Discover Water

Brief Communication

A high degree of phosphorus sediment saturation in a shallow reservoir system

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Abstract
This study sought to establish the degree of phosphorus saturation in sediment within a shallow reservoir that has a history of being hypereutrophic with documented harmful cyanobacteria blooms. Five sites were chosen within the Des Lacs Reservoir System for sediment sampling. At each site a transect with five sampling points had sediment sampled at the 0–15 cm and 15–30 cm depth from the sediment–water interface line. The degree of phosphorus saturation was determined by measuring the amount of solution nitric acid extractable phosphorus adsorbed on the sediments using increasing phosphorus concentrations. Adsorption at low phosphorus concentrations indicates low phosphorus saturation while adsorption at only high concentrations indicates high phosphorus saturation. Sediment soil texture, organic matter, and carbon content was analyzed. Sediment within the lakes were measured to contain a mean total phosphorus concentration of 641 mg/kg (174 mg/kg standard deviation) with sediments being at more than 95% of their phosphorus saturation. With a high degree of phosphorus saturation, sediments are unable to readily sequester P from the water column and will internally load phosphorus which can sustain hypereutrophic conditions and cyanobacteria blooms. The high phosphorus sediment saturation is unique compared to other lakes and reservoirs which have lower phosphorus sediment saturation (20–70%) and some ability to sequester phosphorus. Nearly saturated sediment means there is a high risk of continued hypereutrophic conditions and cyanobacteria blooms in the reservoir and solutions such as reducing external phosphorus loading will be less effective.

Keywords Hypereutrophic · North Dakota · Phosphorus saturation · Reservoir · Sediment

1 Introduction

Shallow lakes are increasingly becoming hypereutrophic, accompanied by an increase in the frequency of cyanobacteria blooms [1–4]. Phosphorus (P) is the limiting nutrient in most freshwater ecosystems and at excessive levels leads to hypereutrophic conditions and persistent cyanobacteria algae blooms [2, 4, 5]. Even though the contribution of P can come from external sources to an aquatic system, internal sediment P can be a controlling factor and poses a risk to continued hypertrophic conditions. Often sediment in aquatic ecosystems can act as a reservoir for P and continues to be an internal source of P into the system [6–8]. Understanding the amount and availability of P in sediment is important to predicting hypertrophic conditions and cyanobacterial blooms.

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The Des Lacs Reservoir System (DLRS) is series of large, shallow, hypereutrophic man-made reservoirs located in North Dakota [9]. Multiple cyanobacteria blooms have been recorded in DLRS and has resulted in the mortality of large mammals. The mortality has occurred in groups of domestic cattle (> 12 animals) with the latest in 2018 [9] and the earliest in the 1940s [10]. A review by Orihel et al. [7] found few reservoirs have been studied in the region for sediment P. This lack of knowledge of sediment P in reservoirs prompted us to study sediment P in DLRS.

If a lake or reservoir has a low capacity for P sorption, then the risk would be higher for P migration out of the sediment [11, 12]. Conversely, if sediment can act as a sink for P, then managing conditions for internal loading, and still expecting some sequestering of P, can reduce hypereutrophic conditions [7]. One way to assess the risk from P loading is to measure the degree of P saturation (DPS) in sediments [12]. As suggested by Huang et al. [12], DPS can be used as an index for the risk of internal P loading to the aquatic system. DPS is being used in the region on terrestrial and stream systems to judge the risk of P movement and eutrophication potential in receiving waters [13]. Because of documented hypereutrophic conditions and cyanobacteria blooms in DLRS, the objectives of this study were to: (a) assess DPS within sediments in DLRS at different sites and depths, and (b) determine what role DPS levels contribute to the potential for hypereutrophic conditions and accompanying cyanobacteria blooms. With few reported studies of DPS in lakes and even less for reservoirs, measuring DPS in the DLRS with a history of harmful cyanobacteria blooms will establish the degree to which DPS can be used to determine the risk of hypereutrophic conditions and cyanobacteria blooms.

