

Video Article

Laboratory-determined Phosphorus Flux from Lake Sediments as a Measure of Internal Phosphorus Loading

Mary E. Ogdahl¹, Alan D. Steinman¹, Maggie E. Weinert¹¹Annis Water Resources Institute, Grand Valley State UniversityCorrespondence to: Mary E. Ogdahl at ogdahlm@gvsu.eduURL: <https://www.jove.com/video/51617>DOI: [doi:10.3791/51617](https://doi.org/10.3791/51617)

Keywords: Environmental Sciences, Issue 85, Limnology, internal loading, eutrophication, nutrient flux, sediment coring, phosphorus, lakes

Date Published: 3/6/2014

Citation: Ogdahl, M.E., Steinman, A.D., Weinert, M.E. Laboratory-determined Phosphorus Flux from Lake Sediments as a Measure of Internal Phosphorus Loading. *J. Vis. Exp.* (85), e51617, doi:10.3791/51617 (2014).

Abstract

Eutrophication is a water quality issue in lakes worldwide, and there is a critical need to identify and control nutrient sources. Internal phosphorus (P) loading from lake sediments can account for a substantial portion of the total P load in eutrophic, and some mesotrophic, lakes. Laboratory determination of P release rates from sediment cores is one approach for determining the role of internal P loading and guiding management decisions. Two principal alternatives to experimental determination of sediment P release exist for estimating internal load: *in situ* measurements of changes in hypolimnetic P over time and P mass balance. The experimental approach using laboratory-based sediment incubations to quantify internal P load is a direct method, making it a valuable tool for lake management and restoration.

Laboratory incubations of sediment cores can help determine the relative importance of internal vs. external P loads, as well as be used to answer a variety of lake management and research questions. We illustrate the use of sediment core incubations to assess the effectiveness of an aluminum sulfate (alum) treatment for reducing sediment P release. Other research questions that can be investigated using this approach include the effects of sediment resuspension and bioturbation on P release.

The approach also has limitations. Assumptions must be made with respect to: extrapolating results from sediment cores to the entire lake; deciding over what time periods to measure nutrient release; and addressing possible core tube artifacts. A comprehensive dissolved oxygen monitoring strategy to assess temporal and spatial redox status in the lake provides greater confidence in annual P loads estimated from sediment core incubations.

Video Link

The video component of this article can be found at <https://www.jove.com/video/51617/>

Introduction

As a growing number of lakes worldwide suffer from cultural eutrophication, determination of the causes of water quality degradation is becoming increasingly important for lake management and restoration. Phosphorus (P) loading to lakes is generally implicated in eutrophication, as it is most often the nutrient limiting algal growth¹. Historically, quantification of P loading to lakes focused on external sources, or P originating in the watershed via point and nonpoint sources. However, internal loading from lake sediments can account for a large portion, if not the majority, of the total P load in eutrophic lakes²⁻⁵. Thus, even substantial reductions in external loading to lakes can fail to result in water quality improvement due to the overriding effect of P release from sediments⁵⁻⁸. Because of the ecological and societal implications of P loading, including the cost and difficulty of P control, it is critical that P loads be accurately identified prior to enacting a management strategy.

At least two different mechanisms are responsible for phosphorus release from sediments. 1) During periods of anoxia or hypoxia, reducing conditions can result in the desorption of phosphate from iron oxyhydroxides at the sediment-water interface, causing diffusion of dissolved phosphate from the sediments into the water column⁹⁻¹¹. 2) Disturbance of the sediment surface, through wind-induced resuspension and bioturbation, can result in the release of P into the water column by either desorption of P from resuspended sediment particles or release of dissolved P from the sediment pore water to the water column, respectively¹¹⁻¹³.

Three principal approaches are available for quantifying internal P loading to lakes^{14,15}. (1) *In situ* measurements of changes in hypolimnetic total phosphorus (TP) over time can be used when monitoring data are available. Internal load estimates based on *in situ* measurements suffer from high variability associated with the inherent spatial and temporal variability of environmental data and can be affected by inadequate monitoring frequency¹⁴. (2) Mass balance can be used to estimate internal loading, when complete P budgets can be constructed. However, it is rare that sufficient data are available on P inputs and exports to construct a complete P budget¹⁶. (3) Experimentally-determined sediment P release rates can be used, in combination with information on areal extent and duration of P release (*i.e.* anoxic period), to calculate internal P load. This is a direct method of internal P load quantification, although it too has limitations (see below).

