Examination of sediment phosphorus fluxes and aluminum sulfate dosage considerations for Bone Lake, Wisconsin

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Objectives

Bone Lake, WI, is relatively large (1,780 ac), shallow (13 m), and polymictic during the summer. Temporary summer stratification coincides with the development of bottom water anoxia that can persist for greater than two months. Although hypolimnetic phosphorus (P) gradients have not been well documented (vertical P profile information is needed), surface water P and chlorophyll concentrations tend to increase in late August and September. Hypolimnetic anoxia, frequent summer water column mixing, and late summer peaks in epilimnetic P and chlorophyll all suggest that internal P loading from bottom sediments may be contributing to WQ impairment in Bone Lake. These investigations are designed to examine rates of phosphorus (P) release from sediment under aerobic and anaerobic conditions and mobile forms of sediment P in order to better understand the magnitude of internal P loading in the lake.

Approach

Laboratory-derived rates of P release from sediment under aerobic and anaerobic conditions

Three intact sediment cores were collected from stations located in the deepest portion of the north, central, and south lake basins for the determination of rates of P release from sediment under controlled laboratory conditions (Fig. 1). Cores were carefully drained of overlying water in the laboratory and the upper 10 cm of sediment was transferred intact to a smaller acrylic core liner (6.5-cm dia and 20-cm ht) using a core remover tool. Surface water collected from each lake was filtered through a glass fiber filter (Gelman A-E), with 300 mL then siphoned onto the sediment contained in the small acrylic core liner without causing sediment resuspension. They were placed in a darkened environmental chamber and incubated at a constant temperature of ~20 ^{o}C to reflect summer conditions. The oxidation-reduction environment in the overlying water was controlled by gently bubbling air (aerobic) or nitrogen (anaerobic) through an air stone placed just above the sediment surface in each system. Bubbling action insured complete mixing of the water column but did not disrupt the sediment.

Table 1. Sediment core collection and analyses.

	P release rates		Sediment chemistry	
Station	Aerobic	Anaerobic	Surface only	Vertical profile
1			1	
2	1	2		1
3			1	
4	1	2		1
5			1	
6	1	2		1
7			1	

Water samples for soluble reactive P were collected from the center of each system using an acid-washed syringe and filtered through a 0.45 µm membrane syringe filter. The water volume removed from each system during sampling was replaced by addition of filtered lake water preadjusted to the proper oxidation-reduction condition. These volumes were accurately measured for determination of dilution effects. Soluble reactive P was measured colorimetrically using the ascorbic acid method (APHA 2005). Rates of P release from the sediment (mg/m^2 d) were calculated as the linear change in mass in the overlying water divided by time (days) and the area (m^2) of the incubation core liner. Regression analysis was used to estimate rates over the linear portion of the data. Anaerobic P release rates were determined for duplicate cores while aerobic P release rates were estimated from one core collected at each station (Table 1).

Sediment chemistry

The objectives of this task were to quantify sediment P fractions that are contributing to internal P loading in Bone Lake. For sediments containing iron (Fe) compounds, P is usually coupled with Fe dynamics and flux to the water column is regulated by oxidation-reduction (i.e., eH) and pH reactions (Mortimer 1971). A thin oxidized microzone exists in the sediment surface layer when the overlying water column is oxygenated. Under these conditions, Fe is in an oxidized state (i.e., Fe^{+3}) in the microzone as Fe~(OOH) and adsorbs P, thereby controlling its diffusion into the overlying water column. Elevated pH and production of hydroxyl ions (OH^-) during periods of intense photosynthesis can also indirectly enhance rates of P release from sediment under aerobic conditions via ligand exchange (i.e., competition for binding sites by OH^-). Under anoxic conditions at the sediment-water interface, anaerobic bacterial reduction of iron from Fe^{3+} to Fe^{2+} results in P desorption and diffusion into the water column for potential uptake by algae. Internal P loading via these recycling pathways can account for a substantial portion of the P economy of aquatic systems and hinder restoration efforts targeted at reducing algal biomass.

P bound to Fe, or redox-sensitive P, can be quantified via extraction with a strong reducing agent (dithionite-bicarbonate; BD; Nürnberg 1988). Additionally, biologically-labile P in the form of bacterial polyphosphates and labile organic P compounds and can be recycled to the overlying water column via mineralization and metabolic breakdown and is extracted with a basic solution (1.0 N NaOH; Psenner and Puckso 1988). Thus, the size of the biologically-labile P pool (i.e., redox-P and labile organic P; subject to recycling and internal P loading) in surface sediment can be quantified for evaluation as a source of P recycling in Bone Lake.

Table 2. Textural-physical variables and biologically-labile sediment phosphorus pools.

Moisture content (%)
Sediment wet and dry bulk density (g/cm^3)
Organic matter content (%)
Loosely-bound P (mg/g)
Iron-bound P (mg/g)
Labile organic P (mg/g)
Aluminum-bound P (mg/g)

Sediment physical-textural characteristics and phosphorus concentrations were examined for the upper 5-cm section of sediment core collected from station 1, 3, 5, and 5 (Table 1). Intact sediment cores collected from station 2, 4, and 6 were sectioned at 1-

to 2.5-cm intervals over the upper 15 cm (Table 1). Sediment sections were analyzed for the variables listed in Table 2. The loosely-bound and iron-bound P fractions are readily mobilized at the sediment-water interface as a result of anaerobic conditions that result in desorption of P from sediment and diffusion into the overlying water column (Mortimer 1971, Boström 1984, Nürnberg 1988). The sum of the loosely-bound and iron-bound P fractions are referred to as redox-sensitive P (i.e., the P fraction that is active in P release under anaerobic and reducing conditions). In addition, labile organic P can be converted to soluble P via bacterial mineralization (Jensen and Andersen 1992) or hydrolysis of bacterial polyphosphates to soluble phosphate under anaerobic conditions (Gächter et al. 1988; Gächter and Meyer 1993; Hupfer et al. 1995). The sum of redox-sensitive P and labile organic P are collectively referred to a biologically-labile P or mobile P. This fraction is generally active in recycling pathways that result in exchanges of phosphate from the sediment to the overlying water column and potential assimilation by algae.

Subsamples were dried at 105 °C to a constant weight and burned at 500 ^{o}C for determination of moisture content, sediment density, and organic matter content (Håkanson 1977). Phosphorus fractionation were conducted according to Hieltjes and Lijklema (1980), Psenner and Puckso (1988), and Nürnberg (1988) for the determination of ammonium-chloride-extractable P (1 M NH_4Cl; loosely-bound P), bicarbonate-dithionite-extractable P (0.11 M BD; iron-bound P), and sodium hydroxide-extractable P (1 N NaOH; aluminum-bound P). A subsample of the sodium hydroxide extract was digested with potassium persulfate to determine nonreactive sodium hydroxide-extractable P (Psenner and Puckso 1988). Labile organic P was calculated as the difference between reactive and nonreactive sodium hydroxide-extractable P.

The thickness of the upper sediment layer that is potentially active in diffusive P flux was determined by evaluating vertical P profiles according to Carey and Rydin (2011). Typically, sediment P concentrations are elevated in the upper layer versus deeper layers of eutrophic lake sediments due to accumulation of sediment P that is recycled in excess of burial and diagenesis. It will be critical to define and treat this layer at a minimum with alum in order to control P release from sediment under anaerobic conditions.