2 Study site

The study took place on the Des Lacs National Wildlife Refuge (DLNWR) near Kenmare, North Dakota, United States which encompasses most of the DLRS. The DLRS is comprised of three naturally formed lakes on the Des Lacs River that were turned into reservoirs. The reservoirs were originally created in 1935 by the construction of dikes and small low head water control structures spanning a 45 km long river valley. The refuge is in the northwest corner of North Dakota, borders the Canadian province of Saskatchewan to the north, and is approximately 145 km east of Montana (Fig. 1).

The Des Lacs River Valley was geologically formed from a sudden and catastrophic drainage of Glacial Lake Regina, resulting in a steep and narrow valley [14]. Slumping and the formation of ephemeral streams eventually widened the valley and deposited coarse alluvial sediments along the valley sides with finer materials in the center of the valley [14]. The area around the Des Lacs Valley is dictated by smooth drift plains that transition to steep slopes descending 15–40 m to the river. Soils in the river valley are mapped as Zahl-Williams Association (Zhal: Fine-loamy, mixed, superactive, frigid Typic Calciustolls; Williams: Fine-loamy, mixed, superactive, frigid Typic Argiustoll) formed from medium to moderately-fine textured glacial till [15]. The DLRS lies in the Northern Glaciated Plains ecoregion and is part of the Northern Dark Brown Prairie [16]. The area is characterized by grasslands and agriculture that is transitional from sub-humid to semiarid conditions.

The DLRS has approximately 2029 ha of open water and 283 ha of marsh. Eight water control structures create the series of reservoir pools and wetlands that comprise the refuge today. Two reservoir pools were selected for sampling: (a) the upper reservoir pool has a surface area of 1736 ha, has an average depth of 3 m over the seasons with a maximum depth of 4.2 m, is 42 km long, and is 0.47 km at its widest, (b) the middle reservoir pool has a surface area of 280 ha, has an average depth of 0.8 m with a maximum depth of 1.2 m, is 4 km long, and is 0.77 km at its widest. The DLRS only receives water from the local tributaries. Water movement through the reservoir system primarily occurs in the spring and varies with snowpack levels and spring precipitation. The DLRS can stagnate after spring flows because there are no other major inflows and summers are semi-arid.

Four sites were selected in the upper reservoir pool: (a) site one (48° 59′ 24.1″ N; 102° 11′ 33.4″ W), (b) site two (48° 52′ 25.2″ N; 102° 04′ 16.8″ W), (c) site three (48° 48′ 37.2″ N; 102° 07′ 11.7″ W), and (d) site four (48° 43′ 37.8″ N; 102° 07′ 49.4″ W). Only one site, site five (48° 40′ 19.6″ N; 102° 05′ 37.0″ W) was selected in the middle reservoir pool (Fig. 1). At each of the five sites a transect was established across the lake. Five equidistant points along a transect from the east to west shore were sampled, with the 1st and 5th points located on the shore and remaining samples collected from the lake bed. The five points along the transect included cores on the east and west shores, a lake sediment core from the middle of the pool or river, and a lake sediment core between the middle and east shore and middle and west shore. This amounted to a total of 25 core samples.

