai and Lam 2008).

## 5. Conclusion

Understanding the fate and mobility of phosphorus (P) in Utah Lake sediments is essential for evaluating P cycling and potential impacts on water quality. In this study, we analyzed lake sediments to determine primary P sinks and the potential for temporary internal P release. Results suggest that most of the P in Utah Lake sediments is disseminated throughout calcium carbonate ( $\text{CaCO}_3$ ) and iron (oxy)hydroxides ( $\text{Fe}(\text{OOH})$ ), with a few highly-concentrated apatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH},\text{F},\text{Cl})_2$ ) crystals. Calcium and silica dominate the sediment composition, of which calcium is one of the primary sinks for P in Utah Lake sediments. Iron (oxy)-hydroxides ( $\text{Fe}(\text{OOH})$ ) are also a major source/sink for P in lake sediments, depending on the redox state of the iron. Batch sorption results indicate Utah Lake sediments have a high capacity to adsorb P from the water column at low surface water concentrations of TP. Our findings suggest that Utah Lake sediments have a high capacity to temporarily adsorb TP from the water column, but also have the potential to be a significant source of temporary P release to surface waters. This study

has implications for water quality restoration plans of an increasing amount of eutrophic lakes near developing cities, where nutrient pollution has become critical.

Table 1. X-ray fluorescence (XRF) results showing weight percentages of major constituents of Utah Lake sediments.

<i>Sample ID</i>	<i>SiO<sub>2</sub></i>	<i>TiO<sub>2</sub></i>	<i>Al<sub>2</sub>O<sub>3</sub></i>	<i>Fe<sub>2</sub>O<sub>3</sub></i>	<i>MnO</i>	<i>MgO</i>	<i>CaO</i>	<i>Na<sub>2</sub>O</i>	<i>K<sub>2</sub>O</i>	<i>P<sub>2</sub>O<sub>5</sub></i>	<i>LOI 1000° C</i>	<i>Total wt.%</i>
<i>October 2015</i>												
	<i>Mass weight %</i>											
1-A	41.40	0.27	5.02	1.85	0.04	1.89	22.81	0.44	1.13	0.27	22.61	97.73
2-A	25.98	0.26	5.46	2.15	0.05	2.64	31.85	0.33	1.12	0.19	29.80	99.84
3-A	30.50	0.29	5.62	2.05	0.05	2.47	29.48	0.42	1.21	0.19	27.57	99.84
<i>May 2016</i>												
4-B	49.38	0.34	5.50	3.06	0.04	2.64	17.70	0.32	1.30	0.22	20.30	100.81
5-B	21.41	0.23	5.00	2.10	0.06	2.65	36.31	0.11	0.98	0.19	32.74	101.78
6-B	21.20	0.23	4.97	2.06	0.06	2.62	36.57	0.11	0.98	0.18	32.75	101.73
7-B	21.15	0.23	4.99	2.03	0.06	2.60	36.22	0.16	0.98	0.18	32.25	100.85
8-B	51.74	0.30	5.52	1.84	0.03	1.75	19.46	0.38	1.31	0.16	18.78	101.27
9-B	51.45	0.21	3.26	1.30	0.02	1.44	22.65	0.13	0.91	0.14	19.57	101.08
10-B	33.58	0.28	5.21	2.01	0.05	2.41	28.77	0.34	1.17	0.18	27.92	101.92
11-B	24.73	0.23	4.51	2.42	0.04	2.10	32.39	0.10	0.89	0.39	31.89	99.69
12-B	66.88	0.29	5.75	1.43	0.02	1.53	10.85	0.65	1.40	0.17	11.16	100.13
13-B	65.72	0.28	5.88	1.76	0.03	1.67	11.45	0.72	1.26	0.16	11.03	99.96
<i>August 2016</i>												
4-C	44.57	0.32	5.89	2.99	0.05	2.65	20.63	0.36	1.32	0.27	22.72	101.76
6-C	20.92	0.24	4.91	2.02	0.06	2.69	36.87	0.15	0.97	0.20	33.22	102.25
12-C	22.13	0.26	5.22	2.17	0.06	2.84	35.63	0.14	1.03	0.19	32.51	102.18
13-C	39.71	0.27	4.97	1.86	0.04	1.88	25.08	0.32	1.11	0.32	24.70	100.26
14-C	70.98	0.31	5.91	1.76	0.03	1.64	8.26	0.79	1.25	0.15	8.15	99.23
15-C	63.47	0.30	4.92	1.38	0.03	1.51	12.60	0.45	1.28	0.17	13.38	99.49
<i>November 2016</i>												
2-D	43.45	0.31	5.64	2.95	0.04	2.61	20.13	0.30	1.28	0.25	22.76	99.72
3-D	20.54	0.24	4.88	2.00	0.06	2.65	36.61	0.13	0.97	0.19	33.32	101.59
4-D	26.06	0.27	5.60	2.24	0.06	2.69	33.20	0.23	1.14	0.21	30.69	102.38
6-D	29.60	0.31	5.60	2.08	0.05	2.53	31.26	0.32	1.20	0.19	28.98	102.12
11-D	22.09	0.14	3.27	2.63	0.04	1.89	33.83	0.07	0.64	0.43	36.43	101.46
12-D	43.31	0.28	5.22	1.86	0.04	1.98	23.11	0.36	1.19	0.29	23.16	100.80
15-D	62.92	0.32	5.97	1.86	0.03	1.77	12.41	0.65	1.31	0.19	12.78	100.21

Notes: Silica, calcium, and organic matter dominate the sediments. LOI, loss on ignition at 1000°C represents organic and carbonate matter; A=October 2015, B=May 2016, C=August 2016, D=November 2016

Table 2. Phosphorus concentrations in sediment samples (units in ppm). TP in sediment determined by x-ray fluorescence (XRF) and total digestion.

<i>Sample ID</i>	<i>TP in Sediment (XRF)</i>	<i>TP in Sediment (Total digestion)</i>
<i>October 2015</i>		
1-A	1183	1022
2-A	847	710
3-A	820	659
<i>May 2016</i>		
4-B	973	818
5-B	812	634
6-B	781	607
7-B	764	595
8-B	676	549
9-B	615	599
10-B	768	323
11-B	1711	1321
12-B	751	631
13-B	707	593
<i>August 2016</i>		
4-C	1165	925
6-C	851	656
12-C	842	644
13-C	1388	1090
14-C	659	566
15-C	755	614
<i>November 2016</i>		
2-D	1091	859
3-D	842	643
4-D	895	656
6-D	838	635
11-D	1894	1137
12-D	1253	1001
15-D	820	679

Notes: TP, Total phosphorus; A=October 2015, B=May 2016, C=August 2016, D=November 2016

Table 3. X-ray diffraction (XRD) results showing mass percentages of major mineral assemblages making up Utah Lake sediments.

