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## Aluminum Sulfate Application to Improve Under-Water Light Condition for Native Submersed Macrophyte Restoration: Alum to Phosphorus Binding Ratio Considerations

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**PURPOSE:** Native submersed macrophyte growth is often limited by underwater light availability in eutrophic aquatic systems. Control of canopy-forming exotic species does not necessarily lead to greater light penetration because eutrophic systems usually sustain high algal biomass in the form of cyanobacterial blooms that can rapidly attenuate light near the lake surface. High phosphorus (P) loading from bottom sediments often represents an important source for algal uptake that needs to be controlled in order to drive aquatic systems toward P-limitation of growth and improve underwater light condition for restoration of native submersed macrophyte populations. Aluminum (Al) sulfate application represents an effective management technique for binding P in sediment and reducing flux into the water column for algal uptake. However, research is needed to determine the dosage of alum required to bind sediment P. The objective of this research was to examine Al:P stoichiometry in relation to extractable forms of sediment P in order to improve the accuracy of alum dosage determination to control sediment P loading in aquatic systems.

**BACKGROUND:** Re-establishment of persistent native submersed macrophyte populations in shallow systems is often complicated by invasions of nonnative canopy-forming species and eutrophic conditions that result in light limitation of growth. Exotic invasive species can often outcompete natives for light and nutrients, resulting in declines or complete elimination of desirable species populations over time (Madsen et al. 1991). However, efforts to control exotic macrophyte species may not improve growth conditions for native submersed macrophyte restoration if excessive cyanobacterial blooms maintain high light attenuation (James 2010). Additional management that targets reduction in algal biomass may be required in order to improve underwater light condition for native macrophyte growth and persistence. Since phosphorus (P) is generally regarded as the nutrient that limits algal growth, strategies to control P offer the most sustainable approach to improving algal-induced light attenuation in aquatic systems.

Bottom sediments usually represent a substantial P source that can subsidize high algal productivity, even when external P loading from the watershed has been reduced. 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.,  $\text{Fe}^{+3}$ ) in the microzone as  $\text{Fe}(\text{OOH})$  and adsorbs P, thereby minimizing its diffusion into the overlying water column. However, high pH driven by algal productivity can promote P release from  $\text{Fe}(\text{OOH})$  via ligand exchange reactions (James et al. 1996). Bacterial

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reduction of dissolved oxygen and iron from  $\text{Fe}^{+3}$  to  $\text{Fe}^{+2}$  in the upper sediment layer results in the development of anoxia, desorption of P, and diffusion out of the sediment. 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.

Extractable forms of sediment P found to be most active in these eH and pH reactions are the loosely bound (i.e., adsorbed P and P associated with calcium carbonate; extracted with 1 M ammonium chloride) and Fe-bound (i.e., P associated with  $\text{Fe}(\text{OOH})$ ; extracted with 0.11 M bicarbonate-dithionate or BD) fractions (Boström et al. 1982; Nürnberg 1988). Nürnberg (1988) found that the BD-extractable P fraction was a good predictor of rates of total P release from sediment under anoxic conditions, supporting the hypothesis of Mortimer (1971). These P fractions can be immobilized via an alum (aluminum sulfate;  $\text{Al}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$ ) application to the sediment to irreversibly bind P to aluminum hydroxides ( $\text{Al}(\text{OH})_3$ ) and inhibit P flux from sediments (Cooke et al. 2005). Alum is usually applied in the surface waters over the active sediment area, where it disassociates to form an  $\text{Al}(\text{OH})_3$  precipitate that then settles to the bottom as a milky white floc that covers the sediment. The hydrolysis of Al leads to the production of hydrogen ions, which lowers the pH of the water column. Al toxicity to the biota can occur if the pH falls below 6 and must be considered in dose determination. The dosage of alum can be estimated by determining the maximum allowable concentration based on the pH and alkalinity of the aquatic system (Kennedy and Cooke 1982). The rationale for this dose determination method is to apply a maximum amount of alum to the hypolimnion and yet maintain the pH above 6.0 to prevent Al dissolution. However, this estimation technique is not suitable for soft water lakes with a low buffering capacity. It also does not directly consider control of reactive P stored in the sediment or the rate of sediment P release.