Alum dosage determination

Vertical profiles of sediment redox and biologically-labile P were evaluated and mean integrated concentrations were estimated over the upper 5 to 10 cm layer. The aluminum sulfate dosage (expressed as Al) required to bind sediment P in the upper layer was determined as described in James and Bischoff (2015). In general, the dry mass concentration of redox-P (mg/g) was converted to an area-weighted concentration (g/m^2) as,

$$\text{Redox-P (g/m}^2\text{)} = \text{Redox-P (mg/g)} \cdot \rho \text{ (g/cm}^3\text{)} \cdot \theta \cdot h \text{ (m)} \cdot 1{,}000{,}000 \text{ (cm}^3\text{/m}^3\text{)} \cdot 0.001 \text{ (g/mg)} \qquad 1),$$

where, ρ is sediment bulk density (g/cm^3), θ is the percentage of sediment solids (100 – percent moisture content; dimensionless), and h is sediment thickness (m). A stoichiometric Al:P binding ratio (i.e., the mass of Al required to bind redox-P) was estimated from regression equations between redox-P and the Al:P binding ratio developed in James and Bischoff (2015). The Al concentration (g/m^2) required to bind P in the upper sediment layer was estimated as,

$$\text{Al (g/m}^2\text{)} = \text{Redox-P (g/m}^2\text{)} \cdot \text{Al:P} \qquad 2).$$

The Al dosage calculated in this manner does not take into account decreased binding efficiency of the Al floc over time. Berkowitz et al. (2006) and De Vicente et al. (2008) found that Al binding efficiency can decrease by > 50% within months in the absence of P adsorption due to crystallization and polymerization. Since the Al floc usually layers on top of the original sediment surface rather than instantaneously mixing or sinking into the sediment, P sequestration proceeds as a result of slower upward diffusion. This scenario leads to a decrease in binding efficiency over time and a potential need for future (and lower dose) maintenance Al applications (see below).

Maximum allowable Al dosage based on alkalinity and pH

Addition of aluminum sulfate to a lake leads to hydrolysis and the liberation of hydrogen ions which lowers the pH of the water column. Since Al toxicity to the biota can occur if the pH falls below ~4, maintaining a pH ≥ 6.0 as a margin of safety should also be considered in dose determination (Cooke et al. 2005). For situations where alkalinity is low or the required dosage exceeds the maximum allowable dosage to maintain pH ≥ 6.0, a buffered aluminum sulfate-sodium aluminate treatment will be needed to maintain pH near neutrality. Surface water collected from each lake was analyzed for total alkalinity and pH according to APHA (2005). A titration procedure was used to determine the maximum allowable dosage of aluminum sulfate that can be added and yet maintain pH above 6.0 (Cooke et al. 2005). A 1.25 g Al/L solution of $Al_2(SO_4)_3 \cdot 18\ H_2O$ was used as the titrant: 1.0 mL additions to 500 mL of lake water were each equivalent to 2.5 mg Al/L. Lake water was titrated with the Al solution until an endpoint of pH 6 was reached. The total volume (mL) of Al solution needed to titrate lake water to pH 6 was multiplied by 2.5 mg Al/L to estimate the maximum allowable concentration. This calculation was then compared with estimates based on sediment redox-P to ensure that the latter was at or below the maximum allowable dosage. Caution needs to be used because a vertical alkalinity and pH profile over the entire vertical water column needs to be estimated in order to more accurately evaluate the maximum allowable dosage.

RESULTS AND INTERPRETATION

Diffusive P flux from sediment

Under anaerobic conditions, P mass and concentration increased rapidly and approximately linearly in the overlying water column of station 2, 4, and 6 sediment incubation systems (Fig. 2). In contrast, P mass and concentration increases in the overlying water column were not detected in sediment cores from these stations that were incubated under aerobic conditions (not shown). Overall, mean anaerobic diffusive P fluxes were moderate to high, ranging between 3.7 and 10.2 mg/m^2 d (Table 3 and Fig. 3). Compared to other lakes in the region, mean anaerobic diffusive P fluxes measured at station 2 fell above the upper 25% quartile while sediment P fluxes at station 4 and 6 fell near the median (Fig. 4).

Table 3. Mean rates of phosphorus (P) release under anaerobic conditions (n = 2) for intact sediment cores collected from various stations in Bone Lake.

Station	Anaerobic diffusive P flux	
	(mg/m^2 d)	(SE)
2	10.21	0.80
4	3.69	1.03
6	5.01	0.41

Sediment characteristics

Bone Lake sediment exhibited very high moisture content (> 94%), high organic matter content (> 30%), and low wet and dry bulk density, primarily in the upper 5-cm layer (Table 4, Fig. 5 and 6). Physical-textural characteristics in the upper 5-cm sediment layer generally fell outside the upper and lower quartiles compared to other lakes in the region, indicating very flocculent sediment with high porosity (i.e., interstitial spaces for porewater, Fig. 7). Newly precipitated Al floc is usually very fluid with low density during the first year of application. Thus, exposure to and binding of redox-P in the upper sediment layer will depend on density differences between the Al floc and surface sediment as briefly introduced in the Methods section.

Table 4. Textural characteristics in the upper sediment layer for sediment cores collected in Bone Lake.

Station	Moisture Content (%)	Wet Bulk Density (g/cm^3)	Dry Bulk Density (g/cm^3)	Organic Matter (%)
1	95.5	1.019	0.046	32.4
2	94.9	1.022	0.053	32.4
3	94.4	1.024	0.058	31.0
4	95.0	1.020	0.051	35.1
5	94.3	1.024	0.059	33.2
6	95.2	1.020	0.050	32.9
7	95.1	1.022	0.051	29.4

Biologically-labile P concentrations in the upper 5-cm layer were relatively high (Table 5, Fig. 8 and 9). Labile organic P dominated this mobile P pool and concentrations were high relative to other lakes in the region (Fig. 10). Iron-bound P, which is closely correlated with anaerobic diffusive P flux (Nürnberg 1988), was also relatively high (Fig. 8 and 9) and fell near the median versus other lakes in the region (Fig. 10).

Vertically in the sediment column, loosely-bound P concentrations were relatively

Table 5. Concentrations of biologically labile and refractory P in the upper 5-cm sediment layer for sediment cores collected in Bone Lake. DW = dry mass, Redox-P = sum of loosely-bound P and iron-bound P, Bio-labile P = sum of redox-P and labile organic P.

Station	Redox-sensitive and biologically labile P					Refractory P
	Loosely-bound P (mg/g DW)	Iron-bound P (mg/g DW)	Labile organic P (mg/g DW)	Redox-P (mg/g DW)	Bio-labile P (mg/g DW)	Aluminum-bound P (mg/g DW)
1	0.012	0.237	0.514	0.249	0.763	0.152
2	0.011	0.269	0.685	0.280	0.965	0.116
3	0.005	0.150	0.448	0.155	0.603	0.075
4	0.021	0.264	0.619	0.285	0.904	0.082
5	0.029	0.334	0.439	0.363	0.802	0.068
6	0.019	0.269	0.659	0.288	0.947	0.120
7	0.025	0.399	0.403	0.424	0.827	0.074

homogeneous with sediment depth and accounted for a minor portion of the biologically-labile P in Bone Lake (Fig. 11). Modest surface peaks in iron-bound P were observed in the upper 8- to 10-cm layer at stations 2, 4, and 6. Other research has linked surface concentration peaks in iron-bound P and total P to internal P loading in eutrophic aquatic systems (Carey and Rydin 2011, Rydin et al. 2012). Concentrations of iron-bound P declined below the 10 cm at these stations and were relatively constant, indicative of buried iron-bound P that was not active in internal P loading. Labile organic P also exhibited modest concentration maxima in the upper 8-cm sediment layer (Fig. 11). As a result, both redox-P and biologically-labile P concentrations were maximal in the upper 8 cm sediment layer (Fig. 11). Aluminum-bound P, relatively inert to recycling pathways and internal P loading, was relatively low in concentration and constant with sediment depth at each Bone Lake station (Fig. 11). The aluminum-bound P concentration in the upper 5-cm layer was also moderate and fell near or below the median concentration compared to other lakes in the region (Fig. 11). Since the aluminum-bound P fraction also reflects P that is bound to added alum, these pre-treatment vertical profiles will be used to evaluate future sediment P binding efficiency and effectiveness of the precipitated Al floc, if an alum treatment is conducted on Bone Lake in the future.