Because management decisions often must be made on compressed time scales due to funding restrictions or societal pressures, experimental determination of internal P load can have greater utility for lake management and restoration since it requires less time and data than the *in*

situ and mass balance approaches. Laboratory incubations of sediment cores, combined with monitoring of external loads, have been used to determine the relative contributions of internal and external P loads, with the goal of guiding management decisions to optimize nutrient source control^{2,4,17}. In two Michigan lakes with extensive shoreline development and high percentages of impervious surface (>25%) in the sub-basins directly adjacent to the lake, internal P load was estimated to account for up to 80% of the total P load, prompting recommendations to focus management efforts on reducing sediment P release^{2,4}. In contrast, experimental studies of sediment from a less developed lake in the same region showed that internal loading composed only 7% of the total P load, prompting a recommendation to focus P management strategies in the watershed¹⁷. Sediment core experiments also have been used in a Michigan lake to determine the potential effectiveness of aluminum sulfate (alum) treatment to reduce sediment P release rates², the most efficient alum dosing concentration and the effects of sediment resuspension¹³, and the efficacy of an *in situ* alum treatment 1 year¹⁸ and 5 years¹⁹ following treatment. Experimental determination of internal P load is an effective approach to providing answers to key management questions in eutrophic lakes.

Protocol

1. Field Sampling

1. Conduct sampling once during each ice-free season (where applicable) for 1-2 years, if possible (*i.e.* 3 times/year in a north temperate climate). If time and/or funds prohibit seasonal sampling, conduct sampling once per year during mid-to-late summer.
2. Select sediment collection sites to cover different geographic regions within the lake. Choosing locations close to historical water quality and/or sediment sampling sites, when available, is often desirable to take advantage of historical data. Otherwise, attempt to select sites that represent different sediment types in the lake.
3. Conduct water quality sampling prior to sediment core collection.
 1. At a minimum, measure water depth and vertical profiles of water temperature and dissolved oxygen. Near-bottom measurements should be taken as close to sediment surface as possible, without disturbing the sediment.
 2. Collect any other water quality data and samples that are desired to fulfill the specific goals of the study. Examples include vertical profiles of pH, conductivity, and turbidity; Secchi depth; photosynthetically active radiation (PAR) profiles; chlorophyll *a*; soluble reactive phosphorus (SRP); total phosphorus (TP); and nitrogen species.
4. At each sampling location, fill a 10 L carboy with water collected 1 m above the sediment surface using a Van Dorn or Niskin bottle. This will be used in the initial set up of sediment cores in the lab and for refilling cores after sampling during incubation. Place the carboy in a cooler with ice.
5. Collect 6 sediment cores per site using a piston corer^{2,20}.
 1. Refer to Fisher *et al.*²⁰ for specific instructions regarding construction of the coring device. Briefly, the coring device consists of a graduated 0.6 m long polycarbonate core tube (7 cm i.d.), a polyvinyl chloride (PVC) attachment assembly for coupling to aluminum drive rods, a piston constructed of two rubber stoppers and an eye bolt, a plastic-coated piston cable with a swivel clip, and aluminum drive rods. Assemble the coring device according to the following steps:
 1. Thread the swivel clip end of the piston cable through the top of the PVC attachment assembly. Orient a core tube with bolt holes facing upward and extend the cable through the length of the core tube. Clip the piston cable to the eye bolt of the piston stopper.
 2. Attach the core tube to the PVC attachment assembly using a wire lock hitch pin. Pull the piston cable to advance the piston 20 cm from the bottom of the core tube to maintain a water layer on top of the sediment surface during core collection.
 3. Attach an aluminum drive rod to the other end of the PVC attachment assembly using a wire lock hitch pin. Lower coring device vertically into the water, adding additional sections of aluminum drive rod as needed.
 2. Position the corer vertically at the sediment-water interface and push downward, with the piston cable remaining stationary. To accomplish this, pull the piston cable taut once the corer is in place at the sediment-water interface, attach vice grips to cable, step on the cable to the inside of the vice grips, and then push downward.
 3. Bring core to the surface and seal with a rubber stopper prior to breaking the water surface. Secure bottom stopper with duct tape.
 4. Bolt the piston to the top of the core tube to keep it stationary during transit. Place the core tube in a vertical rack and maintain at ambient near-bottom lake temperature, using ice if necessary.

