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Connecting sources, fractions and algal availability of sediment phosphorus in shallow lakes: An approach to the criteria for sediment phosphorus concentrations

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A B S T R A C T

Although point and nonpoint sources contribute roughly equal nutrient loads to lakes, their relative role in supporting algae growth has not been clarified. In this research, we have established a quantitative relationship between algae-available phosphorus (P) and P chemical fractions in sediments; the latter indicates the relative contribution of point versus nonpoint sources. Surface sediments from three large shallow lakes in eastern China, namely, the Chaohu, Taihu and Hongzehu Lakes, were sampled to assess their algae-available P and chemically extracted P fractions. The algae-available P primarily comes from iron/aluminium (hydr)oxide-bound P (Fe/Al-bound P), 45% of which is algae-available P. The ratio of Fe/Al-bound P to calcium compound-bound P (Ca-bound P) indicated the relative contribution of point to nonpoint sources, with the point sources contributing the majority of increased Fe/Al-bound P in sediments. Therefore, the reduction of point sources from urbanized areas, rather than nonpoint sources from agricultural areas that primarily contribute to the Ca-bound P fraction, should be prioritized to alleviate cyanobacterial algal blooms (CyanoHABs) in shallow lakes with sediment P as a potential source to support algae growth. With these important results, we proposed a conceptual model for "P-pumping suction" from sediments to algae to aid in the development of the criteria for sediment P concentrations in shallow lakes.

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Introduction

In recent decades, excess phosphorus (P) has been loaded into lakes, causing eutrophication and harmful algae blooms (Elser and Bennett, 2011; Fink et al., 2018). After entering the lake, most P is associated with particulate matter and deposited into sediments, from which P can be released to the water column to increase algal biomass (Søndergaard et al., 2003; Horppila, 2019; Zhang et al., 2020). Compared to deep lakes, P cycles between sediments and the water column in shallow lakes are particularly active because of the short physical distance between sediments and surface water, from which energy can be easily transported to the lake bottom (Tammeorg et al., 2013; Tang et al., 2013; Qin et al., 2020; Zou et al., 2020). In shallow lakes, the water quality improvement and control of cyanobacterial harmful algal blooms (CyanoHABs) is more dependent on the reduction of sediment P pools if the external P load is substantially reduced (Wang et al., 2019; Xu et al., 2021). Therefore, it is necessary to develop criteria for sediment P concentrations to aid in water quality management as well as pollution control (Rydin and Carey, 2011). At present, the criteria for sediment P concentration are still lacking compared to other toxic substances (Burton Jr et al., 2002). At present, sediment P control is mostly associated with the P release flux that is used to estimate the contribution of sediments (Fan et al., 2020). However, estimates of sediment P release potential rely on all kinds of experimental measures, which are site-specific and affected by many factors, e.g., estimate methods, sediment properties and environmental conditions (temperature, oxygen, pH, resuspension, etc.). The criteria for sediment P concentration connecting sediment properties and algae growth are necessary.

It is well known that the P bioavailability of sediments is associated with Fe/Al (hydr)oxides. Iron reduction in subsurface sediments results in P release and diffusion towards the water column through pore water (Ding et al., 2018a; Zhang et al., 2020). More importantly, Fe/Al (hydr)oxide-adsorbed P would be directly desorbed with pH increase during algae growth in shallow lakes, resulting in an effect of “P-pumping suction” from sediments to algae (Xie et al., 2003; Zhang et al., 2020; Deng et al., 2022). This indicates that a direct relationship between algal biomass and sediment P pools could be established in shallow lakes, making the criteria for sediment P concentration possible.

Sediment bioavailable P pools are mainly assessed by bioassay techniques or chemical extraction methods. Bioassays of algal growth potential (AGP) can evaluate the P amount directly available by algae, which has been widely applied worldwide (Skulberg, 1964; Chiaudani and Vighi, 1974; Klapwijk et al., 1982; Vries and Klapwijk, 1987; Klapwijk et al., 1989; Gerdes and Kunst, 1998). The chemical method has been popular because it is simple and time-saving compared to bioassays, but it is difficult to determine which fraction and how much P is exactly available by algae. Therefore, a quantitative relationship between algal available P and chemical fractions of sediments is crucial to develop the criteria for sediment P concentrations in shallow lakes.

Sediment P concentration and its fractional composition are the most important parameters that indicate P pollution level, bioavailability and potential mobility/immobilization under different environmental factors (Kaiserli et al., 2002). Sediment P is generally fractionated into Fe (hydr)oxide-bound P (Fe-bound P), Al (hydr)oxide-bound P (Al-bound P), Ca compound-bound P (Ca-bound P), and organic P. The Fe-bound P tends to be released under anoxia, Al-bound P is sensitive to alkaline environments, and Ca-bound P is stable in circumneutral or alkaline lakes (Kopáček et al., 2005). Moreover, sediment P fractions could indicate P pollution informatics from different sources, which are typically classified as point and nonpoint sources. The former is primarily due to the discharge of sewage effluents or livestock manure. The latter comprises all P entering the surface water system in a diffuse manner, mainly originating from agricultural fertilizer and soil erosion (Yi et al., 2020a). Sediments more impacted by point sources usually have a higher proportion of Fe/Al (hdyr)oxide-bound P (Fe/Al-bound P), while those more impacted by nonpoint sources have a higher proportion of Ca compound-bound P (Ca-bound P, Yi et al., 2017b; Lannergård et al., 2020). Considering the availability of Fe/Al-bound P and unavailability of Ca-bound P to algae, the P from point sources could play a more important role than that from nonpoint sources in providing algae-available P with sediments as P sources in shallow lakes. At this point, sediment informatics that indicates P-origin sources and availability could clarify pollution control strategies at a watershed scale, which is concerned with a balance between the reduction of point sources versus nonpoint sources (Yi et al., 2017a).

On the spatial scale, sediment P presented heterogeneity not only among different lakes but also in different sampling sites in each lake depending on the P load and the relative proportion of each source. Some lakes receive a P load with a dominant contribution from point sources, while some lakes receive a P load with a dominant contribution from nonpoint sources. In a large lake, the P load from different inflowing rivers also presented spatial heterogeneity on the P load with differing contributing sources from their respective subwatersheds (Yi et al., 2017b). Therefore, the indictors reflecting the relative contribution of point versus nonpoint sources could be revealed through a number of sediment samples across several representative large lakes, which usually present differences in P load with its contributing sources.

In this research, we focused on the connection between sources, sediment fractions and algae availability of P in sediments, aiding in the development of the criteria for sediment P concentrations to alleviate CyanoHABs in shallow lakes. We had three objectives in this research: (1) to establish a quantitative relationship between P availability and its chemical fractions in sediments, (2) to clarify the relative importance of point versus nonpoint sources to provide algae-available P with sediments as potential P sources, and (3) to discuss the criteria for sediment P concentrations in shallow lakes.