Aluminum sulfate dosage and cost

Al dosage and sediment thicknesses for stations 2, 4, and 6 is shown in Table 6. Al dosage increased from ~ 60 g/m^2 to greater than 90 g/m^2 at these stations, depending on the mass of mobile P targeted for inactivation. For instance, a mean 93 g/m^2 would be required to theoretically inactivate mobile P in the upper 10 cm sediment layer of stations 2, 4, and 6 (Table 6). A similar mean Al dose was estimated when all stations in Bone Lake were compared (Fig. 12).

Recent Al dosages have ranged between ~94 and 150 g/m^2 (Table 7). A target dosage of ~ 90 to 100 g/m^2 split over several years is recommended with sediment monitoring to evaluate future P binding efficiency and internal P loading control by the alum floc (see below for further description). Because moisture content was very high while sediment bulk density was low, the Al floc is likely to contact a greater portion of the sediment column in Bone Lake sediments.

Table 6. The redox-sensitive phosphorus (P) concentration integrated over the 4-, 6-, 8-, and 10-cm sediment thickness and Al dosage required to treat various sediment thicknesses in Bone Lake.

	Station 2		Station 4		Station 6		Mean
Sediment thickness (cm)	Redox-sensitive P (mg/g)	Alum Dosage (g/m^2)	Redox-sensitive P (mg/g)	Alum Dosage (g/m^2)	Redox-sensitive P (mg/g)	Alum Dosage (g/m^2)	Alum Dosage (g/m^2)
6	0.264	60	0.280	58	0.280	57	**58**
8	0.231	80	0.233	75	0.234	74	**76**
10	0.204	97	0.201	92	0.196	90	**93**

Schieffer (2009, 2017) reported that the depth of anoxia and the potential for anaerobic diffusive P flux in Bone Lake ranged between ~ 8 and 11 m from the surface during the summer, suggesting that the Al application area should at least encompass the 30-ft depth contour. Cost scenarios for various Al dosages to this area are shown in Table 8. Application costs ranged between ~ $957,000 and ~ $1,436,000 for a 60 g/m^2 to 90 g/m^2 Al dose, respectively.

Total alkalinity in Bone Lake at the time of sediment core sampling (5 July, 2017) was moderate at ~102 mg $CaCO_3$/L, suggesting moderate buffering capacity for regulating pH during alum application. Al binding of P is most efficient within a pH range of 6 to 8. As pH declines below 6, Al becomes increasingly soluble (as Al^{3+}) and toxic to biota. The maximum allowable Al dosage that could be applied and yet maintain pH at or above 6, determined via jar tests (Cooke et al. 2005), was moderate at 13.75 mg Al/L (Table 9). Al dosage up to 100 g/m^2 would fall below the maximum allowable dosage, indicating that aluminum sulfate could be safely applied without decreasing the pH to < 6.

Table 7. Recent alum (as Al) dosages for various lakes.

Lake	Al Dose (g Al/m^2)	Reference
Bone Lake, WI	90-100	Present study
Cedar Lake, WI	100-130	(James unpubl. data)
Lake Riley, MN	100	(James unpubl. data)
Bald Eagle, MN	100	(James unpubl. data)
Black Hawk, MN	145	(James unpubl. data)
Tiefwarensee, Germany	137	Wauer et al. (2009)
East Alaska, WI	132	Hoyman (2012)
Half Moon, WI	75-150	James (2011)
Susser See, Germany	100	Lewandowski et al. (2003)
Green, WA	94	Dugopolski et al. (2008)

Other research (Berkowitz et al. 2005, 2006; de Vicente et al 2008a, 2008b; James 2017) has suggested development of an adaptive management approach of applying Al concentrations spread out over a period of years (i.e., 1-3 year intervals) and monitoring lake response for future Al maintenance applications.

Table 8. Cost scenarios for treating the 30-ft contour with aluminum sulfate. Estimates assumed a current cost of $1.76 per gallon of aluminum sulfate.

Al dose (g/m^2)	Gallons	Cost ($)
25	257,202	$452,676
50	514,405	$797,327
60	617,286	$956,796
70	720,167	$1,267,493
80	823,048	$1,275,724
90	925,929	$1,435,189
100	1,028,810	$1,810,705

Table 9. A comparison of the maximum allowable Al dose, based on a titration assay and nomograph estimate presented in (Cooke et al. 2005) and the the areal sediment redox-P based Al dosage converted to a concentration. Al dosages and longevity for other unstratified and stratified lakes are from Cooke et al (2005). Numbers on parentheses denote percent reductions in lake total phosphorus. Longevity = as of publication of Cooke et al. (2005).

Lake		Al Dose ($g\ Al/m^3$)	Observed Longevity (years)
Bone Lake	Maximum allowable	13.75	
	Actual	**<10**	
Unstratified lakes	Long Kitsap County	5.5	11(30%)
	Pickerel	7.3	<1
	Long Thurston County North	7.7	>8 (56%)
	Pattison North	7.7	7 (29%)
	Wapato	7.8	<1
	Erie	10.9	>8 (75%)
	Campbell	10.9	>8 (46%)
Stratified lakes	Eau Galle	4.5	<2
	Morey	11.7	8 (60%)
	Cochnewagon	18	6 (not reported)
	Dollar	20.9	18 (68%)
	Annabessacook	25	13 (41%)
	West Twin	26	18 (66%)
	Irondoquoit Bay	28.7	5 (24%)
	Kezar	30	9 (37%)

The goal of these approaches is to increase overall P binding efficiency and internal P loading control longevity by stabilizing $Al(OH)_3$ polymerization and enhancing P saturation of binding sites. Application of multiple Al concentrations spread out over a period of years may be more effective in saturating binding sites, lowering the Al:P binding ratio, and stabilizing polymerization for longer internal P loading control. Dose splitting can also be used as an adaptive management approach to address slower degradation of labile organic P into mobile forms as well as increased P binding efficiency onto the Al floc.

For instance, a higher Al dosage should be applied during year 1 to control internal P loading while a lower dose should be applied in subsequent years to maintain internal P loading control. In addition, it is recommended that Al application occur within 1 month or less of the development of bottom anoxia to bind P diffusing out of the sediment. For Bone Lake, application in early June would coincide with the development of bottom anoxia. Finally, the Lake District should be made aware that an additional Al application (much lower Al concentration) may be needed several years after these applications as a maintenance measure to ensure complete internal P loading control (see James 2017). Both water column and sediment monitoring will be used to assess control and the need, if any, for another maintenance Al application several years into the future.

Table 9. An example adaptive management scenerio approach in which the Al dose is split into smaller applications. A smaller dose is applied to lake sediments in year 1. The second Al application and dose is determined via annual sediment profile monitoring. In this example, annual sediment core vertical profiling indicated that a second application should occur in year 3 at a dose estimated from similar core analysis.