Dosage can also be estimated by considering the mass of BD-extractable P in the upper layers of the sediment. These calculation methods more specifically address the control of sediment P flux and reactive sediment P pools. Unlike the alkalinity-based dosage estimations, calculations based on sediment P require knowledge of the amount of alum required to bind the P fraction extracted with BD. An assumption used in this calculation is that P binds to alum at or near a 1:1 mass ratio. However, Rydin and Welch (1999) recently reported that the Al:P binding ratios can be considerably greater than 1:1. If so, a low ratio assumption could result in substantial under-dosage and short-lived effectiveness. More information is needed regarding stoichiometric relationships between alum and BD-extractable P in order to more accurately estimate the amount of alum required to control sediment P flux. The objectives of this research were to examine Al:P ratios as a function of the concentration of BD-extractable P in sediment and to demonstrate dosage calculations based on these ratios.

**STUDY SITE:** Half Moon Lake was chosen as a study site because it exhibits high concentrations of BD-extractable P and high rates of P release from sediment (James et al. 2002). The lake is a relatively small ( $0.5 \text{ km}^2$  and  $8.9 \times 10^5 \text{ m}^3$ ), shallow (mean depth = 1.6 m, maximum depth = 4 m) urban lake located in Eau Claire, Wisconsin (Figure 1). An oxbow of the Chippewa River, the bathymetry resembles that of a river channel. The eastern portion of the lake is isolated by a causeway that restricts water exchange with the rest of the lake. Forty percent of the surface area is less than 2 m deep. External inputs to the lake occur primarily via a storm sewer system that drains residential, commercial, and industrial land uses from a  $2.3\text{-km}^2$  watershed. The P budget has been

dominated by internal loading from sediment (42%), decomposition of *P. crispus* and recycling to the water column (20%), and sediment resuspension by motor boat activity (17%). In contrast, external P loading accounts for only 21% of the P budget (James et al. 2002). The lake is classified as eutrophic (Carlson  $TSI_{CHLA} = 74$ ); mean summer trophic state indicators are  $0.110 \text{ mg}\cdot\text{L}^{-1}$  total P,  $82 \text{ mg}\cdot\text{m}^{-3}$  viable chlorophyll, and 1.1 m Secchi transparency.

The macrophyte community is dominated by *P. crispus*. Turions germinate in late March and the population can occupy greater than 75% of the lake surface area by early June, attaining peak biomass levels greater than  $100 \text{ g dry mass}\cdot\text{m}^{-2}$ . Native submersed and emergent macrophyte species are dominated primarily by *Ceratophyllum* and *Elodea* sp.

## METHODS:

### Sediment P fractionation.

Sediment cores were collected at 29 stations in Half Moon Lake in July 2009 for determination of BD-extractable P concentrations in the upper 4-cm layer (Figure 1). Subsamples were dried at  $105 \text{ }^\circ\text{C}$  to a constant weight and burned at  $500 \text{ }^\circ\text{C}$  for determination of moisture content, sediment density, and organic matter content (Håkanson 1977). To determine BD-extractable P, 12.5 mL of a solution of  $0.11 \text{ M NaHCO}_3 - 0.11 \text{ M Na}_2\text{S}_2\text{O}_4$  (sodium bicarbonate-sodium dithionate; BD) was added to 25 mg dry mass equivalent of fresh sediment contained in 50-mL centrifugation tubes. The tubes were gently shaken, incubated in a  $40 \text{ }^\circ\text{C}$  water bath for 30 minutes, and centrifuged for 45 min at 500 g to clarify the mixture (Nürnberg 1988). The extract was filtered through a  $0.45\text{-}\mu\text{m}$  membrane syringe filter and gently bubbled with air for 1 hr to reoxidize the solution. Filtrate was analyzed for soluble reactive P (SRP) using standard colorimetric techniques (American Public Health Association (APHA) 2005).

