Gypsum as a Phosphorus and Sediment Control Agent in Cranberry Floodwaters

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Summary

Gypsum, a calcium sulfate mineral, was identified as a potential surface water amendment for reducing phosphorus in cranberry flood water. In our preliminary research, prior to this project, relatively modest additions (~500 lb/acre) of gypsum removed P from cranberry harvest water. Despite those preliminary findings, which formed the basis for this project, subsequent lab experiments showed that gypsum additions had very little effect on reducing P. Geochemical modeling showed that these results were most likely due to the slightly acidic pH and relatively low P concentration of harvest floodwater. A second set of experiments was conducted to test the efficacy of calcite, a calcium carbonate mineral. Amending cranberry harvest floodwater with calcite reduced the dissolved P concentration up to 62%, from 350 to 140 µg P L⁻¹. For field-scale implementation, however, model results showed that a calcite application rate of ~2,000 lb acre⁻¹ would be required for 62% removal of P. Given its high application rate, we concluded that calcite would not represent a practical option for reducing P in cranberry floodwater.

Notably, model results also showed that removal of 35% of P could be explained by adsorption and flocculation ("sorption") processes, as opposed to calcium phosphate precipitation. Because P appeared to be removed by sorption rather than precipitation processes, a third set of lab experiments were conducted to evaluate the P sorbing potential of aluminum sulfate ("alum") and iron sulfate.

These materials were also chosen based on their potential to be effective in the slightly acidic environment of cranberry floodwaters. Keeping with the original objective of the proposal, the experiments also included gypsum, calcite, and calcium hydroxide ("slaked lime") for comparison against alum and iron sulfate.

The results from these experiments showed that application of 10-15 mg L⁻¹ of either alum or iron sulfate would effectively remove all P from cranberry bog water, reducing total P concentration from 49 to <5 μ g P L⁻¹. For a 1-m flood, these application rates equate to 100 lb acre⁻¹, making alum and iron sulfate feasible options for many growers. Modest reductions in the initial pH of 5.9 were observed for alum treatments of 10 mg L⁻¹, declining to 4.7, whereas pH responded more strongly to iron sulfate applications at that rate, declining to 4.2 and even lower as treatment rate was increased. Given that pH less than 4.0 could potentially impact cranberry production, alum represented a better option for reducing P in cranberry floodwater, particularly if rates greater than 10 mg L⁻¹ were to be used.

A detailed survey of the A.D. Makepeace holding pond at White Island Pond, the original proposed site for a field demonstration of a selected compound, showed large spatial variation in P concentration (65 to $157 \ \mu g \ L^{-1}$). As a result, a smaller holding pond, also on A.D. Makepeace property, that displayed less variable P concentration was identified. The site, known as "Edwards Pond West", is a 1.78-ha irrigation pond that is connected to a second pond of similar size by a culvert (Fig. 1). Alum applied to Edwards Pond West at a rate of 8 mg L⁻¹ resulted in a 73% reduction in total P concentration, from 71 to 16 $\mu g \ L^{-1}$.

We view the results of the field demonstration as clear evidence of the effectiveness of alum to reduce P in cranberry irrigation water. While it may be possible to extend these results to applications into flood water on bogs (prior to flood release), further testing is needed, particularly with respect to the horticultural impact of such an alum treatment. In addition to any horticultural implications to cranberry, the well-known side effects of alum treatments, applied to ponds, on organisms in those ecosystems (i.e., Al toxicity) must be taken into consideration when proposing any wide-scale adoption of alum as a control agent in cranberry floodwater that would then be released out of the bog system. However, the alum treatment seemed to be a very effective short-term remedial strategy for reducing P in storage ponds within a farm. Again, the implications of repeated treatments of stored water on the cranberry bogs when that water is then reused remains to be determined.