2. Laboratory Incubation

1. Upon return from the field, adjust cores to contain the desired depth of sediment and overlying water column. Excess sediment can be carefully let out the bottom of the core tube by removing the bottom stopper; add water from the carboy collected at the corresponding site, if needed. Commonly-used sediment and water column depths are 20 cm of sediment with a 25 cm overlying water column^{2,4,13,17-19}, but these amounts can be modified as desired.
2. Place sediment core tubes into a darkened environmental growth chamber, with the temperature maintained to match ambient bottom water temperatures measured in the field.
3. Expose the cores to redox treatments. For the oxic treatment, bubble the water column of 3 cores/site with air. Bubble the water column of the remaining three cores per site with N₂ (with ~350 ppm CO₂ to buffer pH) for the anoxic treatment. Ensure a slow and consistent bubble rate that is nondisruptive to the sediment surface.
4. On day 1 of core incubation, filter each 10 L carboy containing near-bottom water collected from each site in the field. Using a peristaltic pump and filter cartridge housing, filter water first through a 1 μm filter, followed by a 0.2 μm filter. Store filtered water at 4 °C for the duration of the core incubation.
5. Sample cores for P release rate over the duration of the incubation period^{2,3}. Because this is a redox-sensitive experiment, take precautions to maintain redox treatment conditions whenever possible.
 1. With a syringe, remove a 40 ml water sample through the sampling port of each sediment core on days 0 (*i.e.* at the time cores are placed in the growth chamber), 1, 2, 4, 6, 8, 12, 20, 24, and 28 of core incubation. (Note: if changes over very short time frames are

desired, the sampling regime can be modified to sample at hr 1, 2, 4, 8, etc. However, the system is often still equilibrating through the first 12 hr, so P release dynamics can be quite variable at the start of incubations.)

2. Immediately after removal, dispense a 20 ml subsample into a scintillation vial and refrigerate for analysis of TP. Filter the other 20 ml subsample through a 0.45 µm membrane filter and into a scintillation vial and freeze for analysis of SRP.
3. Replace the 40 ml subsample with an equal volume of filtered water (see step 2.4) from the corresponding site.

3. P Release Rate Calculation

1. Calculate flux (release rate) based on the change in water column TP or SRP using the following equation²:

$$P_{rr} = (C_t - C_0) \times V / A$$

where P_{rr} is the net P release (positive values) or retention (negative values) rate per unit surface area of sediment ($\text{mg P/m}^2/\text{d}$), C_t is the TP or SRP concentration in the water column at time t , C_0 is the TP or SRP concentration at time 0, V is the volume of water in the water column of the core tube, and A is the planar surface area of the sediment cores. Calculate P release rate using the linear portion of the concentration vs. time curve to give the maximum apparent release rate^{4,13,18,19}. To avoid potential short-term bias, choose nonconsecutive sampling dates for C_t and C_0 ^{18,19}.

4. Internal P Load Calculation

1. Calculate annual P flux.
 1. For each season during which sampling occurred, multiply anoxic and oxic flux individually by the number of days in that season. Sum the seasonal values to yield annual anoxic and oxic flux ($\text{mg/m}^2/\text{year}$). If multiple sites in the same lake were sampled, this calculation can be performed either separately for each site or using the mean flux values for all sites (see Section 4.2.2).
 2. P release from sediments is generally very low during winter due to low water temperatures. If sampling was not conducted during winter, assume that P flux was 0 for that season^{14,15}.
 3. Because the majority of internal P release occurs during summer, annual internal P flux can be coarsely estimated from summer measurements alone in the absence of seasonal data^{2,15,17}. For this approach, calculate the P flux according to Section 4.1.1 and assume 0 flux for all seasons except summer. Recognize that this will be a conservative estimate of annual P release.
 4. If available, dissolved oxygen data may be used to refine the annual P flux calculation^{2,4}. Such data may reveal that a lake experiences hypoxia or anoxia for a certain percentage of the year, or during specific seasons. In those cases, use anoxic and oxic flux according to the appropriate proportion or season and sum the values to calculate annual internal P flux.
 1. For example, if hypoxia or anoxia was measured only during summer, calculate Section 4.1.1 using anoxic flux for summer and oxic flux for the remaining seasons. Sum the values to obtain annual internal P flux. Similarly, if routine dissolved oxygen monitoring data indicate that the lake experiences hypoxia or anoxia 35% of the year, multiply annual anoxic flux from Section 4.1.1 by 0.35 and annual oxic flux from Section 4.1.1 by 0.65 and sum the values to calculate annual internal P flux.
 5. Polymictic lakes pose a particular challenge to internal P load calculation, due to their frequent mixing and spatial and temporal variability in redox condition¹⁴. Nürnberg *et al.*¹⁶ developed a model to calculate the number of anoxic days a polymictic lake may experience during a season or year. The active sediment release area and time (AA), which represents the length of time (days/season) that an area similar to the lake surface area is actively releasing P, can be calculated as follows:

$$AA = -36.2 + 50.2 \log(P_{\text{season}}) + 0.762 z/A^{0.5}$$
 where P is the average water column TP concentration during a given season, z is mean depth, and A is lake surface area. To calculate annual internal P flux, multiply AA by anoxic flux and the number of oxic days by oxic flux for each season, and then sum all values.
2. Scale up internal P flux to the entire lake area.
 1. Multiply the annual P flux from step 4.1 by the entire lake surface area to calculate annual internal P load. Unless annual P flux was calculated according to Sections 4.1.4 or 4.1.5, use the anoxic annual flux to calculate annual internal P load. Otherwise, use the flux calculated in Sections 4.1.4 or 4.1.5.
 2. If multiple sites in the same lake were sampled, the lake can be divided into geographic zones associated with each site. Multiply the annual anoxic P flux (or annual flux from Sections 4.1.4 or 4.1.5) for each site by the surface area of the zone, then sum the values to obtain annual internal P load for the entire lake^{4,17}. Alternatively, the mean annual P flux of all sites can be used in Section 4.2.1.
 3. Detailed dissolved oxygen data may indicate that specific areas of the lake experience hypoxic or anoxic conditions (e.g. deep areas), while other areas remain oxic year round. If available, use this information to refine the flux \times area calculation (Steinman *et al.*, in prep). Multiply the anoxic surface area by the annual anoxic flux and multiply the oxic surface area by the annual oxic flux, and sum the two values to calculate annual internal P load.

Representative Results

Internal P release was measured from sediment cores collected in Mona Lake, Michigan, to identify the relative contribution of internal versus external P loads⁴. Four sites were sampled over three seasons to estimate annual internal P load, accounting for spatial variation in P flux. Sediment cores were incubated for 20-28 days under anoxic and oxic conditions, and the overlying water column was sampled for SRP and TP concentrations at regular intervals during the incubation period. The anoxic treatment triggered SRP and TP release from the sediments; however, we are presenting only the TP flux results for illustrative purposes. TP concentrations were highest during summer in anoxic treatments, and spatial variability in TP release was evident during all seasons (**Figure 1**). Mean internal TP flux was less than 1.4 $\text{mg P/m}^2/\text{day}$ in all oxic cores; negative flux values at 3 of the 4 sites during fall indicated that oxic sediments were acting as a sink rather than a source of P during that season⁴ (**Table 1**). TP release rates were considerably higher in anoxic cores, with flux as high as 15.56 $\text{mg P/m}^2/\text{day}$ in the summer

and as low as 0.80 mg P/m²/day in the spring⁴ (Table 1). These flux values were used to calculate seasonal internal P flux based on dissolved oxygen conditions measured at the time of sediment core collection⁴. Seasonal internal P load was calculated by scaling up the flux at each site to the surface area of the corresponding geographic zone⁴; seasonal values were summed to estimate annual internal P load, assuming 0 flux during winter. Annual internal P load was estimated to be 3.4 metric tons, with the majority of the load occurring during summer (Table 2). Comparing these results with concurrent external P load estimates, it was estimated that the sediments in Mona Lake contribute between 9-82% of the total annual P load⁴ (Table 2).

A series of experiments was conducted in Spring Lake, Michigan, to determine 1) the potential effectiveness of aluminum sulfate (alum) treatment in reducing internal P loading² and 2) the efficacy of an *in situ* alum treatment^{18,19}. Laboratory experiments simulating a lake-wide application of alum demonstrated a dramatic decline in internal P release with treatment² (Figure 2). Similar to the example above, we are presenting only TP release from these experiments as representative results. In anoxic cores without alum treatment (simulating natural summer conditions in Spring Lake sediments), mean TP concentrations in the overlying water column reached more than 1.2 mg/L (Figure 2). In contrast, anoxic cores dosed with alum had virtually no P release and concentrations were not different from either of the oxic treatments² (Figure 2). A sediment core incubation conducted 1 year following lake-wide application of alum in Spring Lake revealed that the treatment was highly effective at reducing sediment P release, with release rates similar between anoxic and oxic treatments¹⁸ (Figure 3A). When the experiment was repeated 5 years following alum treatment, TP release remained substantially lower than pretreatment but was greater than that measured 1 year following treatment, suggesting a slight decline in alum efficacy¹⁹ (Figure 3B).