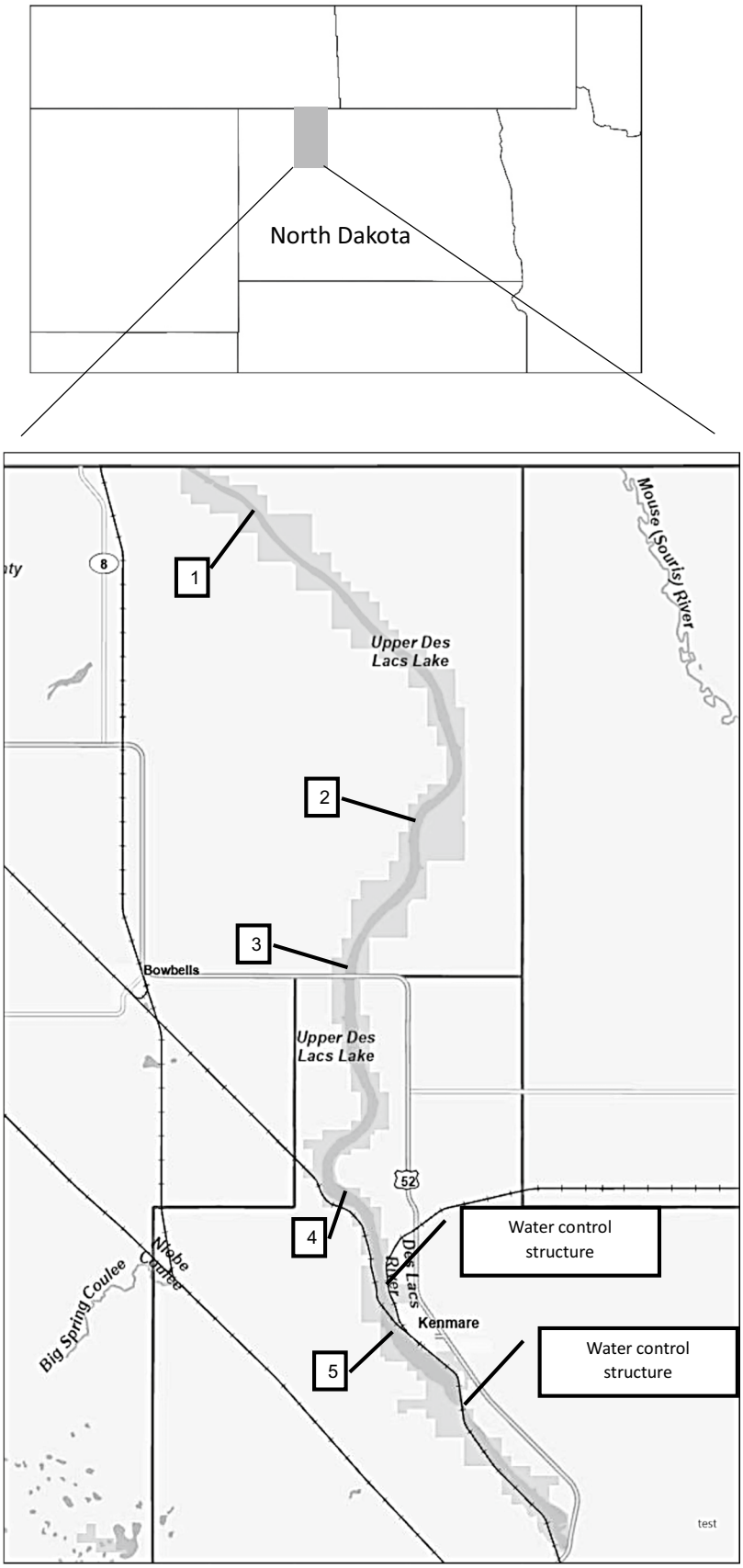

Fig. 1 Map showing sites one through five where samples were collected in the Des Lacs Reservoir System within the Des Lacs National Wildlife Refuge in North Dakota, United States. The darker gray denotes the reservoir pools. The lighter gray denotes the refuge area

3 Materials and methods

3.1 Sediment sampling and characterization

Soils along the shore and lake sediment samples were collected in June and July of 2019. Lake sediment cores were collected using a universal percussion corer (Aquatic Research Instruments, Hope, Idaho, United States). Shore soil samples were taken using a 10 cm diameter bucket auger. The top 30 cm of each sample was collected and split into two samples, one spanning from the 0–15 cm depth and the other from the 15–30 cm depth. Cores were stored in glass jars, kept on ice in the dark, and shipped to the North Dakota Department of Environmental Quality laboratory (Bismarck, North Dakota, United States) to be analyzed for total phosphorus (TP). Duplicate field samples were collected that were air dried, ground to pass through a 2 mm sieve, and stored at 20 °C before being analyzed for P sorption isotherms, nitric acid extractable Fe, Ca, and P, total carbon (TC), inorganic carbon (IC), organic carbon (OC), soil pH, and particle sizes as described in [17, 18].

