

Article

Sediment Phosphorus Storage and Release After Aquaculture Cessation: A Case Study of Datong Lake

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Abstract

Reducing external phosphorus (P) inputs is central to eutrophication control, but sediment P release can sustain internal loading and delay lake recovery. This study assessed sediment P composition, vertical distribution, and short-term release potential in Datong Lake, a shallow lake formerly affected by intensive aquaculture. Total phosphorus (TP), dissolved total phosphorus (DTP), and particulate phosphorus (PP) were measured in surface and overlying waters during the dry and wet seasons. Surface-sediment P fractions were characterized in both seasons, whereas dry-season sediments were used for adsorption and release tests, vertical profiling, and depth-specific incubations. Wet-season samples had higher TP, DTP, and PP concentrations in surface water and higher TP and PP concentrations in overlying water. Surface-sediment P was predominantly inorganic, with Ca-P as the largest inorganic fraction; bioavailable P accounted for about 32% of TP. In the 24-h batch tests, the adsorbed P amount exceeded the released amount. Total P and its NaOH-extractable and organic fractions were enriched in the upper sediment. Across the 1–5 to 21–25 cm intervals, 15-d cumulative P release decreased from 22.9 ± 1.9 to 7.2 ± 1.0 mg·kg^{−1}, and the initial 3-d release rate decreased from 1.13 ± 0.18 to 0.24 ± 0.07 mg·kg^{−1}·d^{−1}. Overall, Datong Lake sediments contained high P concentrations but exhibited limited short-term release, with the greatest release amount and rate occurring in the upper sediment.

Keywords: internal phosphorus loading; sediment phosphorus; phosphorus fractions; bioavailable phosphorus; sediment–water interface; phosphorus release



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1. Introduction

Eutrophication is a major global threat to aquatic ecosystems because it degrades water quality through harmful algal blooms and threatens aquatic life through oxygen depletion [1]. In freshwater lakes, phosphorus (P) enrichment is a major driver of eutrophication [2]. For decades, efforts to control lake eutrophication have focused on reducing external P inputs from wastewater discharges and catchment runoff [3]. However, a fraction of the P delivered from external sources accumulates in lake sediments and can later be remobilized to the water column [4]. Sediment P release can contribute substantially to water-column P supply and persist for decades after external inputs decline, slowing the decrease in lake-water P concentrations [4,5]. Effective eutrophication control must therefore account for internal P loading.

Sediment P is distributed among fractions with contrasting potential mobility, commonly described as loosely bound P, Fe- and Al-associated P, Ca-associated P, organic P, and residual P [6]. Loosely bound P is readily exchangeable with porewater, whereas P associated with redox-sensitive Fe(III) (oxyhydr)oxides can be mobilized by reductive dissolution [6,7]. Microbial mineralization can convert the labile portion of organic P to phosphate [8]; Al-associated P is comparatively insensitive to redox change, while the stability of Ca-associated P depends on mineralogy and saturation state [6,9]. Phosphate sorption to mineral surfaces can retain dissolved P and regulate its partitioning between sediment solids and porewater [10]. In shallow lakes, wind-driven resuspension can bring surface sediments into contact with the overlying water, thereby transporting particulate P and modifying phosphate exchange between particles and water [10,11]. Burial and early diagenesis redistribute P among sediment fractions, producing vertical changes in P concentration and composition [8]. Sediment depth also controls transport to the overlying water: P mobilized near the surface has a shorter path to the sediment–water interface, whereas P mobilized deeper in the profile must first be transported upward [12]. The short-term release potential of legacy sediment P therefore depends on the relative abundance of potentially mobile P fractions and their distribution with sediment depth.

Intensive lake aquaculture can intensify and prolong eutrophication because feed and fertilizers deliver P directly to the water, while low recovery through harvest allows much of this P to remain within the lake [13,14]. Datong Lake provides a representative case of a lake transitioning from intensive aquaculture to ecological restoration. Its eutrophication history has been reconstructed from sediment records [15], and water-column P responses to submerged-macrophyte restoration have been documented at the whole-lake scale [16]. Sediment studies have further shown that dredging, sediment amendment, and macrophyte establishment alter P forms and sediment–water exchange, and that P enrichment can remain concentrated in the upper sediment [17,18]. These studies clarify how water-column P and sediment–water P exchange respond to restoration. However, it remains unclear how much of the P remaining in the sediment is available for short-term release to the water column, and how this release potential changes with sediment depth.

Combining sediment P composition and short-term adsorption and release responses with vertical P profiles and depth-specific release measurements can describe the forms in which P is retained and how its short-term release potential changes with depth. In this study, we combined water P measurements with surface- and depth-resolved sediment analyses to investigate sediment P remaining in Datong Lake after the cessation of intensive aquaculture. We aimed to (1) characterize surface-sediment P composition and its short-term adsorption and release responses under standardized conditions, (2) determine how sediment P concentration, composition, and short-term release vary with depth and whether P-enriched layers show greater release, and (3) compare the total, dissolved, and particulate P between surface water and overlying water in the dry- and wet-season surveys. Linking vertical P profiles with depth-specific release responses helps identify the sediment layers most relevant to P management.

2. Materials and Methods

2.1. Study Area and Sample Collection

This study was conducted in Datong Lake, a shallow lake in Datonghu District, Yiyang City, Hunan Province, China (112°26′28″–112°35′02″ E, 29°05′19″–29°16′16″ N; Figure 1). Datong Lake has a surface area of 82.67 km² and mean and maximum water depths of 2.58 and 2.94 m, respectively [19]. The region has a humid monsoon climate, with approximately 68% of annual precipitation occurring from April to September [19]. Following restrictions on large-scale aquaculture beginning in 2017, fish removal and whole-lake

submerged-macrophyte restoration were implemented [15,16], and submerged vegetation was restored over approximately 60 km² during the first year of restoration [20]. We conducted two lake-wide surveys in January 2024 (dry season) and August 2024 (wet season). During each survey, surface water and overlying water were collected at 27 sites for TP, DTP, and PP analyses. Nine of these sites (S1–S9) were selected for sediment sampling to provide spatial coverage across the lake (Figure 1). Surface-sediment P fractions were analyzed in both seasons. Dry-season surface sediments and cores were selected for adsorption–release kinetic tests and for vertical profiling and depth-specific static incubations, respectively, because this lower-runoff period was less directly affected by monsoon-driven runoff and associated particulate inputs [15,19].