### Alum dosage assays.

Sediments from six of the sampling stations were used for determination of the dosage of alum (as Al) required to immobilize BD-extractable P using a modification of the Rydin and Welch (1999) assay method. These samples were chosen to represent the range of BD-extractable P concentration observed in the lake sediments. Alum (as aluminum sulfate;  $\text{Al}_2(\text{SO}_4)_3 \cdot 18 \text{ H}_2\text{O}$ ) was diluted with  $0.1 \text{ M}$  sodium bicarbonate ( $\text{NaHCO}_3$ ) to a concentration of  $1.4$  to  $2.5 \text{ g Al L}^{-1}$  (at pH 7.0). Aliquots of this solution (diluted to a final volume of 10 mL with distilled water) were added to centrifuge tubes containing the equivalent of  $0.025 \text{ g}$  dry mass of fresh sediment to obtain Al concentrations ranging from 0 (i.e., control) to greater than  $300 \text{ mg Al g}^{-1}$  sediment. The assay tubes were shaken for a minimum of 2 hr at  $20 \text{ }^\circ\text{C}$  in a darkened environmental

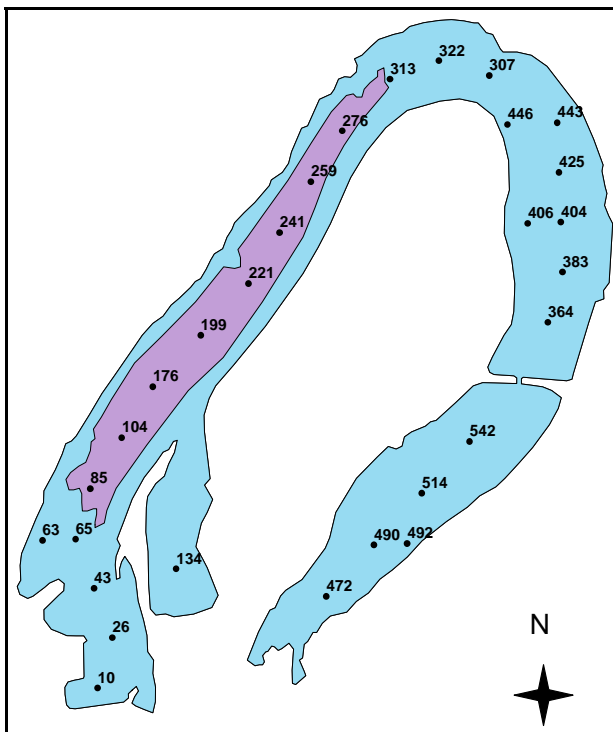


Figure 1. Sediment sampling stations on Half Moon Lake, Wisconsin. Lavender area represents the 3-m contour.

chamber and centrifuged at 500 g to concentrate the sediment. The treated sediments were then extracted with BD using procedures described above.

Al:P ratios were estimated as concentration of alum (as Al) required to bind 90% of the BD-extractable P. This calculation differed from Rydin and Welch (1999) in that they used the concentration of aluminum-bound P (as extracted in 0.1 N sodium hydroxide) formed versus BD-extractable P lost to the alum floc. Earlier studies on Half Moon Lake sediment found that an interference in the form of a precipitate developed during the NaOH extraction, which rendered determination of Al-bound P formed on the alum floc untenable. This precipitate was believed to be the outcome of reaction between organic acids and alum during the NaOH extraction procedure.

Al dosage was estimated as the concentration ( $\text{g}\cdot\text{m}^{-2}$ ) required to bind BD-extractable P in the upper 4-cm sediment layer. This sediment depth was chosen based on observations of the position of the alum floc in sediments subjected to an alum application in field enclosures deployed in Half Moon Lake. The alum floc was located within the upper 4-cm layer one year after application (W.F. James, unpublished data). The dry mass concentration of BD-extractable P ( $\text{mg}\cdot\text{g}^{-1}$ ) was converted to an areal concentration ( $\text{g}\cdot\text{m}^{-2}$ ) as

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

where  $\rho$  is sediment bulk density ( $\text{g}\cdot\text{cm}^{-3}$ ),  $\theta$  is sediment porosity (100-percent moisture content; dimensionless), and  $h$  is sediment thickness (m). The Al concentration ( $\text{g}\cdot\text{m}^{-2}$ ) was estimated as

$$Al (\text{g}\cdot\text{m}^{-2}) = BD - P (\text{g}\cdot\text{m}^{-2}) \cdot Al : P_{90\%} \quad (2)$$

where  $Al:P_{90\%}$  was the binding ratio required to adsorb 90% of the BD-extractable P in the sediment.