Introduction

The cranberry industry occupies a unique place in the history of southeastern Massachusetts (MA), where commercial production of cranberries has existed for close to two centuries. More than 200 million pounds of cranberry are grown each year across 13,000 acres of farmland in southeastern MA. Although no longer the leading producer of cranberries, MA still accounts for 26% of the U.S. cranberry supply. Seasonal flooding of cranberry farms is essential for long-term sustainability of cranberry production in southeastern MA, with roughly 90% of growers using a flood to harvest the fruit in the fall. Discharge of harvest floodwaters has been associated with relatively high export of P to surface water (Howes and Teal, 1995). In southeastern MA, elevated levels of P in many ponds and lakes have been linked to eutrophication, the biological enrichment of a water body, which can lead to harmful algal blooms that make recreational water resources unsafe. While cranberry agriculture is far from the only source of P to regional surface waters (septic systems, lawn fertilizers, and storm water have also been implicated), the association of cranberry production with wetland areas and the extensive use of water resources in the production of the crop, including flood harvesting, make it one of the most prominently noted sources. In order to reconcile the demand for P fertilizer by the cranberry industry with regulatory pressure to reduce environmental impacts associated with agricultural runoff, there is a need to implement science-driven management practices and new technologies that reduce P and sediment losses associated with cranberry production.

The goal of this project was to improve environmental water quality in southeastern MA through implementation of gypsum, a naturally occurring soluble salt of calcium (Ca) and sulfur that has historically been used as a source of Ca in blueberry and cranberry production and as a substitute for other amendments such as alum to lower dissolved P and sediment in freshwater lakes and ponds. Specifically, the research project was to develop gypsum as a floodwater amendment for reducing P concentrations in water to be discharged following cranberry harvest. In addition to demonstrating the effectiveness of gypsum as a P control agent, we planned to show that gypsum application to cranberry floodwaters did not adversely affect levels of P in soils or plant tissues.

Several factors came into play that led to a change in that research plan. The laboratory experiment (objective 1), designed to refine a rate for gypsum application that could then be tested, first in a holding pond (objective 2) and later on a flooded bog (objective 3), did **not** result in significant removal of total P from cranberry bog flood water (collected from the 2014 harvest). Additional laboratory experiments were necessary to find an alternative material. This took enough time that the holding pond treatment could not be scheduled prior to the 2015 harvest. Therefore, the earliest that an on-bog demonstration could have been conducted would have been during the 2016 harvest, subsequent to the end of this project. As a result, we here report the outcomes of two of our three original objectives and discuss potential implications and next steps.

Methods

Lab and Modeling Experiments with Gypsum, Calcite, and Slaked Lime In 2014, harvest discharge was sampled from a cranberry farm managed by Federal Furnace at White Island Pond. Initial analyses indicated that dissolved P represented ~90% of total P. As a result, the water was filtered (0.45 µm) and transferred to 200-ml borosilicate beakers. (Note that references to total P for this study are, therefore, actually total dissolved P). In a series of experiments, samples were amended in triplicate with 0-10,000 mg L⁻¹ of reagent-grade calcite (CaCO₃), gypsum (CaSO₄), or slaked lime (Ca(OH)₂) powder and mixed for exactly 30 min with a magnetic stirrer set to 350 rpm. The beakers were not exposed to direct sunlight, but were open to CO₂ gas exchange with the atmosphere. After 4 d, samples were collected from the beakers by slowly pulling on a plastic syringe and analyzed for major and trace elements (Cl⁻, NO₃⁻, NH₄⁺, Al, Ca, Fe, Mg, Mn, P, Si, S), including dissolved organic carbon (DOC) and dissolved inorganic carbon (DIC). Samples were not preserved with the exception of those collected for analysis of DOC, which were acidified with trace-metal grade sulfuric acid.

Inverse mass-balance modeling with the computer program PHREEQC v.3 was used to interpret the geochemical reactions in calcium phosphate precipitation. For the mass-balance models, calcite (CaCO₃), carbon dioxide (CO₂), and hydroxyapatite $(Ca_5(PO_4)_3(OH))$ were allowed to enter or leave the control water to produce the concentrations of P, Ca, DIC, and H⁺ (pH) observed in the treated water. Model uncertainty of 5% in the control water was specified based on the geochemical variation in the three replicate samples, meaning the elemental concentrations (i.e., model constraints) of the control water were allowed to vary up to, but not more than, 5% in order to satisfy the geochemical mass balance between the control and treated samples. Uncertainty in the treated sample was set to 2% and then increased by intervals of two (i.e., 2%, 4%, 6%) until a solution could be reached for the geochemical mass balance. Charge imbalance error was calculated as the sum of positive charges minus sum of negative charges, divided by the average of the two sums (Parkhurst, 1995). Total CO₂ was approximated as the modeled CO₂ flux plus the initial CO₂ in the control sample, which was determined based on measured Ca and the 1:1 molar ratio between Ca and CO_2