Variable	Year 1	Year 2	Year 3	Year 4	Year 5
Al application	60 g/m^2		25-40 g/m^2		25 g/m^2
Assessment[1]		▨	▨	▨	▨

[1] Sediment core collection and vertical profile monitoring

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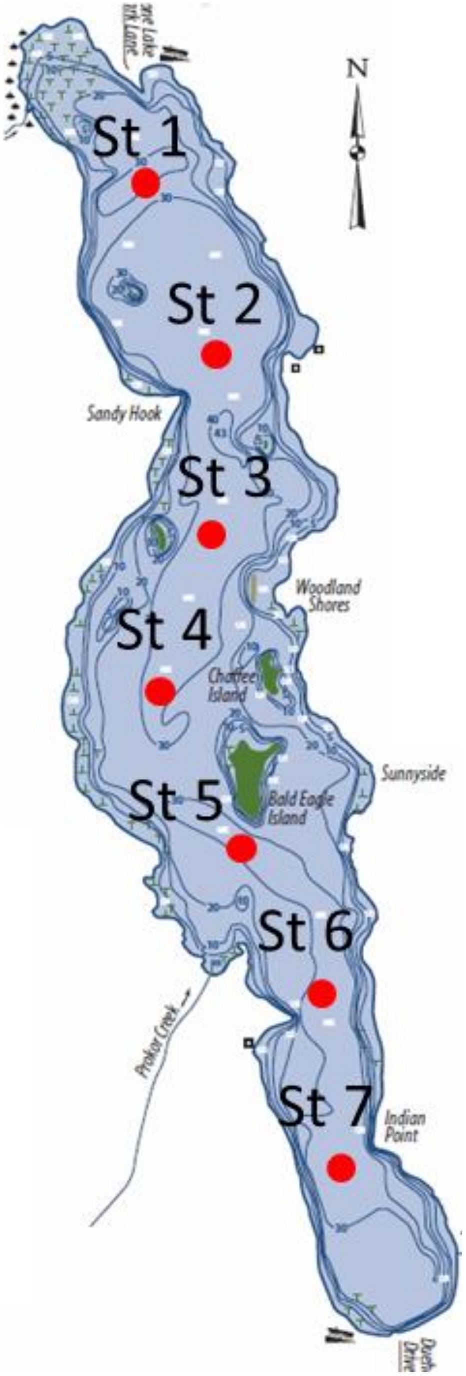

Figure 1. Station locations in Bone Lake.

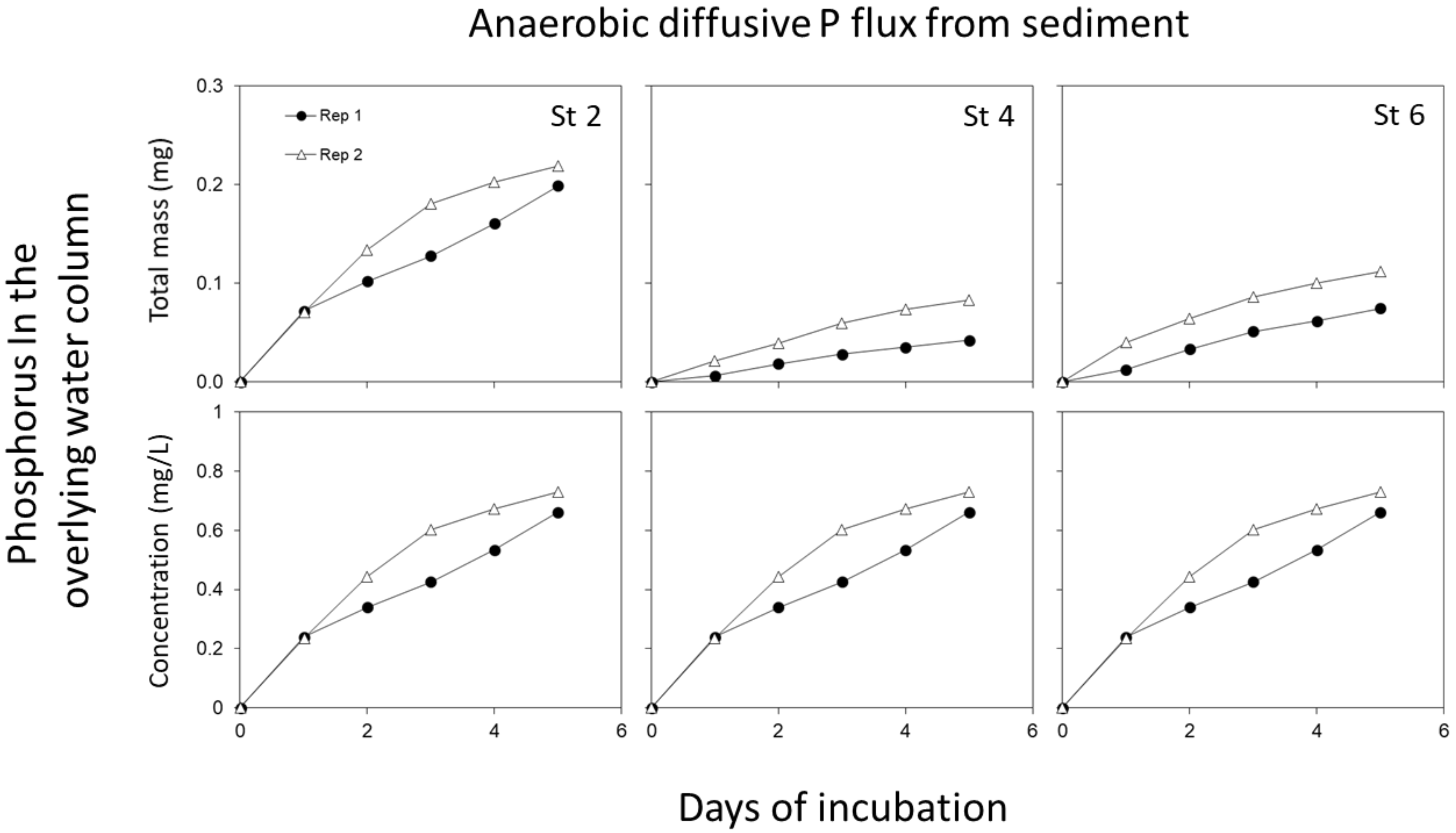

Figure 2. Changes in soluble reactive phosphorus mass (upper panels) and concentration (lower panels) in the overlying water column under anaerobic conditions versus time for sediment cores collected in Bone Lake.

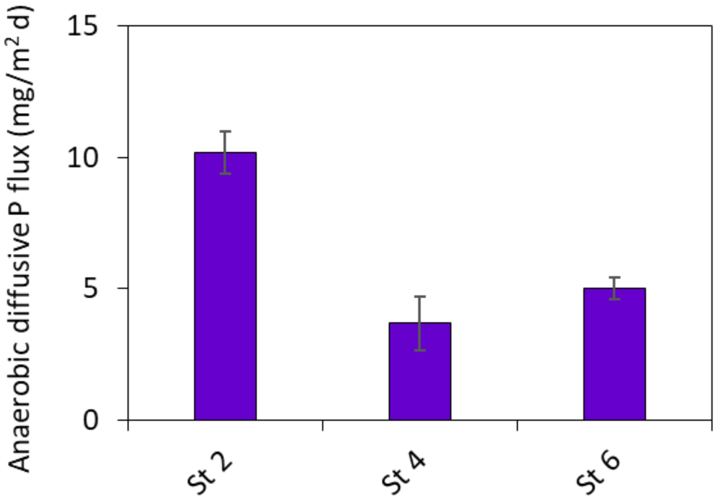

Figure 3. Mean rates of diffusive phosphorus (P) flux under anaerobic conditions at various stations in Bone Lake.