1. Materials and methods

1.1. Study areas, lakes and sampling sites

The division of shallow lakes in eastern China is one of five geographical divisions across China; these lakes are mainly dis-

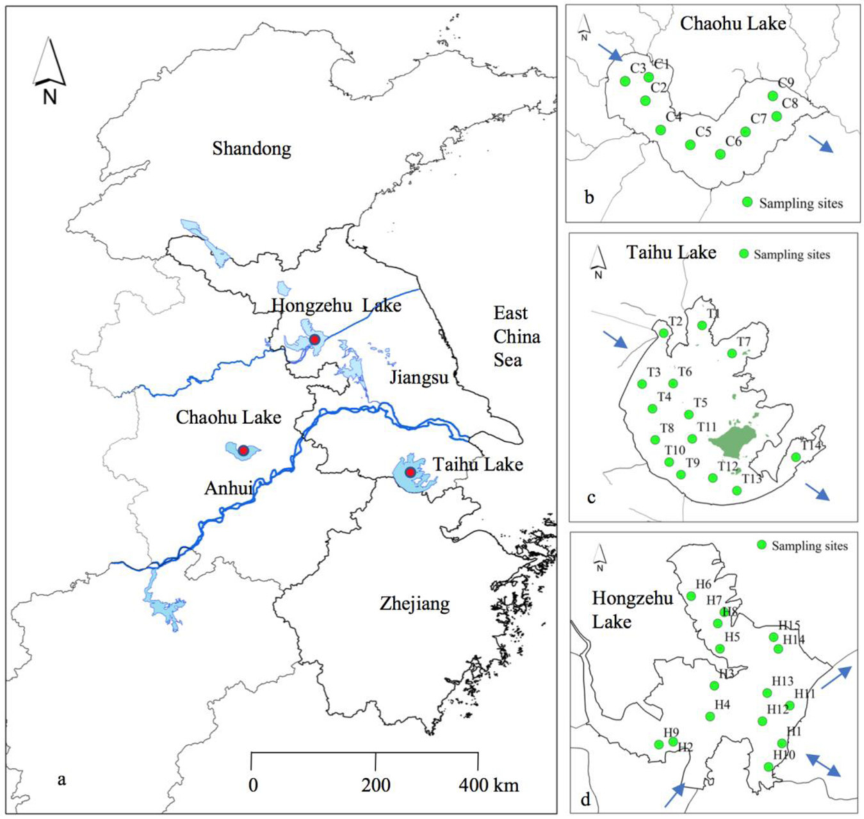

Fig. 1 – Study area (a) and location of the sampling sites in Chaohu Lake (b), Taihu Lake (c) and Hongzehu Lake (d). The blue arrows indicate the directions of inflow and outflow in each lake.

tributed in the flood plains of the Yangtze River and Huaihe River (Zhang et al., 2019). The total area of these lakes is approximately 21,000 km^2, accounting for 25% of the lake area in China. Due to heavy nutrient loads with rapid economic developments in the last four decades since 1980 (Liu et al., 2012a; Liu et al., 2012b; Liu et al., 2013), these lakes have been subjected to eutrophication (Guan et al., 2020a; Wang et al., 2020). The P released from sediments is thought to make a major contribution to sustainable CyanoHABs in several large lakes (Qin et al., 2020; Wang et al., 2019; Zhang et al., 2020; Deng et al., 2022).

We choose three large lakes, Chaohu Lake, Taihu Lake, and Hongzehu Lake, downstream of the Yangteze River and Huaihe River (Fig. 1), which are three of the ten largest rivers in China. These three lakes receive different P loads from their watersheds, with spatial heterogeneity of sediment P not only among these three lakes but also inside each lake (Yi et al., 2007a; Wan et al., 2020; Deng et al., 2022). Chaohu Lake is the ninth largest freshwater lake in China, with an area of 787 km^2 and a water depth of 2.9 m on average. Taihu Lake is the third largest freshwater lake, with an area of 2246 km^2 and a water depth of 1.8 m on average. Hongzehu Lake is the fourth largest lake in China, with an area of 1960 km^2 and a water depth of 1.8 m. Taihu Lake and Chaohu Lake belong to the Yangtze River watershed, which is experiencing severe CyanoHABs during warm seasons (Wan et al., 2019; Guan et al., 2020b). Hongzehu Lake belongs to the Huaihe River watershed, which accepts the inflow of the Huaihe River and discharges the outflow to the Yangtze River and East China Sea.

These three lakes are located in a region with a subtropical monsoon climate, with air temperatures of 15.5-16.5°C and a flooding season from April to August. We set 9 sampling sites in Chaohu Lake (C1-C9, as shown in Fig. 1b), 15 sampling sites in Taihu Lake (T1-T15, as shown in Fig. 1c), and 14 sampling sites in Hongzehu Lake (H1-H14, as shown in Fig. 1d), covering the main ecological zones in space from the inflow to outflow areas of these large lakes.

1.2. *Water quality analysis and P fractionation in the sediments*

Water quality samples and surface sediment samples at each sampling site in the three lakes were collected in the summer of 2019 using a 5-L Plexiglas water sampler and a Peterson gravity sampler, respectively. The water samples were packed in an ice box, brought to the laboratory and analysed following standard methods (APHA et al., 1998). The parameters for water quality analysis included total nitrogen (TN), nitrate (NO_3-N), nitrite (NO_2-N), ammonium (NH_4-N), total phosphorus (TP), and soluble reactive phosphorus (SRP). The TN samples were digested by alkaline reagent and determined via the UV–visible colorimetric method, and the TP samples were digested with persulfate and determined via the acid–molybdenum-blue colorimetric method. NO_3-N and SRP were

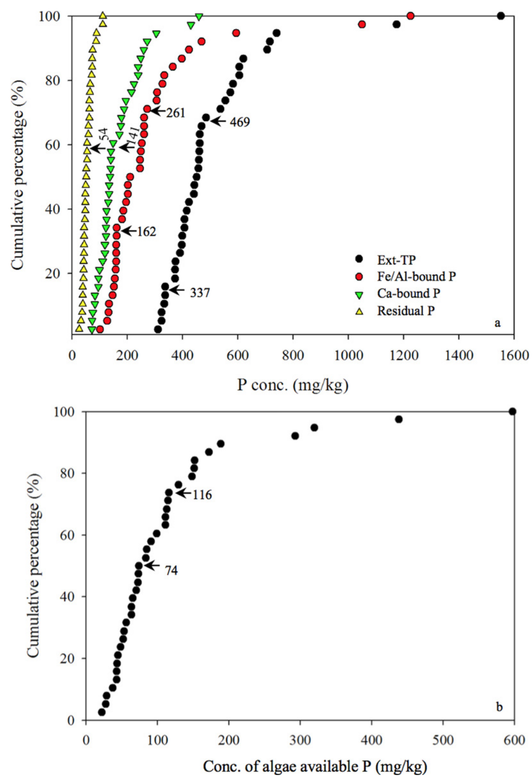

Fig. 2 – Cumulative frequency curve of Ext-TP and its fractions (a) and algae-available P (b) in sediment samples from three lakes.

examined via the UV–visible colorimetric method and acid–molybdenum-blue colorimetric method, respectively. NH_4-N was determined using the phenol–hypochlorite method, and NO_2-N was determined using diazotization with sulfanilamide dihydrochloride. To present the algae biomass across the three lakes, we mapped a spatial pattern of averaged chlorophyll *a* (Chl *a*) concentrations during the summer (July, August and September) of 2019, which was calculated from MODIS-derived daily Chl *a* concentration data.