Lab Experiments with Alum and Iron Sulfate

In spring of 2016, pond water was collected from Edwards Pond West (the location identified as a potential site for the field demonstration) at a depth of 0.5 m. In the laboratory, the water was divided into aliquots and transferred to 200-ml borosilicate beakers (93 in total). The aliquots were amended in triplicate with 0 to 50 mg L⁻¹ of reagent-grade alum, ferrous sulfate, gypsum, calcite, or slaked lime. The solutions were then stirred at 100 rpm for 5 min. After 48 hr, samples from each beaker were collected with a syringe, digested with alkaline persulfate, and analyzed for total P (see next paragraph). The remaining water in the beakers was analyzed for pH by the electrometric method.

Preliminary analysis of water collected from Edwards Pond, as well as that collected from many other holding ponds used in cranberry farming, showed concentrations of total P less than 1 mg L⁻¹, the detection limit of the analytical method described in the QAPP. As a result, these water samples were analyzed for P using an alkaline persulfate digestion (Patton and Kryskalla, 2003) rather than aqua regia digestion, and by colorimetry (Lachat QuikChem Method 31-115-01-1-I) rather than

inductively coupled mass spectrometry. The colorimetric method reports a measurement range of 1-100 μ g P L⁻¹ for orthophosphate.

Refining Rate for Alum Treatment

Because alum substantially reduced P at the lowest application rate in the experiment described above, an additional round of lab experiments was conducted to evaluate P removal for alum application rates of 1, 2, 3, 4, 5, 7 and 10 mg L⁻¹. Using the methods described above, but with a new sample collected from Edwards Pond West, water was transferred to 1-L beakers and treated with alum using the above application rates. After 48 hr samples were collected for analysis of total P, total dissolved P, and orthophosphate as in the previous round.

Field Implementation

After selecting Edwards Pond for a field application site, we conducted a bathymetric analysis that showed a uniform pond depth of 3.2 (\pm 0.2) m and a volume of 55,545 m³ for the Pond (Fig. 1).

Field application of alum to Edwards Pond West took place on June 15, 2016. A custom designed barge, generally used for the practice of bog sanding (application of a sand layer into a flood that then settles onto the plants, stimulating growth), was obtained from a local cranberry grower, who also operated the barge (Skid Whipple, pers. comm. May, 2016). Alum was applied uniformly across Edwards West Pond, using the sand dispersal mechanism of the barge as it traversed the entire pond surface (Fig. 7). The application of 454 kg alum to the pond resulted in an alum rate of 8 mg L⁻¹. Prior to the alum application, 1-L samples were collected from the West pond at nine locations at two depths (0.5 and 2 m from the surface) along north-south transects, which were marked by wooden stakes on the banks. We also collected 0.5 m depth samples along the edge of the adjacent East Pond (untreated). Two and seven days following the alum application, we returned to those same locations and collected post-treatment samples. All samples were analyzed for total P, total dissolved P, and orthophosphate using the same methods as in the laboratory experiments.

Results and Discussion

Treatments with Gypsum, Calcite, and Slaked Lime

With respect to Ca-based amendments, lab studies showed that up to 28%, 62%, and 98% P removal with additions of gypsum, calcite, and slaked lime, respectively (Fig. 2). Relationships between application rate and P removal varied widely for treatments of these three materials. For an application rate of 500 mg L⁻¹, slaked lime reduced the total P concentration to <10 μ g L⁻¹ from 350 μ g L⁻¹, whereas calcite and gypsum applied at 500 mg L⁻¹ reduced total P to roughly 320 μ g L⁻¹ (Fig. 2). The poor P removal result with gypsum was not consistent with results obtained in preliminary research (prior to this project). For this reason, the use of other

materials was subsequently explored. By comparison, slaked lime was far more effective at reducing P in floodwater than either gypsum or calcite, but it also significantly raised floodwater pH. At 500 mg L⁻¹, resultant pH was 8.2 and 10.9 for calcite and slaked lime, respectively (gypsum did not significantly alter pH). Standard pH for cranberry bog soil is 4.0 to 5.5. Therefore, materials that significantly increase water pH in a bog flood raise concerns for potential impact on soil pH. The relatively high application rates (i.e., 500 mg L⁻¹ equates to about 22 tons for a 10-acre bog with 1-m flood), as well as the resultant high pH, especially for slaked lime, pose serious practical and environmental limitations on the use of any of these Ca-amendments for reducing P in cranberry floodwaters.