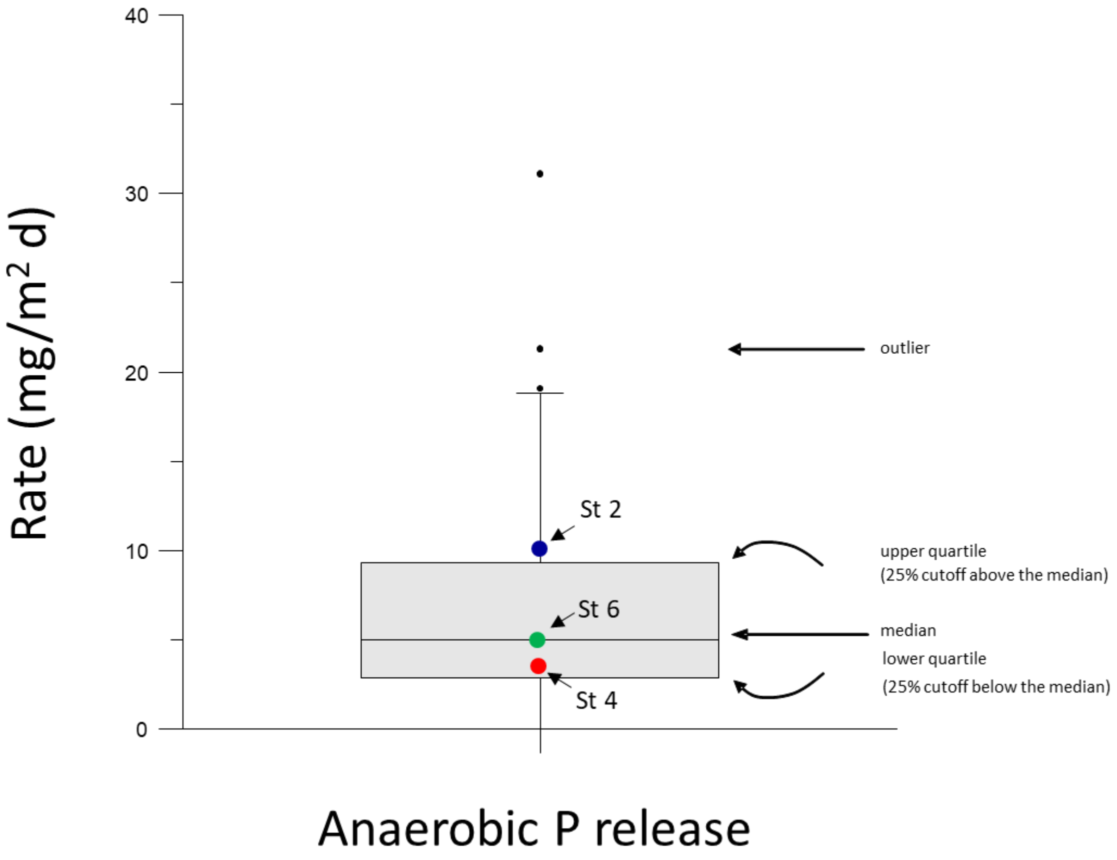

Figure 4. Comparison of rates of diffusive phosphorus (P) flux under anaerobic conditions at various stations in Bone Lake versus other lakes in the region.

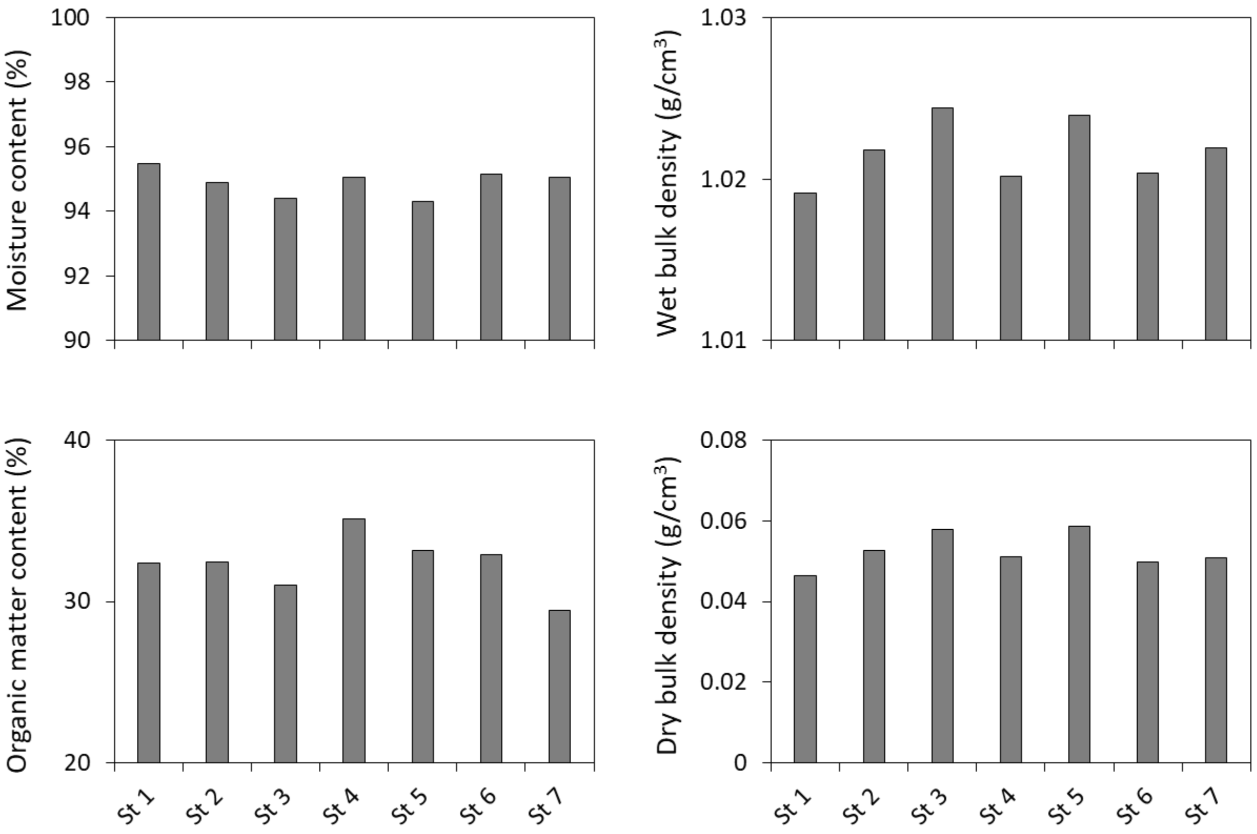

Figure 5. Variations in moisture content, wet and dry bulk density, and organic matter content in the upper 5-cm sediment section for various stations in Bone Lake.