Nitric acid extractable Fe, Ca, and P were determined using the EPA3050B method, which uses a hot nitric acid digestion with 30% H_2O_2 to extract the total Fe, Ca, and P from outside the silicate mineral lattice which are considered environmentally available [17]. Thus, the extractable contents represent a pool of elements relevant to environmental fluxes and transformations, such as P sorption. Total carbon (TC) and soil inorganic carbon (IC) were evaluated using a Primacs SLC TOC Analyzer (Skalar Analytical B.V., Breda, The Netherlands); soil organic carbon (OC) was calculated as the difference between TC and IC [18]. Soil pH was determined with a 1:1 suspension [18]. Particle size analysis was determined using the pipette method [19].

3.2 Phosphorus sorption isotherms

Phosphorus sorption isotherms were measured using the SERA-IEG 17 standard procedure [20]. From each sample 1.2 g of air-dry soil was decanted into 50 mL polyethylene centrifuge tubes with 30 mL of the corresponding concentrations of P: 0, 0.01, 0.1, 0.5, 10, 25, 50 and 100 mg P/L as potassium phosphate (KH_2PO_4) prepared using 0.01 M $CaCl_2$. These concentrations represent four levels below and above the highest recorded P concentration within the water column from the DLRS (i.e., 1.08 ppm) [9]. Samples were shaken on a horizontal shaker for 24 h at 20 °C then centrifuged at a relative centrifugal force of 650×*g* for 10 min. The supernatant was filtered through 0.45 µm glass fiber filters (GE Healthcare Life Sciences, Marlborough, Massachusetts, USA). Soluble reactive P of the supernatant was measured at the North Dakota State University Soils Testing Lab (Fargo, North Dakota, United States).

Phosphorus sorption isotherms were developed by fitting either the Langmuir or Freundlich equations. Specifically, (mg P/kg soil) was used as the independent variable and P solute remaining in solution after adsorption equilibrium (mg P ml solution) was used as the dependent variable. In these equations, P not detected in solution is assumed to be adsorbed by adsorption sites on the soil's mineral and organic matter fractions [20]. The curve of the Langmuir equation plateaus when a homogeneous set of sorption sites with the same free energies are saturated, whereas the Freundlich equation continues to gradually increase due to a presence of heterogeneous sorption sites that are diverse acting simultaneously and spontaneously [21, 22]. The degree of phosphorus saturation of the sorption sites was estimated by using antecedent nitric acid extractable P in the soils and either the P sorption isotherm at a plateau (Langmuir type) or at the highest solution concentration (100 mg/L; Freundlich type). The Solver Add-in for Microsoft Excel (Microsoft Corporation) was used to minimize the sum of squared errors between the measured and predicted values with the generalized reduced gradient method by adjusting equation parameters for the Langmuir and Freundlich equations. The sediment's water-soluble P (WSP) fraction was calculated as the P desorbed into the 0 mg/L solution after equilibrium.

3.3 Statistical analysis

The coefficient of determination and inspection for correlated residual errors were used to assess the Langmuir and Freundlich fitted equations to the observed soil P values (Microsoft Excel version 2008, Microsoft Corporation). The Analysis Toolpak in Excel (Microsoft Corporation) was also used to create a correlation matrix for all tested soil parameters. Since all adsorption isotherms showed Freundlich types curvature, DPS was determined as the difference between the antecedent P level and adsorbed P at 100 mg P/L of the isotherm. Clay percent, sediment Fe, sediment Ca, IC, OC,

antecedent sediment P, and sorption of additional P were then tested for differences among the sites (5 sites), transect positions (5 positions), and soil depth (two depths). The test used the PERMANOVA + procedure [23, 24] which is a non-parametric, permutation statistical technique. The analysis had transect position nested under site and depth nested under transect position. All three factors were designated as random effects.