Sediment samples were air-dried at room temperature, homogenized and sieved through a 150 μm stainless steel sieve. Sediment P fractions were extracted by using the method proposed by Psenner et al. (1988), with slight modifications as suggested by Hupfer et al. (1995). Briefly, the sediment samples were extracted sequentially using 1 mol/L NH_4Cl, 0.11 mol/L bicarbonate-dithionite (BD) at 40°C, 1 mol/L sodium hydroxide (NaOH) at 25°C, 0.5 moL/L HCl and then 1 mol/L NaOH at 85°C, resulting in fractions of ion-exchangeable P (Ex-P), metal (hydr)oxide-sensitive P (mainly referred to as Fe-bound P), P bound to Al (hydr)oxides (Al-bound P), P bound to carbonates and apatite-P (Ca-bound P), and residual P, respectively. Residual P is mainly referred to as refractory organic P. The total extractable P (Ext-TP) is the sum of the P measured in all extracted fractions. We also used 0.1 mol/L NaOH to extract the P adsorbed onto the surface of Fe/Al (hydr)oxides. The P in the above extracts was determined by using a spectrophotometer with the acid–molybdenum-blue method.

1.3. *Bioassay of algal growth potential with sediment particles as the sole P source*

A bioassay with sediment particles as the sole P source was conducted using *Microcystis aeruginosa* (FACHB1343) as a test organism, which was purchased from the Freshwater Algae Culture Collection at the Institute of Hydrobiology (FACHB) in China. The BG11 culture medium for blue–green algae was used; the specific composition is detailed on the official website of the FACHB collection (http://algae.ihb.ac.cn/English/). The species of *Microcystis aeruginosa* causes cyanobacterial blooms in freshwater ecosystems worldwide and are also dominant species in the shallow lakes of eastern China during warm seasons. *Microcystis aeruginosa* presents high P utilization efficiency with sediments as the sole P source (Deng et al., 2022).

The linear calibration of algae maximum cell yield (counted in Chl *a*) against the known P concentration was preliminarily established to calculate the algae-available P (AAP) in sediment samples, detailed in the reference of (Deng et al., 2022). Taking one sampling site (Site C5) as an example, a set of sediment particle concentrations was used as the sole P source to validate the linear response of the algal maximum cell yield to the concentration of sediment particles that contained AAP. Bioassays on AAP in sediments, with 500 mg/L dry particles and 5×10^5 cells/mL *Microcystis* as initial conditions, were conducted with no inoculation of algal cells as a control. Before biomass, the algae were starved for 7-10 days in the BG11 culture of P-depletion to consume the luxury P that was stored inside algal cells. The algae were incubated in triplicate under the following conditions: 12 hr of darkness and 12 hr of light at a light intensity of 25 μmol /(m^2 sec) and a temperature of 25°C. The incubation lasted for 2 to 3 weeks, and the concentration of Chl *a* in the culture was determined every day by using a molecular fluorescence spectrometer (RF5310PC, Shimadzu Corporation, Japan).

1.4. *Data analysis*

Cumulative frequency curves for Ext-TP, P chemical fractions and AAP were drawn by plotting P concentrations on the X-axis and the cumulative frequency on the Y-axis. Generally, inflexions in cumulative frequency curves indicate the natural, anthropogenic and anomalous concentration boundaries of elements and Wen(Wei and Wen, 2012), which were employed to analyse potential contributory sources of sediment P and their concentration ranges. The inflexions were determined using a linear regression of the cumulative frequency method under the condition of $p<0.05$ and $R^2>0.9$, with the maximum value of slope in the linear regression line indicating inflexion. Pearson correlation coefficients at a confidence level of 99% ($p< 0.01$) were used to analyse the correlation between the concentrations of Ext-TP, its chemical fractions and AAP.

In this research, we employed enrichment factor (EF), which is usually used to differentiate the increases in trace metals and P above natural background concentrations (natural detrital origin) and to quantify the level of pollution (Liu et al., 2012a), to indicate the relative contribution of point sources (sewage and manure) versus nonpoint sources (chem-

Table 1 – Statistics for water quality and sediment phosphorus in the three lakes.

Lakes	Water	TN (mg/L)	NH_4-N (mg/L)	NO_2-N (mg/L)	NO_3-N (mg/L)	TP (mg/L)	SRP (mg/L)	Chl *a* (μg/L)
Chaohu Lake	Range	0.68-4.74	0.10-0.30	0-0.01	0.25-0.50	0.05-0.45	0-0.15	12.6-151.4
	Mean	1.62	0.14	0	0.34	0.17	0.05	48.6
Taihu Lake	Range	1.20-5.24	0.09-0.41	0.01-0.24	0.25-1.17	0.03-0.57	0-0.07	5.8-143.5
	Mean	2.42	0.20	0.07	0.52	0.23	0.02	53.5
Hongzehu	Range	1.00-2.45	0-0.09	0.02-0.94	0-0.02	0.06-0.39	0-0.08	6.0-24.9
Lake	Mean	1.55	0.02	0.26	0.01	0.22	0.03	14.4
Lake	Sediments	Ex-P (mg/kg)	Fe-bound P (mg/kg)	Al-bound P (mg/kg)	Ca-bound P (mg/kg)	Residual P (mg/kg)	Ext-TP (mg/kg)	AAP (mg/kg)
Chaohu Lake	Range	0-2.9	80.0-527.9	79.0-518.8	71.7-459.4	26.0-86.7	399.5-1175.6	66.4-422.2
	Mean	0.6	205.5	213.3	130.6	47.1	597.1	174.6
Taihu Lake	Range	0.1-12.1	48.2-678.8	69.6-534.5	110.7-459.4	38.5-111.4	312.1-1551.7	7.6-590.1
	Mean	2.4	146.2	158.8	170.9	56.0	534.3	123.6
Hongzehu	Range	0.1-5.5	22.4-133.2	50.9-200.6	95.4-304.6	38.5-111.0	324.9-606.3	37.4-246.3
Lake	Mean	2.0	75.6	113.4	190.2	61.7	442.8	90.9

TN: total nitrogen; NH_4-N: ammonium; NO_2-N: nitrite-N; NO_3-N: nitrate-N; TP: total phosphorus; SRP: soluble reactive phosphorus.

ical fertilizer) with Eq. (1):

$$EF = (Fe/Al - boundP/Ca - boundP)_s/(Fe/Al - boundP/Ca - boundP)_b \quad (1)$$

where (Fe/Al-bound P/Ca-bound P)$_s$ and (Fe and Al-bound P/Ca-bound P)$_b$ are the ratios of Fe/Al-bound P to Ca-bound P in the surface sediments and background sediments, respectively. The EF values of <=1 indicate more contribution by nonpoint sources, whereas EF values of >1 suggest more contribution by point sources in sediment P. The (Fe/Al-bound P/Ca-bound P)$_b$ was set to be 0.733, achieved from the average ratio of Fe/Al-bound P/Ca-bound P in the low section of a sediment core sampled from Chaohu Lake (C-2007, Liu et al. 2012a). This low section of 64–90 cm in core C-2007 was deposited before ca. 1540, which was used for assessing the background of trace metals and P pollution in Chaohu Lake. There is an increase in the P concentration and the ratio of Fe/Al-bound P to Ca-bound P over the past half-millennium in the sediment record, clearly indicating increased P pollution from human activities, with point sources from urbanization as the major contributor in recent decades (Liu et al., 2012a).