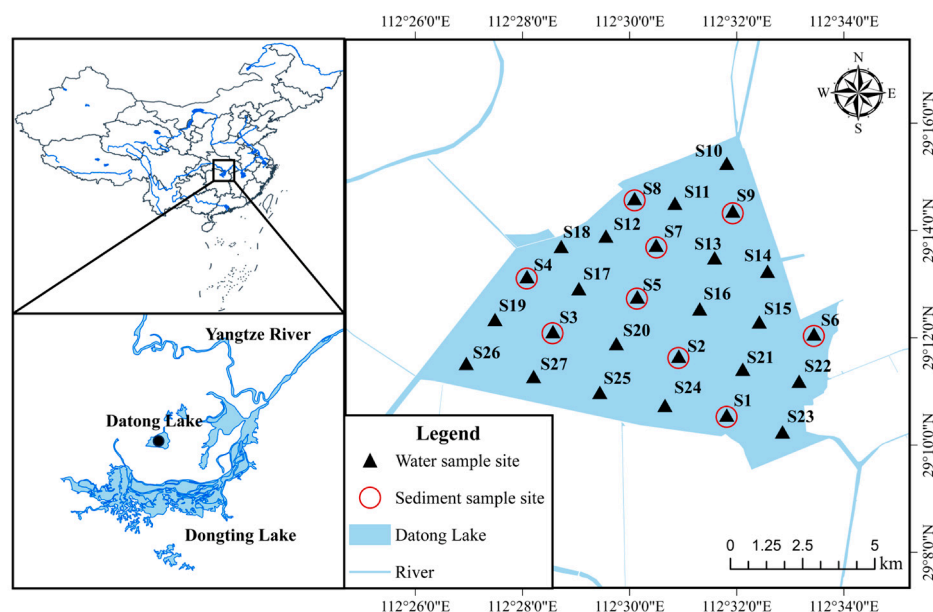


Figure 1. Location of Datong Lake and distribution of water and sediment sampling sites. Black triangles indicate water sampling sites, and red open circles denote sites where sediment samples were also collected. The inset maps show the location of Datong Lake in China and its position relative to Dongting Lake and the Yangtze River.

2.2. Water and Sediment Sampling and Pretreatment

Water sampling and sample handling followed the general guidance of HJ 494–2009 and HJ 493–2009, respectively, whereas sediment sampling followed ISO 5667-12:2017 [21–23]. Surface water (SW) and overlying water (OW) were collected at all 27 sites. SW was collected approximately 20 cm below the water surface using a 1 L cylindrical acrylic water sampler (TL-CYQ10, Tianlian Group Technology Co., Ltd., Beijing, China). At each site, three equal-volume SW samples were combined and mixed thoroughly. OW was gently siphoned from the water layer 1–5 cm above the visually intact sediment–water interface (SWI) in each of three sediment cores. Equal volumes of OW from the three cores were combined and mixed thoroughly. Aliquots for dissolved total phosphorus (DTP) analysis were filtered in the field through 0.45 µm mixed cellulose ester membranes (Sterlitech Corporation, Auburn, WA, USA), while unfiltered aliquots were retained for total phosphorus (TP) analysis. The filtered and unfiltered aliquots were stored separately in precleaned polyethylene bottles, protected from light, transported to the laboratory at approximately 4 °C, and analyzed promptly after arrival.

Surface sediments were collected from S1–S9 using a Petersen grab sampler (CN-150, 25 × 20 cm sampling aperture, Guangzhou Ruibin Technology Co., Ltd., Guangzhou, China). At each site, the upper 0–1 cm of sediment from three grab samples was combined

and homogenized after visible stones, plant fragments, and other coarse materials were removed. Samples collected during both surveys were used for P fractionation and for total Fe and TOC analyses, whereas dry-period samples were used for the adsorption and release experiments. Sediments used for chemical analyses and adsorption–release experiments were freeze-dried, ground with an agate mortar (Shanghai Lichen Bangxi Instrument Technology Co., Ltd., Shanghai, China), and passed through a 150 μm sieve (100SS8F, Advantech Manufacturing, Inc., New Berlin, WI, USA). During the dry-period survey, three sediment cores were collected at each of S1–S9 using a gravity corer (Rigo Co., Ltd., Saitama, Japan) fitted with acrylic tubes (90 mm in diameter and 500 mm in length). After OW collection, the cores were sectioned horizontally in the field at 1 cm intervals to a depth of 30 cm. Sediment from the corresponding depth in the three cores was combined and homogenized. Subsamples used for vertical-profile analysis were freeze-dried, ground, and sieved, while fresh sediment was retained for static incubation. For incubation, five consecutive 1 cm sections were combined into the following depth groups: 1–5, 6–10, 11–15, 16–20, and 21–25 cm. Additional details on sample collection and preparation are provided in Text S1, and equipment specifications are listed in Table S1.

2.3. Phosphorus Analysis and Fractionation

TP and DTP in surface water and overlying water were determined after potassium persulfate digestion [24]. TP was measured in unfiltered samples, whereas DTP was measured in samples filtered through 0.45 μm membranes. Particulate phosphorus (PP) was calculated as the difference between TP and DTP. Orthophosphate in the digested water samples and sediment extracts was determined by the molybdenum blue method using a UV–visible spectrophotometer (UV-1800, Shimadzu, Kyoto, Japan) [25].

Sediment TP, inorganic phosphorus (IP), and organic phosphorus (OP) were determined using the harmonized Standards, Measurements and Testing (SMT) protocol [26,27]. TP was determined by ignition followed by HCl extraction, whereas IP was extracted directly with HCl. OP was determined by igniting and extracting the residue remaining after IP extraction.

Surface-sediment IP was further fractionated using a modified sequential extraction procedure adapted from Hupfer et al. [8,9]. The operationally defined fractions comprised loosely sorbed P ($\text{NH}_4\text{Cl-P}$), bicarbonate–dithionite-extractable redox-sensitive P (Fe-P), NaOH-extractable metal-oxide-associated P (Al-P), HCl-extractable Ca-associated P (Ca-P), and residual inorganic P (Res-P). The first four fractions were extracted sequentially with NH_4Cl , bicarbonate–dithionite, NaOH, and HCl, respectively. Res-P was calculated as the difference between IP and the sum of the four extracted fractions. Potentially bioavailable inorganic P (BAP) was estimated as the sum of $\text{NH}_4\text{Cl-P}$, Fe-P, and Al-P, following Zhou et al. [28]. Detailed procedures for water and sediment P analyses, including sediment P fractionation, are provided in Text S2. Total Fe and TOC were measured in the 0–1 cm surface sediments by ICP-MS following microwave-assisted acid digestion and by potassium dichromate oxidation spectrophotometry, respectively. Detailed analytical procedures and quality-control information are provided in Text S3.

For the sediment cores, TP, IP, OP, NaOH-P, and Ca-P were determined following the SMT protocol [26,27]. NaOH-P represents the NaOH-extractable non-apatite inorganic P associated primarily with Fe and Al phases, whereas Ca-P represents the HCl-extractable apatite and other Ca-associated P. Other IP was calculated as the difference between IP and the sum of NaOH-P and Ca-P.

Sediment P concentrations were reported on a dry-mass basis as $\text{mg}\cdot\text{kg}^{-1}$. Each TP and DTP determination for the field-collected surface-water and overlying-water samples and each sediment P determination was performed in triplicate. The mean of the three

determinations was used in subsequent analyses. Detailed analytical procedures are provided in Text S2.

2.4. P Adsorption and Release Kinetic Experiments

Dry-season surface sediments from sites S1–S9 were used to characterize P adsorption and release kinetics under standardized batch conditions. The experimental procedure was adapted from Jin et al. [29], and equipment specifications are provided in Table S1.