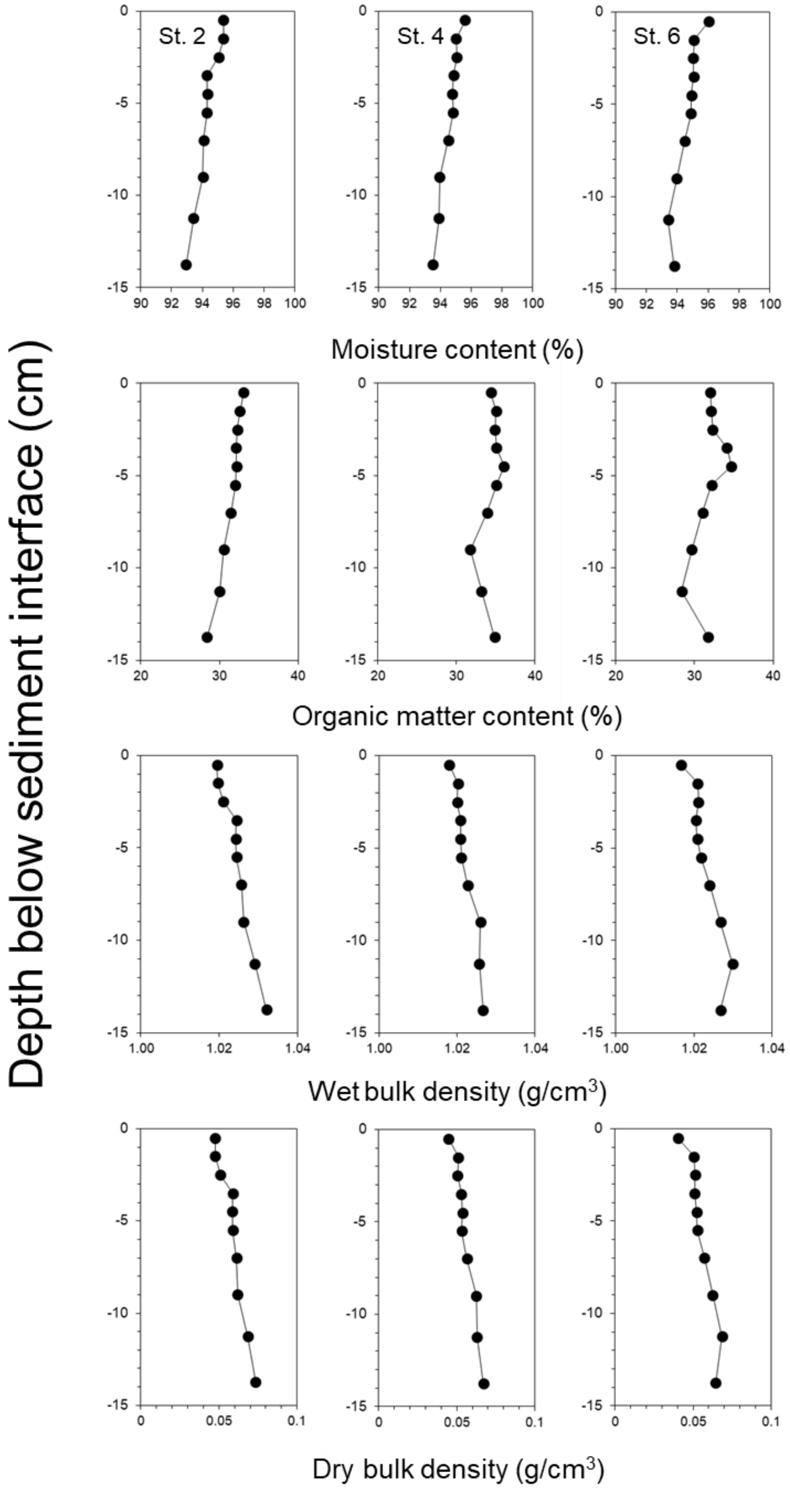

Figure 6. Vertical variations in sediment moisture content, solids content, porosity, wet bulk density, dry bulk density, and organic matter content as a function of depth below the sediment surface for a sediment cores collected in Bone Lake.

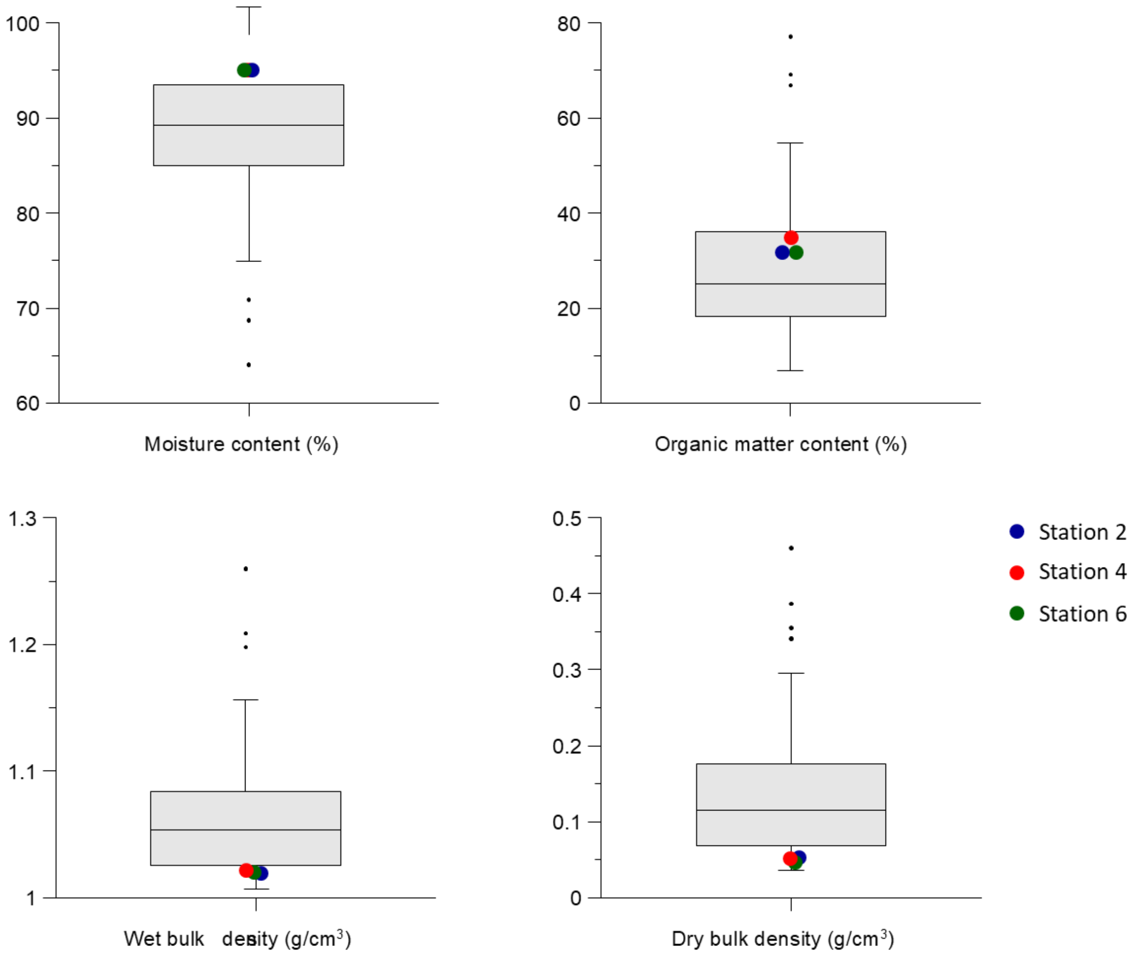

Figure 7. Comparison of moisture content, wet and dry bulk density, and organic matter concentration in Bone Lake versus other lakes in the region.