**RESULTS AND DISCUSSION:** An example of changes in BD-extractable P as a function of alum concentration is shown in Figure 2. BD-extractable P declined in an exponential pattern as a function of increasing alum concentration due to irreversible transfer of P from Fe compounds to Al (Figure 2a). Over 60% of an initial BD-extractable P concentration of  $0.149 \text{ mg}\cdot\text{g}^{-1}$  was bound with a modest Al concentration addition of  $2.8 \text{ mg}\cdot\text{g}^{-1}$  (Figure 2b). However, P binding diminished exponentially at higher Al concentrations. This pattern was probably due to increased competition for binding sites on the alum floc by other constituents as Al concentration increased. A target level of 90% BD-extractable P inactivation was chosen as an alum dosage goal. However, this target may need to be set at greater than 90% for sediments with very high BD-extractable P concentrations and/or high rates of P release from sediment. The concentration of Al required to bind 90% of the BD-extractable P was  $\sim 22.8 \text{ mg}\cdot\text{g}^{-1}$  for this particular example (Figure 2b), resulting in an  $Al:P_{90\%}$  of 170 (i.e.,  $22.8 \text{ mg Al}\cdot\text{g}^{-1}$  required to bind  $0.134 \text{ mg P}\cdot\text{g}^{-1}$ ). This ratio was considerably higher than the 1:1 ratio that has been typically used for alum dosage estimation.

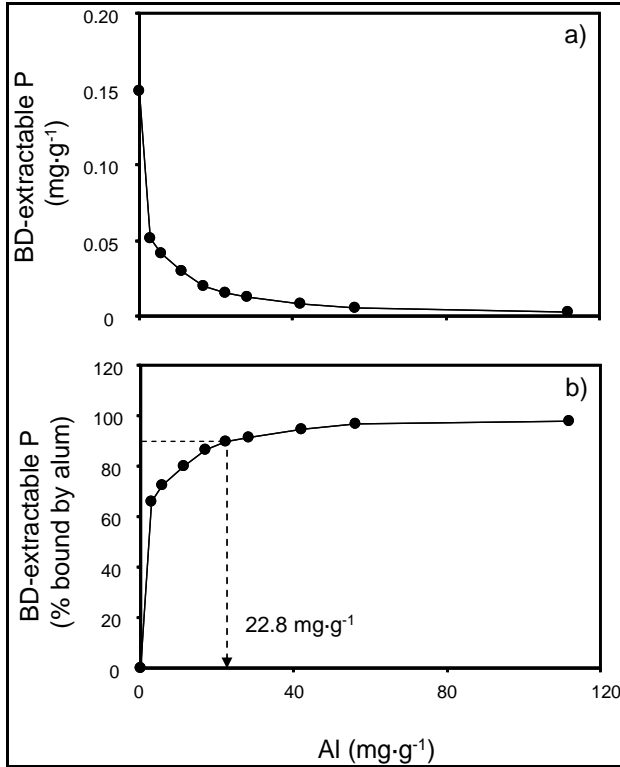


Figure 2. Relationships between aluminum (Al) concentration and the concentration of BD-extractable phosphorus (P) remaining in sediment (a) and the percentage of BD-extractable P bound by alum versus the Al concentration for sediment collected at station 404 (see Figure 1). Dotted lines designate the concentration of Al required to bind 90% of the BD-extractable P.

