Phosphorus Release from Bog Lake Muds[1]

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ABSTRACT

The paper reports the results of some laboratory investigations concerning the mud-water phosphorus relationship of some Northern Wisconsin and Upper Michigan bog lake muds as influenced by lime, acid, and mechanical agitation.

Calcium carbonate added to the water phase of mud-water systems reduced the amount of soluble phosphorus from 8.0 to 0.8 parts per billion. The lime formed a thin crust at the mud-water interface, while the original pH of the bottom material prevailed at a depth of one-fourth inch below the mud surface. Agitation of the mud cores by stirring resulted in complete lime penetration throughout a three-inch core.

Experimental apparatus in which it is possible to obtain a 12°C temperature differential for stratifying water is described.

Radioactive ordinary superphosphate fertilizer was placed at various depths below the mud surface, and the percentage as well as the amount of phosphorus released to the water was indicated to be very small with virtually no phosphorus released from depths greater than one-fourth inch below the mud surface. Agitation by stirring of the muds resulted in approximately twice the concentrations of soluble phosphorus in the water phase of the agitated systems as compared to the undisturbed systems. The most effective means of releasing fertilizer phosphorus placed one-fourth inch below the mud surface was found to be the acidification of previously limed mud-water systems.

The mechanism of phosphorus suppression by the addition of calcium compounds to mud water systems is suggested to be adsorption of phosphate ions onto the surface of the calcium particles in a similar manner as that described by Boischot *et al.*

Large quantities of phosphorus are consistently found deposited in lake muds, but often the quantities of soluble phosphorus found in the waters are very small. If a mechanism could be discovered to increase the release of the phosphorus from the bottom muds so that it could contribute to the production of algae, and hence fish, it would be a valuable contribution. This research reports the results of some laboratory investigations concerning the mud-water phosphorus relationship as influenced by lime, acid, and mechanical agitation.

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LITERATURE REVIEW

A number of investigators have reported on the mud-water relationships of phosphorus in lakes. Einsele (1936) found that an insoluble ferric phosphate is precipitated when ferrous iron and phosphate occur together in the hypolimnion of eutrophic lakes and oxygen is added and the reaction is alkaline. The excess iron is precipitated out as ferric hydroxide. When oxygen is lacking in the muds, iron is reduced from the ferric to the ferrous state and phosphorus is free to go into solution. Thus in small shallow lakes phosphorus in the hypolimnion is in a cycle consisting of precipitation during the period of circulation and solution during the period of stagnation.

Ohle (1935, 1938) and Einsele (1938) have pointed out the significance of colloidal gels as nutrient regulators in lakes. Precipitated ferric hydroxide gel suspended in water containing phosphate was found to adsorb the phosphate onto the ferric hydroxide gel. The adsorbed phosphate was again brought into solution by washing the gel with phosphate-free water.

Gessner (1939) found that the addition of calcium carbonate to natural waters caused a removal of phosphorus from solution. Gessner observed an approximately linear relationship between the addition of calcium carbonate and the decline in phosphorus content. Gessner also claimed that the removal of bicarbonate from solution by plants under conditions of high calcium content leads to the deposition of calcium carbonate and a reduction in soluble phosphorus content of the water.

Mortimer (1941, 1942) recorded the events in the hypolimnion after the onset of stratification and noted the accumulation of various nutrients in the water. The accumulation of nutrients in the water coincides with the increases in solubility accompanying reduction at the mud surface. The autumn oxidation period was accompanied by precipitation of the nutrients from the water.

Hutchinson and Bowen (1947, 1950) using radioactive phosphorus in Linsley Pond, Connecticut, noted that on introduction practically the whole of the phosphorus enters the phytoplankton and is sedimented. These investigators also noted that phosphorus passes into the littoral vegetation and from such vegetation into the free water again. A phosphorus cycle was postulated whereby the phosphorus liberated from the muds and entering the illuminated layers of water is taken up by phytoplankton and later sedimented out as a fine rain of particulate matter consisting of partly dead plankton and feces of zooplankton. The resultant movement of phosphorus is believed to be horizontal into the free water as phosphate ions and vertically as seston.