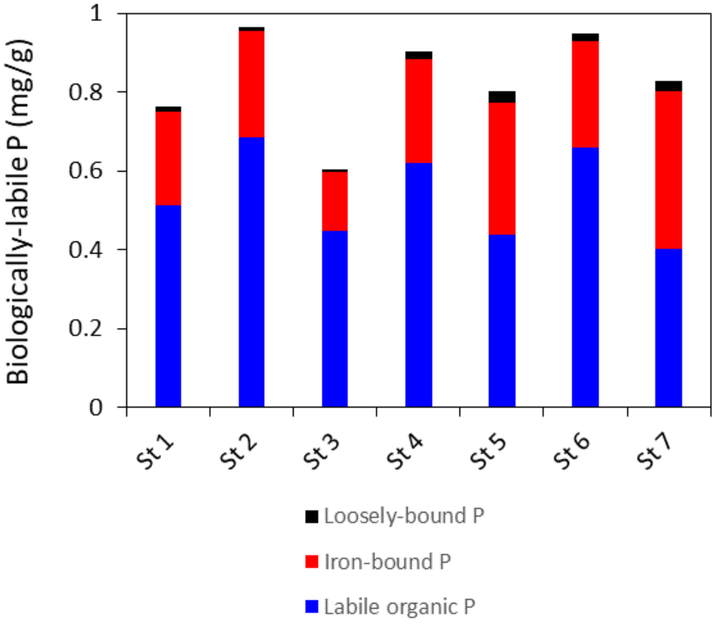

Figure 8. Variations in the composition of biologically-labile phosphorus (P; i.e., subject to recycling with the overlying water column; sum of the loosely-bound, iron-bound, and labile organic P) in the upper 5-cm sediment layer for cores collected in Bone Lake.

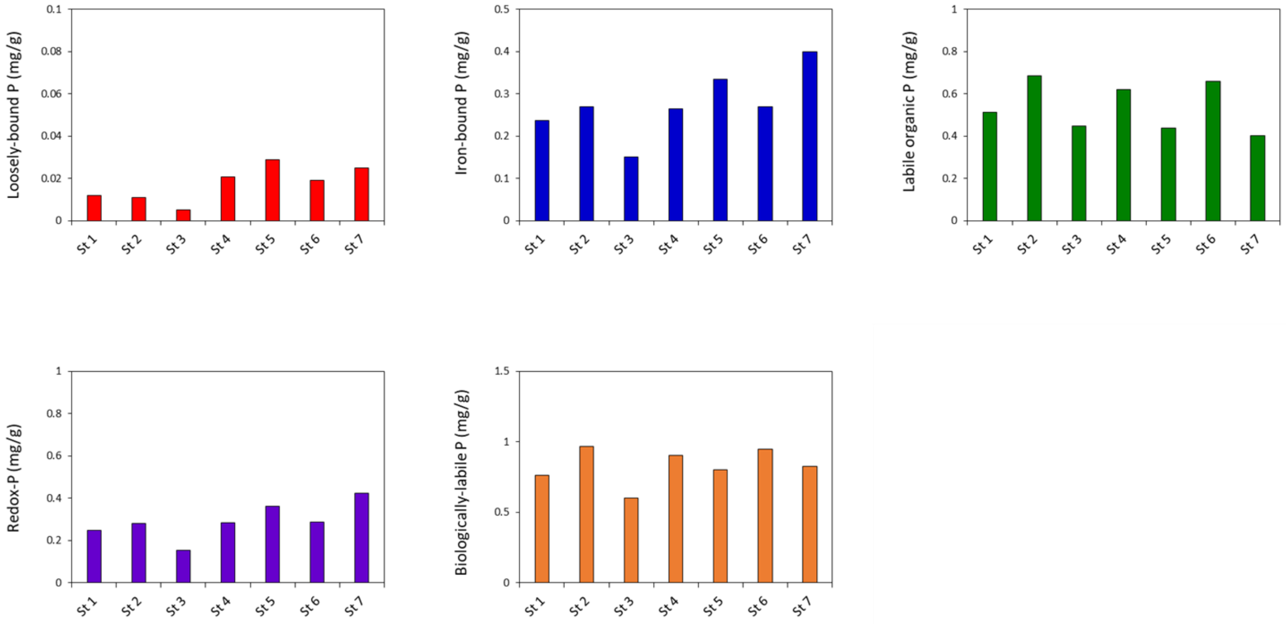

Figure 9. Concentrations of various phosphorus (P) fractions in the upper 5-cm sediment layer.

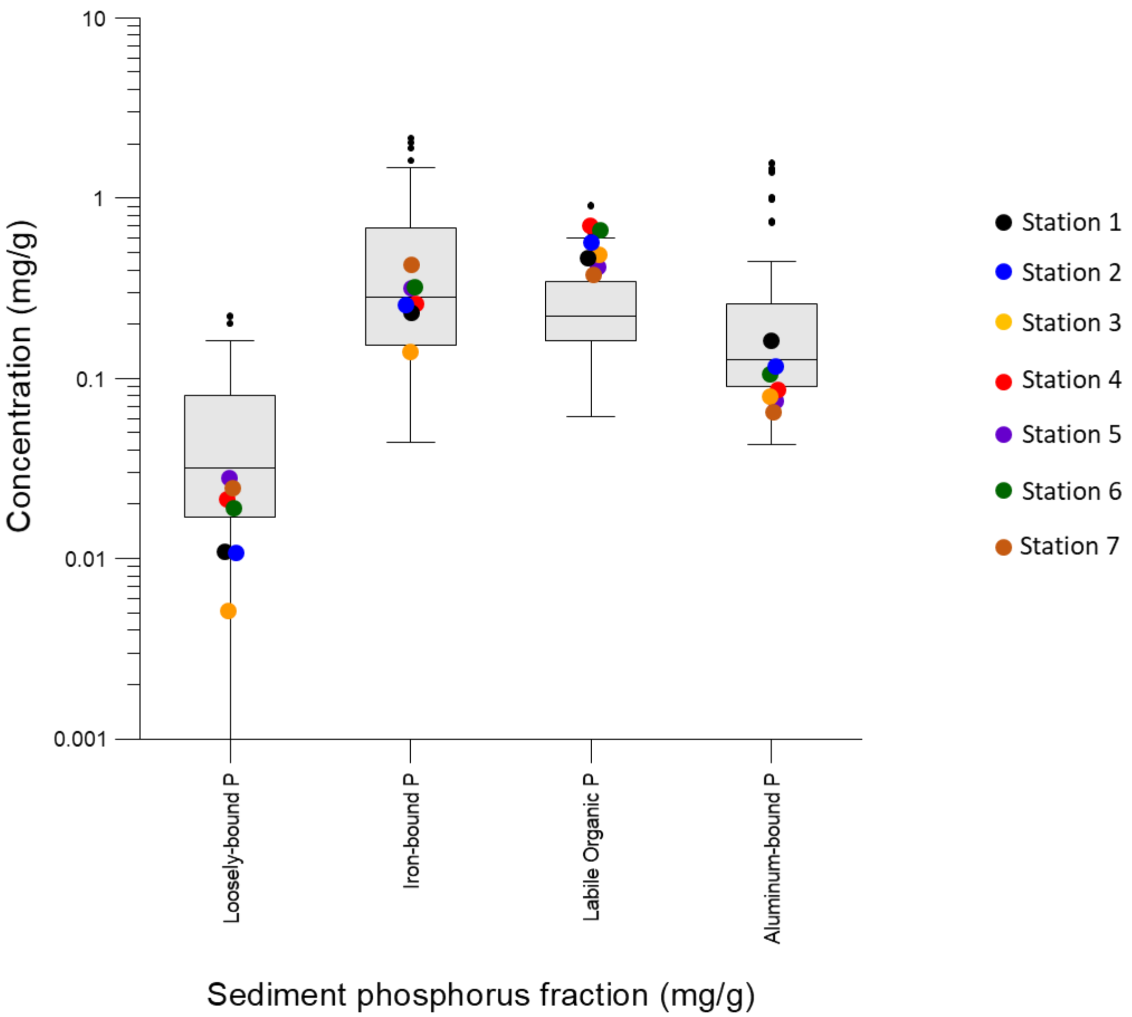

Figure 10. Comparison of sediment phosphorus (P) concentrations in Bone Lake versus other lakes in the region.