Sample ID	Carbonates			Clays			Silica oxides	Feldspars			Other			
	Calcite	Aragonite	Dolomite	Kaolinite	Montmorillonite	Illite		Plagioclase		K-Feldspar	Goethite	Ankerite	Hornblende	Pyrite
								Albite	Anorthite	Microcline				
May 2016														
	Mass %													
4-B	32.0	0.2	3.1	3.2	0.3	6.7	44.3	0.4	0.3	4.5	2.3	2.7	0.1	-
5-B	61.3	3.2	2.7	4.8	0.6	10.6	8.0	2.2	1.2	2.5	0.7	0.3	1.9	-
6-B	69.9	-	0.2	1.6	1.3	7.2	10.5	-	3.4	2.0	0.7	1.8	-	1.4
7-B	57.8	4.0	2.2	1.5	0.8	13.1	8.1	1.3	2.1	6.2	2.1	0.6	-	0.2
8-B	23.2	12.1	5.0	1.1	0.7	11.3	29.1	12.8	0.9	1.6	0.2	0.5	0.7	0.7
9-B	31.4	4.4	0.4	1.0	0.1	5.4	42.4	7.1	0.4	3.9	1.3	1.9	0.4	-
10-B	44.4	0.1	1.1	2.0	1.0	12.3	17.4	6.9	10.2	1.0	-	2.8	0.1	0.8
11-B	55.7	4.7	2.1	2.4	0.4	8.9	14.9	4.8	2.7	1.4	-	0.2	-	1.7
12-B	14.9	3.2	0.2	0.8	0.3	4.9	45.9	21.0	-	4.3	1.2	1.2	2.2	-
13-B	14.2	12.5	6.4	1.3	0.5	6.7	42.8	11.4	0.6	1.6	0.1	-	0.9	1.0
August 2016														
4-C	26.2	1.1	0.9	2.5	1.0	8.4	25.6	25.5	0.2	0.1	1.5	3.3	0.6	3.2
6-C	60.8	1.1	1.4	2.0	0.7	3.1	8.4	10.6	5.4	3.1	0.8	2.0	-	0.7
12-C	37.8	1.1	1.3	1.1	0.6	-	32.7	21.3	0.5	-	2.1	0.2	-	1.3
13-C	8.9	0.9	3.5	0.7	0.3	0.5	49.7	31.5	1.0	0.5	1.4	0.1	-	1.0
14-C	59.0	3.3	-	1.7	1.7	8.2	7.4	6.4	8.3	0.1	0.4	2.2	-	1.4
15-C	18.1	0.6	1.9	-	0.2	0.8	46.2	12.6	16.2	1.5	0.6	0.5	0.1	0.8
November 2016														
2-D	52.3	7.5	-	6.3	0.4	13.0	10.4	4.3	-	2.5	0.2	2.4	0.6	0.1
3-D	41.8	16.4	0.8	2.7	0.6	12.9	11.2	4.8	0.7	5.3	0.7	1.6	-	0.3
4-D	25.5	18.6	1.6	5.4	0.3	7.5	26.0	6.8	0.4	2.6	1.0	3.4	0.5	0.4
6-D	65.0	3.5	-	2.2	1.2	6.4	9.7	6.4	1.0	1.1	1.1	1.5	-	0.9
11-D	55.1	2.9	0.4	2.4	0.2	7.8	14.0	11.2	-	2.0	-	0.5	2.4	1.1
12-D	41.3	-	2.0	2.9	0.5	0.9	34.0	8.1	3.0	4.2	0.8	0.2	1.4	0.4
15-D	16.0	-	5.8	2.0	0.4	2.8	42.1	25.3	0.1	0.9	2.5	0.1	1.2	0.9

Notes: A=October 2015, B=May 2016, C=August 2016, D=November 2016

Table 4. Analyses for major sediment samples.

<i>Sample Dates</i>	<i>XRF</i>	<i>XRD</i>	<i>Total Digestion</i>	<i>ESEM/ Helios</i>	<i>Sequential Extraction</i>	<i>Batch Sorption (High)</i>	<i>Batch Sorption (Low)</i>
October 2015 (n=3)	X	-	X	X	X	X	-
May 2016 (n=10)	X	X	X	X (1 of 10)	X	X (7 of 10)	-
August 2016 (n=6)	X	X	X	-	-	-	-
November 2016 (n=7)	X	X	X	-	-	-	X

Notes: XRF, X-ray fluorescence; XRD, X-ray diffraction; ESEM, Environmental scanning electron microscope; Helios, Helios scanning electron microscope



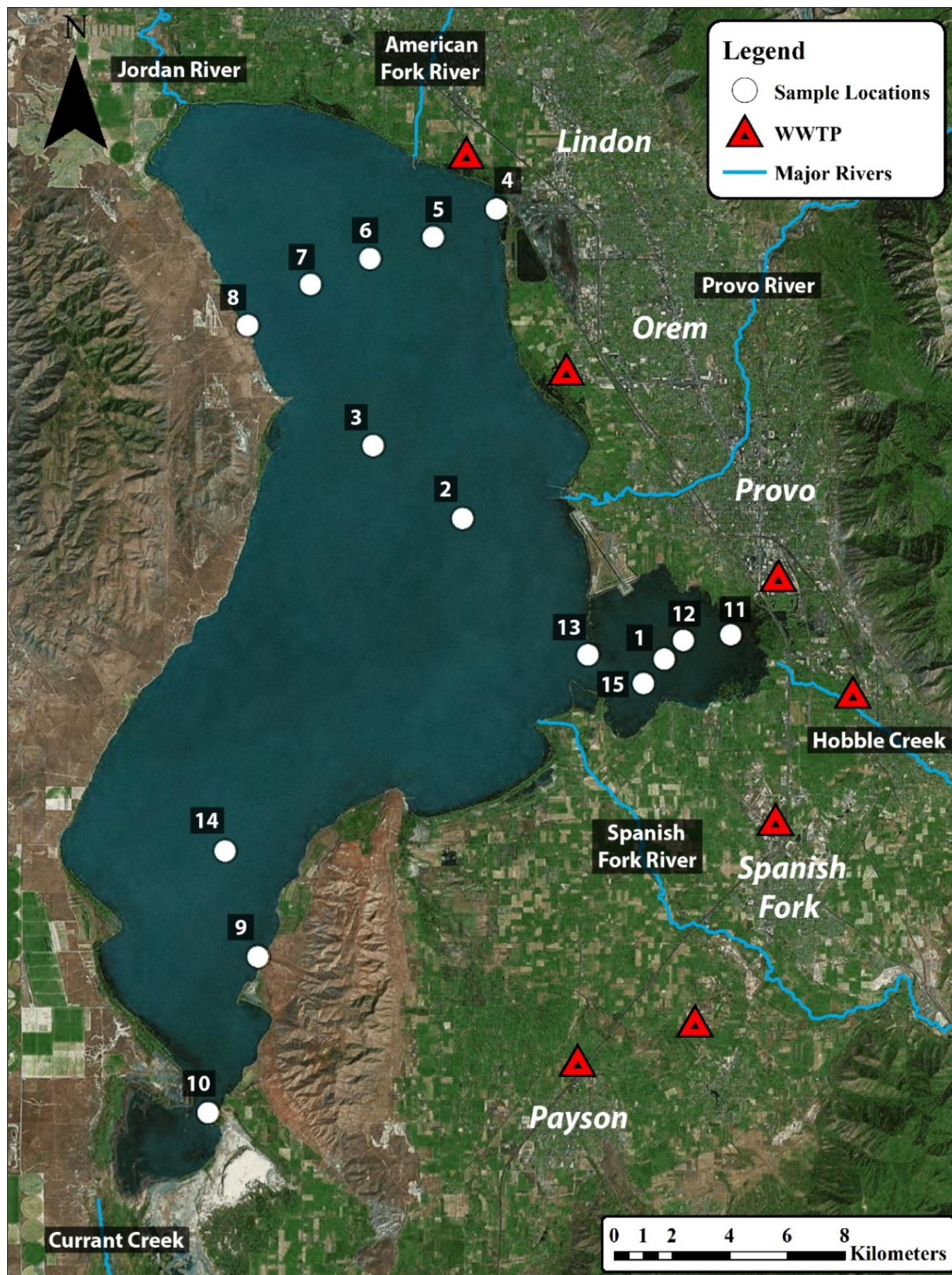


Figure 1. Location map of sampling sites across Utah Lake. Major tributaries and local wastewater treatment plants (WWTP) that discharge to the lake are labeled. The Jordan River is the only effluent from Utah Lake.