Hayes *et al.* (1952) added radioactive phosphorus to a small unstratified lake in Nova Scotia. These investigators postulated an active exchange between the water phosphorus and participating phosphorus in the lake solids, which together make up a single system. They hold untenable the proposition that some enhancement of plant or animal growth takes place.

EXPERIMENTAL APPARATUS

Pyrex glass tubing in units four feet long and 100 mm in diameter was used as containers for the mud-water systems. The bottom end of each tube was sealed with a large rubber stopper fastened to the tube with electrical "Scotch" tape. Two-quart cylindrical ice cream containers were placed in each tube to serve as containers for the bottom muds. The tubes were mounted in a vertical position in an aquarium, and a 15-watt electric light bulb was fastened in the top of each tube to serve as a heating unit to produce thermal stratification.

Thermal gradients were applied to the systems to reduce the amount of turbulent and convectional diffusion. A 12°C temperature-difference between the water above the mud surface and the water at the top of the column was produced by circulating cold lake water in the aquarium and employing the electrical heating system.

EXPERIMENTAL PROCEDURE

Natural hypolimnetic mud was procured with an Ekman dredge from Hummingbird and Moccasin lakes. The pH of the muds in the dry state as determined by the Hellige-Truog method ranged from 5.2 to 5.5.

To measure the depth of lime penetration into the bottom mud and the soluble phosphorus content of the water above the mud the following procedure was used. Bottom mud from Moccasin Lake was placed in a four-liter beaker to an approximate depth of three inches. Three liters of phosphorus-free distilled water were placed over the bottom mud with a minimum of disturbance to the mud surface. Lime in the form of calcium carbonate was applied at the rate of 0, 250, and 500 pounds per acre to the surface of the water and allowed to settle for a period of six weeks. The water surfaces of the mud-water systems were exposed to the air but protected from dust. At the end of the settling period the water was removed with a minimum of disturbance to the mud surface. The bottom mud was air dried to a firm consistency after which it was divided into a thin surface section and three one-inch depth sections. The pH of the muds was determined by the Hellige-Truog method and the pH of the water by the glass electrode method. The soluble phosphorus was determined by the development of the molybdenum-blue color according to the method of Truog and Meyer (1929).

To measure the depth of lime penetration and its effect upon the suppression or release of phosphorus in an agitated system the same procedure was followed as that described for the undisturbed system with the following exceptions. Calcium carbonate was applied at the rate of 5 tons per acre to the surface of the water in one container at the time of agitation and to the surface of the water in the second container 72 hours after agitation. The third container served as a control and was agitated but did not receive an application of calcium carbonate. Agitation consisted of vigorously stirring the entire mud-water system with a glass rod for a period of two minutes. The bottom mud used in this experiment was from Hummingbird Lake.

Experiments designed to measure the percentage of ordinary superphosphate fertilizer in the water at various levels above the mud surface as related to placement of the fertilizer at various depths beneath the mud surface were carried out using the glass tube systems already described. P^{32} in the form of ordinary superphosphate fertilizer was obtained from the Plant Industry Station at Beltsville, Maryland. The fertilizer analyzed 20.4 per cent P_2O_5 and had a specific activity of 0.15 millicuries per gram of P_2O_5. Specific amounts of P^{32} superphosphate fertilizer were placed on the surface of the mud and at depths of one-fourth, one-half, and one inch below the surface in the four experimental tubes. Natural hypolimnetic mud from Hummingbird Lake was used in all tubes. Each tube was then filled with seven liters of phosphorus-free distilled water and allowed to stand for a period of 15 days, after which the water was sampled in one-liter aliquots at heights of 3, 18, and 32 inches above the mud surface. The water samples were then analyzed for radioactive phosphorus content.