4 Results

Soils along the shore and lake sediments on DLRS ranged from sand to silty clay (Fig. 2). There was large variation in the amount of sand in the sediment, with levels ranging from 5% to over 95%. However, loamy categories were dominant which consisted of sandy loam, loam, clay loam, and silty clay loam. The majority of silty categories (silty clay loam, silty clay) were from the three points taken from the lakebed (west middle, middle, and east middle). Most of sandy sediments were on the east shore. Clay percentages (Table 1) significantly differed by site and transect location ($P = 0.015$ for site; $P < 0.001$ for transect).

The OC component of TC was the dominant fraction of TC (Table 1) with a mean and median of 84% of the TC within each sample. Significant differences were evident in IC and OC content by site (IC, $P < 0.001$; OC, $P = 0.019$) and by transect

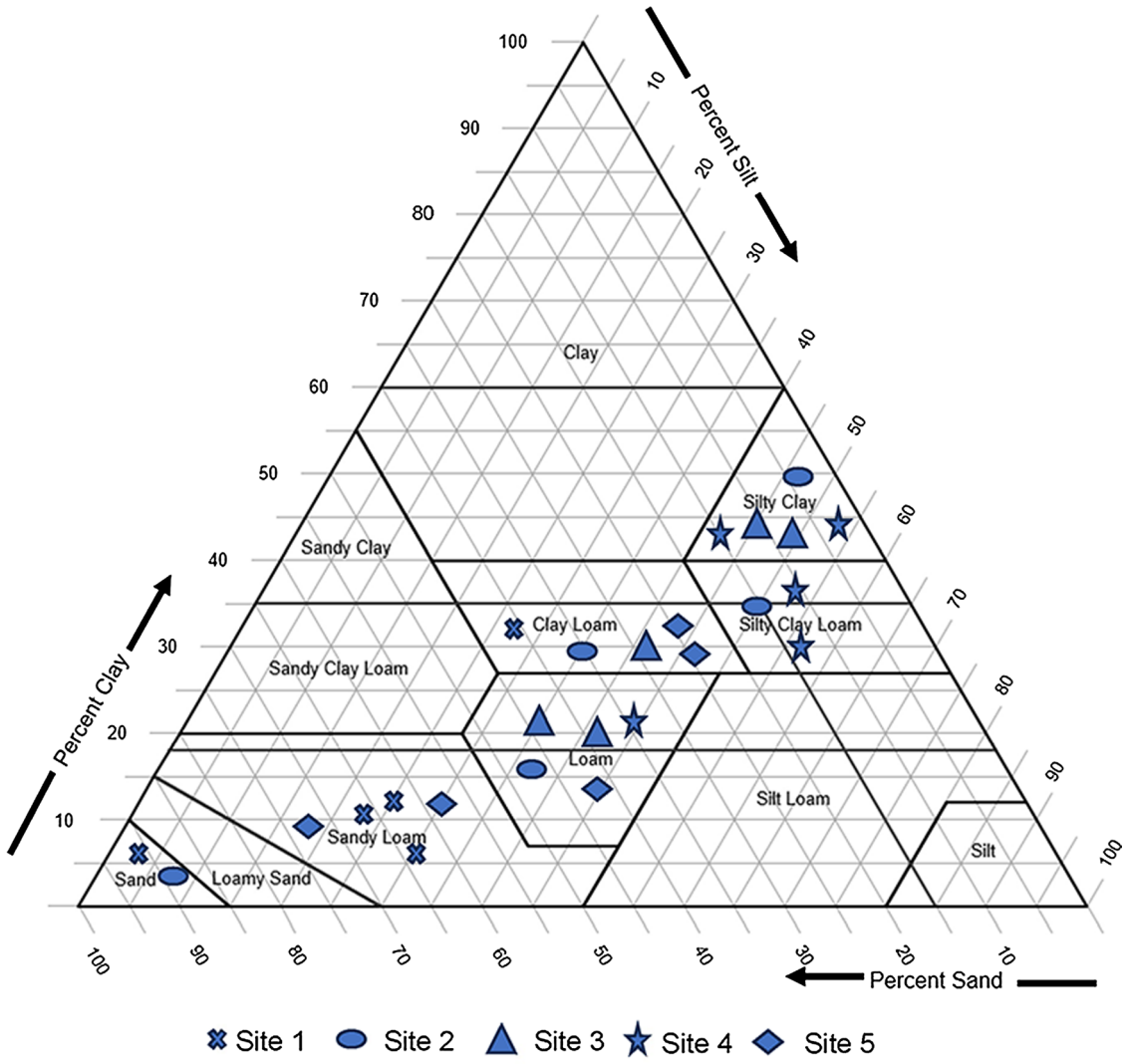

Fig. 2 Soil texture chart depicting sediment samples taken within each of the five sites (only the top 15 cm sample shown) from the Des Lacs Reservoir System within Des Lacs National Wildlife Refuge

Table 1 Statistics of soil properties in the sediment samples taken from the Des Lacs Reservoir System within Des Lacs National Wildlife Refuge

Soil property	Minimum	Mean	Median	Maximum	Standard deviation
Clay %	4.1	25.5	25	44.6	12.4
TC %	0.71	5.35	3.86	15.6	3.68
IC %	0	0.86	0.91	2.2	0.55
OC %	0.28	4.5	3.23	14.4	3.5
FE mg/kg	2719	12,562	13,410	21,004	4601
Ca mg/kg	2326	20,683	18,547	67,126	14,008
Antecedent P mg/kg	296	641	605	1042	174
WSP mg/kg	0.005	0.077	0.066	0.295	0.052
DPS %	94.7	97.6	97.8	99.5	1.0
Sorption of P mg/kg	2.4	15.4	13.6	34.1	8.1
Additional P adsorbed at 100 mg P/L solution mg/kg	3	19	16	60	12