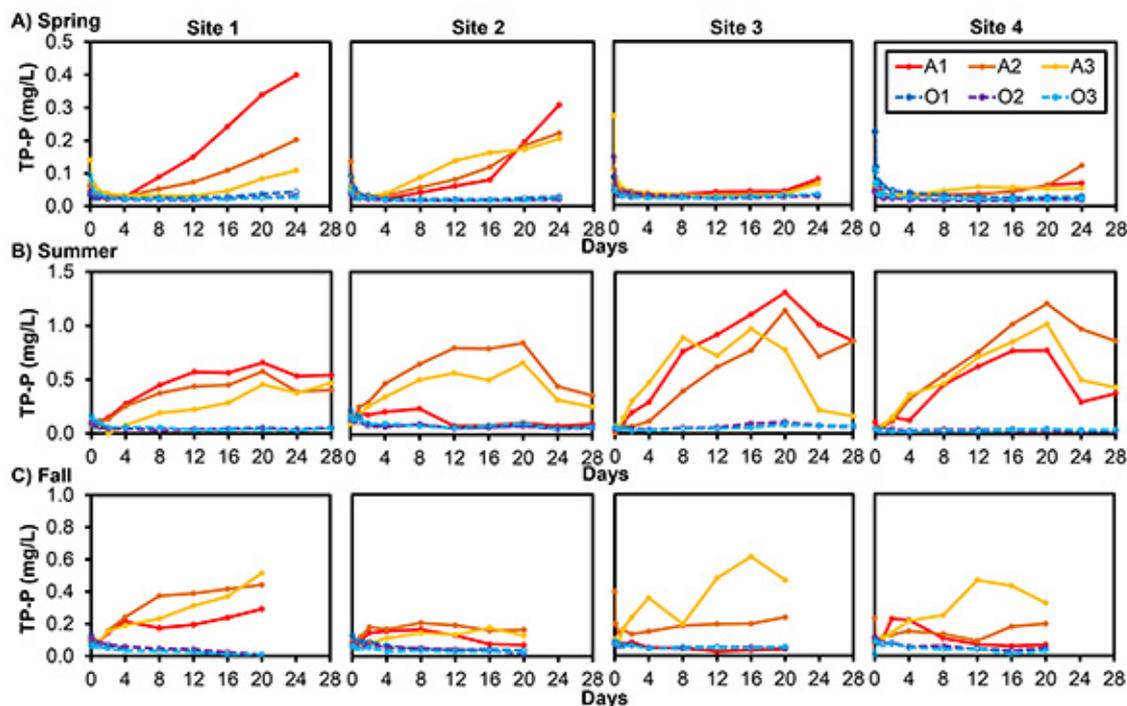


Figure 1. Total phosphorus (TP) concentrations (mg/L) measured during laboratory incubations of sediment cores collected from Mona Lake, Michigan, during spring (A), summer (B), and fall (C)⁴. TP was measured in the water overlying sediment cores from 4 lake sites over a 20- to 28-day incubation. The letter in the legend refers to redox state (A = anoxic treatment; O = oxic treatment); the number refers to replicate number (1-3). Note the different scales on the y-axes among seasons. [Click here to view larger image.](#)

Season	Site	Anoxic flux, mg P/m ² /day	Oxic flux, mg P/m ² /day
Spring	1	2.77 ± 1.53	0.25 ± 0.01
	2	2.82 ± 0.83	0.26 ± 0.23
	3	0.80 ± 0.07	0.17 ± 0.07
	4	1.15 ± 0.71	0.12 ± 0.04
Summer	1	7.06 ± 2.57	0.46 ± 0.24
	2	9.27 ± 5.99	1.36 ± 0.73
	3	15.56 ± 1.00	0.90 ± 0.29
	4	13.63 ± 1.82	0.59 ± 0.41
Fall	1	4.48 ± 1.56	-0.66 ± 0.22
	2	2.87 ± 0.97	-1.14 ± 0.93
	3	3.10 ± 4.08	0.51 ± 0.13
	4	6.46 ± 4.66	-0.79 ± 0.23

Table 1. Mean (±SD) maximum apparent TP flux (mg P/m²/day) in sediment cores collected from Mona Lake, Michigan, and incubated under anoxic and oxic conditions⁴. Flux was calculated from the change in TP concentrations over time, shown in Figure 1.