One-liter samples of water were evaporated to a volume of less than 100 ml on a hot plate, filtered through Whatman 42 filter paper, and made up to volume in a 100-ml volumetric flask. An aliquot of the solution such that it would give less than 300 mg of $MgNH_4PO_4 \cdot 6H_2O$ precipitate was taken, and the radioactive phosphorus along with any additional phosphorus present was precipitated as magnesium ammonium phosphate according to a procedure adapted from Kolthoff and Sandell (1947). The magnesium ammonium phosphate precipitate was collected on an aluminum filter ring and prepared for counting in the Geiger-Mueller counter according to the method proposed by MacKenzie and Dean (1948).

An experiment designed to obtain information pertaining to the relationship between the amounts of phosphorus released by the fertilizer placed at various depths beneath the mud surface and the amount of mud and fertilizer surface exposed to the water was set up in a similar manner to the previously described experiment with the following exception. After the phosphorus-free water was added to the tubes each mud-water system was agitated with a glass rod for a period of two minutes after which the systems were allowed to stand for 15 days. The agitation as in the previous experiments consisted of vigorous stirring.

The tube experiments were repeated a third time to investigate the release or suppression of fertilizer phosphorus in the bottom muds as effected by applications of lime and sulphuric acid to the water phase of the mud-water systems. In the third experiment the radioactive phosphate fertilizer was placed one-fourth inch below the mud surface in all four tubes. The tubes were then treated as follows: No. 1 served as a control; No. 2 was limed with calcium hydroxide at the rate of 160 pounds per acre, and after 15 days was acidified to pH 3.4 with sulphuric acid; No. 3 received an application of 80 pounds per acre of calcium hydroxide; and No. 4 was limed at the rate of 40 pounds per acre calcium hydroxide. The calcium hydroxide form of lime was added in preference to the calcium carbonate form because of the higher neutralizing value of the hydroxide form.

RESULTS

Applications of lime in the form of calcium carbonate to the surface water of undisturbed mud-water systems formed thin crusts of lime at the mud-water interface. The data presented in Table 1 show the increased pH value in the mud surface layer. At the 500-pound-per-acre level of calcium carbonate the surface crust had an alkaline pH value, while the pH values of the deeper layers remained acidic.

The amount of phosphorus (8.0 parts per billion) released by the mud and present as soluble phosphorus in the water phase of the control system was of the magnitude found by Juday and Birge (1931) in northeastern Wisconsin lakes. Calcium carbonate added to the water phase of the mud-water systems reduced the amount of soluble phosphorus in the water to a trace—0.5 and 0.8 parts per billion.

Agitation or stirring of the mud-water systems following applications of calcium carbonate resulted in penetration of lime throughout the three-inch core of mud, as shown in Table 2. There was no apparent difference in the depth of lime penetration if

TABLE 1. *The pH values of bottom mud from Moccasin Lake at various depths below the mud-water surface, and the water soluble phosphorus content of limed and unlimed undisturbed systems in a laboratory experiment*

Treatment (lbs. $CaCO_3$ per acre)	Bottom mud			Water		
	Depth (inches)	pH		pH		Soluble phosphorus (parts per billion)
		Before treatment	6 weeks after treatment	Before treatment	6 weeks after treatment	
Control	surface	5.5	5.5	5.65	5.65	8.0
	1/4	5.5	5.5			
	1–2	5.5	5.5			
	2–3	5.5	5.5			
250	surface	5.5	6.8	5.65	6.80	0.5
	1/4	5.5	5.5			
	1–2	5.5	5.5			
	2–3	5.5	5.5			
500	surface	5.5	7.5	5.65	7.05	0.8
	1/4	5.5	6.0			
	1–2	5.5	5.5			
	2–3	5.5	5.5			