For the adsorption experiment, 0.5000 ± 0.0002 g of freeze-dried sediment was mixed with 80 mL of phosphate solution containing $1.0 \text{ mg P} \cdot \text{L}^{-1}$. One drop of 0.1% chloroform solution was added to each suspension to suppress microbial activity. The suspensions were shaken at 150 rpm and 25 ± 1 °C. Contact times were 0.5, 1, 1.5, 3, 5, 8, 12, and 24 h.

For the release experiment, 0.5000 ± 0.0002 g of freeze-dried sediment was mixed with 100 mL of $0.02 \text{ mol} \cdot \text{L}^{-1}$ KCl solution without added phosphate. The suspensions were shaken at 200 rpm and 25 ± 1 °C. Contact times were 0.5, 1.5, 3, 5, 8, 12, and 24 h. At each contact time, the suspensions were centrifuged at $2000 \times g$ for 15 min. An unfiltered aliquot of the supernatant was subjected to potassium persulfate digestion, and TP was determined by the molybdenum blue method as described in Section 2.3. Sediment-free blanks containing the corresponding experimental solutions were processed in parallel. All adsorption and release experiments were conducted in triplicate.

The adsorbed or released P amount at time t was calculated as:

$$Q_t = \frac{\Delta C_t V}{m} \quad (1)$$

where Q_t is the adsorbed or released P amount at time t ($\text{mg} \cdot \text{g}^{-1}$), V is the solution volume (L), m is the sediment dry mass (g), and ΔC_t is the concentration difference between C_0 and C_t .

The pseudo-first-order (PFO) and pseudo-second-order (PSO) models were fitted to the original time-series data using nonlinear regression [30,31]. The PFO model is written as:

$$Q_t = Q_e (1 - e^{-k_1 t}) \quad (2)$$

and the PSO model is written as:

$$Q_t = \frac{k_2 Q_e^2 t}{1 + k_2 Q_e t} \quad (3)$$

where Q_e is the fitted equilibrium adsorption or release amount ($\text{mg} \cdot \text{g}^{-1}$), k_1 is the PFO rate constant (h^{-1}), and k_2 is the PSO rate constant ($\text{g} \cdot \text{mg}^{-1} \cdot \text{h}^{-1}$). The model-derived initial rate, h_0 , is defined as the instantaneous slope of the fitted kinetic curve at $t=0$ and is calculated as $h_0 = k_1 Q_e$ for the PFO model and $h_0 = k_2 Q_e^2$ for the PSO model.

2.5. Static Incubation of Sediment Cores

Fresh sediments from sites S1–S9 were incubated under static conditions using a method from previous studies [32,33]. Five depth intervals were examined: 1–5, 6–10, 11–15, 16–20, and 21–25 cm. For each site and depth interval, 100 g of fresh sediment (wet mass) was placed in a 1 L beaker, and 900 mL of distilled water was gently added along the beaker wall. The beakers were maintained in the dark at 20 °C for 15 d without agitation. A 10 mL water sample was collected from mid-depth on each of days 1–15 without disturbing the sediment, and no replacement water was added. To characterize release timing, cumulative P release for each site and depth interval was fitted using a three-parameter logistic model. The parameter t_{50} represents the time required to reach 50% of the final observed cumulative release on day 15. The early-stage release rate was

calculated as the slope of cumulative P release during the first 3 days. TP was determined as described in Section 2.3, and the measured TP concentration was corrected for the fivefold dilution and the corresponding blank.

Sediment water content was determined gravimetrically on a wet-mass basis and used to convert the fresh sediment mass to dry mass for release normalization (Table S2). Cumulative P release was corrected for the decrease in water volume caused by repeated sampling. At the n th sampling time, the cumulative released P mass is calculated as:

$$M_n = C_n \cdot V_n + \sum_{i=1}^{n-1} (C_i \cdot V_s) \quad (4)$$

Cumulative TP release per unit dry sediment mass is calculated as:

$$Q_n = \frac{M_n}{m} \quad (5)$$

where M_n is the cumulative P mass released by the n th sampling time (mg), C_n is the P concentration in the overlying water at the n th sampling time ($\text{mg} \cdot \text{L}^{-1}$), V_n is the actual overlying-water volume before the n th sampling (L), C_i is the P concentration in the i th previously removed water sample ($\text{mg} \cdot \text{L}^{-1}$), V_s is the sampling volume (L), Q_n is the cumulative P release per unit dry sediment mass ($\text{mg} \cdot \text{kg}^{-1}$), and m is the dry sediment mass represented by the 100 g wet sediment used in each incubation (kg).

2.6. Data Analysis and Statistics

Differences between the wet- and dry-period surveys in water P variables and surface-sediment TP, IP, OP, and BAP were assessed using paired t -tests, with sampling site as the pairing unit. Differences in individual inorganic P fractions and selected sediment properties (total Fe and TOC) between the two surveys were assessed using paired Wilcoxon signed-rank tests. Differences among the five sediment depth intervals in final cumulative TP release and early-stage release rate were assessed using Friedman tests, with sampling site as the blocking factor, followed by pairwise Wilcoxon signed-rank tests. For comparisons between the wet- and dry-period surveys, Benjamini–Hochberg correction was applied separately to the p values for water P variables, sediment P pools, individual inorganic P fractions, and selected sediment properties (total Fe and TOC) [34]. For comparisons among sediment depths, pairwise p values were adjusted separately for final cumulative TP release and early-stage release rate. Adjusted p values are reported as p_{adj} . PFO and PSO models were fitted separately to the adsorption and release time-series data from each site by nonlinear least-squares regression, and model fit was evaluated using R^2 . The early-stage release rate was calculated as the slope of the linear regression fitted to cumulative TP release over the first 3 days. All tests were two-sided, and $p < 0.05$ or $p_{\text{adj}} < 0.05$ was considered statistically significant. Statistical analyses and figures were produced using R software (version 4.4.1; R Foundation for Statistical Computing, Vienna, Austria) with the stats, readxl, dplyr, tidyr, tibble, ggplot2, patchwork, ragg, and svglite packages.

3. Results

3.1. Phosphorus Concentrations and Composition in Surface Water and Overlying Water

Phosphorus concentrations were higher in the wet season than in the dry season in both surface and overlying waters, but the changes in TP, DTP, and PP differed between the two water layers (Figure 2a). In surface water, TP, DTP, and PP were all significantly higher in the wet season than in the dry season (TP, 0.218 ± 0.024 and $0.176 \pm 0.013 \text{ mg} \cdot \text{L}^{-1}$; DTP, 0.121 ± 0.015 and $0.097 \pm 0.009 \text{ mg} \cdot \text{L}^{-1}$; PP, 0.098 ± 0.012 and $0.079 \pm 0.007 \text{ mg} \cdot \text{L}^{-1}$; all

$p_{\text{adj}} < 0.001$). The wet-season concentrations were 23.1–24.5% higher than the corresponding dry-season concentrations.