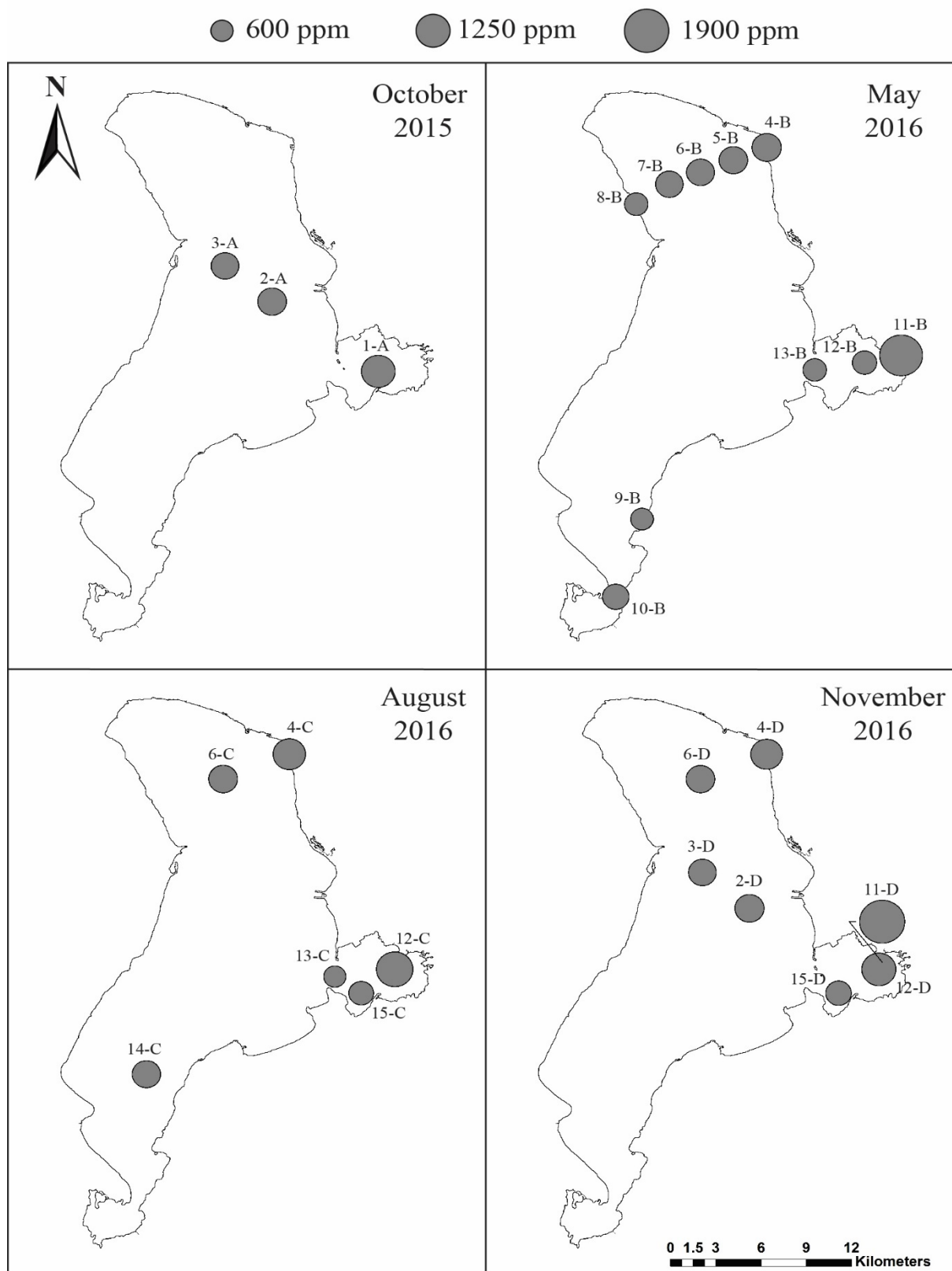


Figure 2. Total phosphorus (TP) concentrations in Utah Lake sediments measured by XRF on October 2015, May 2016, August 2016, and November 2016.

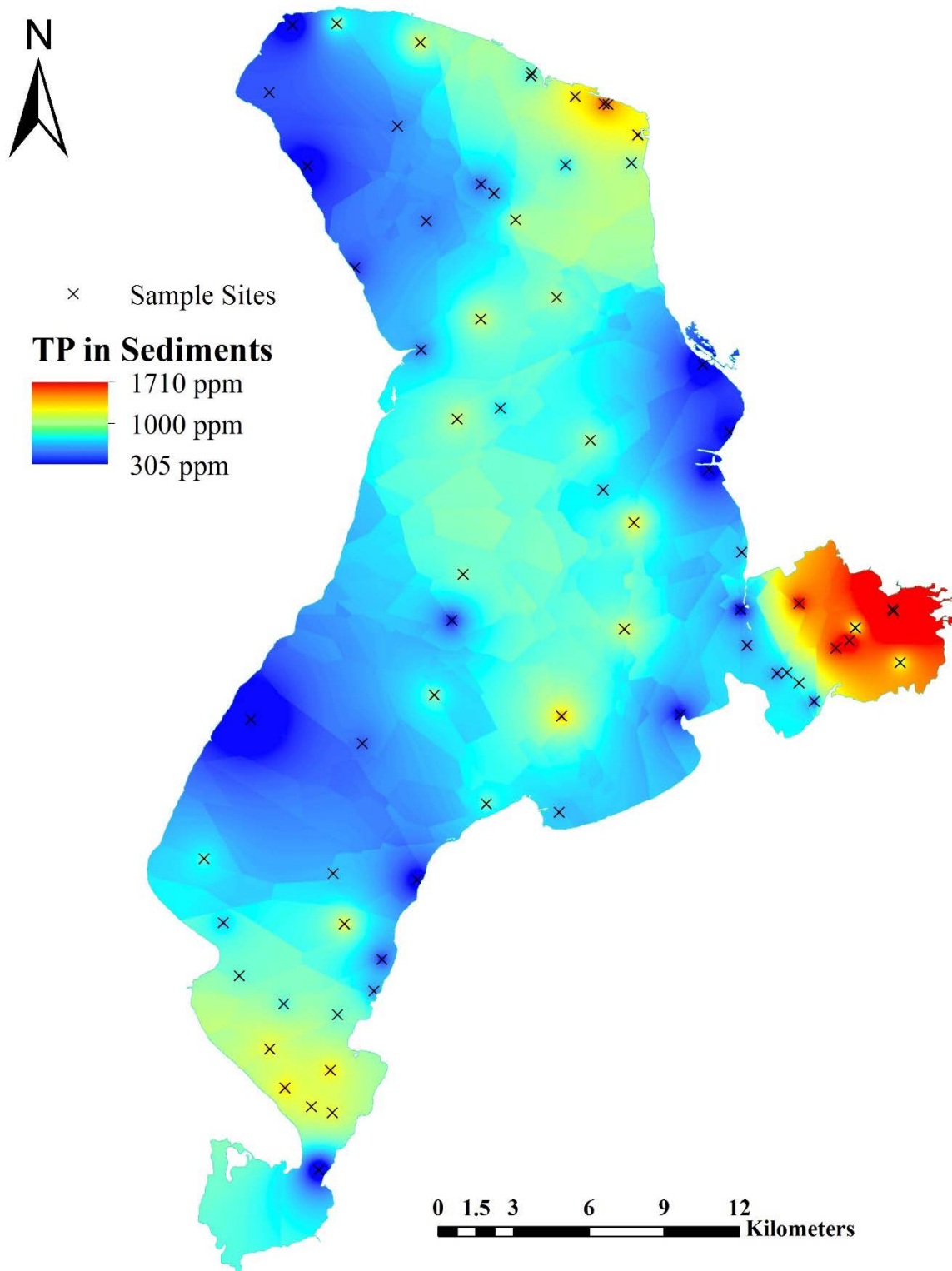


Figure 3. Interpolated surface map of total phosphorus (TP) concentrations in Utah Lake sediments, measured by total digestion from 81 samples collected from August 2015 through November 2016.