location (IC, $P > 0.001$; OC, $P < 0.001$). Depth did not significantly differ for both IC and OC (IC, $P = 0.053$; OC $P = 0.093$). Mean sediment pH was 7.5 with 20% of samples being above 7.8, whereas the mean water column pH was 9.1 [9].

The Fe levels along the shore and lake sediments significantly differed across sites and transect position ($P = 0.023$ for site; $P < 0.001$ for transect) but did not differ between depths ($P = 0.8$). Site one (i.e., the most upstream site) had the lowest Fe value (7783 mg/kg averaged over the transect locations) while site four on average had the highest (16,643 mg/kg averaged over the transect locations). The center of each transect had the highest lake sediment Fe concentrations. Th Ca levels along the shore and in lake sediments only significantly differ across transects ($P = 0.642$ for site; $P = 0.029$ for transect), and did not differ between depths ($P = 0.257$).

Antecedent nitric acid extractable P in the soils along the shore and lake sediments significantly differed by site and transect but not depth ($P < 0.001$ for site; $P = 0.002$ for transect; $P = 0.67$) with site one being the lowest (439.3 mg/kg averaged over the transect locations) and site three on average being highest (774 mg/kg averaged over the transect locations). Soils along the shore and lake sediment P desorbed into solutions initially containing 0.1, 0.01, and 0 mg P/L treatments, with the mean equilibrium P in solution after desorption being 0.061 mg/L. Of these solution treatments, 22, 60, and 100% of sediments desorbed P into the 0.1, 0.01, and 0 mg P/L treatments. The mean Fe/P ratio (by mass) was 19.8 with sites ranging from 15.9 (site five) to 27.8 (site one). The mean Ca/P ratio (by mass) was 33.1 with sites ranging from 24.7 (site two) to 38.4 (site four).