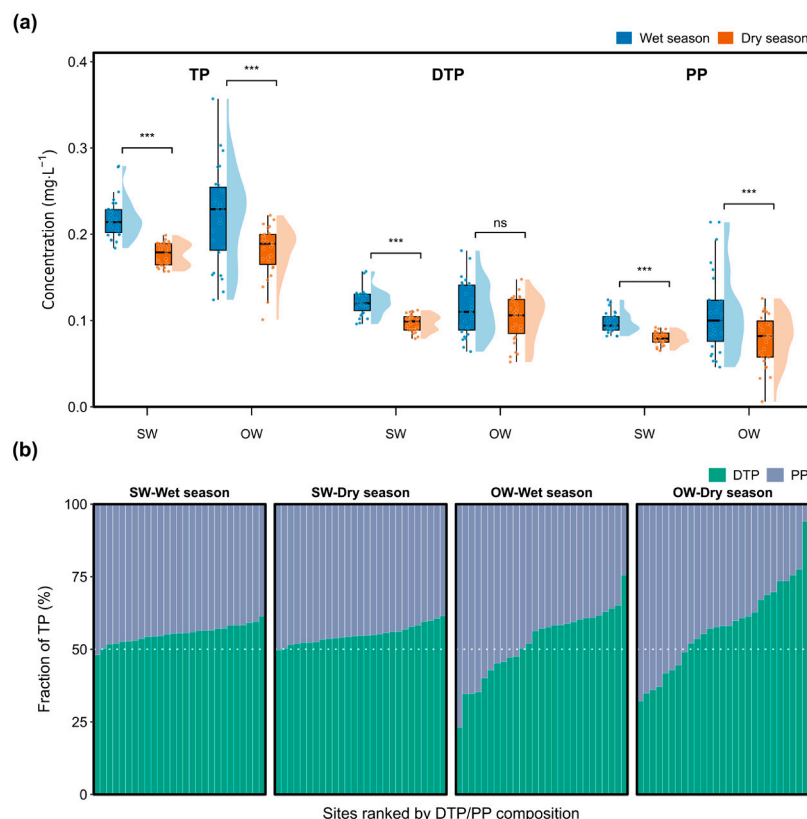


Figure 2. Concentrations and composition of P forms in surface water and overlying water of Datong Lake during the wet- and dry-season surveys. (a) Concentrations of total phosphorus (TP), dissolved total phosphorus (DTP), and particulate phosphorus (PP) in surface water (SW) and overlying water (OW). (b) Relative contributions of DTP and PP to TP at each sampling site. Sites in panel (b) were ranked by DTP/PP composition within each water layer and survey. The white dotted line in panel (b) marks 50%, indicating equal contributions of DTP and PP to TP. Half-violin plots show the data distributions, boxes show the interquartile ranges and medians, and colored points represent individual sampling sites ($n = 27$ paired sites for each water layer). Asterisks indicate significant differences between the wet- and dry-season surveys after Benjamini–Hochberg adjustment (***) indicates $p_{\text{adj}} < 0.001$; ns indicates not significant).

In overlying water, the seasonal difference was mainly observed for TP and PP. TP and PP were significantly higher in the wet season than in the dry season (TP, 0.222 ± 0.057 and 0.180 ± 0.029 mg·L⁻¹; PP, 0.108 ± 0.048 and 0.078 ± 0.029 mg·L⁻¹; both $p_{\text{adj}} < 0.001$), whereas DTP did not show a significant seasonal difference (0.113 ± 0.031 and 0.102 ± 0.026 mg·L⁻¹, $p_{\text{adj}} = 0.098$). PP had the largest wet-season increase in overlying water, followed by TP and DTP (39.4%, 23.2%, and 10.8%, respectively).

The DTP/PP composition also differed between the surface and overlying waters (Figure 2b). In surface water, DTP accounted for a stable proportion of TP in the wet and dry seasons ($55.3 \pm 3.0\%$ and $55.0 \pm 3.0\%$, respectively). In overlying water, the DTP proportion showed larger among-site variation ($52.4 \pm 11.8\%$ in the wet season and $57.6 \pm 14.9\%$ in the dry season). This indicates that the relative contributions of DTP and PP were more variable in overlying water than in surface water.

3.2. Phosphorus Pools and Inorganic P Fractions in Surface Sediments

Surface sediments contained high phosphorus concentrations, with IP as the dominant P pool (Figure 3a). TP and IP were significantly higher in the dry season than in the wet season. TP increased from $1911.07 \pm 162.94 \text{ mg} \cdot \text{kg}^{-1}$ in the wet season to $2088.23 \pm 131.94 \text{ mg} \cdot \text{kg}^{-1}$ in the dry season, and IP increased from 1712.29 ± 147.69 to $1873.08 \pm 117.22 \text{ mg} \cdot \text{kg}^{-1}$ (both $p_{\text{adj}} < 0.05$). In contrast, OP showed no significant seasonal difference (198.79 ± 19.15 and $215.15 \pm 24.27 \text{ mg} \cdot \text{kg}^{-1}$ in the wet and dry seasons, respectively; $p_{\text{adj}} > 0.05$). IP accounted for nearly 90% of TP in both seasons ($89.59\% \pm 0.55\%$ and $89.70\% \pm 0.88\%$).

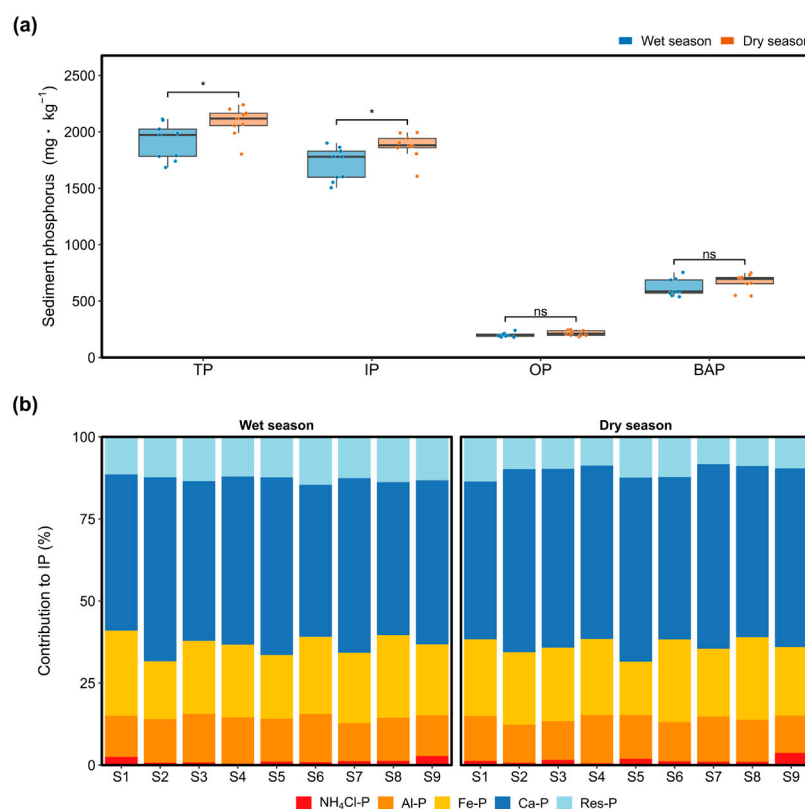


Figure 3. Seasonal differences in sediment phosphorus pools and inorganic P composition in surface sediments of Datong Lake. (a) Concentrations of TP, IP, OP, and BAP in the wet and dry seasons. (b) Contributions of $\text{NH}_4\text{Cl-P}$, Al-P, Fe-P, Ca-P, and Res-P to IP at each sediment site. Asterisks indicate significant seasonal differences between wet and dry seasons (* indicates $p_{\text{adj}} < 0.05$; ns indicates not significant).