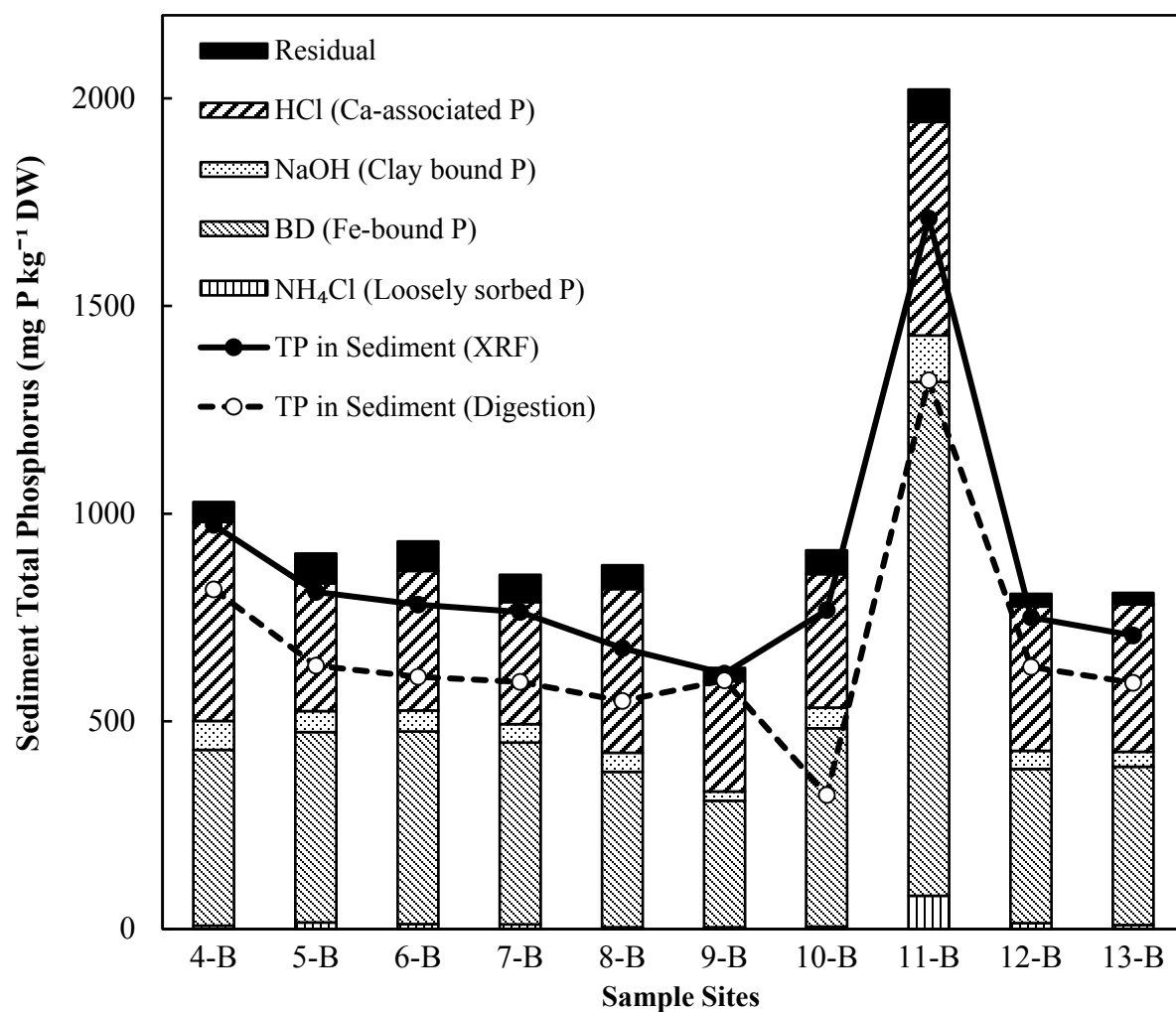


Figure 4. Sediment total phosphorus (TP) fractions from May 2016 Utah Lake sample sites, adjacent to comparable x-ray fluorescence and total digestion data.

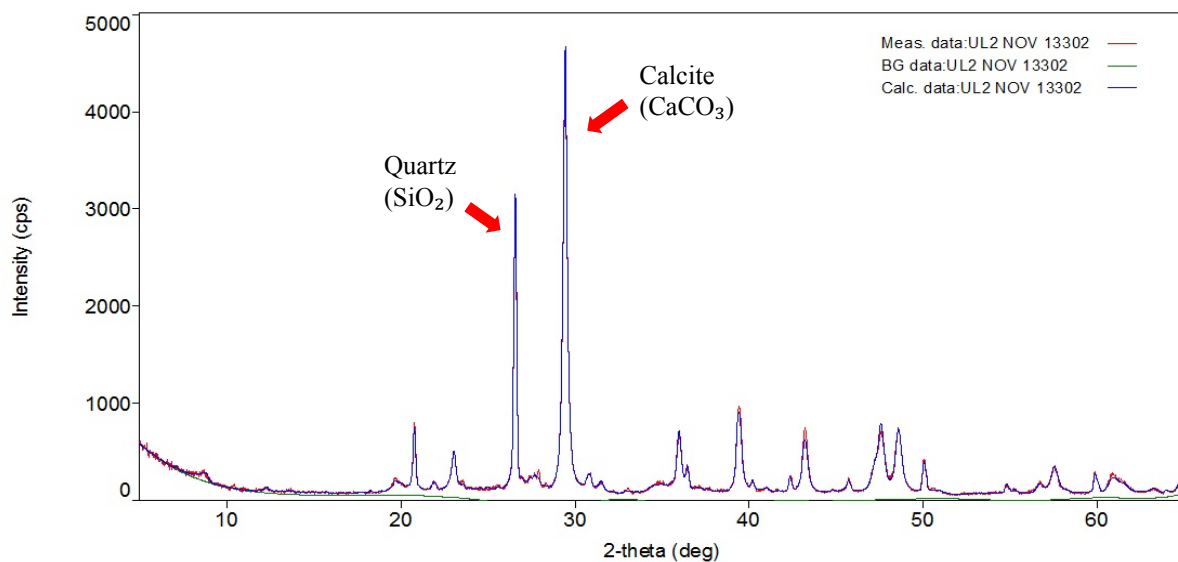


Figure 5. X-ray diffraction (XRD) graph of the top 5 cm of Utah Lake sediments from sample site 3-D taken November 2016. The height of the mineral peaks represents the abundance of minerals present in the sample.

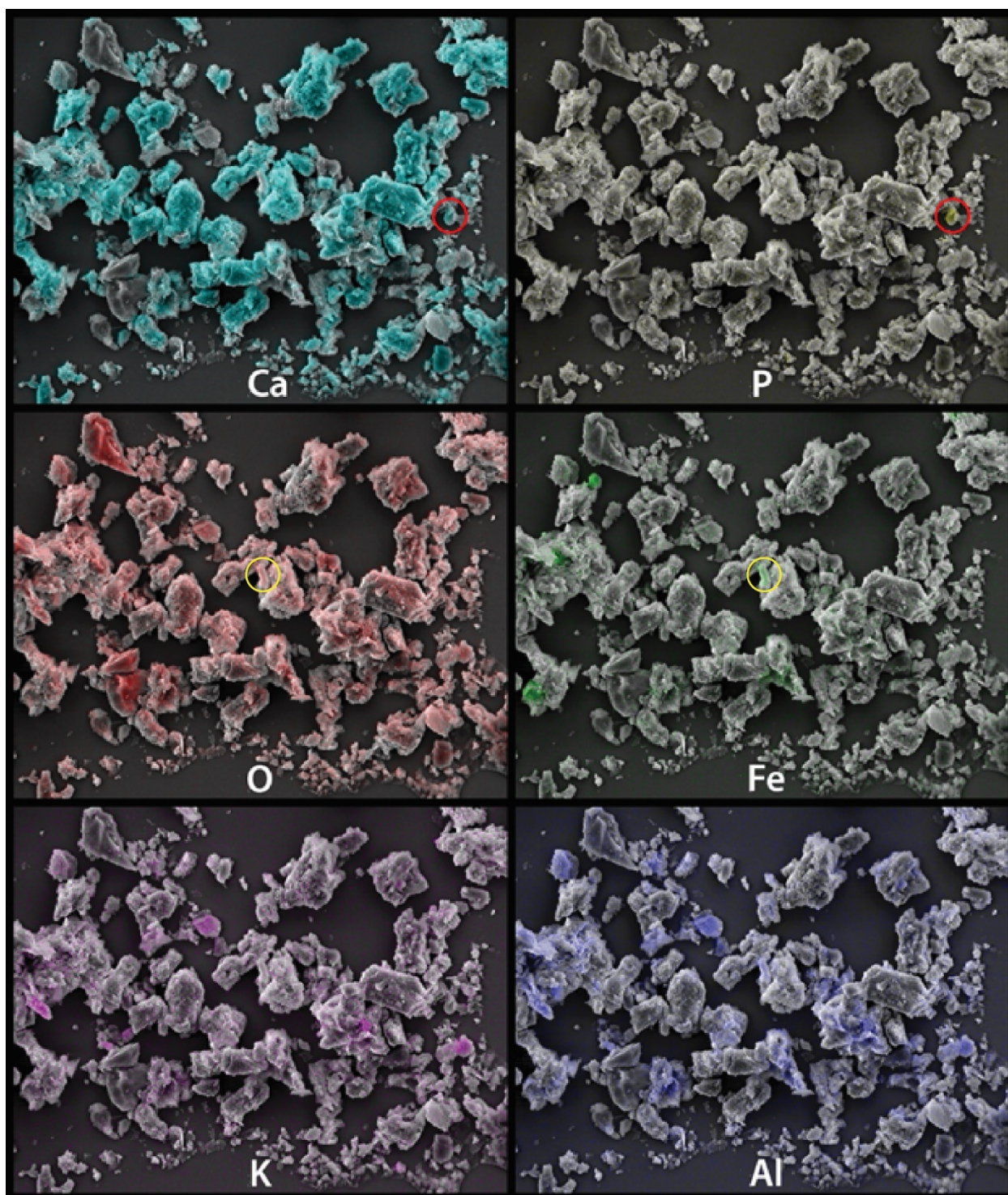


Figure 6. Backscattered electron image overlain with energy dispersive spectroscopy dot maps of Ca, P, O, Fe, K, and Al of sample 2-A collected October 2015. The colored portions on the dot maps indicate the presence of Ca, P, O, Fe, K, and Al. The red circle highlights a grain with both highly concentrated Ca-associated P and the yellow circle highlights a Fe-O grain.



