

Potential for sediment phosphorus release in coal mine subsidence lakes in China: perspectives from fractionation of phosphorous, iron and aluminum

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Abstract Few studies have been conducted on how the quantitative relationship between phosphorus (P) and iron (Fe) and aluminium (Al) compounds in the sediments of coal mine subsidence lakes influence sediment P release. Four representative lakes, characterized by sedimentary environments of soil inundation, were selected in the Huainan and Huaibei coal mine areas of China. Their ages, pollutant loading patterns and nutrient levels were assessed to evaluate the potential for sediment P release based on the fractional composition of P, Fe and Al. Sediment P, Fe and Al were extracted sequentially using ammonium chloride (NH₄Cl), bicarbonate-dithionite (BD) and sodium hydroxide (NaOH) at 25 °C, followed by HCl, and then NaOH at 85 °C. The resulting fractions were considered as environmental indicators for P, Fe and Al, including ion-exchangeable forms (NH₄Cl-P, NH₄Cl-Fe, NH₄Cl-Al), associated fractions with reducible metal hydroxides (BD-P, BD-Fe, BD-Al) and amorphous hydroxides (NaOH₂₅-P, NaOH₂₅-Fe, NaOH₂₅-Al), acid-soluble fractions (HCl-P, HCl-Fe,

HCl-Al) and residual species (NaOH₈₅-P, NaOH₈₅-Fe, NaOH₈₅-Al), respectively. The potential for sediment P release was related to the concentrations of Al and Fe compounds in the presence of soil inundation. Calcareous soils in the Huaibei area were influential in regulating sediment P release, whereas soil Fe and Al were influential in the Huainan area. The results agreed with a common empirical model that predicts low P flux if the molar ratio of $[\text{NH}_4\text{Cl-Al} + \text{BD-Al} + \text{NaOH}_{25}\text{-Al}]:[\text{NH}_4\text{Cl-Fe} + \text{BD-Fe}] > 3$ or $[\text{NaOH}_{25}\text{-Al}]:[\text{NH}_4\text{Cl-P} + \text{BD-P}] > 25$ in sediments when anoxia develops. Increased loading of oxidizable matter (OM) or enriched P bound to Fe oxides tends to change these ratios and increase the potential for sediment P release.

Keywords Aluminium · Iron · Phosphorus · Sediment · Soil inundation · Subsidence lakes in China

Introduction

Coal-based energy accounts for over 70 % of the total primary energy production in China. Annual coal production has consistently exceeded 3 billion tons in recent years as a consequence of increasing demand to support rapid economic growth. Regions with coal mines face major environmental challenges, such as pollution of air, soil and water, and the generation of solid wastes (Bian et al. 2010). Among these issues, land subsidence and submergence is of particular

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concern in the Huainan and Huaibei coal fields (traditionally called the H–H coal mine areas) located in the North China Plain. This region is one of the largest coal producing areas in China, generating 100 million tons of raw coal per year. The hydrogeology of these mine areas is characterised by thick Quaternary sediments overlying coal deposits with high underground water tables (Xie et al. 2013). Underground exploitation of coal resources has led to the formation of water-rich landscapes, where water accounts for 30–50 % of the subsided land in the H–H coal mine areas. At present, pockets of land subsidence with a total area of 250 km² have formed in the H–H coal mine areas, and over 100 km² is submerged permanently. The area of land subsidence is increasing by 3–5 % per year due to continuous mining activity.

Subsidence creates a variety of aquatic ecosystems, including ponds, reservoirs, wetlands, and lakes, all of which exhibit potential in terms of water resources and ecological benefits. There are many examples of successful ecological restoration/rehabilitation, conducted by local governments, coal mine operators, and community members (Xie et al. 2013). Taking into account site-specific hydrology and other characteristics, these water bodies are generally regulated as fishponds, wetland parks, plain reservoirs or ecological lakes in China (Xie et al. 2013).