2. Results

2.1. *Nutrients in the water column and sediment P fractions in the three lakes*

The TP concentrations among the three lakes were comparable, ranging from 0.17 to 0.23 mg/L. However, the TN concentration was higher in Taihu Lake, with an average value of 2.42 mg/L, than in Chaohu Lake (1.62 mg/L) and Hongzehu Lake (1.55 mg/L). The concentration of sediment Ext-TP ranged from 399.5 to 1175.6 mg/kg with an average value of 597.1 mg/kg in Chaohu Lake and from 312.3 to 1551.7 mg/kg and 534.3 mg/kg in Taihu Lake (Table 1). The sediment Ext-TP in Hongzehu Lake ranged from 324.9 mg/kg to 606.3 mg/kg, with an average value of 422.8 mg/kg. The Fe/Al-bound P accounted for 70.1% of Ext-TP in Chaohu Lake, followed by 57.1% in Taihu Lake and 42.7% in Hongzehu Lake. The Ca-bound P in Hongzehu Lake accounted for 43.3% of the Ext-TP, followed by 32.0% in Taihu Lake and 21.9% in Chaohu Lake.

The cumulative frequency curves for Ext-TP and Fe/Al-bound P had two inflexions, with values of 337.5 mg/kg and 469.1 mg/kg for Ext-TP and 161.7 mg/kg and 261.3 for Fe/A-bound P. The fractions of Ca-bound P and residual P presented one inflexion, with values of 140.6 mg/kg and 54.2 mg/kg, respectively (Table 2 and Fig. 2a).

Sediment P showed spatial heterogeneity across the three large lakes (Fig. 3). In Chaohu Lake, the highest concentration of Ext-TP occurred at the western sampling sites, with Fe/Al-bound P accounting for 83.6% at C1, C2 and C3 on average, while lower Ext-TP concentrations were found at the middle and eastern sites, with Fe/Al-bound P accounting for 61.4% at C4 to C9. The Ext-TP concentration was high in the northwest area, which is upstream of Taihu Lake; the highest concentration of 1551.7 mg/kg occurred at Site T1. The percentage of Fe/Al-bound P exceeded 50% of Ext-TP at the sampling sites along the western coastline of the lake. In Hongzehu Lake, the highest concentration of Ext-TP occurred at Sites H10 (606.3 mg/kg) and H15 (573.6 mg/kg), and the lowest concentration occurred at Sites H1, H5 and H8 (<350 mg/kg). The percentage of Fe/Al-bound P in Hongzehu Lake ranged from 29.6% to 60.6%.

2.2. *Algae available P in the three lakes*

With K_2HPO_4 as the sole P source, the growth phase of *M. aeruginosa* lasted for 5 to 14 days depending on the amount of P added (Fig. 4a). The Chl *a* concentration increased with the concentration of added P from 0 to 0.5 mg/L. The relationship of the concentration of Chl *a* versus P was linear until 0.1 mg/L in 200 mL of culture medium, at which point the Chl *a* concentration was 329.3 μg/L on average from triplicate determinations (Fig. 4b). With sediment particles (Site C5) as the sole P source, the algal growth was as stable as it was with K_2HPO_4 (Fig. 4c). The maximum cell yield increased with the concentration of sediment particles, with a linear response until a particle concentration of 1000 mg/L, at which point the con-

Table 2 – Inflexion and averages of the phosphorus concentration-based cumulative frequency approach in the sediment samples of three lakes.

P fractions	First inflexion (mg/kg)	Ave_first (mg/kg)	Second inflexion (mg/kg)	Ave_second (mg/kg)
Ext-TP	337.5	326.7	469.1	417.8
Fe/Al-bound P	161.7	146.1	261.3	219.8
Ca-bound P	140.6	111.1	—	—
Residual P	54.2	43.5	—	—
AAP	74.2	50.6	116.6	98.2

Ave_first: the average value before the first inflexion; Ave_second: the average value between the first and second inflexions; Ext-TP: extractable total phosphorus; Fe/Al-bound P: Fe/Al (hydr)oxide-bound P; Ca-bound P: Ca compound-bound P; AAP: algae-available P.

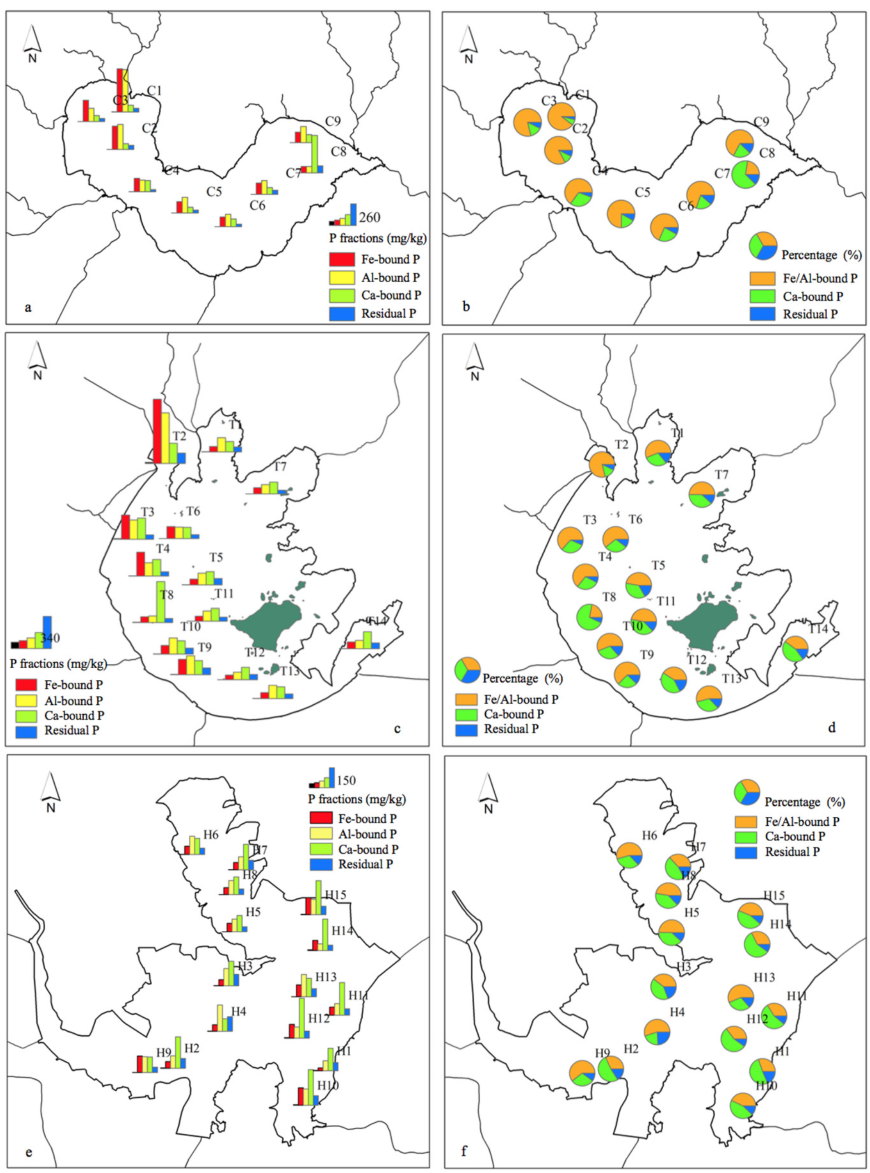

Fig. 3 – Spatial patterns of the sediment P fractions and their percentages in Chaohu Lake (a, b), Taihu Lake (c, d) and Hongzehu Lake (e, f).