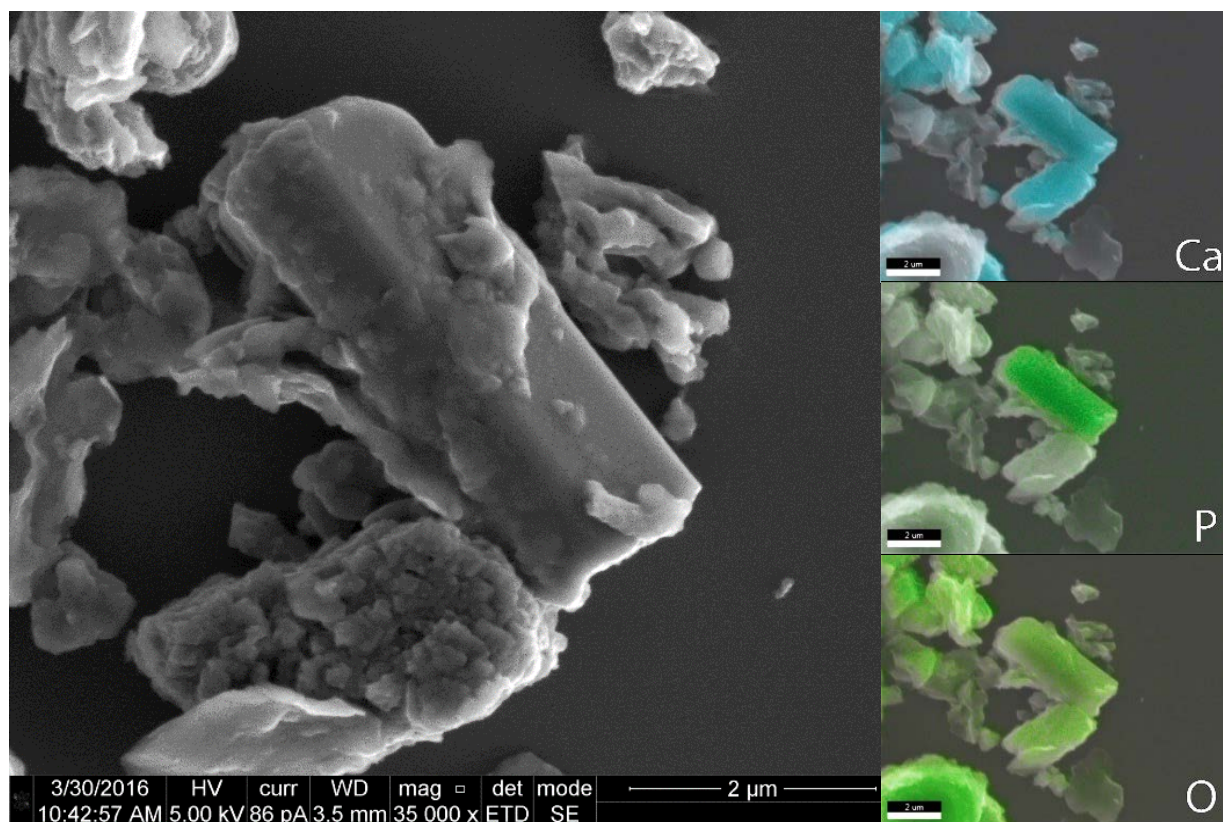


Figure 7. Backscattered electron image (left) and energy dispersive spectroscopy dot maps of Ca, P, and O (right) of an apatite crystal ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH},\text{F},\text{Cl})_2$ ) from sample site 1-A in Provo Bay collected October 2015. The colored portions on the dot maps indicate the presence of Ca, P, or O.

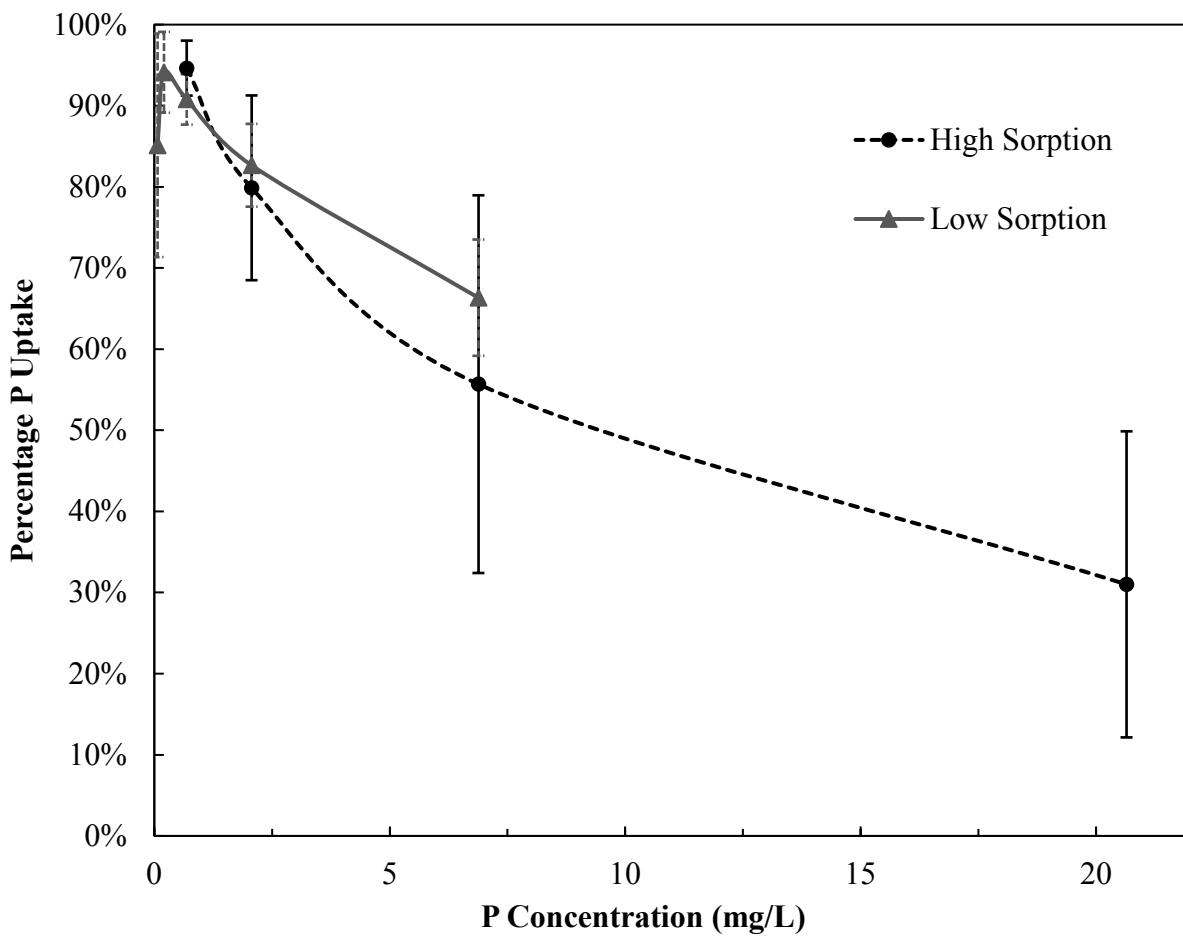


Figure 8. Total phosphorus (TP) sorption averages of Utah Lake sediments from samples (n=14) collected in May 2016 (high concentration sorption values) and November 2016 (low concentration sorption values). Error bars show mean and standard deviation.