No distinct plateau in P adsorption was evident for any sample, therefore, the Freundlich equation best fit the isotherms and was used for all isotherms. The largest amount of additional P adsorbed onto the soils along the shore and lake sediments occurred at the highest solution dose of 100 mg P/L. The additional P adsorbed onto samples with the dose of 100 mg P/L solution was 3, 19, 16, 60, and 12 mg/kg for the minimum, mean, medium, maximum, and standard deviation, respectively, and are a magnitude lower than the sample's antecedent P levels (i.e., 296 to 1042 mg/kg). Sediment DPS was antecedent P level divided by adsorbed P at the 100 mg P/L solution level of the isotherm (adjusted to percent). The DPS along the shore and within lake sediments varied little with most at 100% and 94.7% being the lowest level (Tables 1 and 2). The sorption of additional P differed by site, but not by transect location ($P < 0.001$ for site; $P < 0.001$ for transect). The 15–30 cm depth P sorption values were significantly lower than the 0–15 cm depth ($P = 0.021$).

5 Discussion

The soils along the shore and lake sediments throughout the DLRS are at or near P saturation. Only a minimal amount of P could be adsorbed on sediments when solutions were at or above 0.5 mg/L. This is still somewhat below the highest total P concentrations measured in the lake's water column (1.08 mg/L), but similar to the water column's mean annual total P concentrations (0.48 mg/L) [9]. Sediments displayed sorption characteristics that suggest they have multiple heterogenous sorption sites with differing free energies (i.e., Freundlich type isotherms) [21, 22]. This could be due to the sediment having both large contents of minerals and organic matter, which may have contrasting sorption isotherms [15]. The high contents of both Fe and Ca in the sediments could also result in various organo-metal complexes and

Table 2 Phosphorus saturation of soils with differing concentrations of solution phosphorus (P): 0, 0.01, 0.1, 0.5, 10, 25, 50 and 100 mg P/L as potassium phosphate (KH_2PO_4) prepared using 0.01 M $CaCl_2$

Solution P Concentration	Mean % Saturation	Median % Saturation	Min. % Saturation	Max % Saturation
0 mg P/L	100	100	99	100
0.01 mg P/L	100	100	100	101
0.1 mg P/L	100	100	99	100
0.5 mg P/L	100	100	100	100
10 mg P/L	99	99	97	100
25 mg P/L	98	98	95	100
50 mg P/L	98	98	95	100
100 mg P/L	98	98	95	99

Percent saturation was estimated by dividing antecedent P values by absorbed P values of the different soil samples (n = 50, 5 sites, 5 points along a site transect, and 2 depths) at the different solution concentrations. Values at 100 percent indicate no sorption of solution P onto soils. Values below 100 percent indicate sorption of solution P onto soils (i.e., a partial P sink)

bridging with mineral and organic colloids. Differing clay mineral composition may also be a source for contrasting sorption site characteristics because the soils in the river valley have mixed clay mineralogy [15]. Whatever the source of heterogeneous sorption sites, the small overall capacity to adsorb additional P suggests that contrasting sorption sites may all be near or at saturation.

Compared to other reservoirs and lakes with reported DPS, DLRS is the only reservoir system at or near 100% P saturation. The Lake of the Woods, just 500 km east of DLRS, also has persistent cyanobacterial blooms, however, it was found to have a DPS of just 16–23% across four sites [25]. In Poland, Augustyniak et al. [26] reported on a lake that had P inactivation actions and DPS values of 25 to 72%. They reported that those values were similar to other lakes in the region without inactivation efforts. In eastern Canada, Cyr et al. [27] reported P sorption values ranged widely depending on if the sediments were in shallow or deep water, but all locations had some P sorption capacity. A large reservoir in western Canada, Lake Diefenbaker, in a similar landscape as DLRS was found to still be a sink for P [28]. In China, where lakes are affected by urbanization and agricultural P inputs, lakes still had low DPS values and high capacities for P sorption [12, 29]. In Florida lakes, Belmont et al. [30] found that lakes had high P sorption capacities and continued to be P sinks. In the same region as DLRS, Badiou et al. [31] found intact wetlands with similar environmental influences had high P sorption capacities such that wetlands would act as sinks for P. Comparing the DLRS DPS with near 100% saturated condition with all the aforementioned lakes and wetlands finds none were at the same high level of saturation and were still able to absorb P. These comparisons make the DLRS system unique in the region and outside the region. This unique status increases the likelihood that this system will act differently from other systems with the likelihood of continued hypereutrophic conditions and cyanobacteria blooms.