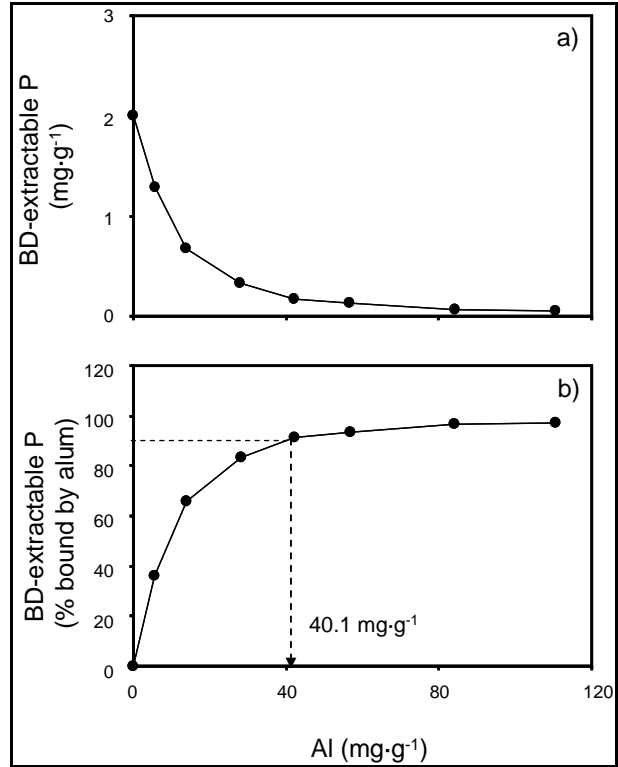


Figure 3. Relationships between aluminum (Al) concentration and the concentration of BD-extractable phosphorus (P) remaining in sediment (a) and the percentage of BD-extractable P bound by alum versus the Al concentration for sediment collected at station 241 (see Figure 1). Dotted lines designate the concentration of Al required to bind 90% of the BD-extractable P.

In contrast, sediment with a much higher BD-extractable P concentration of 2.0 mg·g<sup>-1</sup> required only a slightly greater concentration of Al to achieve 90% inactivation (Figure 3). In this example, 40.1 mg Al·g<sup>-1</sup> were needed to bind 90% of the BD-extractable P, resulting in an Al:P<sub>90%</sub> of only 22. For all sediments assayed, the Al:P<sub>90%</sub> ratio varied in a negative exponential pattern as a function of increasing initial BD-extractable P concentration (Figure 4). Thus, the Al:P<sub>90%</sub> ratio exceeded 150 for initial BD-extractable P concentrations less than 0.15 mg·g<sup>-1</sup>, and it approached ~ 20 for concentrations greater than ~ 2.0 mg·g<sup>-1</sup>. Competition for binding sites by other constituents may be responsible for this pattern. At lower initial BD-extractable P concentrations, a higher Al:P<sub>90%</sub> ratio is required because other constituents are competing with BD-extractable P for binding sites. At higher concentrations of BD-extractable P, a lower stoichiometric ratio is needed because there is more P relative to other constituents to compete for binding sites on the alum floc.

Because sediment P concentrations can vary spatially as a result of bathymetry and delta formation, variations in Al:P stoichiometry need to be considered for accurate alum dose calculation on a lakewide scale. For instance, an Al:P ratio of 10:1 would inactivate ~ 60% of the BD-extractable P for sediments depicted in Figure 3 (Table 1). However, the same ratio would result in an under-dosage by 77% for sediments with a BD-extractable P concentration of 0.149 mg·g<sup>-1</sup> (Figure 2). Although Al:P ratio of 100:1 would be effective in binding BD-extractable P at both stations, it would result in substantial overdosing and be cost ineffective for sediments with the higher BD-extractable P concentration (Table 1).

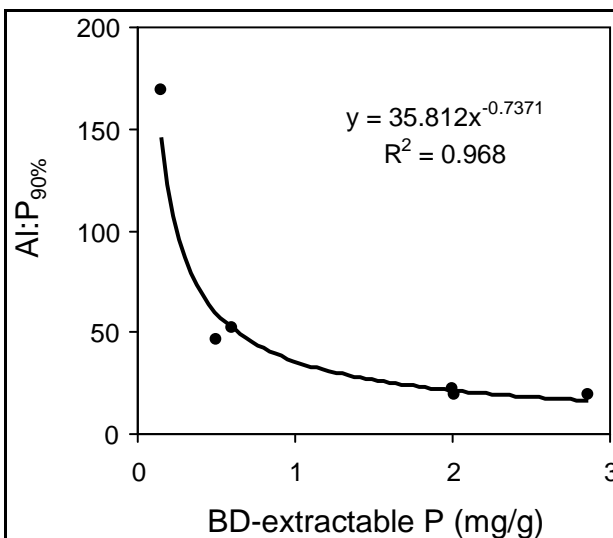


Figure 4. Relationships between the alum (as aluminum; Al) to phosphorus (P) binding ratio needed to inactivate 90% of the BD-extractable P (Al:P<sub>90%</sub>) and the initial BD-extractable P concentration in sediment.