The IP pool was dominated by Ca-P at all sediment sites (Figure 3b). Ca-P accounted for $50.97 \pm 3.43\%$ of IP in the wet season and $53.96 \pm 3.05\%$ in the dry season, and its concentration was significantly higher in the dry season (873.02 ± 97.99 and $1009.68 \pm 70.39 \text{ mg} \cdot \text{kg}^{-1}$ in the wet and dry seasons, respectively; $p_{\text{adj}} < 0.05$; Table 1). Fe-P, Al-P, and Res-P were secondary fractions, whereas $\text{NH}_4\text{Cl-P}$ contributed less than 0.3% of IP. Among the five inorganic P fractions, only Ca-P showed a significant seasonal difference (Table 1). This result indicates that the dry-season increase in IP was mainly associated with Ca-P rather than a broad increase in all inorganic P fractions.

BAP showed no significant seasonal difference between the wet and dry seasons (616.70 ± 76.23 and $667.31 \pm 74.44 \text{ mg} \cdot \text{kg}^{-1}$, respectively; $p_{\text{adj}} > 0.05$; Figure 3a). The BAP/TP ratio also remained similar between seasons, accounting for $32.26 \pm 2.56\%$ of TP in the wet season and $31.90 \pm 2.29\%$ in the dry season. Thus, although the sediment P pool was dominated by IP and Ca-P, approximately one-third of TP was still present as bioavailable P. Total Fe did not differ significantly between the wet- and dry-period

surveys (53.34 ± 1.63 and 54.54 ± 3.20 $\text{g} \cdot \text{kg}^{-1}$, respectively; $p_{\text{adj}} = 0.129$; Table S3). TOC increased by 33.1%, from 16.6 ± 5.6 $\text{g} \cdot \text{kg}^{-1}$ in the wet-period survey to 22.1 ± 7.7 $\text{g} \cdot \text{kg}^{-1}$ in the dry-period survey ($p_{\text{adj}} = 0.008$; Table S3).

Table 1. Seasonal differences in inorganic phosphorus fractions in surface sediments of Datong Lake.

Inorganic Phosphorus Fraction	Wet Season Concentration ($\text{mg} \cdot \text{kg}^{-1}$)	Dry Season Concentration ($\text{mg} \cdot \text{kg}^{-1}$)	Wet Season Contribution to IP (%)	Dry Season Contribution to IP (%)	p_{adj} for Concentration Difference
$\text{NH}_4\text{Cl-P}$	4.43 ± 2.72	5.24 ± 2.97	0.26 ± 0.16	0.29 ± 0.19	0.161
Al-P	230.14 ± 30.71	242.16 ± 24.79	13.41 ± 1.04	12.93 ± 1.13	0.426
Fe-P	382.13 ± 56.07	419.91 ± 65.57	22.34 ± 2.67	22.35 ± 2.70	0.161
Ca-P	873.02 ± 97.99	1009.68 ± 70.39	50.97 ± 3.43	53.96 ± 3.05	0.039
Res-P	221.79 ± 22.31	196.23 ± 38.73	12.97 ± 0.97	10.47 ± 1.92	0.137

3.3. Depth Profiles of Phosphorus Fractions in Sediment Cores

TP differed significantly among depth intervals in the dry-season sediment cores (Friedman test across depth intervals, $p < 0.001$; Figure 4a), and the mean TP concentrations decreased with depth. High TP concentrations were mainly distributed in the upper sediment layers, with most high values occurring within the upper 5 cm. The mean TP concentration was 2081 ± 151 $\text{mg} \cdot \text{kg}^{-1}$ in the 1–5 cm interval and decreased to 1563 ± 199 $\text{mg} \cdot \text{kg}^{-1}$ in the 6–10 cm interval. TP decreased rapidly by about 12 cm and reached approximately 1000 $\text{mg} \cdot \text{kg}^{-1}$ at around 12 cm. Below this depth, TP remained at much lower concentrations, with mean values of 970 ± 63 , 849 ± 68 , and 776 ± 66 $\text{mg} \cdot \text{kg}^{-1}$ in the 11–15, 16–20, and 21–25 cm intervals, respectively. The deepest intervals had low TP concentrations and showed only small changes with depth. The depth distribution shows that TP was mainly concentrated in the upper part of the sediment profiles.

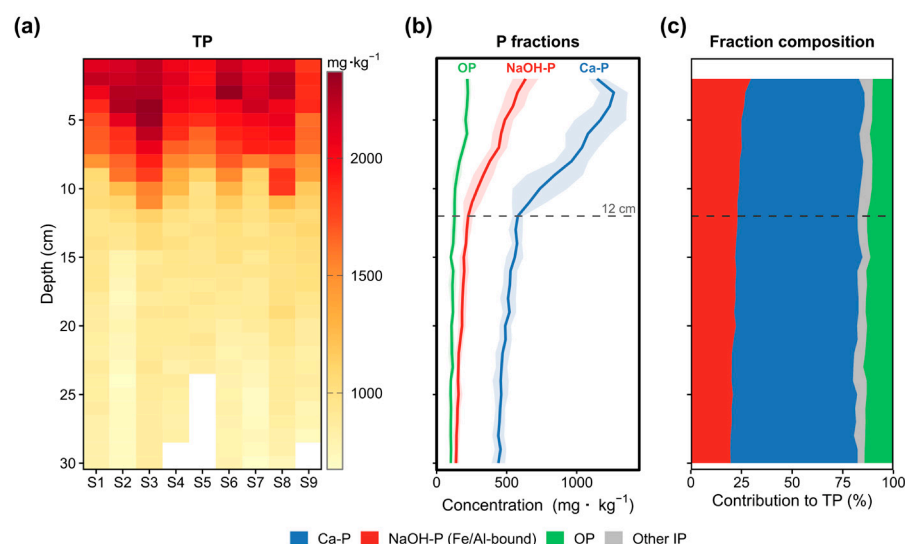


Figure 4. Vertical distribution of total phosphorus and phosphorus fractions in sediment cores from Datong Lake. (a) Depth–site distribution of total phosphorus (TP). (b) Depth profiles of major phosphorus fractions, including Ca-P, NaOH-P, and OP. (c) Relative contribution of phosphorus fractions to total phosphorus. The dashed line indicates the approximate depth boundary at 12 cm. The number of site-specific sediment cores represented at each depth was $n = 9$ at 1–25 cm, $n = 8$ at 26–28 cm, and $n = 6$ at 29–30 cm; the P-fraction profiles began at 2 cm.