Season	Internal P Load, t	External P Load, t	Internal Load Contribution, %
Spring	0.055	0.557	9.0%
Summer	2.272	0.862	72.5%
Fall	1.127	0.242	82.3%
Winter	0.000		
Annual	3.454		

Table 2. Annual and seasonal internal P load estimates (metric tons, t) for Mona Lake, Michigan, calculated based on maximum apparent TP flux⁴ (shown in Table 1). Seasonal internal P load estimates are compared to external P load estimates to determine the contribution of internal load to total P load.

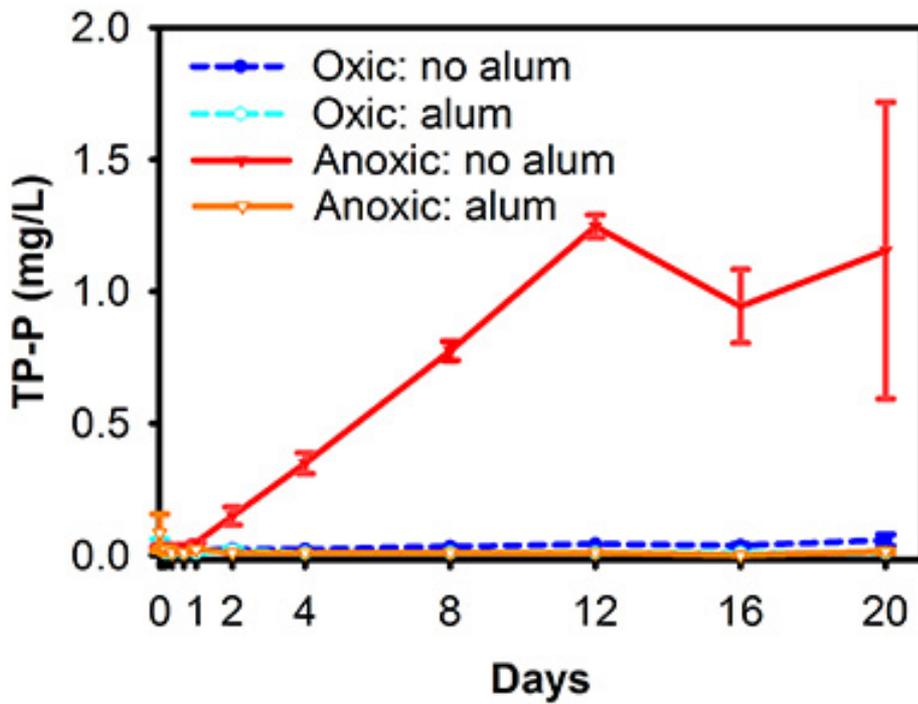


Figure 2. Mean (\pm SD) TP concentrations (mg/L) measured during laboratory incubations of sediment cores collected from Spring Lake, Michigan, and experimentally treated with aluminum sulfate (alum) under oxic and anoxic conditions². TP was measured in the water column overlying sediment cores over a 20-day incubation period. This figure has been modified from Steinman *et al.*² Reprinted by Permission, ASA, CSSA, SSSA. [Click here to view larger image.](#)