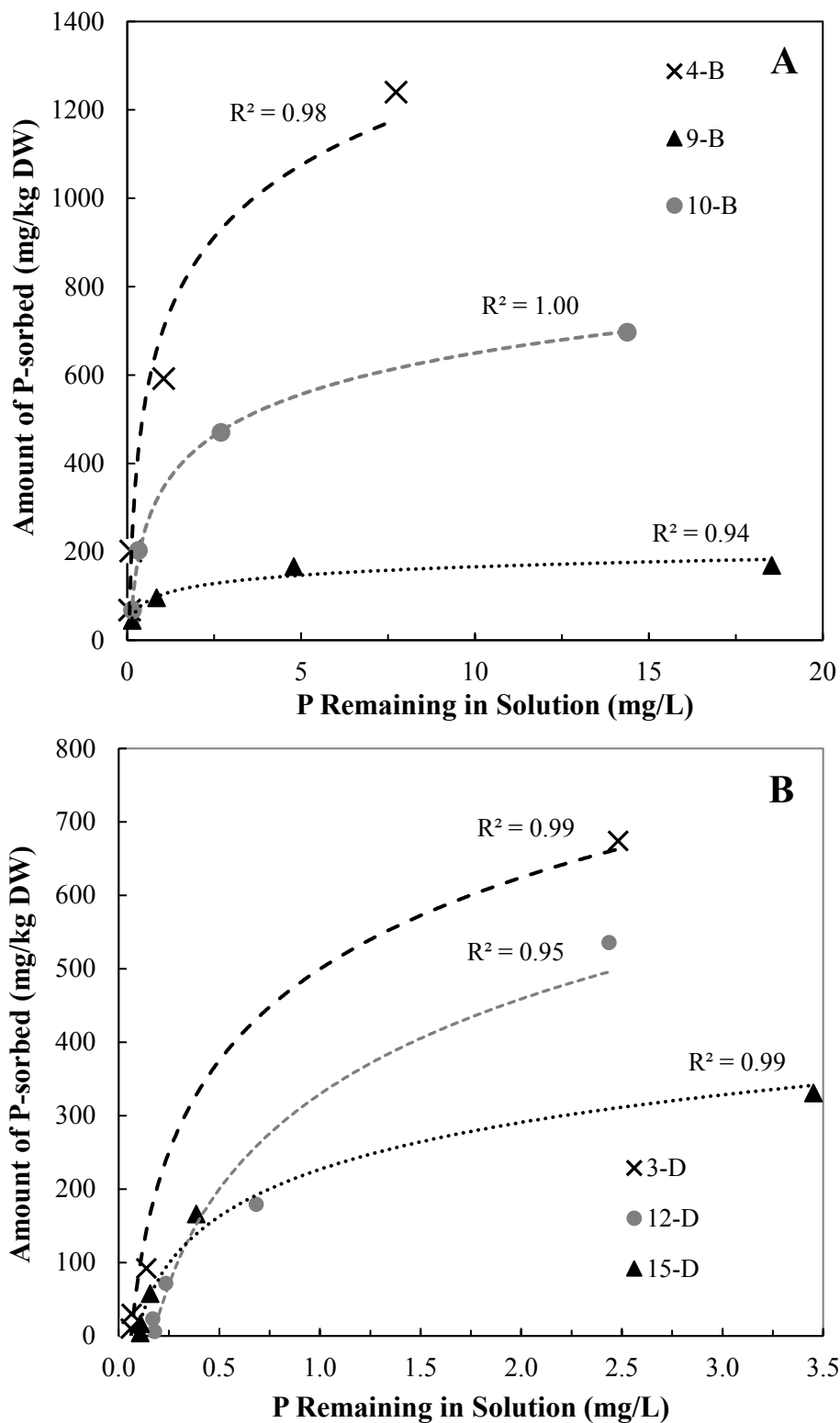


Figure 9. Representative total phosphorus (TP) sorption isotherms on different Utah Lake sediments. (A) Low sorption values of sediments collected November 2016 (B) High sorption values of sediments collected May 2016.

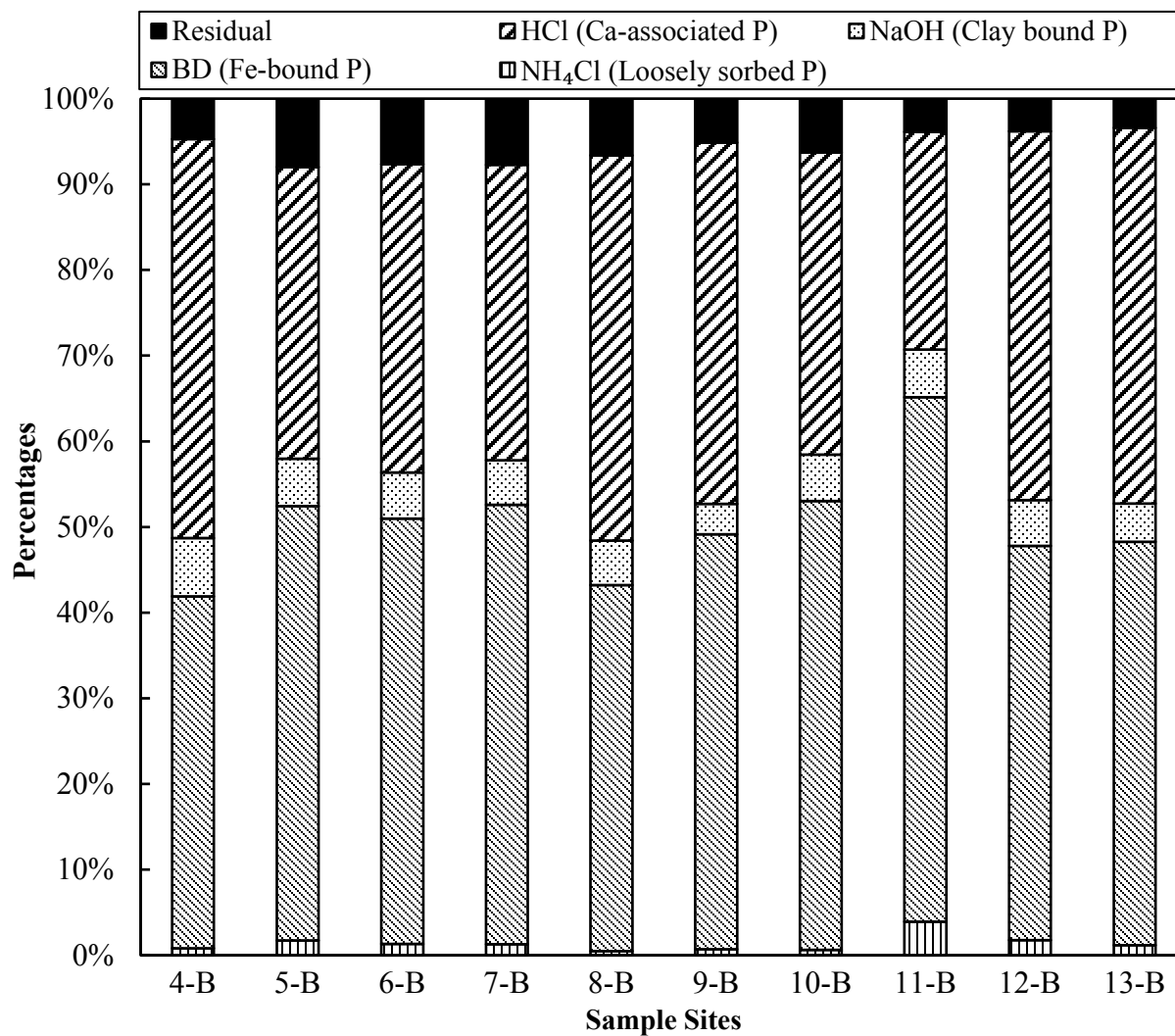


Figure 10. Percentage distribution of total phosphorus (TP) fractions in Utah Lake sediments (n=10) collected May 2016. Average values are shown for the ten samples.