<b>Table 1. The aluminum:phosphorus ratio (Al:P), areal concentration of Al, and percentage of bicarbonate-dithionate-extractable P (BD-P) bound by alum for sediments with a relatively low (left column) versus high (right column) BD-P concentration.</b>					
BD-P = 0.149 mg g <sup>-1</sup>			BD-P = 2.00 mg g <sup>-1</sup>		
Al:P	Al (g m <sup>-2</sup> )	BD-P bound (%)	Al:P	Al (g m <sup>-2</sup> )	BD-P bound (%)
170 (measured)	95.5	90	22 (measured)	177	90
1	0.6	2	1	8	5
10	5.6	23	10	80.5	60
100	56.2	83	100	805	100

For Half Moon Lake, there were distinct spatial variations in BD-extractable P concentrations (Figure 5). Concentrations were greatest in the slightly deeper western arm and declined to lower values in the northern, eastern, and southern portions of the lake. In particular, concentrations in excess of 1.5 mg·g<sup>-1</sup> were very high relative to those reported for lakes in North America (Nürnberg 1988), and coincided with very high rates of P release from anoxic sediment on the order of 10 to 15 mg·m<sup>-2</sup>·d<sup>-1</sup> (W.F. James, unpublished data).

Regression equations developed for Al:P<sub>90%</sub> versus BD-extractable P (Figure 4) were used to estimate spatial variations in the Al:P<sub>90%</sub> (Figure 6). The ratio approached 20 in the western arm of the lake and exceeded 150 in other regions (the maximum Al:P<sub>90%</sub> was set to 150 for calculation purposes). To estimate the alum dosage for various regions of the lake, Equations 1 and 2 were used with an *h* value of 4 cm (i.e., binding of BD-extractable P in the upper 4-cm layer). As shown in

Figure 7, a higher alum dosage would be needed for the western and northern arm versus other areas of the lake. A lakewide areal alum dosage of  $\sim 115 \text{ g}\cdot\text{m}^{-2}$  and a volumetric dosage of  $\sim 40 \text{ g}\cdot\text{m}^{-3}$  were estimated. The volumetric dosage estimated for Half Moon Lake fell in the high range relative to alum dosages reported for other systems (Table 2; see Cooke et al. 2005). However, this observation was not surprising given the high concentrations of BD-extractable P observed in the sediments of Half Moon Lake. In general, higher volumetric dosages coincided with greater longevity of effective sediment P control, which is an important goal in alum dosage strategy (Table 2).

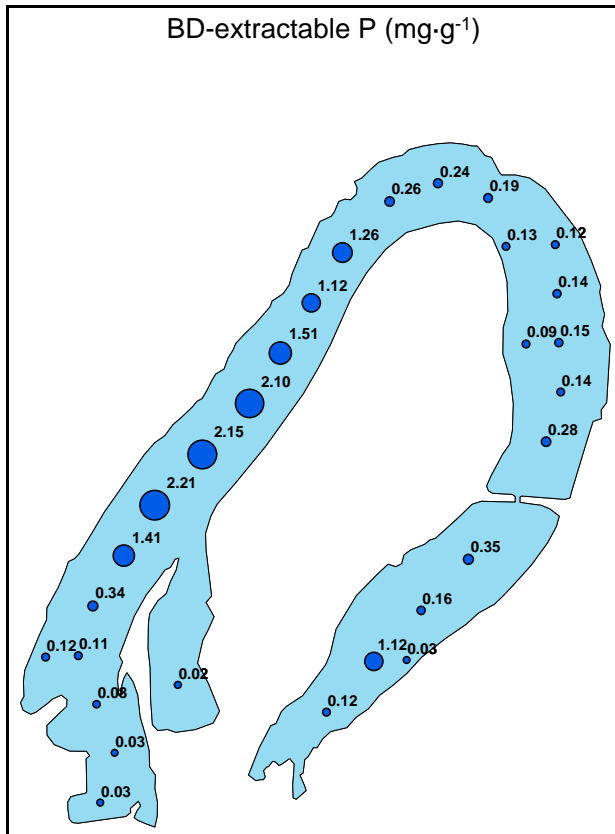


Figure 5. Spatial variations in the BD-extractable phosphorus (P) concentration in the upper 4-cm sediment layer.