TABLE 2. *The pH values of bottom mud from Hummingbird Lake at various depths below the mud-water surface, and the water soluble phosphorus content of limed and unlimed agitated systems in a laboratory experiment*

Treatment (5 tons $CaCO_3$ per acre)	Bottom mud			Water		
	Depth (inches)	pH		pH		Soluble phosphorus (parts per billion)
		Before treatment	6 weeks after treatment	Before treatment	6 weeks after treatment	
Control	surface	5.3	5.3	5.65	5.55	6.0
	1–2	5.3	5.3			
	2–3	5.3	5.3			
Added at time of agitation	surface	5.3	8.3	5.65	7.52	0.3
	1–2	5.3	8.3			
	2–3	5.3	8.3			
Added 72 hrs. after agitation	surface	5.3	8.3	5.65	7.63	0.4
	1–2	5.3	8.3			
	2–3	5.3	8.3			

the lime was applied directly before agitation or 72 hours later. This lack of difference can possibly be attributed to the slow rate of sedimentation following the vigorous stirring procedure. Approximately the same concentrations of soluble phosphorus were present in the water of the control and experimental systems for both the unagitated and agitated procedures (compare Tables 1 and 2).

The percentage as well as the amount of phosphorus released to the water from radioactive superphosphate fertilizer placed at various depths below the mud surface in an undisturbed mud-water system was indicated to be very small, as shown by the data presented in Table 3 and as previously reported by Hayes *et al.* (1952) in their studies of undisturbed cores taken with a Jenkins sampler. There was virtually no release of phosphorus from fertilizer placed at depths greater than one-fourth inch below the mud surface. There was a higher percentage of soluble phosphorus contained in the water samples taken near the mud surface than in water samples taken at greater distances above the mud surface. The radiophosphorus placed one-half inch below the mud surface showed only a very slight tendency to diffuse into the water, while the radiophosphorus placed at the one inch depth did not diffuse into the water at all. The concentrations of soluble phospho-

TABLE 3. *Diffusion and convection of P^{32} superphosphate fertilizer in an undisturbed mud-water system*

Water samples collected after 15 days.

Placement of fertilizer below mud surface (inches)	Fertilizer added (grams)	Relative specific activity of fertilizer (counts per second)	Height of water sample above mud surface (inches)	Sample (zero count per second)	Per cent uptake by water
surface	2.0052	130,739	32	1539	1.177
			18	1333	1.020
			3	1838	1.406
0.25	2.0193	131,658	32	579	0.440
			18	667	0.506
			3	1392	1.058
0.50	1.8385	119,870	32	76	0.063
			18	66	0.055
			3	177	0.148
1.00	1.9963	130,159	32	0	0.000
			18	0	0.000
			3	0	0.000

Note: A thermal gradient of 8°C was established and maintained between the mud surface and a point 12 inches above the mud surface.

rus in the water phase of each treatment based upon the average of the three water samples taken from each treatment were 2.1, 1.2, 0.1, and 0.0 parts per billion arranged in order of increasing depth of fertilizer placement below the mud surface.

Agitation or stirring of the bottom muds containing radioactive ordinary superphosphate fertilizer at the same depth placements as in the undisturbed systems resulted in approximately twice the concentrations of soluble phosphorus in the water phases of the agitated mud-water systems, as shown by a comparison of Tables 3 and 4. The concentrations of soluble phosphorus in the water phase of each treatment based upon the average of the three water samples taken from each treatment were 3.9, 2.9, 2.7, and 1.2 parts per billion arranged in order of increasing depth of fertilizer placement below the mud surface.

Acidification of previously limed mud-water systems proved to be the most effective means of releasing fertilizer phosphorus "planted" one-fourth inch below the mud surface. As shown in Table 5 the acidification of pH 3.4 with sulphuric acid of the water phase of a mud-water system that had previously been limed with calcium hydroxide at the rate of 160 pounds per acre caused a greater release of phosphorus to the water than the control or limed systems. The percentage of phosphorus released from mud to water decreased as the rate of liming increased.