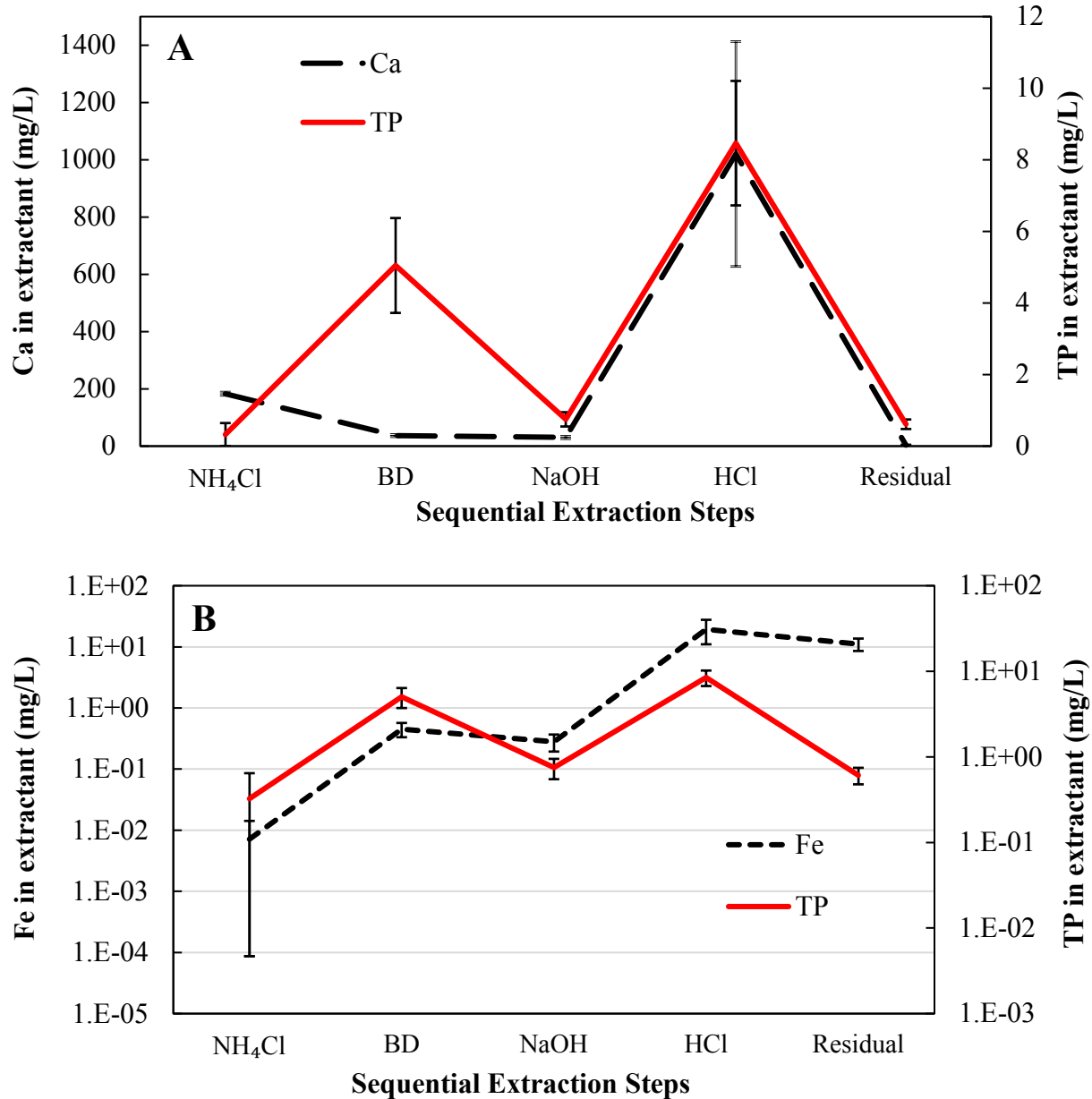


Figure 11. Total phosphorus (TP) release in sequential extraction (A) TP release associated with the release of Ca in the HCl extractant step, signifying Ca-associated P release. (B) TP release associated with the release of Fe in the BD extractant step, signifying Fe-bound P release from reducible iron (Fe(OOH)). The association of TP and Fe seen in the HCl step is the release of unreducible iron.

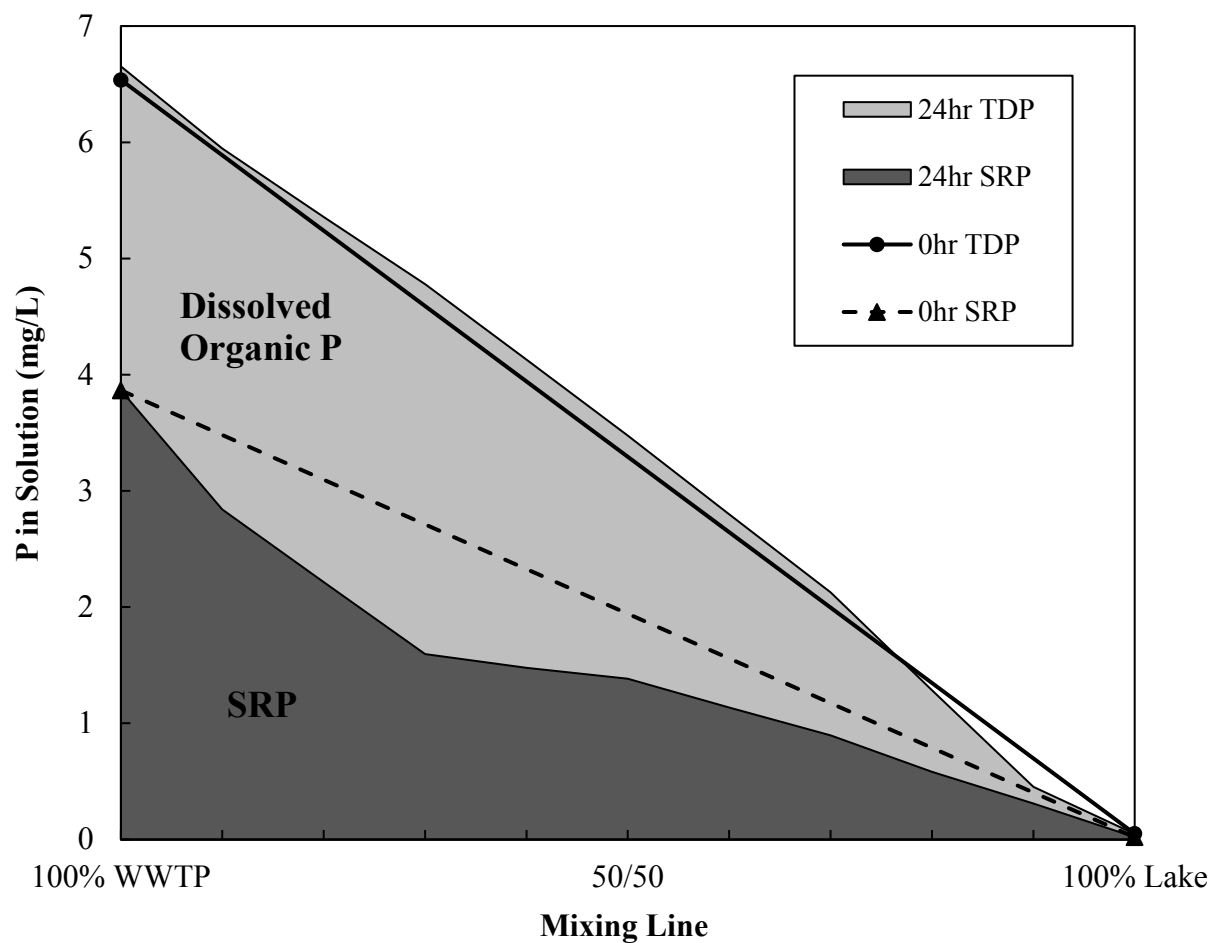


Figure 12. Mixing experiment done between Utah Lake water and Provo City wastewater (WWTP). Samples labeled 0hr represent pure Utah Lake and WWTP waters before mixing and samples labeled 24hr represent different mixtures of lake and WWTP waters shaken lightly for 24h and re-analyzed. A mixing line was then created with the known 0hr values; this mixing line shows that no substantial deviation occurred to TDP (Total dissolved P) after 24h, but there is a substantial deviation from SRP (Soluble reactive P) to dissolved organic P after 24h.