Ca-P, NaOH-P, and OP also decreased with depth, but their decreases were not the same (Figure 4b). In the 2–5 cm interval, Ca-P, NaOH-P, and OP averaged 1203 ± 107 , 561 ± 69 , and 216 ± 14 $\text{mg} \cdot \text{kg}^{-1}$, respectively. In the 20–30 cm interval, their mean

concentrations decreased to 464 ± 38 , 156 ± 18 , and 107 ± 13 $\text{mg} \cdot \text{kg}^{-1}$, respectively. NaOH-P showed the largest proportional decline from the upper layers to the middle part of the profiles. Ca-P was the largest measured P fraction throughout the profiles, although its concentration also decreased with depth. OP was also higher in the upper layers and lower in deeper sediments.

The contribution of each fraction to TP changed with depth (Figure 4c). Ca-P accounted for the largest proportion of TP throughout the sediment profiles, increasing from $57.9 \pm 2.8\%$ in the 2–5 cm interval to $61.4 \pm 3.1\%$ in the 26–30 cm interval. In contrast, the contribution of NaOH-P decreased from $26.9 \pm 2.2\%$ to $20.0 \pm 2.0\%$ over the same depth range. OP accounted for $10.4 \pm 0.6\%$ of TP in the 2–5 cm interval and $14.0 \pm 0.4\%$ in the 26–30 cm interval. This relative increase occurred because OP concentration decreased less proportionally than TP with depth. Other IP accounted for a small proportion of TP, generally around 4–5%.

3.4. P Adsorption and Release Kinetics in Surface Sediments

The PFO and PSO models were fitted to the original time-series data using nonlinear regression. The PSO model fitted both P adsorption and P release better than the PFO model (Table 2). For adsorption, the mean R^2 was 0.972 ± 0.014 for PSO and 0.833 ± 0.038 for PFO. For release, the mean R^2 was 0.957 ± 0.014 for PSO and 0.899 ± 0.040 for PFO. The differences between the two models were significant for both processes (both $p < 0.001$). Therefore, the PSO parameters were used to describe the adsorption and release kinetics.

Table 2. Nonlinear kinetic parameters of P adsorption and release in surface sediments.

Process	Model	Q_e ($\text{mg} \cdot \text{g}^{-1}$)	k_1 (h^{-1})	k_2 ($\text{g} \cdot \text{mg}^{-1} \cdot \text{h}^{-1}$)	Initial Rate, h_0 ($\text{mg} \cdot \text{g}^{-1} \cdot \text{h}^{-1}$)	R^2
P adsorption	PFO	0.133 ± 0.003	1.886 ± 0.296	—	0.251 ± 0.041	0.833
	PSO	0.141 ± 0.003	—	24.06 ± 5.22	0.477 ± 0.103	0.972
P release	PFO	0.028 ± 0.003	0.633 ± 0.174	—	0.017 ± 0.004	0.899
	PSO	0.031 ± 0.003	—	31.77 ± 13.30	0.028 ± 0.007	0.957

P adsorption increased rapidly during 0.5–3 h and then approached equilibrium (Figure 5a). The mean adsorbed P reached 0.126 ± 0.006 $\text{mg} \cdot \text{g}^{-1}$ at 3 h and increased only slightly thereafter, reaching 0.140 ± 0.003 $\text{mg} \cdot \text{g}^{-1}$ at 24 h. The PSO-fitted Q_e for adsorption was 0.141 ± 0.003 $\text{mg} \cdot \text{g}^{-1}$, which was close to the observed value at 24 h. The PSO initial rate, h_0 , was 0.477 ± 0.103 $\text{mg} \cdot \text{g}^{-1} \cdot \text{h}^{-1}$.

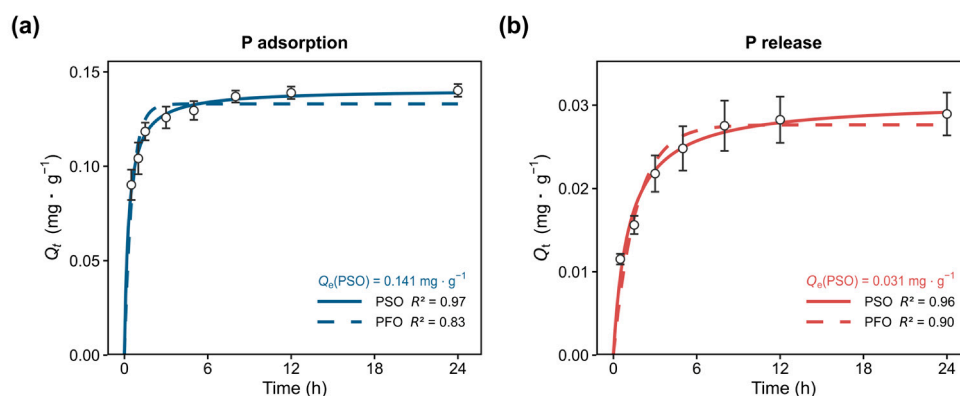


Figure 5. P adsorption and release kinetics in surface sediments of Datong Lake. (a) P adsorption kinetics. (b) P release kinetics. Points and error bars represent the mean $\pm$ SD of the nine sediment sites. Solid and dashed lines represent pseudo-second-order (PSO) and pseudo-first-order (PFO) model fits, respectively.

P release also showed a rapid increase followed by a slower change, but the released amount was much lower than the adsorbed amount (Figure 5b). Released P increased from $0.0115 \pm 0.0006 \text{ mg} \cdot \text{g}^{-1}$ at 0.5 h to $0.0248 \pm 0.0027 \text{ mg} \cdot \text{g}^{-1}$ at 5 h, and then increased slowly to $0.0289 \pm 0.0026 \text{ mg} \cdot \text{g}^{-1}$ at 24 h. The release curve was close to equilibrium after 8–12 h. The PSO-fitted Q_e for release was $0.031 \pm 0.003 \text{ mg} \cdot \text{g}^{-1}$, and h_0 was $0.028 \pm 0.007 \text{ mg} \cdot \text{g}^{-1} \cdot \text{h}^{-1}$.

The fitted parameters showed a clear difference between adsorption and release. The PSO-fitted Q_e for release was about 22% of that for adsorption, and the initial rate for release was about 6% of that for adsorption. Thus, both the fitted equilibrium amount and initial rate were lower for P release than for P adsorption under the tested conditions.

3.5. Depth-Dependent P Release from Sediment Cores

Cumulative P release increased throughout the 15-d incubation at all five depth intervals, but the released amounts differed among depths (Figure 6a). The release curves separated early and maintained the same order throughout the incubation: 1–5 cm > 6–10 cm > 11–15 cm > 16–20 cm > 21–25 cm. The 1–5 cm and 6–10 cm intervals increased more rapidly during the first 8–10 d, and their slopes decreased near the end of incubation. The deeper intervals also released P continuously, but their cumulative release remained much lower than that of the upper intervals.