Modeling results for the calcite experiments showed that 27% of P removal was the result of calcium phosphate precipitation, whereas 62% of P removal could be associated with Fe-P flocculation that was facilitated by the calcite (i.e., sorption of P onto particles of calcite or newly form calcium phosphate precipitate). The feasibility of calcite as a P control agent was constrained by the low solubility of calcite in water (i.e., 13 mg L⁻¹ at 25°C). Calcite additions resulted in ~ 15% decrease in the concentration of Fe and DOC. Adsorption of humic acids onto calcium phosphate growth sites has been implicated in the reduction of DOC and, consequently, in the formation of amorphous calcium phosphate. With respect to Fe, the concentration decreases were consistent with the electrostatic interactions involved in the coagulation and flocculation of humic colloids, a mechanism cited in the removal of dissolved Fe, as well as DOC and P, in estuarine mixing of river water and sea water. Overall, these results suggested that the low pH and P concentration of floodwater limited the precipitation of calcium phosphate, and that flocculation of P represented a more efficient method for reducing P in flood water.

Treatments with Alum and Iron Sulfate

In a round of experiments comparing calcium and non-calcium materials for P removal, alum and iron sulfate were far more effective at reducing P in cranberry water compared to gypsum, calcite, or slaked lime. With a maximum application rate of 50 mg L⁻¹ (the upper limit of the rate that is considered feasible when scaled up to a field application), the Ca-based amendments reduced total P concentration by 18% or less, while alum and iron sulfate lowered total P concentration by ~90%, from 49 to <1 μ g P L⁻¹ (Fig. 3). These results were in agreement with the previous experiments that indicated low P removal in response to Ca-based amendments. Model results suggested that the low pH of the floodwater may be inhibiting the precipitation of calcium phosphate from solution, but the presence of high fractions of organic phosphorus may have also contributed.

In contrast, rapid reduction in total P was achieved with alum and iron sulfate (Fig. 3). Concentrations of total P decreased below the detection limit (<1 μ g P L⁻¹) following applications of either material in excess of 10 mg L⁻¹. Relatively high variability in the total P concentration of replicate samples was also observed following alum and iron sulfate treatments. For example, the treatment of 1 mg L⁻¹

of alum yielded total P concentrations of <1 μ g P L⁻¹ for two of the three replicates, and 23 μ g P L⁻¹ for the third replicate sample. We suspect greater variation in TP concentration following treatment was associated with the suspension of flocculate matter; this material was likely bound-P (Fig. 4). We carefully extracted water from the beakers by slowly pulling on a syringe, but could visually observe the collection of some of the small flocculant matter (Fig. 4). Given the apparent non-uniform distribution of the flocculant, it was not surprising that post-treatment replicate samples for the alum and iron sulfate treatments exhibited higher variability in total P concentration than those for the Ca-based amendments, where flocculants did not form.

Aqueous pH, Laboratory Studies

An important component of our evaluation was the effect of the various treatments on aqueous pH, since high pH water could be detrimental to cranberry agriculture. Values of pH were relatively constant for gypsum, as would be expected for a neutral salt, and increased to pH 7.5 for calcite and pH 8.6 for slaked lime (Fig. 5). Generally, increases in pH were minor and within the range of most environmental waters with the exception of the slaked lime treatment. Increases in pH in excess of 8 were observed for treatments of 7 and 10 mg L⁻¹ of slaked lime. Values of pH greater than 8 could prove detrimental to the production of cranberry, which is a wetland plant adapted to acidic soils. As a result, we conclude that slaked lime is not a suitable amendment for reducing P in cranberry floodwaters.

For alum and iron sulfate, values of pH decreased with increasing application rate. Values of pH ranged from 4.3 to 4.8 for alum treatments, and from 3.3 to 4.5 for iron sulfate treatments (Fig. 5). The standard range of cranberry soil pH is 4.0 to 5.5. Cranberry growers are familiar with iron sulfate, which has been used in fertility programs. However, values of soil pH that are much less than 4.0 could pose a threat to production and might be promoted by the acidified water. Since alum had less impact on water pH, it is potentially a less risky choice for reducing P in flood water. Application of alum in excess of 10 mg L⁻¹ resulted in concentrations of total P that were below the detection limit of 1 μ g P L⁻¹ for all replicate samples, as well as pH values that were within the standard range of cranberry soils (4.5 to 4.8; Fig. 5). Therefore, we concluded that alum treatments represent the best amendment to test for reducing P in cranberry floodwater because (1) it effectively lowers P in cranberry drainage water and (2) it does not lower pH to levels that could adversely effect cranberry production.