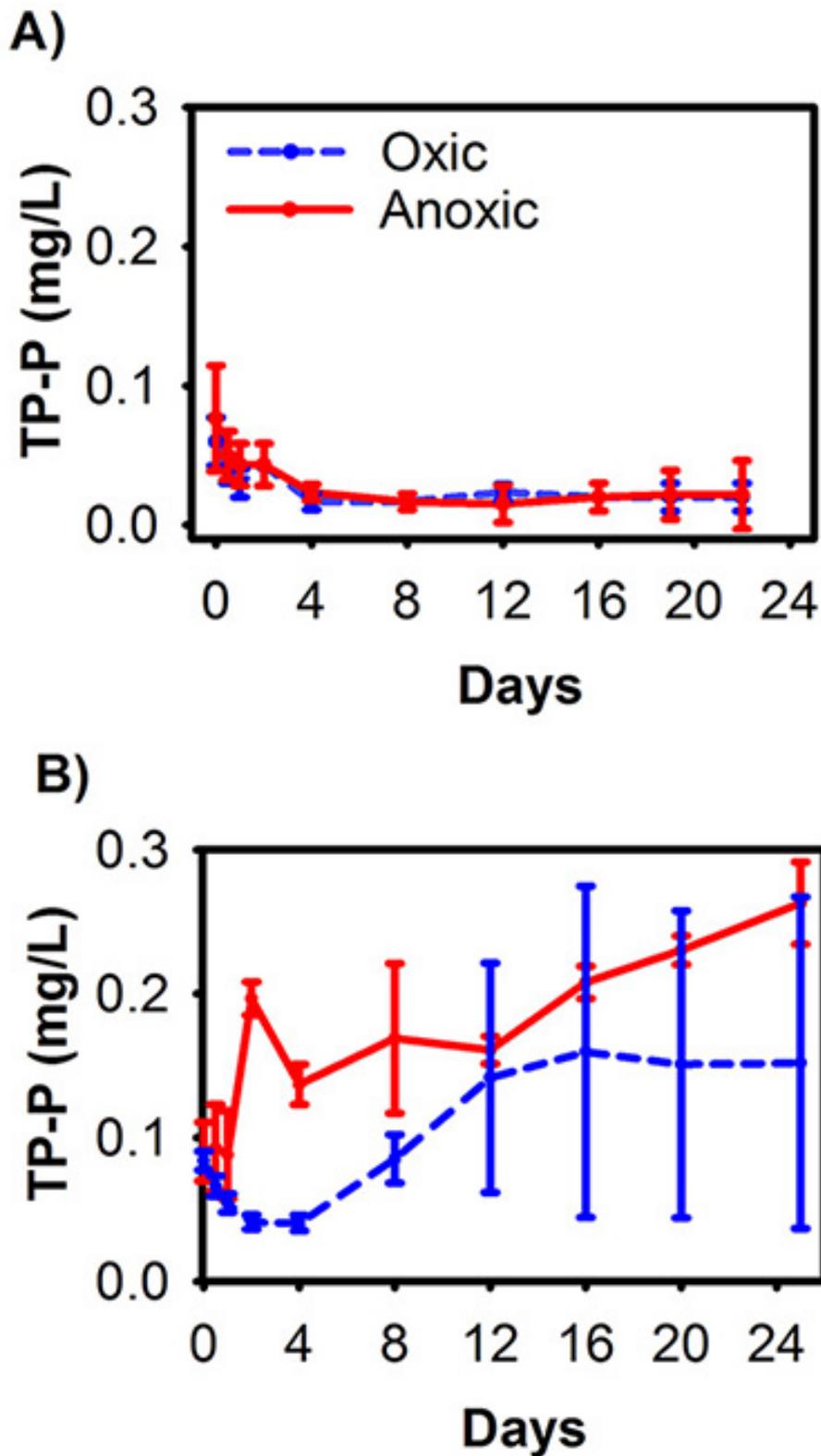


Figure 3. Mean (\pm SD) TP concentrations (mg/L) measured during laboratory incubations of sediment cores collected from Spring Lake, Michigan 1 year after¹⁸ (A) and 5 years after¹⁹ (B) a lake-wide application of alum. Sediment cores were subjected to oxic and anoxic treatments and the overlying water column was sampled for TP concentration over a 22-day (A) to 25-day (B) incubation. This figure has been modified from Steinman *et al.*¹⁸; panel A and Steinman *et al.*¹⁹; panel B. Reprinted by Permission, ASA, CSSA, SSSA. [Click here to view larger image.](#)

Discussion

Nutrient loading to lakes can result in both environmental and economic impairments²¹⁻²³; therefore, it is crucial that society understands the nature of the nutrient sources and how to manage them. Costly attempts to reduce nutrient loading may not improve water quality if the appropriate contributing source (*i.e.* lake sediments or watershed inputs) is not targeted for management action, thereby resulting in setbacks in lake restoration and frustration on the part of stakeholders. Particularly in shallow eutrophic lakes, quantification of internal phosphorus load is a critical step in identifying a management strategy to improve water quality conditions. Even when sediments are implicated as a major source of nutrients, reductions in external P load must be included in any lake management strategy for alleviating eutrophication, since external inputs of P ultimately accumulate in the sediments and fuel future internal loading^{24,25}.