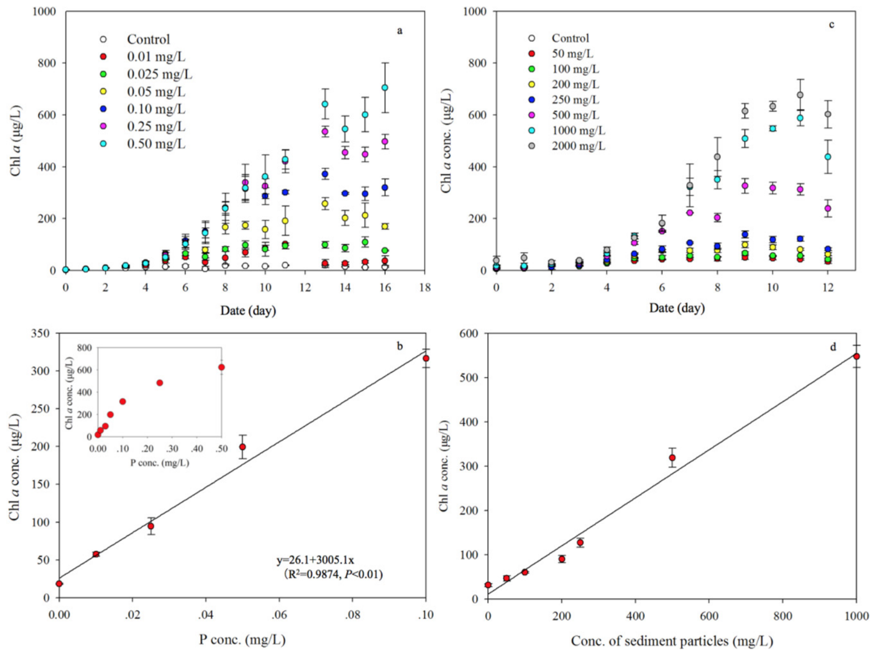

Fig. 4 – The growth of *M. aeruginosa* with K_2HPO_4 and particulate P as the sole P source and the linear calibration for maximum algal cell yield against the concentration of P and particles. The error bars show the standard errors of cultures in triplicate.

centration of Chl *a* was 547.4 mg/L from triplicate determinations (Fig. 4d).

The growth of *M. aeruginosa* with sediment particles as the sole P source in Chaohu Lake, Taihu Lake and Hongzehu Lake is shown in Appendix A Figs. S1, S2 and S3, respectively. The spatial patterns of AAP in the three lakes showed patterns similar to those of Ext-TP and Fe/Al-bound P, with high concentrations occurring at the sampling sites in the upstream areas of Chaohu Lake and Taihu Lake (Fig. 5).

The average concentrations of AAP in Chaohu, Taihu and Hongzehu Lakes were 174.6 mg/kg, 123.6 mg/kg, and 90.9 mg/kg, with the highest concentrations of 422.2 mg/kg at Site C2, 530.6 mg/kg at Site T2 and 246.3 mg/kg at Site H15, respectively (Figs. 5a, 5c and 5e). The spatial patterns of Chl *a* (Figs. 5b, 5d and 5f) were consistent with those of Ext-TP, Fe/Al-bound P and AAP, with Chl *a* concentration of 50.4 μg/L in Taihu Lake, 37.1 μg/L in Chaohu Lake and 14.0 μg/L in Hongzehu Lake (Table 1).

2.3. *The relationships between Ext-TP, P chemical fractions, and algae-available P connected to P sources*

The Ext-TP concentration presented a significantly positive correlation with Fe/Al-bound P but not with Ca-bound P (Appendix A Table S1). Overall, the linear equation [Ext-TP]=0.94[Fe/Al-bound P]+242.2 (R^2=0.8355, p<0.01) was achieved across the three lakes (Fig. 6a). The P adsorbed onto the surface of Fe/Al (hydr)oxides, which was extracted by 0.1 mol/L NaOH accounted for 66% of Fe/Al-bound P (Fig. 6b). The AAP concentration is linearly correlated to Fe/Al (hydr)oxide-adsorbed P, of which 68% is indicated to be directly available by algae (Fig. 6c). Consequently, the linear equation [AAP]= 0.45[Fe/Al-bound P] (R^2=0.8356, p<0.01) was achieved across the three lakes (Fig. 6d).

The percentage of Fe/Al-bound P against Ca-bound P in Ext-TP presented a decreased linear trend, with a linear equation Fe/Al-bound P (%) =90.9(%)-1.1 Ca-bound P (%) (R^2=0.9412, p<0.01) across the three lakes (Fig. 7a). The enrichment factor (EF) of Fe/Al-bound P to Ca-bound P, which was calculated according to Eq. (1), were highest in Chaohu Lake (6.6), moderate in Taihu Lake (2.5) and lowest in Hongzehu Lake (1.6), with greater variability in Chaohu and Taihu Lake than in Hongzehu Lake (Fig. 7b).

3. Discussion

3.1. *Algae availability of sediment P and its connection to sediment P chemical fractions*

Our results showed that *M. aeruginosa* is applicable for the assessment of algae-available P in sediments, and algal cell maximum cell yield linearly responded not only to the sole source of K_2HPO_4 but also to the sole source of particulate P (Fig. 4). *M. aeruginosa* can tolerate low P concentrations in the water column (Guan et al., 2020b), and they desorbed P from particles very effectively. The P concentration in the culture of suspended particles remained at zero during algae growth periods, establishing a diffusion gradient of P concentration from sediment particles to *Microcystis* cells until depletion of AAP.

The quantitative linear relationship between AAP and the chemical P fractions clearly revealed that algae availability of P came from Fe/Al (hydr)oxide-adsorbed P that was ex-