An additional experiment was conducted to further evaluate lower alum application rates, from 1-10 mg L⁻¹. Results showed >50% reduction in total P with alum additions in excess of 5 mg L⁻¹ (Fig. 6). For alum rates lower than 5 mg L⁻¹, results were not consistent with a decrease in total P. Notably, a slight rise in the concentration of particulate P was evident at alum rates from 1-5 mg L⁻¹, followed by a sharp decrease in particulate P for alum application rates of 7 and 10 mg L⁻¹.

This suggested flocculation of sediment and P at all application rates, but the genesis of larger floc sizes that could easily settle out at rates of 7 and 10 mg L⁻¹.

Field Application of Alum

On June 15, 2016, twenty 50-lb bags (454 kg) were applied to Edwards Pond West. Alum was applied from a sand barge, with one person operating the barge and a second applying the alum (Fig. 7). Alum was metered through an 8-ft-wide hopper that was located in the center of the barge, that was powered by a paddle wheel system. Mixing of alum in the water could be observed visually from the barge, largely owing to the turbulence created by the paddle wheels.

Prior to the treatment, water samples were collected from the nine sampling locations in Edwards West at depths of 0.5 and 2 m and from 9 stations in Edwards East at 0.5 m depth. Total water depth was also recorded at the Edwards Pond West sample locations, showing a mean depth of 3.2 (\pm 0.2 - 1 standard deviation) m. Mean concentrations of total P were 78 and 64 µg P L⁻¹ at depths of 0.5 and 2 m, respectively, and exhibited less stratification than concentrations of particulate P, orthophosphate, and dissolved organic P (Fig. 8). Using the mean value of total P, Edwards Pond West stored approximately 4 kg of P, of which 70% was particulate P, 20% was orthophosphate, and 10% was dissolved organic P.

Two days following the alum application, we returned to the same sampling locations and repeated the collection and analysis of pond water samples. Total P was reduced by 87% and 65% at depths of 0.5 and 2 m, respectively in the treated Edwards Pond West (Fig. 8). By comparison, total P in the untreated Edwards Pond East decreased by only 33%. At both depths in Edwards Pond West, dissolved P and orthophosphate were lowered by ~85% and ~94%, respectively and the concentration of particulate P decreased by 87% and 48% at 0.5 and 2 m, respectively. Overall, total P load in Edwards Pond West had declined from 4 to 1 kg P two days following the alum treatment.

On day 7 following the treatment, the P concentrations were very still low in Edwards Pond West. Total P concentrations were 10 and 17 μ g P L⁻¹ at depths 0.5 and 2 m, respectively (compared to pre-treatment concentrations of 78 and 64 μ g P L⁻¹), and orthophosphate and organic P were similarly low (Fig. 8). In contrast, P concentrations increased in Edwards Pond East, from 72 to 123 μ g P L⁻¹ for total P.

Total sediment concentration in Edward Pond West was measured before and after the alum treatment. A slight decrease in mean total sediment concentration from 0.067 to 0.051 g L⁻¹ was observed following the treatment, but differences in mean values were not statistically significant (p = 0.2; two-tail t-test). This finding was probably related to the uncertainty in the analysis, as variation among replicate samples was, on average, 60%, about twice the difference in mean sediment concentration before and after the alum treatment. Other factors, such as a significant decrease in particulate P (Fig. 8), suggested a coagulating effect in response to the alum treatment. We suspect that the decrease in P was driven by the formation of floc material, which then settled out of solution, similarly to what we observed in laboratory treatments (Fig. 6).

Conclusions and Lessons Learned

Alum represents an effective and potentially feasible option for lowering P levels in cranberry irrigation ponds. Based on the results of this study, the optimal alum dosage rate was between 5 and 10 mg L⁻¹. However, we recommend that site-specific dosage rates should be developed for any future treatments in cranberry production systems, where significant variation in phosphorus concentration is known to exist (Kennedy et al. 2015). The approach used here could easily be applied to most cranberry irrigation ponds, including the bathymetric survey and the use of a sand barge. However, consideration must be taken regarding the potential adverse impacts of alum on organisms in the ponds and any potential impacts as the water moves from the pond either back onto the bog or downstream.