Water quality and eutrophication are important issues for the subsidence water bodies because they are subject to the same environmental disruptions as other freshwater ecosystems, such as point source pollution by sewage or nonpoint source pollution by agricultural activities. A wide range in nutrient levels and primary production is observed in these water bodies due to variation in the patterns and amounts of pollutant loads (Qu et al. 2013; Yi et al. 2014a). Previous research revealed that limitation in phosphorus (P) availability, relative to nitrogen (N) content, is a common constraint to primary production (Wang et al. 2013; Yi et al. 2014a). An alternative explanation for this P limitation to phytoplankton growth may be the relatively high nitrate concentrations in wastewater and agricultural effluent (Elser et al. 2009a, b; Elser and Bennett 2011). This pattern of P limitation may be also related to the abundance and reactivity of Fe oxides in the sedimentary environment of inundated agricultural soils, which limits P migration under oxic conditions. Hypoxic and anoxic environments enhance P release from sediment Fe-

(hydr)oxides according to the classical model of sediment P release (Mortimer 1941; Sah et al. 1989; Slomp et al. 1996; Roden and Wetzel 2002; Loeb et al. 2008; Mort et al. 2010).

Other mechanisms also play important roles in controlling sediment P release. Elevated concentrations of Al(OH)₃ in the non-calcareous sediments of low-pH lakes can prevent P release under anoxic conditions by adsorbing the P liberated from Fe hydroxides due to the high sorption capacity and stability of Al(OH)₃ under both oxic and anoxic conditions (Kopáček et al. 2000; Rydin et al. 2000; Reitzel et al. 2005). Phosphorus sorption capacity, and thus the potential magnitude of release, has been linked with the concentrations of calcium carbonate and ferric hydroxide in high-pH lakes (Kopáček et al. 2000). A pH increase and CO₂ removal during algal blooms may result in oversaturation with respect to CaCO₃ in hard water lakes, and consequent calcite precipitation. Increases in the CaCO₃ sedimentation rate and P co-precipitation with calcium compounds are the most important processes that result in P immobilization in sediment and thus prevent eutrophication (Koschel et al. 1983; Dittrich et al. 2011).

Building on these principles and mechanisms, research has been conducted on the relationships among Al, Fe and P in lake sediments and the related implications for sediment P release and lake environmental restoration (Jensen, et al. 1992; Kopáček et al. 2005; Huser and Pilgrim 2014). Kopáček et al. (2005) developed an empirical relationship, by analysing 43 studies on lake sediment processes, to determine a threshold for sediment P release potential based on the molar ratios of Al, Fe, and P fractions as determined by the extraction procedure of Psenner et al. (1988). Sediment P, Fe and Al in surface sediments are extracted sequentially using water (H₂O), bicarbonate-dithionite (BD), sodium hydroxide (NaOH) at 25 °C, and HCl and NaOH at 85 °C, with the resulting fractions considered as environmental indicators of P, Fe and Al status. These comprise loosely bound and pore-water forms, fractions associated with reducible metal hydroxides (BD-P, BD-Fe, BD-Al) and amorphous hydroxides (NaOH₂₅-P, NaOH₂₅-Fe, NaOH₂₅-Al), acid-soluble (HCl-P, HCl-Fe, HCl-Al) and residual fractions (NaOH₈₅-P, NaOH₈₅-Fe, NaOH₈₅-Al), respectively. The model indicates that P is unlikely to be released from sediment to the water column during hypolimnetic anoxia if either the molar [NaOH₂₅-

Al]:[H₂O-Fe + BD-Fe] ratio (often denoted as Al:Fe) > 3 or the molar [NaOH₂₅-Al]:[H₂O-P + BD-P] ratio (denoted as Al: P) > 25.

The aforementioned empirical model has been tested in several studies of lake sediments (Lake et al. 2007; Wilson et al. 2008; Norton et al. 2008) and its role has been verified in indicating the potential for sediment P release. However, there have been few studies conducted on the topic of soil inundation, where soil undergoes periodic or permanent flooding along hydro-fluctuation belts, a common phenomenon in littoral zones of wetlands, lakes and reservoirs. In this study, we characterized the fractions of Al, Fe, and P in inundated sediments, quantified sorption capacity through development of P isotherms, and applied the Kopáček (2005) model in evaluating the potential magnitude of sediment P release for several lakes in the H–H coal mine areas.