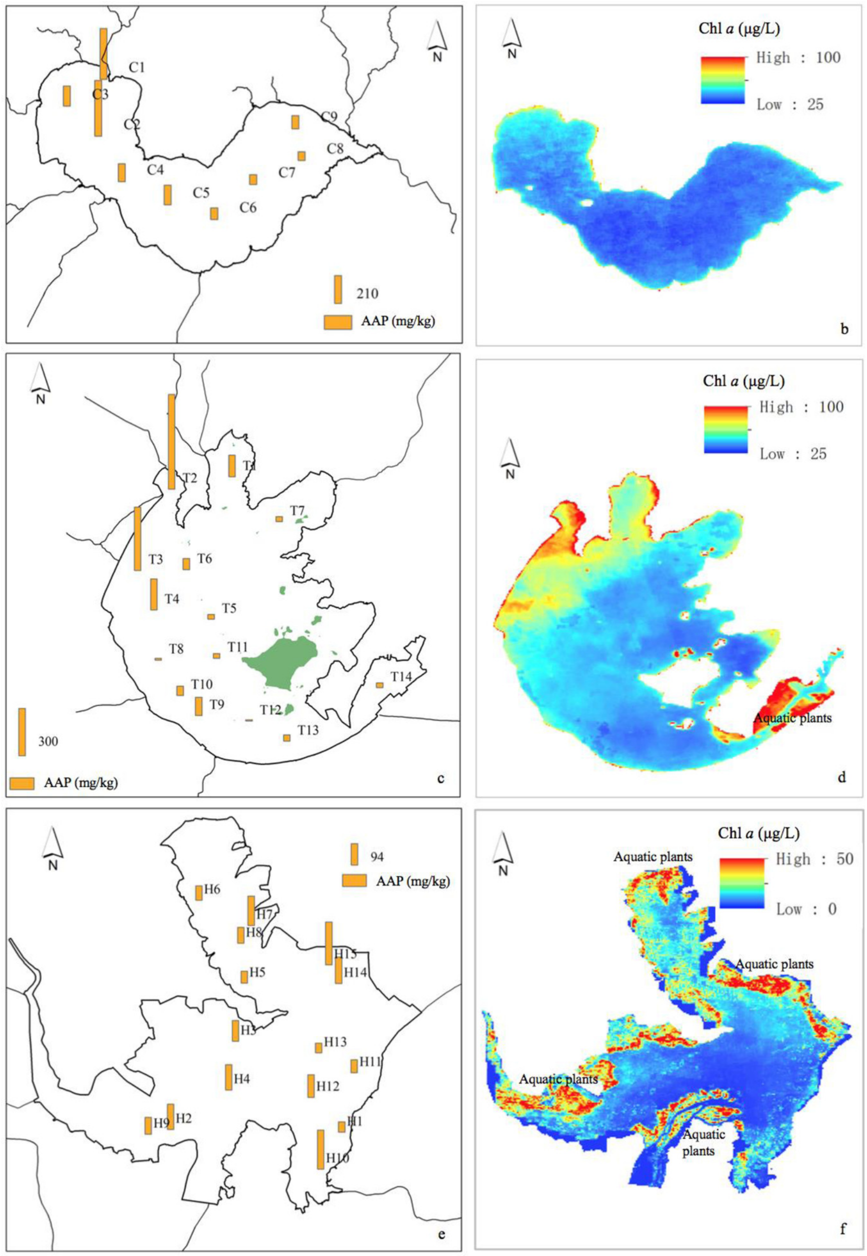

Fig 5 – Spatial patterns of sediment alga-available P and Chl *a* concentrations across the sampling sites in the three lakes.

tracted by 0.1 mol/L NaOH, of which 68% is AAP (Fig. 6b). The Fe/Al (hydr)oxide-adsorbed P accounted for 66% of the Fe/Al-bound P extracted by BD, followed by 1 mol/L NaOH (Fig. 6b). Overall, approximately 45% of Fe/Al-bound P was estimated to be AAP. The mechanisms of P bound to Fe/Al (hdyr)oxides mainly include adsorption, coprecipitation, and inclusion (Wang et al., 2021), and the BD reagent and 1 mol/L NaOH can dissolve both amorphous and crystalline Fe/Al (hydr)oxides and their associated P (Jan et al., 2013). However, only 68% of Fe/Al-(hydr)oxide-adsorbed P was estimated to be algae-available with the bioassay of *M. aeruginosa*. The Fe/Al (hydr)oxides in sediments include a number of minerals, e.g., ferrihydrite, goethite, haematite, magnetite, amorphous Al (hydr)oxides, gibbsite, and corundum, which show different P adsorption/desorption abilities depending on the P-complexation structure with minerals (Wu et al., 2020). Particularly, the amorphous or noncrystal forms of Fe/Al (hydr)oxides have some fixed adsorption sites inside their micropores of minerals for P adsorption, and some of Fe/Al (hydr)oxides can bind P through surface coprecipitation (and Sparks, Arai and Sparks, 2001; Khare et al., 2005; Wu et al., 2020); it hinders P desorption for algae availability. Therefore, only the part of P that is absorbed onto the surface of Fe/Al (hydr)oxides could be directly available by algae, which is desorbed from sediments/particles through ligand exchange of hydroxyl, whose concentration increases with the growth of algae (Xie et al., 2003; Deng et al., 2022; Zhang et al., 2020).

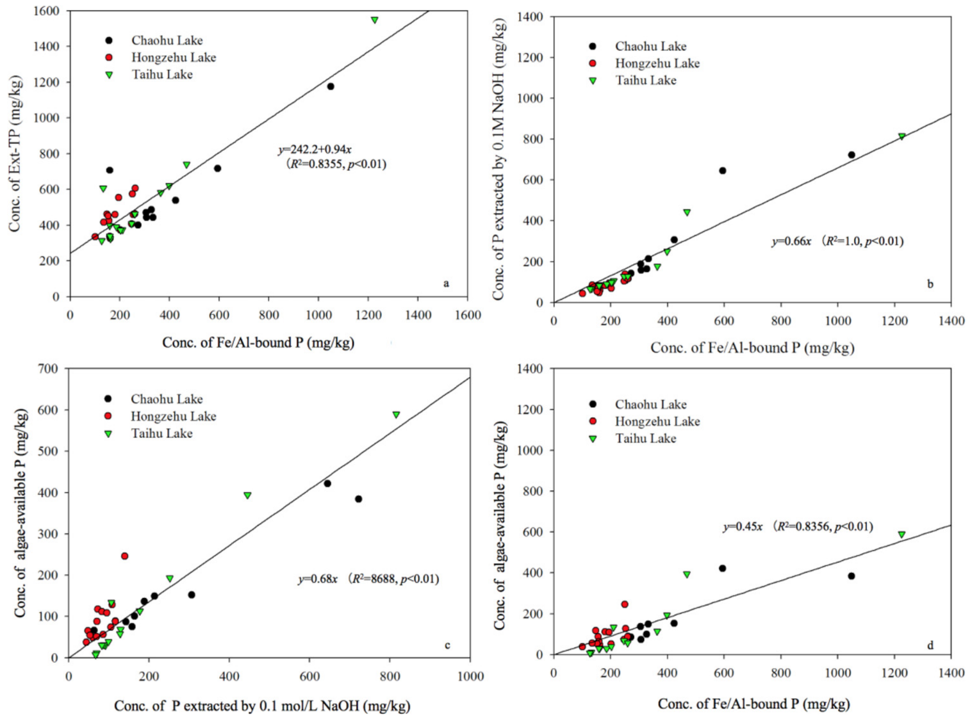

Fig. 6 – The linear plots of Ext-TP against Fe/Al-bound P (a), Fe/Al (hydr)oxide-adsorbed P against Fe/Al-bound P (b), AAP against Fe/Al (hydr)oxide-adsorbed P (c) and AAP against Fe/Al-bound P (d) achieved across the three lakes.