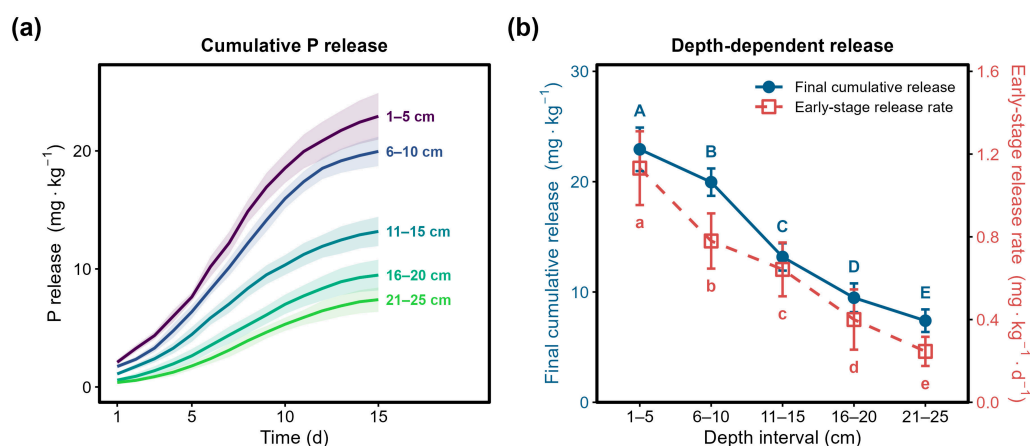


Figure 6. Depth-dependent static release of phosphorus from sediment cores of Datong Lake. (a) Cumulative phosphorus release over time for five sediment depth intervals (1–5, 6–10, 11–15, 16–20, and 21–25 cm); solid lines and shaded bands denote the mean and standard deviation across sites ($n = 9$). (b) Final cumulative release (left axis) and early-stage release rate (the initial slope over the first three days; right axis) as a function of depth interval. Different letters indicate significant differences among depth intervals (Wilcoxon signed-rank test with Benjamini–Hochberg correction, $p_{\text{adj}} < 0.05$), with uppercase letters (A–E) denoting final cumulative release and lowercase letters (a–e) denoting the early-stage release rate. Phosphorus release is expressed per unit dry sediment mass.

Day-15 cumulative P release differed significantly among depth intervals (Friedman test, $p < 0.001$; Figure 6b). The highest release occurred in the 1–5 cm interval, with a mean value of $22.9 \pm 1.9 \text{ mg} \cdot \text{kg}^{-1}$. The 6–10 cm interval had the second highest release, with a mean value of $19.9 \pm 1.2 \text{ mg} \cdot \text{kg}^{-1}$. Release then decreased to 13.1 ± 1.2 , 9.5 ± 1.0 , and $7.2 \pm 1.0 \text{ mg} \cdot \text{kg}^{-1}$ in the 11–15, 16–20, and 21–25 cm intervals, respectively. Relative to the 1–5 cm interval, final cumulative P release decreased by 68.4% in the 21–25 cm interval (Table 3). The upper 10 cm showed the greatest P release per unit dry sediment mass.

The early-stage release rate also decreased significantly with depth (Friedman test, $p < 0.001$; Figure 6b). The 1–5 cm interval had the highest rate, averaging $1.13 \pm 0.18 \text{ mg} \cdot \text{kg}^{-1} \cdot \text{d}^{-1}$, followed by the 6–10 cm interval at $0.78 \pm 0.13 \text{ mg} \cdot \text{kg}^{-1} \cdot \text{d}^{-1}$. The rate decreased to $0.64 \pm 0.13 \text{ mg} \cdot \text{kg}^{-1} \cdot \text{d}^{-1}$ in the 11–15 cm interval and to 0.40 ± 0.15

and $0.24 \pm 0.07 \text{ mg} \cdot \text{kg}^{-1} \cdot \text{d}^{-1}$ in the 16–20 and 21–25 cm intervals, respectively. The early-stage release rate in the 21–25 cm interval was 78.8% lower than that in the 1–5 cm interval (Table 3). The upper sediment intervals therefore had both higher final cumulative release and faster early-stage release.

Table 3. Depth attenuation and timing parameters of P release during static incubation.

Depth Interval (cm)	Decrease in Final Cumulative P Release Relative to 1–5 cm (%)	Decrease in Early-Stage Release Rate Relative to 1–5 cm (%)	Time to 50% of Final Cumulative Release, t_{50} (d)	Curve-Fitting R^2
1–5	0	0	6.7 ± 0.4	0.979 ± 0.007
6–10	13.2	31.0	6.9 ± 0.3	0.977 ± 0.005
11–15	42.6	43.4	6.7 ± 0.6	0.981 ± 0.008
16–20	58.5	64.6	7.4 ± 0.7	0.977 ± 0.011
21–25	68.4	78.8	7.8 ± 0.4	0.969 ± 0.009

The time required to reach half of the final cumulative release varied less among depth intervals. The t_{50} values ranged from 6.7 ± 0.4 to 7.8 ± 0.4 d, with slightly longer values in the 16–20 and 21–25 cm intervals (Table 3). The absolute range of t_{50} was small compared with the decreases in final cumulative release and early-stage release rate. The main depth-related difference was therefore in the amount and early-stage rate of P release.

4. Discussion

4.1. Seasonal Differences in Phosphorus Between Surface and Overlying Waters

In surface water, TP, DTP, and PP were all higher in the wet season than in the dry season, whereas the DTP/TP ratio remained almost unchanged at 55.3% and 55.0%, respectively. This stable partitioning indicates that the wet-season increase in TP involved proportional enrichment of both dissolved and particulate P rather than a pronounced shift toward either form. The pattern likely reflects increased hydrological inputs during the wet season. Monsoon rainfall increases catchment runoff, which can transport dissolved P together with P associated with eroded soil, drainage material, and organic particles [35]. The resulting P input may have outweighed dilution associated with the higher lake-water volume. Earlier monitoring of Datong Lake during 2018–2020 showed a similar pattern: TP increased with water level, declined markedly from summer to autumn [20], and reached its highest concentrations in July and August [19].

In overlying water, the wet-season increase was more strongly associated with PP. Based on the mean concentrations, PP accounted for approximately 71% of the wet-season increase in TP, whereas DTP changed only slightly. This particulate phosphorus may derive from settling algae and detritus and from the resuspension of recently deposited material. Resuspension is common here because the lake is shallow. At a mean depth of 2.58 m, wind mixing reaches the bed and lifts surface sediment back into the water column [36]. The same particle-dominated pattern occurs in other eutrophic shallow lakes. There, organic and externally derived particles dominate near-bottom PP (over 88% in Lake Taihu [37]), and wind reworking can release both legacy and bioavailable phosphorus [11].

The dissolved pool rose only slightly. DTP increased by 10.8% and did not differ significantly between seasons ($p_{\text{adj}} = 0.098$) compared with the 39.4% rise in PP. This weak response may reflect a balance between dissolved-P supply and removal. Organic-matter mineralization, Fe-bound P dissolution, and upward porewater transport can replenish dissolved P near the sediment surface [38], while biological uptake [36] and sorption to suspended particles [39] remove it from solution. As a result, the dissolved pool may be continuously replenished without showing substantial net accumulation. Part

of the released phosphate may therefore enter the particulate pool instead of building up in solution.