Materials and methods

Site description

The H–H coal fields in Anhui Province of China (Fig. 1a) cover an area of 15,000 km², of which the Huainan coal field occupies 3600 km² and the Huaibei coal field has an area of 9600 km², with a total reserve of 37 billion tons of coal. Two typical coal mining areas exhibiting the presence of water bodies formed from subsidence were selected as representative research zones (Fig. 1a). One is located in the Panxie district (PX) of Huainan City, and the other is in the Zhahe district (ZH) of Huaibei City. The former is the largest in the Huainan mining area, covering an area of 870 km² (Fig. 1b). Three small lakes were selected in the eastern, central and western parts of PX and were designated as PJ, GQ, and XQ, based on the names of the corresponding coal mines. Meanwhile, ZH is located in northeastern Anhui Province, covering an area of 75 km² (Fig. 1c). Lake Nanhu (NH), located in the southern part of this district, was selected as the representative research lake for this zone.

For the sake of comparison, the four lakes were characterized by their formation histories, nutrient status, and level of impact by human activities. Lake PJ (32.793167°N, 116.542333°E; WGS-84 coordinate system) is 20 years old, with an area of 3.5 km². This water body is connected with the Ni River, which

receives mine drainage, rural nonpoint source pollutants, and domestic sewage from surrounding villages. Lake GQ (32.839000°N, 116.553667°E) has an area of 2.5 km² and was formed within the last 5 years; it lacks any connection with rivers. Lake XQ (32.795167°N, 116.357000°E) has an area of 4 km² and has existed for at least 10 years; it receives storm surges from the southern Ji River during seasonal flooding periods. Finally, Lake NH (33°54.37'N, 116°48.84'E), with an area of 2.6 km², is 30 years old and is the first national urban wetland park that has been built on a coal mine subsidence area.

The four lakes have average depths in the range of 4.0–5.0 m. In addition, the lakes PJ, GQ, and XQ are used by local farmers for aquaculture, albeit without the addition of fish food or fertiliser, whereas Lake NH serves as the source for Huaibei City's water supply. Prior to subsidence, the land was used for rice or dryland farming. The regional soil is classified as a yellow fluvo-aquic soil, a common soil species in the Huaibei floodplains. The soil texture ranges from silty clay in PX to silty loam with calcareous properties in ZH.

Water sampling, sediment collection and analysis

Five sediment sampling sites were selected in each lake to reflect the spatial heterogeneity in the soil properties of the sedimentary environments. Sampling was conducted in July 2013 with surface water and sediment sampled at the five sampling sites in each lake. Surface water samples were collected at 0.5–1.0 m depth using a 5 L Plexiglas water sampler at each sampling site. Temperature, pH, and dissolved oxygen (DO) were measured on site using portable YSI electrodes (models pH100 and DO200, Xylem Company, New York, USA). The water samples were transported on ice to the laboratory and analyzed immediately. Water parameters, including total phosphorus (TP), soluble reactive phosphate (SRP), total nitrogen (TN), and concentrations of individual N species, namely, nitrate (NO₃-N), nitrite (NO₂-N), and ammonium (NH₄-N), were analyzed according to standard methods (APHA et al. 1998). Briefly, water samples were filtered through pre-rinsed 0.45-μm cellulose ester filters, after which TP and SRP analyses were conducted using an acid–molybdenum–blue colorimetric method (after digestion with potassium persulphate for TP). The TN and NO₃-N analyses were conducted using a UV