3.2. *The P accumulation in large shallow lakes with its contributing sources*

The spatial patterns of sediment P in a large-scale space provided information on P accumulation in large shallow lakes with different contributing sources. The ratio of Fe/Al-bound P and Ca-bound P is confirmed to be an effective indicator to distinguish the relative contribution of point versus nonpoint sources among lakes and between specific sediment samples in each lake, with all samples pooled towards a similar linear line (Fig. 7a). Many studies have documented that P in soils treated with manures or biosolids is dominant in the fraction of Fe/Al-bound P, whereas P in soils treated with chemical fertilizer is dominant in the fraction of Ca-compound P (Negassa and Leinweber, 2009; Kahiluoto et al., 2015). The chemical fractionation conducted on the inflowing rivers of the three lakes also suggested that the areas more impacted by point sources (urbanized area) showed higher concentrations of P with dominant Fe/Al-bound P, while the areas more impacted by nonpoint sources (agricultural area) showed the lowest concentrations of P with dominant Ca-bound P (Liu et al., 2012b; Yi et al., 2017b; Lannergård et al., 2020; Ni et al., 2020; Wan et al., 2020; Yang et al., 2020a). In the case of Taihu Lake, the sediment Ca-bound P in the upstream rivers of agricultural areas accounted for 69%, while sediment Fe/Al-bound accounted for 70% in the downstream rivers of urbanized areas (Yi et al., 2017b).

As indicated by the EF values of Fe/Al-bound P to Ca-bound P (Fig. 7b) and P spatial patterns (Fig. 3), point sources contributed the dominant proportion to sediment P in Chaohu Lake and Taihu Lake, with average EF values of 6.6 and 2.5, respectively. However, the contribution from point and nonpoint sources was comparable to sediment P in Hongzehu Lake, with an averaged EF value of 1.6. In Chao Lake and Taihu Lake, sediment P was more impacted by point sources in their upstream areas, while it was more impacted by nonpoint sources in their downstream areas.

The records of core sediments in Chaohu Lake and Taihu Lake revealed that the ratio of Fe/Al-bound P to Ca-bound P was lowest in the preanthropogenic period, gradually increasing in the period of historical agricultural expansion and reaching a maximum in the period of urbanization in their basins since 1980 (Liu et al., 2012a; Liu et al., 2012b; Liu et al., 2013; Yi et al., 2017a). The linear equation of [Ext-TP]=0.94 [Fe/Al-bound P]+242.2 (Fig. 6a), which was achieved from the 5-cm surface sediments across the three lakes, indicated that P accumulation in recent decades is predominantly contributed from point sources rather than nonpoint sources. The intercept of 242.2 mg/kg could indicate the background of Ext-TP, which numerically approximated the background and revealed the record of C-2007 in Chaohu Lake (Liu et al., 2012b). The two inflexions for Fe/Al-bound P in cumulative percentage curves, 161.7 mg/kg and 261.3 mg/kg (Table 2 and Fig. 2), could divide sediment P pollution by point sources into three levels: high pollution, moderate pollution and low pollution.

Although the ratio of Fe/Al-bound P to Ca-bound P is a simple indicator to distinguish the relative contribution of point versus nonpoint sources in the studied shallow lakes, its ex-

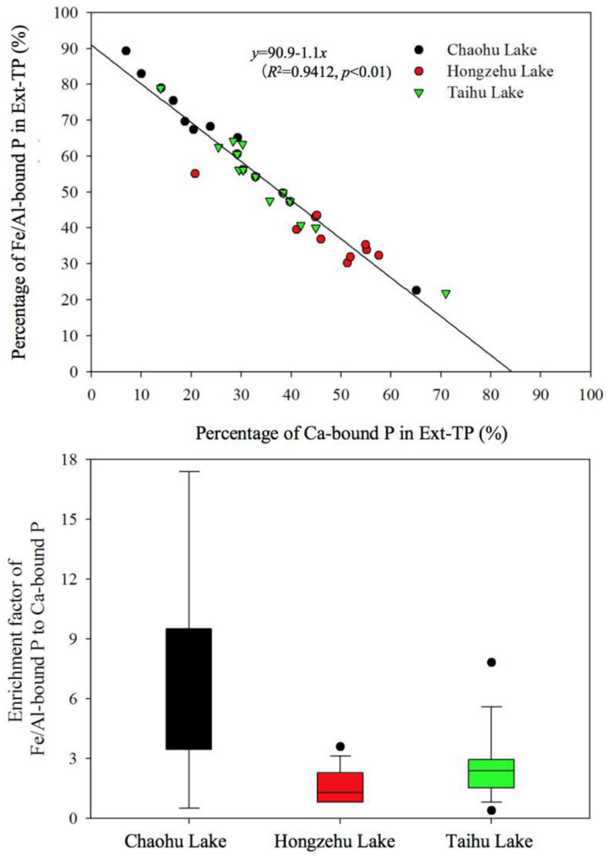

Fig. 7 – The linear plot for the percentage of Fe/Al-bound P against Ca-bound P in Ext-TP (a) and statistical distribution of enrichment factors of Fe/Al-bound P to Ca-bound P (b) in the three lakes.

tensive application to other areas should be done with caution. Potential transformation between P fractions and the transfer of Fe-bound P from the deeper layer to the surface layer of sediments occurs during short or long time periods (Yang et al., 2022), resulting in a possible change in this ratio. The mineralization of organic P could be transformed to Fe/Al-bound P in the long term (Katsev et al., 2006), Fe-bound P could be transformed to Ca-bound P in lakes with hard water chemistry (Markovic et al., 2019), and Fe-bound P could be transformed to Al-bound P with a high ratio of Al to Fe in sediments (Kopáček et al., 2005). Nevertheless, the amount of P through transformation and transfer should account for a small part of their fractions to change the ratio of Fe/Al-bound P to Ca-bound P in the study area because the 5-cm surface sediments usually reflect a fast accumulation of P in recent decades. Moreover, the reference value of Fe/Al-bound P to Ca-bound P is important to improve the accuracy in indicating the relative proportion of point sources versus nonpoint sources, which could be variable in different lakes and even among sampling sites in each lake due to the spatial heterogeneity of sediment properties. Therefore, how to characterize the reference value and its uncertainty need further research.

3.3. *The criteria for sediment P concentrations with connection of sediment P and algal biomass in shallow lakes*

The spatial patterns of algal biomass in the three lakes were consistent with Ext-TP, Fe/Al-bound P and AAP in sediments (Fig. 3 and Fig. 5), indicating that sediments contribute a major source for algae growth during warm summer. This is supported by multiple lines of evidence from field observations of P mass balance in the lakes. There were high P release fluxes from pore water in core sediments in the upstream areas of Chaohu Lake and Taihu Lake and low P release fluxes in Hongzehu Lake (Yao et al., 2016; Ding et al., 2018a; Yang et al., 2020b). More importantly, algal blooms can directly pump a large amount of P from surface sediments or suspended particles to the water column in shallow lakes (Xie et al., 2003; Spears et al., 2008; Liu et al., 2015; Zhang et al., 2020; Deng et al., 2022), and the amount of P released from sediments depends on the demand for algae growth (Tong et al., 2020; Xu et al., 2021). As revealed by our research, the response of *Microcystis* to particulate P was fast, reaching the maximum cell yield with particulate AAP depletion in 7-10 days (Fig. 4 and Appendix A Figs. S1, S2, S3).