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## APPENDIX

### *Sequential Extraction Procedure*

*(Step 1 of extraction)* 2.5 g of wet sediment was measured and added to a 50mL polyurethane centrifuge tube. 25 mL of 1 M  $\text{NH}_4\text{Cl}$  (deoxygenated  $-\text{N}_2$  purged) was added to the centrifuge tube under anoxic conditions and shaken end to end on a (shaker table) for 0.5 h. The tube was then centrifuged for 10 min at 3000 rpms and the supernatant was collected and filtered through a 0.45-micron syringe filter into a separate 50 mL polyurethane centrifuge tube. Then the residue was rinsed with 25 mL of  $\text{NH}_4\text{Cl}$  and the centrifugation step was repeated. The supernatant was again collected and filtered, then added to the previous filtrate. The sample was then acidified with 1 mL 1M  $\text{HNO}_3$  and stored at  $4^\circ\text{C}$  until analysis for TP on ICP-OES.

*(Step 2)* to the sediment from the previous sample, 25 mL of 0.11 M BD (bicarbonate/dithionite-buffered to a pH 7 using  $\text{NaHCO}_3$ ) was added under anoxic conditions to extract Fe and Mn bound P. After shaking for 1 h the supernatant was collected and two BD washes and 1 M  $\text{NH}_4\text{Cl}$  were applied and supernatant was collected and filtered into a 125-mL polyurethane bottle and acidified with 8 mL 1M  $\text{HNO}_3$ , totaling to 108 mL collected. The sample was then stored at  $4^\circ\text{C}$  until analysis for TP on ICP-OES.

*(Step 3)* 25 mL of 1M  $\text{NaOH}$  was added to the previous sediment sample under aerobic conditions and shaken for 16 h. The supernatant was collected and filtered into a 125-mL polyurethane bottle, the sediment sample was then washed once with 1 M  $\text{NaOH}$  and once with 1 M  $\text{NH}_4\text{Cl}$  and supernatants were recovered and added to the 125-mL sample bottle. The sample was then acidified using 3mL 1 M  $\text{HNO}_3$ , totaling to 78 mL. Sample was stored at  $4^\circ\text{C}$  until analysis for TP on ICP-OES.

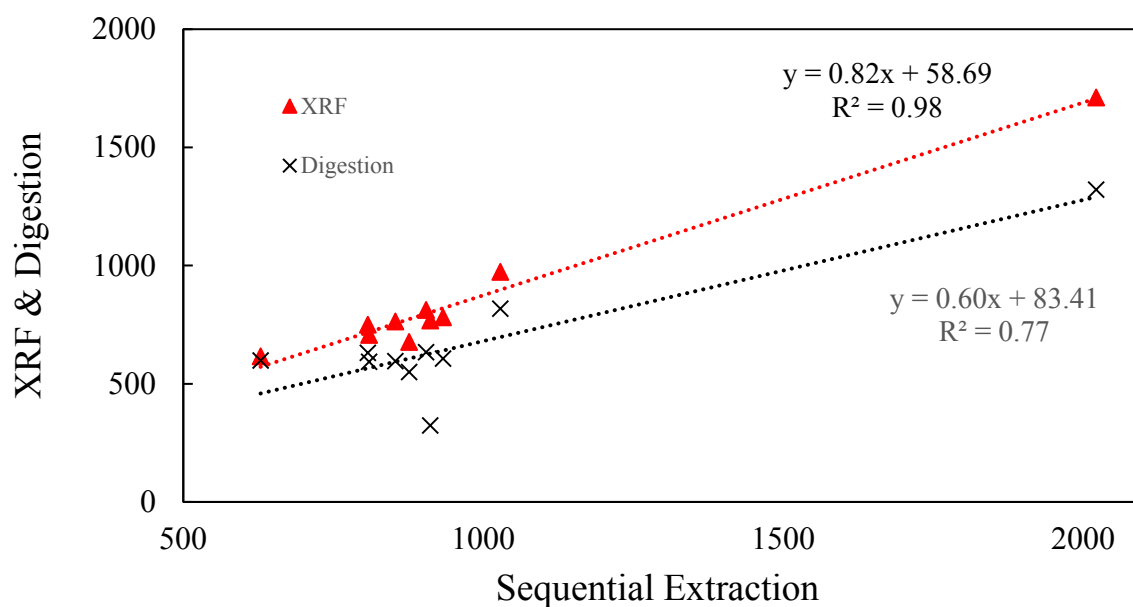


*(Step 4)* To the sediment from the previous sample, 25 mL of 0.5 HCl was added and shaken for 16 h. Supernatant was collected and a 1 M NH<sub>4</sub>Cl wash was applied and collected in a 50-mL centrifuge tube, totaling to 50 mL. Sample was then stored at 4°C until analysis for TP on ICP-OES.

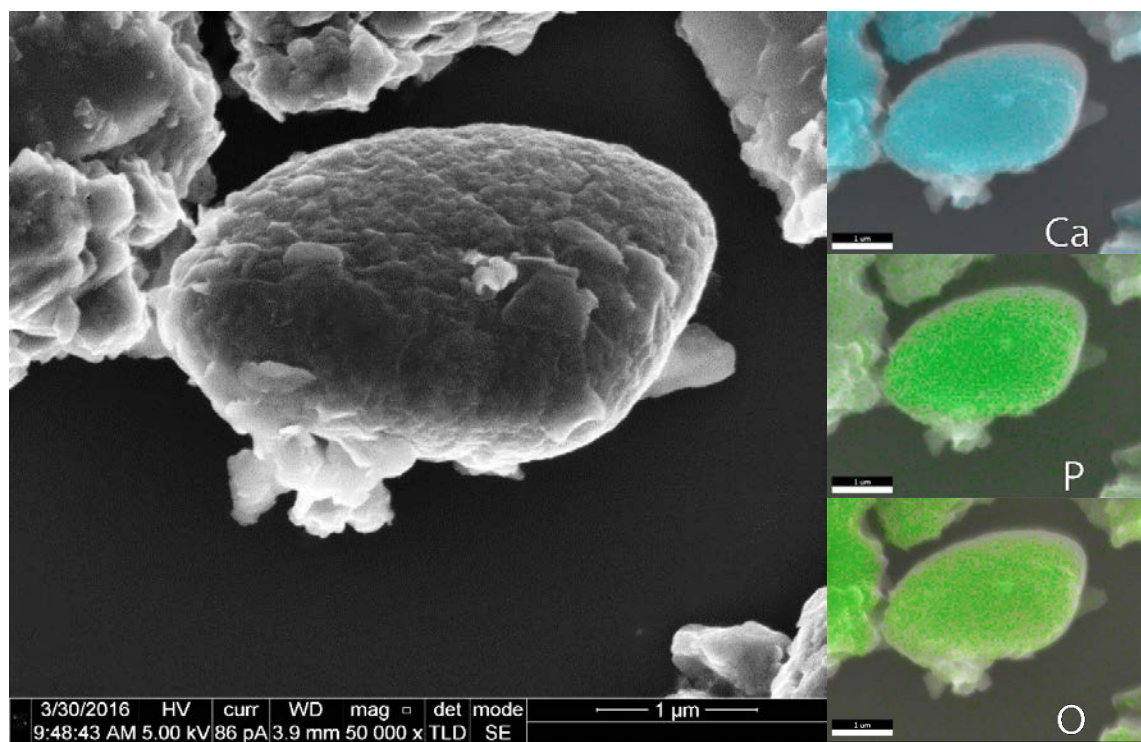
*(Step 5)* Sediment residue was dried at 105°C for 24 h and then ignited in a muffle furnace at 550°C for 2 h. To the remaining residue 25 mL of 1 M HCl was added and boiled for 10 min. Supernatant was collected and DI water was added until 100 mL was reached and sample was stored at 4°C until analysis for TP on ICP-OES.

After TP analysis on the ICP-OES, concentrations of TP were multiplied by the fraction of sample analyzed (51 mL, 108 mL, 78 mL, 50 mL, and 100 mL)

*Supplementary Figures*



Supplementary Figure 1. Statistical correlations of x-ray fluorescence (XRF) vs. sequential extraction and total digestion vs. sequential extraction for extractable TP from Utah Lake sediment samples collected in May 2015.



Supplementary Figure 2. Backscattered electron image (left) and energy dispersive spectroscopy dot maps of Ca, P, and O (right) of an amorphous apatite crystal.