4.2. Storage Forms and Vertical Distribution of Legacy Sediment Phosphorus

Surface sediments in Datong Lake contained a large legacy P pool dominated by inorganic forms. IP accounted for nearly 90% of TP, and Ca-P alone for 45.7–48.4%. Ca-P similarly accounted for 43.7–50.8% of TP in a 2021 survey [18]. A whole-lake restoration study reported that macrophyte restoration approximately halved the sediment P concentrations, particularly Ca-P [16]. Although its concentration declined, Ca-P remained the dominant fraction in the 2021 field survey [18] and in the present study, suggesting that lake recovery reduced its concentration without changing the principal storage form. Ca-associated P is generally less susceptible to redox-driven remobilization than Fe-associated P, which may favor its persistence during burial [8]. Between-survey variation was concentrated in Ca-P, which accounted for approximately 77% of the mean TP difference. BAP, total Fe, and Fe-P did not differ significantly between surveys. TOC was 33.1% higher in the dry-period survey, but OP showed no significant increase. Thus, the higher dry-period TP mainly reflected Ca-P enrichment; no corresponding increase was detected in the bioavailable, Fe-associated, or organic-P pools.

The Ca-P-dominated sediment also contained substantial BAP. BAP reached 617–667 mg·kg^{−1} and represented about 32% of TP. More than 99% of BAP was operationally assigned to Fe-P and Al-P, with NH₄Cl-P contributing less than 1%. In Datong Lake, warm, low-oxygen conditions have been linked to elevated porewater DRP and Fe reduction [18]. In Qinglan Lake, another retired aquaculture lake analyzed using a comparable fractionation scheme, BAP averaged 362 mg·kg^{−1} and accounted for 45% of TP [40]. Datong Lake had a lower BAP/TP ratio but a substantially higher BAP concentration because its sediment TP was higher. Thus, a high concentration of potentially responsive Fe/Al-associated P coexisted with the dominant Ca-P fraction.

The P-rich upper sediment is consistent with the aquaculture history of Datong Lake. TP declined from 2081 mg·kg^{−1} in the upper 5 cm to about 1000 mg·kg^{−1} by 12 cm and remained lower beneath this depth. A ²¹⁰Pb/¹³⁷Cs-dated core indicated that the 10 cm horizon corresponded to approximately 1980, after which aquaculture intensified and reconstructed lake-water TP increased [15]. An undredged site in the lake also contained an approximately 7 cm P-enriched surface layer [17]. Together, these records suggest that the upper 12 cm integrates aquaculture-era P accumulation with subsequent deposition and early-diagenetic redistribution.

The major sediment P fractions declined to different degrees with depth. Between 2–5 and 20–30 cm, NaOH-P declined by 72.2%, compared with about 61% for Ca-P and 50.5% for OP. The steeper NaOH-P decline is consistent with early-diagenetic redistribution of Fe/Al-associated P as migrating redox fronts dissolve and re-form Fe oxides, whereas Ca-associated P is retained more efficiently during burial [8]. Across the profile, Ca-P declined less strongly than TP, so its contribution increased from 57.9% at 2–5 cm to 61.4% at 26–30 cm. OP also declined in concentration but increased in contribution from 10.4% to 14.0%. Because organic P compounds mineralize at different rates, labile forms are preferentially lost, whereas slower-turnover compounds associated with minerals or humic matter persist, which may explain the increasing relative contribution of OP in deeper layers [41].

4.3. Sediment Phosphorus Exchange and Depth-Dependent Release Potential

Surface sediment had a much greater short-term capacity to adsorb dissolved P than to release stored P. The fitted adsorption amount was approximately 4.5 times the fitted

release amount. Adsorption and release were both rapid at first and then slowed, with most of each process occurring during the early stage. Sediments from nine lakes in the middle and lower Yangtze region showed the same pattern, with P release proceeding more slowly than adsorption [42].

The amount released was small relative to the BAP pool. The fitted batch release represented approximately 1.5% of surface-sediment TP and 4.6% of BAP, while the depth incubations released 0.93–1.35% of TP. BAP was therefore much larger than the amount of P released over days. A similar difference was found in shallow Lake Ormstrup, where only 11% of the P classified as potentially mobile in the upper 10 cm was lost over 50 d [43]. BAP in Datong Lake represents a larger, longer-term release potential, whereas short-term release involved only a small fraction.

Depth mainly affected the amount of P released. Cumulative release and the early release rate decreased by 68.4% and 78.8%, respectively, from the uppermost to the deepest layer, following the vertical decreases in TP and NaOH-P. The proportion of TP released remained within 0.93–1.35%, and t_{50} varied only from 6.7 to 7.8 d. Higher release from the upper sediment was associated with its higher TP and NaOH-P concentrations. Other depth-resolved lake-sediment incubations also found that P release decreased with depth and was positively related to TP and NaOH-extractable P, with most release originating from the upper 15 cm [33].

4.4. Implications for Sediment Phosphorus Control

The results indicate that the management of legacy sediment P in Datong Lake should focus on the upper sediment rather than the entire sediment P stock. Sediment TP and NaOH-P declined markedly by approximately 12 cm, whereas the greatest cumulative release and early-stage release rates occurred in the 1–10 cm intervals, while the difference between the wet- and dry-season surveys was driven mainly by Ca-P and BAP remained similar. Moreover, only a small fraction of TP was released during the incubations. Bulk sediment TP therefore overstates the portion of the sediment P stock involved in short-term exchange, whereas depth-resolved P composition and release measurements more clearly identify the layer relevant to management.

Previous work in Datong Lake identified an approximately 7-cm P-enriched surface layer, although dredging extended to approximately 1.1 m [17]. Dredging reduced surface Ca-P and summer SRP diffusion, but a newly deposited 14-cm layer enriched in Fe-P and OP developed within two years [17]. This comparison suggests that the depth of sediment intervention should be matched to the vertical extent of the P-enriched layer and that deep removal may not provide durable control where rapid deposition leads to renewed surface enrichment. Long-term effectiveness also depends on limiting continued P inputs and sediment reworking [44,45]. Wet-season increases in dissolved P and overlying-water PP are consistent with continued catchment delivery and enhanced particle transport within the lake. Management focused only on dissolved release would therefore overlook an important particulate pathway.

5. Conclusions

Datong Lake sediments retained high P concentrations after aquaculture ceased, but only a small proportion of sediment P was released during the short-term experiments. Ca-P was the dominant sediment P fraction. P release was highest in the sediment from the surface to approximately 12 cm depth and decreased markedly below this layer. Therefore, bulk sediment TP alone does not indicate short-term release potential; sediment P fractions and their vertical distribution must also be considered. The wet-season increase in overlying-water P was mainly associated with PP, indicating that particulate P contributed

substantially to the difference between the wet- and dry-season surveys. These results indicate that sediment management should focus on the shallow P-enriched layer. Continued external P control is also needed to limit renewed P accumulation in surface sediments.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/w18151857/s1>, Text S1: Details of sampling, sample preparation, and laboratory equipment; Text S2: Procedures for phosphorus analysis and sediment fractionation; Text S3: Determination of total Fe and total organic carbon in surface sediments; Table S1: Principal equipment and materials; Table S2: Water content of sediments used for static incubation (%); Table S3: Site-specific total Fe and total organic carbon (TOC) concentrations in the 0–1 cm surface sediments collected during the wet- and dry-period surveys.

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