Therefore, the criteria for sediment P concentrations in shallow lakes could be achieved if a conceptual model for “P-pumping suction” from sediments to algae was coupled with the benthic boundary layer of lakes. According to the theory of the benthic boundary layer, a diffusive boundary layer (DBL) exists, through which the solute exchanges between sediments and overlying water (Boudreau and Jorgensen, 2001). In the case of P, orthophosphate-P released from the reduction of Fe-bound P under anoxia in subsurface sediments would diffuse towards the surface through porewater. However, the uplifting P could be reabsorbed by Fe/Al (hydr)oxides at the aerobic surface, hindering P release to the water column (Zhang et al., 2020). Therefore, Fe/Al (hydr)oxide-adsorbed P in the DBL could be directly desorbed in the alkaline water chemistry of shallow lakes, with algae growth accelerating P desorption through pH regulation, known as “P-pumping suction” (Fig. 8).

A direct connection between algae and sediment DBL in shallow lakes can help quantify the linear relationships between algal biomass and sediment P, the latter of which was correlated to P sources, as presented in Fig. 7. Assuming P desorption from a 0.5 mm-deep diffusive boundary layer (DBL) (δ_{DBL} shown in Fig. 8) with a bulk sediment concentration of 200 g/L, it would have a 100 g mass of sediment particles per square metre in the diffusive layer. This mass approaches the flux of suspended solids when simulating wave disturbance on sediments of shallow lakes, producing a 0.1 mm-depth of bulk sediments disturbed (Ding et al., 2018b). If the AAP concentration of the diffusive layer is 100 mg/kg, it would produce a maximum *Microcystis* cell yield of 15.0 µg Chl *a*/L throughout a 2-m water column, which is some average algal biomass for algal blooms (Zhang et al., 2016). At this point, 222.2 mg/kg of Fe/Al-bound P should be limited according to the linear relationships between AAP and Fe/Al-bound P (Fig. 6d). If this threshold of Fe/Al-bound P is subtracted from its background (102.4 mg/kg of Fe/Al-bound P in 242.2 mg/kg of Ext-TP), a con-

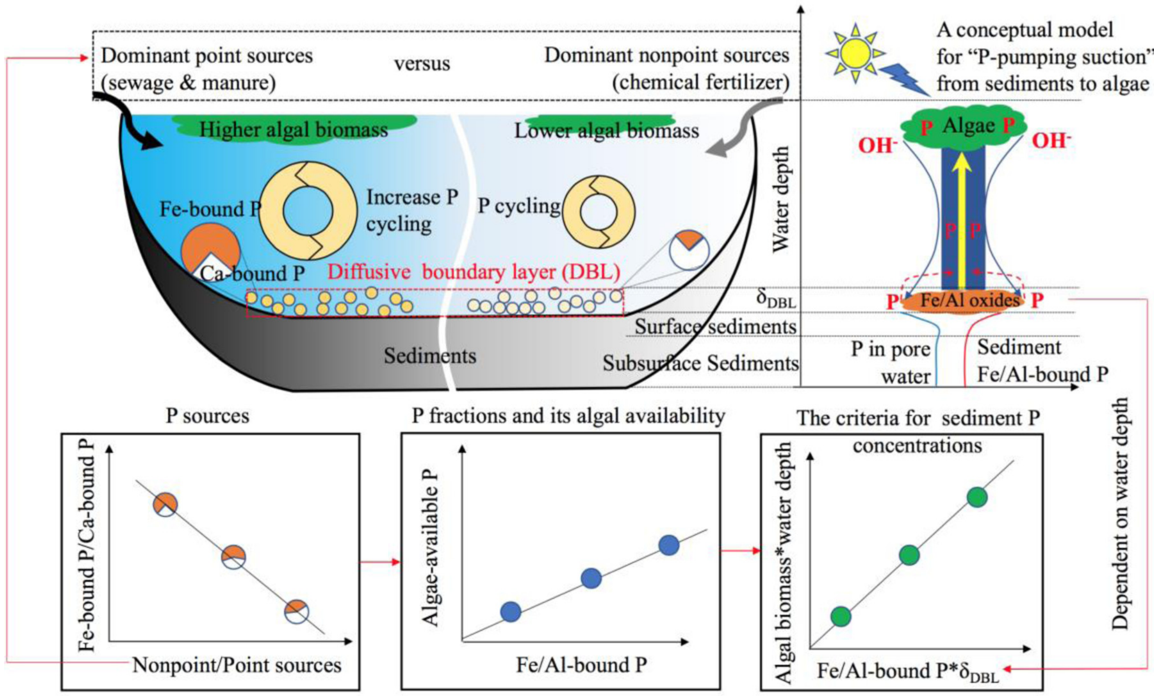

Fig. 8 – A conceptual model for "P-pumping suction" from sediments to algae coupled with the benthic boundary layer to establish the criteria for sediment P concentrations in shallow lakes. (The red arrows indicate the relationships between P sources, algal availability and lake physical conditions (mainly referring to water depth).

tribution of 119.8 mg/kg of Fe/Al-bound P with 53.9 mg/kg of AAP is from increased human activities. With the above principles and processes, the criteria for sediment P concentration in shallow lakes could be made and connected to the reduction in P load from point and nonpoint sources in watersheds (Fig. 8).

However, the Chl *a* concentration in field observations should not respond linearly to the sediment AAP in each lake itself or among the lakes due to a series of other factors affecting algae biomass, such as lake physical conditions (hydrology and water retention, hydrodynamic disturbance and water depth, etc.), local climatic conditions and the N:P ratio dependent on their loads from watersheds (Paerl et al., 2015; Yi, et al., 2017b; Xu et al., 2015; Paerl et al., 2019). Hongzehu Lake showed the lowest concentrations of Chl *a* and sediment P, although rather high TN and TP concentrations occurred in the water column due to its shorter water retention time (35 days) and resuspension of sediment particles (Ma et al., 2017, Lei et al., 2020). Chaohu Lake had a lower N concentration (Table 1) and deeper water depth with less disturbance on sediments compared to Taihu Lake, which produced a lower Chl *a* concentration in the water column, although it had a higher concentration of sediment P than Taihu Lake. Therefore, the depth of the diffusive layer and its relationship to algal biomass in actual environments should be well clarified and defined in future studies. The criteria for sediment P concentrations to alleviate CyanoHABs in shallow lakes require more detailed knowledge of the relationships between algae and sediment P in a specific lake. Despite these factors, our research establishes a fundamental principle by connecting algae growth to sediment P and its sources.

4. Conclusions

Our research has established a quantitative relationship between algae-available P and chemical fractions in sediments and has clarified the relative importance of point versus nonpoint sources to provide algae-available P with sediments as potential P sources in large shallow lakes of Eastern China. The external P load from point sources is mainly deposited in sediments with an Fe/Al-bound fraction, approximately half of which is directly available by algae. Therefore, the reduction of point sources from urbanized areas, rather than nonpoint sources from agricultural areas that primarily contribute to the Ca-bound P fraction, should be prioritized to alleviate CyanoHABs in shallow lakes. The direct connection of algae growth to sediments in shallow lakes makes the criteria for sediment P concentrations possible, whereas more detailed knowledge of the relationship between sediments and algae in actual environments of specific lakes is needed.

Acknowledgments

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Appendix A Supplementary data

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jes.2022.03.038.

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