We suspect that similar reductions in P could be achieved by barge application of alum on a flooded cranberry bog, but the extent to which elevated levels of Al would effect crop growth have not been determined and no such application would be advisable in the absence of that information. Based on these somewhat limited results (i.e., one pond, no horticultural analysis), we would propose the use of alum in only extreme cases and only on irrigation ponds that do not immediately discharge into other water bodies. The potential for an on-bog treatment remains to be determined. Depending on the amount of horticultural risk, such a treatment might be viable if there was a compelling need to reduce P in flood discharge.

Although alum can reduce P in ponds, the side effects of its application may be harmful to the environment. In the case of its application to flooded bogs, alum additions could significantly increase the flux of Al to lakes and streams that receive flood discharge. Although a single flood release treated with alum would likely have very little environmental consequence, widespread and continued use of alum to treat flood discharge could impact the aquatic ecology of lakes connected to cranberry farms. For this reason, even if further study were to show minimal horticultural risk to the bogs, we would not recommend alum additions on any more than a limited basis.

Cranberry growers have very few options for further reducing P in flood discharge once best fertilizer practices (DeMoranville, 2015) have been adopted. Diverting discharge away from nutrient sensitive lakes was adopted by two cranberry growers as part of the White Island TMDL(MOA, 2009). However, many growers do not have the land area or resources required to divert flood discharge. With further study to assure safety to the crop and surrounding environment, alum treatment may become a short-term option for reducing P in flood discharge.

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Fig. 1. Location of Edwards Pond in Carver, MA. Edwards Pond West was the site of the alum treatment.

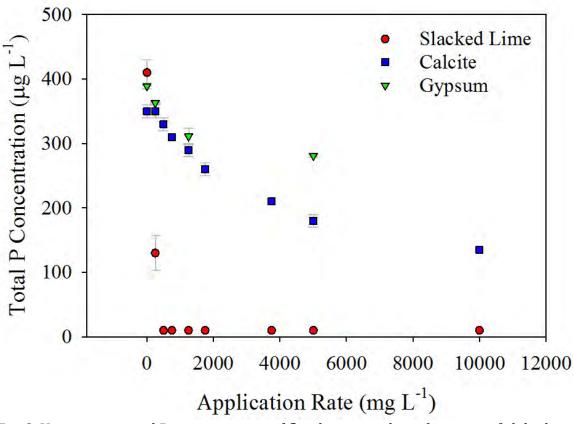


Fig. 2. Variation in total P concentration of floodwater with applications of slaked lime, calcite, and gypsum. Error bars equal the standard deviation of replicate samples.

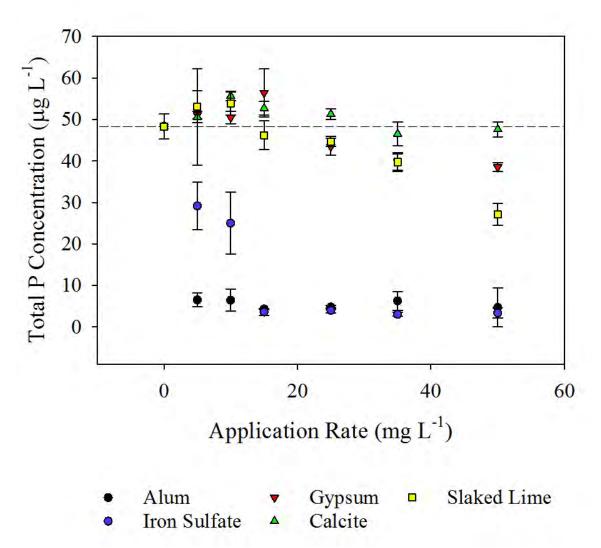


Fig. 3. Total P concentration versus application rate for alum, iron sulfate (FeSO₄), gypsum (CaSO₄), calcite (CaCO₃), and slaked line (Ca(OH)₂). Dashed line is the TP concentration of the control (untreated) sample water. Error bars are one standard deviation of triplicate samples.