TABLE 4. *Diffusion and convection of P^{32} superphosphate fertilized in an agitated mud-water system*

Water samples collected after 15 days.

Placement of fertilizer below mud surface (inches)	Fertilizer added (grams)	Relative specific activity of fertilizer (counts per second)	Height of water sample above mud surface (inches)	Sample (zero count per second)	Per cent uptake by water
surface	1.9883	129,637	32	2771	2.137
			18	2805	2.164
			3	3065	2.364
0.25	2.2174	144,574	32	2107	1.458
			18	2144	1.483
			3	2331	1.612
0.50	1.9944	130,035	32	1891	1.454
			18	1828	1.406
			3	2025	1.557
1.00	1.9545	127,433	32	855	0.671
			18	932	0.732
			3	875	0.687

TABLE 5. *The effect of liming and acidification upon the diffusion and convection of P^{32} superphosphate fertilizer in a mud-water system*

Treatment	Fertilizer added (grams)	Relative specific activity of fertilizer (counts per second)	Height of water sample above mud surface (inches)	Sample (zero count per second)	Per cent uptake by water
Control	1.1909	77,595	32	485	0.625
			18	555	0.715
			3	573	0.739
160 lbs. $Ca(OH)_2$ per acre for 15 days; acidified to pH 3.4 for 15 days	1.6374	106,758	32	1102	1.033
			18	1274	1.193
			3	1192	1.117
80 lbs. $Ca(OH)_2$ per acre for 30 days	1.0143	66,132	32	39	0.059
			18	31	0.047
			3	48	0.073
40 lbs. $Ca(OH)_2$ per acre for 30 days	1.1128	72,555	32	104	0.143
			18	122	0.168
			3	155	0.214

DISCUSSION

Located near the Upper Michigan-Wisconsin boundary in Vilas County, Wisconsin, and Gogebic County, Michigan, are a large number of soft-water bog lakes. These lakes are dystrophic in nature, low in nutrient content as well as in soluble and total iron content, and they have a high content of organic matter which lends a dark color to the water.

Lime has been added to a number of these lakes in an effort to increase the depth of light penetration into the water. In addition the lime prevents summer-kill due to high temperatures by providing oxygen in the cooler water of the thermocline, and prevents winter-kill in marginal cases by reducing the amount of oxygen consuming colloidal organic matter (Hasler *et al.* 1951).

In pursuing the above management procedure further, changing the reactions of the water and bottom muds by the addition of lime or some other chemical might have the effect of liberating plant nutrients such as phosphorus into the water. This would ultimately increase the growth of phytoplankton and zooplankton thus increasing the fish carrying capacity of the lakes.

Phosphorus occurs in soils in both the organic and inorganic form. There has been evidence presented that some mineralization of organic phosphorus in the soil occurs (Schollenberger 1920, Robinson 1937), and that this phosphorus is of value in the nutrition of higher plants only when it is changed to the inorganic form (Eid, Black, and Kempthorne 1951, 1953). In the case of lower plants Chu (1946) has reported that the growth of *Phaeocystis Pouchetii* was a little better and lasted longer when phytin was supplied to natural sea water as a source of phosphorus than when orthophosphate was supplied.

The depressing effect of calcium upon the solubilities of the various phosphates has been pointed out by Larson (1935), Gaarder (1930), McGeorge and Breazeale (1931), McGeorge (1939), and Gessner (1939). The mechanism of this reaction has been described by McGeorge and Breazeale (1931) as the formation of a new compound of lower solubility such as hydroxylapatite. Another possible mechanism of this reaction has been described by Boischot *et al.* (1950), who stated that a new compound is not necessarily formed, but instead the phosphate ions are adsorbed on the surface of the calcium carbonate crystals as an initial reaction. Cole *et al.* (1953) confirmed this theory and advanced the idea of monolayer adsorption of phosphate ions by calcium particles.