The sediments on DLRS are, for practical purposes, fully saturated with P throughout the summer and fall. These sediments, once saturated, will be unable to sequester further P. Phosphorus entering the system will therefore remain in the water column and available for uptake by phytoplankton. The effects of high P saturation in the sediments on DLRS may have been seen as early as 1997, where a previous study by Wax [32] found the lakes on DLRS were eutrophic to hypereutrophic. Furthermore, Young [9] found that a Carlson's Trophic State Index [33] created from the results of Chlorophyll-*a*, TP in the water column, and Secchi disk sampling between 2016 and 2019 showed that the water column on DLRS was eutrophic to hypereutrophic nearly the entirety of each sampling season. This is likely influenced by the sediments no longer being able to sequester new sources of P, and instead the likelihood that sediment P is a source P for the system. This implies that eutrophication on DLRS is not a recent phenomenon and that sediments may have been heavily to fully saturated for a long time.

In a typical system, the sediments can act as a sink for external P with calcium (Ca), iron (Fe), and aluminum (Al) forming less soluble cations, which in turn reduces the amount of soluble P available for uptake by phytoplankton [6, 8]. When sediments are unable to sequester further P, all new P entering the system remains in the water column and, depending on the form, available for phytoplankton uptake. Additionally, the P that is already sequestered in the system can internally load available P into the water column [34]. The high likelihood of P moving from the sediments to the water column is in part dependent on the Fe availability. The Fe/P ratio is just above the value where Fe does not have the ability to control P mobility (> 15) according to [35]. Lake of the Woods had Fe/P ratios of 26 to 42 and these ratios are where P mobilization should be reduced [25]. The low Fe at DLRS compared to Lake of the Woods [25] (DLRS 12,562

vs Lake of the Woods 23,750 mg/kg) could be a function of the underlaying sedimentary bedrock which can have low Fe availability [7].

According to Orihel et al. [7] lakes in the same region and environmental setting can have internal P loading from sediment can be much higher than external P inputs. With the high DPS, the ability to reduce P levels through management actions can be limited. With the potential of cyanobacteria blooms continuing in the DLRS, due in part to high DPS, one management option is to reduce domestic livestock access to the DLRS thus averting mortality events. Options such as fencing off access to DLRS and moving cows to other areas when cyanobacteria blooms are known to occur would be an effective short-term option.

6 Conclusions

The high DPS found in the DLRS is unique in and outside the region with no other lakes or reservoirs with comparable high levels. The high DPS was found at all locations and water depths within the DLRS so no location can act as sink for external P coming into DLRS. With high DPS being an indicator of the risk of P migration from sediments, DLRS has an elevated risk of continued hypereutrophic conditions and accompanying cyanobacteria blooms. The high DPS will need to be accounted for in management options, such as control of external P inputs, or control of internal P by capping sediments or using flocculating agents [36–39]. It is unknown what the effectiveness of these management options will be given that high DPS is an unusual circumstance that has not been encountered in previous studies. Researchers and managers should be aware of the possibility of high sediment DPS in other systems and that management options for P will need to incorporate the consequences of having high sediment DPS in a reservoir or lake.

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Data availability The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests The authors report there are no competing interests to declare. The views expressed in this article are the authors' own and do not necessarily represent the views of the United States Fish and Wildlife Service.

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