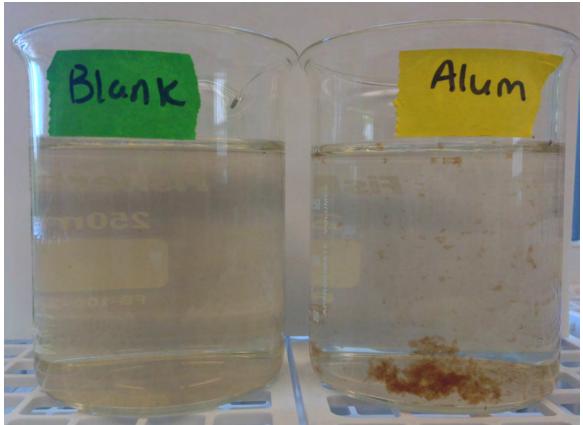


Fig. 4. Photographs of bog water untreated (left) and treated with 5 mg L⁻¹ alum (right). Formation of flocculent matter is apparent in alum treated sample.

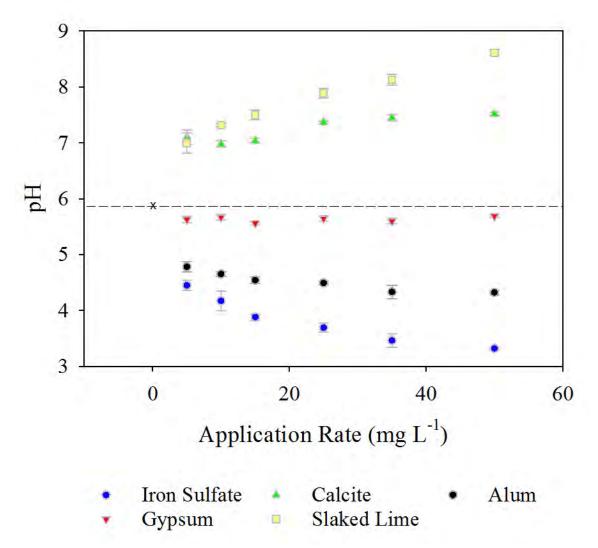


Fig. 5. Aqueous pH versus application rate for alum, iron sulfate (FeSO₄), gypsum (CaSO₄), calcite (CaCO₃), and slaked lime (Ca(OH)₂). Dashed line and point marked "x" is mean pH of the control (untreated) sample water. Error bars are one standard deviation of triplicate samples.

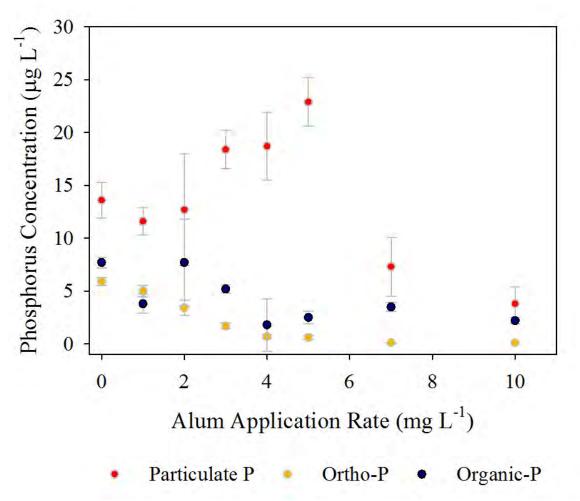


Fig. 6. Concentrations of particulate P, ortho-phosphate, and dissolved organic P versus alum application rate. Error bars are one standard deviation of triplicate samples.



Fig. 7. Sand barge used in the application of alum to Edwards Pond West on June 15, 2016.

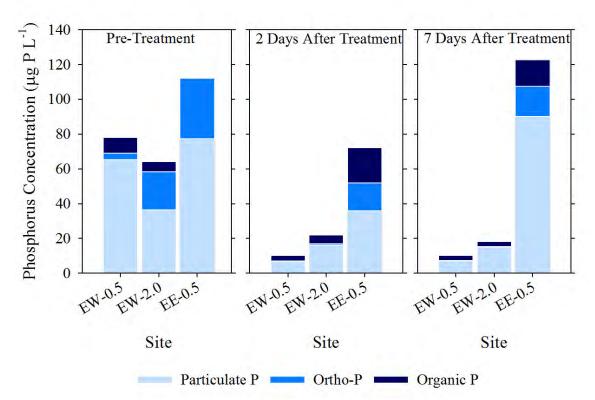


Fig. 8. Phosphorus concentrations in Edwards Pond West ("EW") at the depths of 0.5 and 2 m, and in Edwards Pond East ("EE") at the depth of 0.5 m.