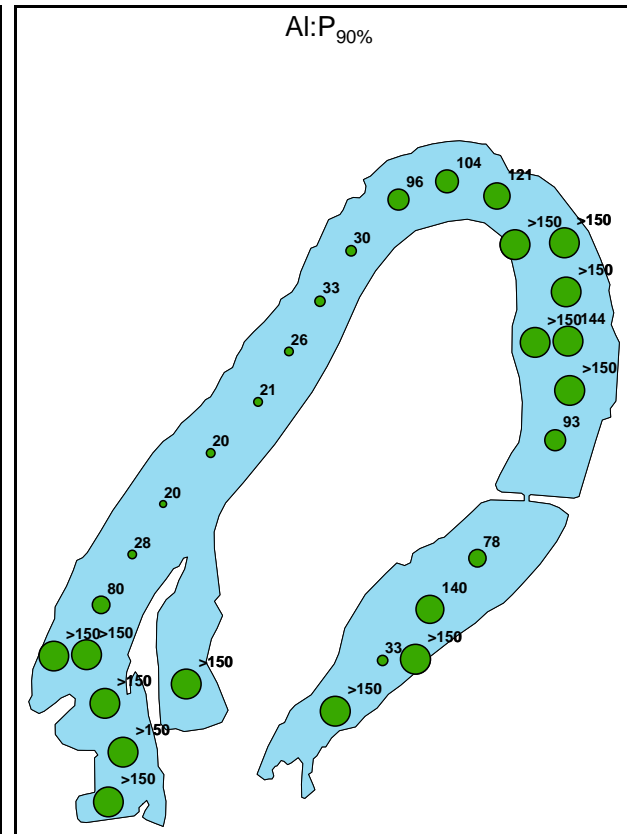


Figure 6. Spatial variations in the alum (as aluminum; Al) to phosphorus (P) binding ratio required to inactivate 90% of the BD-extractable phosphorus (P) concentration ( $\text{Al:P}_{90\%}$ ) in the upper 4-cm sediment layer.

**Table 2. A comparison of alum dosage rates for different lakes in North America and the observed longevity of treatment success based on Welch and Cooke (1999). Longevity was defined as the number of years since treatment that total phosphorus has been reduced in the surface waters by the percentages listed in parentheses. Estimated alum dosage for Half Moon Lake is  $\sim 40 \text{ g}\cdot\text{m}^{-3}$ .**

Lake	Alum Dose ( $\text{g Al m}^{-3}$ )	Observed Longevity (years)
<b>Unstratified</b>		
Long Kitsap Co.	5.5	11 (30%)
Pickerel	7.3	<1
Long Thurston Co. 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%)
<b>Stratified</b>		
Eau Galle	4.5	<2
Morey	11.7	8 (60%)
Cochnewagon	18	6 (0%)
Dollar	20.9	18 (68%)
Annabessacook	25	13 (41%)
West Twin	26	18 (66%)
Irondoquoit Bay	28.7	5 (24%)
Kezar	30	9 (37%)