In an alkaline or neutral medium calcium phosphates are virtually insoluble; however, in soils the calcium phosphate compounds represent an easily available form of phosphorus for plant nutrition because of the solubility of the phosphorus compounds in dilute acids of a strength corresponding to that produced by plant roots.

Based on the previously stated theories it is therefore conceivable that when calcium in the form of lime is added to the surface water it adsorbs the phosphate ions onto the calcium particles as they fall through the water. The portion of the calcium particles that settle into the bottom material and upon the mud surface is believed to act in two ways. The calcium particles that settle on the mud surface might in effect form a seal that prevents or greatly restricts the diffusion of any phosphate ions released by the muds to the water phase of the system. It is also suggested that a portion of the calcium particles entering the mud phase of the system converts some of the iron and aluminum held phosphorus compounds to calcium phosphates. Subsequent acidification of the water phase of the mud-water system releases some of the calcium held phosphorus to the water in the same manner as plant root acids act upon soils. Likewise rooted aquatics growing in the littoral zone would have more phosphorus available for their nutrition from calcium phosphates than from iron and aluminum phosphates.

The results obtained by the use of radioactive phosphorus in agitated or stirred muds suggests the possibility of raking or harrowing the muds of small bays or ponds as a method of releasing more phosphorus into the water. Hayes *et al.* (1952) suggest that the thickness of the mud layer participating in the exchange is approximately one millimeter. Therefore it would seem feasible that the release of phosphorus from mud is a function of the amount of surface exposed to water rather than thickness of the mud layer. Thus by harrowing, discing, or raking the bottom muds the amount of mud surface exposed to water and therefore able to participate in the exchange process would be greatly increased.

It should be pointed out that Ohle (1935, 1937) measured considerable amounts of phosphorus leeching out of alkalized bog lake muds. The lack of acid production by bacterial action might be attributed to the low bacterial population supported by the impoverished sandy soils that predominate in the region where these lakes occur. It should also be noted that leaching is a process where liquids are actively moving through a solid medium and is quite unlikely to occur in lakes while diffusion is one of the established processes by which nutrients are released from mud to water in mud-water systems.

SUMMARY

1. The results of some laboratory investigations concerning the mud-water phosphorus relationship as influenced by lime, acid, and mechanical agitation are reported.

2. Experimental apparatus in which it is possible to obtain a 12°C temperature gradient in water is described.

3. Applications of lime in the form of calcium carbonate to the surface water of undisturbed mud-water systems formed thin crusts of lime at the mud-water interface, while the original pH of the bottom material prevailed at a depth of one-fourth inch below the mud surface.

4. Calcium carbonate added to the water phase of mud-water systems reduced the amount of soluble phosphorus from 8.0 to 0.8 parts per billion.

5. Agitation of mud-water systems following applications of lime resulted in complete lime penetration throughout a three inch mud core.

6. The percentage as well as the amount of phosphorus released to the water from radioactive ordinary superphosphate fertilizer placed at various depths below the mud surface in an undisturbed mud-water system was indicated to be very small, with virtually no phosphorus released from depths greather than one-fourth inch.

7. Agitation of the bottom muds containing radioactive ordinary superphosphate fertilizer at the same depth placements as in the undisturbed systems resulted in approximately twice the concentrations of soluble phosphorus in the water phases of the agitated systems as compared to the undisturbed systems.

8. Acidification of previously limed mud-water systems proved to be the most effective means of releasing fertilizer phosphorus placed one-fourth inch below the mud surface.

9. The percentage of phosphorus released from mud to water decreased as the rate of liming increased.

10. The mechanism of phosphorus suppression by the addition of calcium compounds to mud-water systems is suggested to be adsorption of phosphate ions onto the surface of the calcium particles in a manner similar to that described by Boischot *et al.*

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