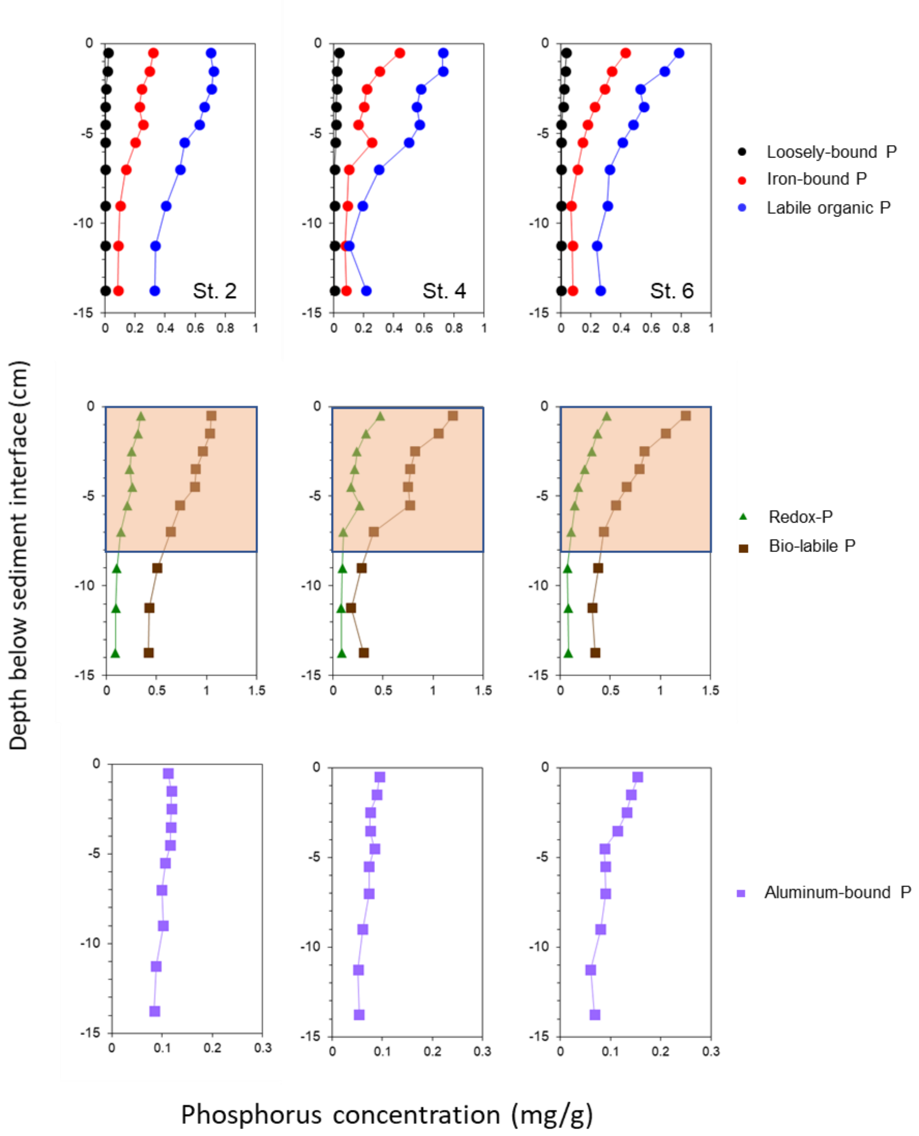

Figure 11. Vertical variations in loosely-bound phosphorus (P), iron-bound P, labile organic P, and aluminum-bound P concentrations for sediment cores collected from various stations Bone Lake. Orange boxes in middle panel denote the sediment thickness where redox-P concentrations are maximal and likely contribute to internal P loading.

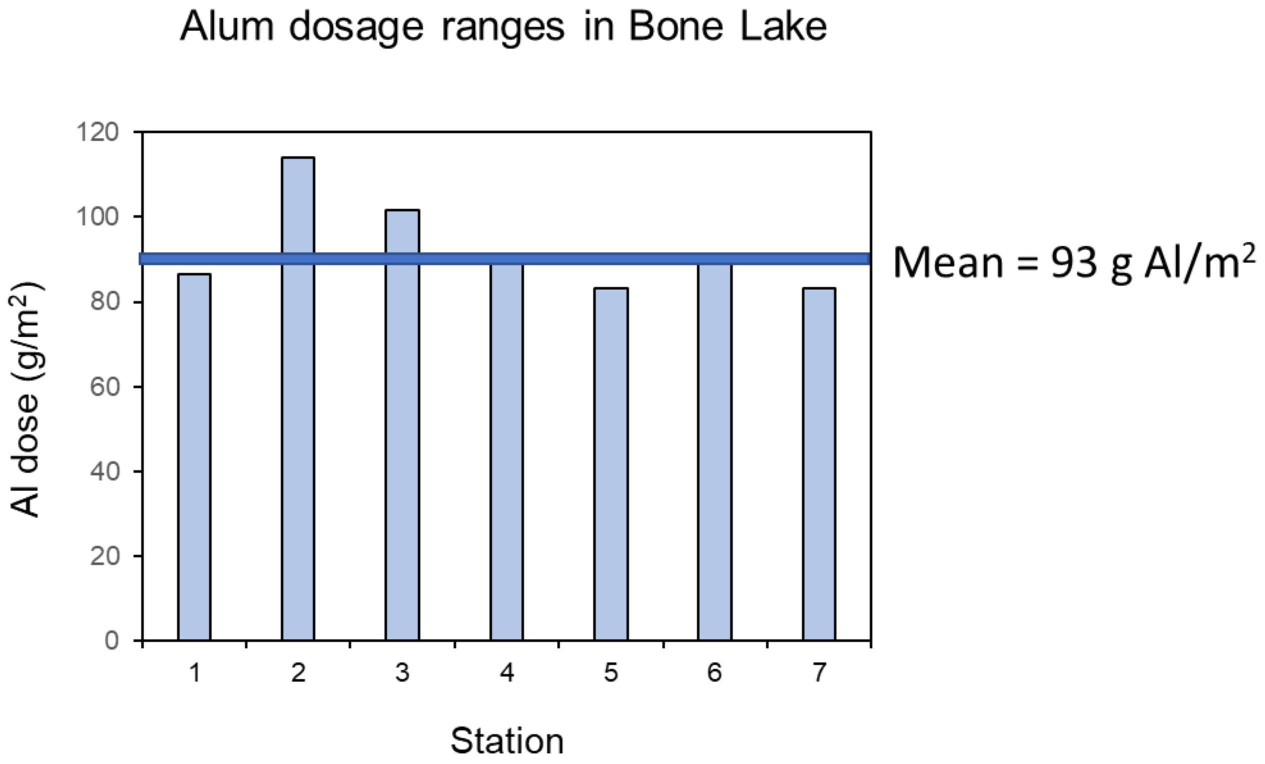

Figure 12. Variations in estimated alum (Al) dose for various stations in Bone Lake. The mean Al dose was 93 g/m^2.