One unknown in the approach to estimating alum dosage presented here is the effect of aging on Al:P stoichiometry. The current assay approach to estimating Al:P ratios addressed immediate control of sediment P. However, Rydin et al. (2000) reported that Al:P stoichiometry approached  $\sim 11$  in lake sediments that had been treated with alum 6 to 21 years previously. Reitzel et al. (2003) suggested that a molar ratio of 4:1 was sufficient to immobilize sediment P in Lake Sonderby, Denmark. Dugopolski et al. (2008) found that alum binding efficiency was low shortly after alum application, implying that full binding saturation at an Al:P ratio of  $\sim 11$  may not be reached for months or years. If so, use of higher ratios based on assay results could lead to over-dosage in the long term and cost overruns. In contrast, inefficient binding could result in poor control of internal P loading for many years until binding saturation is finally achieved, suggesting that higher ratios may be needed to counteract short-term inefficiencies in binding P by the alum floc. For Half Moon Lake, attainment of a long-term Al:P binding ratio of  $\sim 11$  at the suggested dosage of  $115 \text{ g}\cdot\text{m}^{-2}$  would be equivalent to treating the upper  $\sim 20 \text{ cm}$  sediment layer, assuming that the BD-extractable P concentration was relatively constant with sediment depth. If this projected binding ratio was indeed reached in Half Moon Lake, the alum treatment would still be considered a success rather than a cost overrun because BD-extractable P concentrations are high in the deeper sediment layers as well (W.F. James, unpublished data). Exchangeable P forms can migrate upward from deeper sediment layers, accumulate near the sediment surface, and contribute to diffusive P flux (Carignan and Flett 1981). Field case studies on lake sediments treated with high doses of alum are needed in order to verify laboratory assay findings and to more accurately evaluate optimal (and cost-effective) Al:P binding ratios for use in sediment P-based dosage calculations.

Another unknown is the depth of the active sediment layer contributing to internal P loading that needs to be controlled by the alum floc (i.e.,  $h$  in Equation 1). This active sediment layer may be on the order of millimeters to several centimeters and can be influenced by bioturbation. Research on sediment cores collected from systems that had been treated with alum years ago found that the floc was often located several centimeters below the sediment surface (up to 10 to 12 cm). This finding suggested that the alum floc may be either slowly settling through the sediment and that new sediment inputs may also be burying the alum layer over time. However, definitive information is lacking regarding the exact mechanism causing the depth of the alum layer to change relative to the sediment surface over time. If the alum floc is settling over time, the depth of settling may need to be considered in dosage calculation. Finally, diagenetic processes that result in the breakdown of sediment organic P to soluble inorganic forms can represent an additional source that needs to be controlled. For sediments exhibiting high organic P concentrations relative to BD-extractable P, alum dosage estimates may need to factor in control of additional P originating from diagenetic transformation.

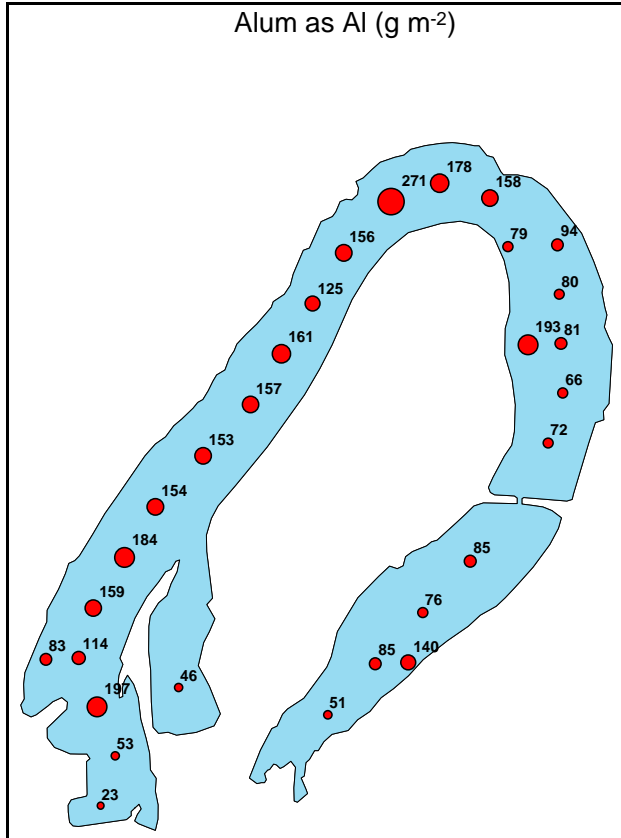


Figure 7. Spatial variations in the alum (as aluminum; Al) dosage concentration required to inactivate 90% of the BD-extractable phosphorus (P) concentration (Al:P<sub>90%</sub>) in the upper 4-cm sediment layer.

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