Although other approaches exist to estimate internal P load, experimental determination of P release rates is a direct method that can be adjusted to answer a variety of management and research questions. Laboratory incubations of sediment cores collected from Spring Lake, Michigan, were used to determine the potential effectiveness of an alum treatment² and the most efficient application concentration¹³. As a result of the findings from these laboratory-based studies, the stakeholders developed confidence that the alum treatment could control P release in Spring Lake sediments. Consequently, they approved a 10-year assessment to fund an alum treatment; subsequent sediment core incubations revealed that the treatment was effective at reducing sediment P flux 1 year¹⁸ and 5 years¹⁹ following treatment. Sediment core incubations have also been used to evaluate the effects of sediment resuspension¹³ and bioturbation (G. Nogaro and A. Harris, unpublished data) on P release.

Several additional sediment analyses can be performed in conjunction with core incubations to provide information that is useful in interpreting sediment P release results. The top 5 or 10 cm of sediment can be extruded from cores for analysis of sediment TP, porewater SRP, sequential P fractionation, and metals^{4,18,19}. An example of sequential P fractionation²⁶ that can be useful in internal loading studies involves determining the amount of P bound to 1) aluminum (Al-P) or iron (Fe-P), which represents a redox insensitive (Al-P) and a redox-sensitive (Fe-P) mineral association that can become soluble under anoxic conditions, and 2) calcium (Ca-P) or magnesium (Mg-P), which are both stable mineral associations. Further, sediment Fe:P ratios can be calculated to provide insight on the potential P-binding capacity of sediments. Iron-rich sediments that remain oxidized have been shown to release very little P when Fe:P ratios are above 15 (by weight)²⁷. These additional sediment analyses can be performed on cores following internal load incubation^{4,18,19}, or on replicate cores taken at the time of internal load core collection but not used for release rate measurements.

Despite the benefits of experimental determination of sediment P flux, the approach is not without limitations. A number of assumptions must often be made that can add uncertainty to the results:

- One assumption is that release rates from the sediment cores are representative of conditions in the study lake. To minimize the impact of this assumption, sampling strategies should be designed to represent as much of the spatial and temporal variability as possible in sediment P release. Sampling sites should cover as much geographic range as possible within a lake to capture spatial variation in sediment characteristics². If available, bathymetric maps can be used to select sites that are representative of the range of bottom depths in the lake. Other considerations for capturing spatial variation include the location of major tributary inputs and the presence of distinct lake basins. When possible, laboratory incubations should be conducted during each ice-free season and over multiple years to capture temporal variation in release rates.
- A second assumption is that the incubation conditions are representative of natural conditions. A constant anoxic condition creates an optimal situation for the release of P, which may not naturally occur in the study lake. Thus, anoxic treatments may overestimate sediment P release; therefore, it may be best to think of release rates measured in anoxic treatments as maximum potential rates.
- In order to calculate annual internal P load, assumptions about the timing, duration, and spatial extent of hypolimnetic anoxia must be made. For example, in strongly stratified lakes with relatively consistent water depth and confirmed hypolimnetic anoxia, some studies have assumed that the entire lake area is anoxic during stratified periods for the purpose of annual internal P load estimation^{2,4}. However, this may result in an overestimation of load due to oxic sediments in shallow littoral areas⁴. Thus, a comprehensive dissolved oxygen monitoring strategy that captures diel, seasonal, and spatial variability in redox condition is highly recommended for accurate annual internal load estimation.
- Finally, laboratory incubations may introduce experimental artifacts due to the inability to completely simulate natural conditions. For example, because the sediments are enclosed in core tubes, water exchange through permeable sediments is precluded; however, it is possible to design flow-through core tubes that mitigate this issue²⁸. Other artifacts include the inability to mimic major mixing events or wind-wave action, which could disrupt sediment integrity in natural systems.

Given that the sediment core incubation approach can be used to generate reasonable internal P load estimates in as little as one year (although multiple years of data provide more robust information), it is a valuable tool for informing lake management decisions. When used to develop lake management or restoration plans, it can help ensure wise use of financial resources. In lakes where internal P load management has already occurred, sediment core incubations can verify the efficacy of treatment and be used to modify the trajectory of management, if warranted.

Disclosures

The authors have nothing to disclose.

Acknowledgements

The authors gratefully acknowledge the field and lab assistance provided by James Smit and Kurt Thompson. Funding for original studies for which this protocol was developed was provided by the Spring Lake-Lake Board^{2,13,18,19}; the Michigan Department of Environmental Quality⁴; and Jim Duncan, Dave Farhat, and the President's Office at Grand Valley State University¹⁷.

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