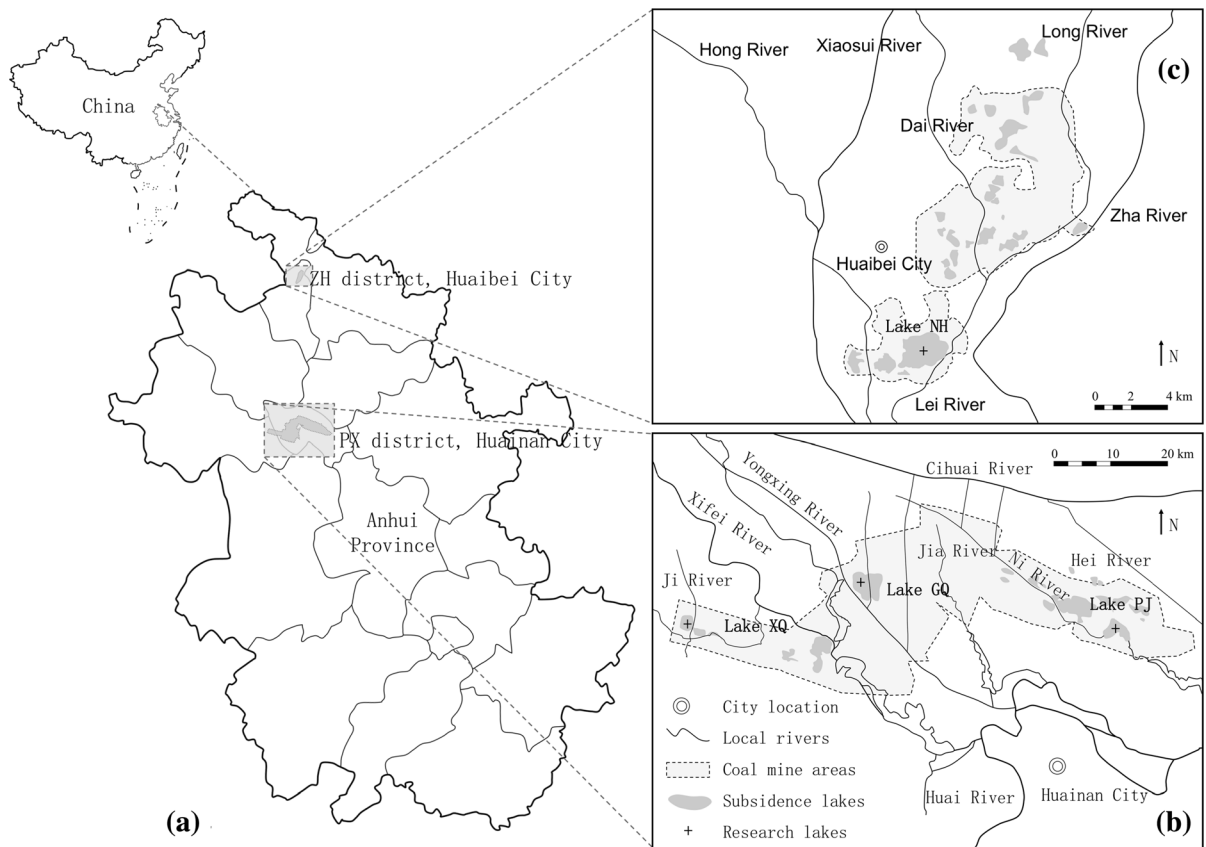


Fig. 1 Locations of the two coal mine areas selected for study (a) and four lakes in Anhui Province, China. Lake NH is located in ZH district of the Huaibei coal mine area (c), while lakes PJ, GQ and XQ are located in PX district of the Huainan coal mine area (b)

colorimetric method (after digestion with alkaline potassium persulphate for TN). The $\text{NH}_4\text{-N}$ and $\text{NO}_2\text{-N}$ concentrations were determined using phenol–hypochlorite and diazotization with sulfanilamide dihydrochloride, respectively. Chlorophyll-a (Chl-a) concentration was determined through fluorescence spectrophotometry after extraction with methanol for 24 h in the dark at $-20\text{ }^\circ\text{C}$. Calibration was performed using a Sigma Chl-a standard. The reproducibility of the measurements was better than 95 %.

Sediment samples were collected using a gravity corer (Lenz, Hydro-Bios of Germany). This study focused on the potential for sediment P release from surface sediment; thus, the top 5 cm of each sediment sample was retained as being representative of the surface sediment and stored directly in air-sealed plastic bags for transportation to the lab in a portable freezer set at $4\text{ }^\circ\text{C}$. Sediment samples were air-dried at room temperature, homogenised and

sieved through a $150\text{ }\mu\text{m}$ stainless steel-sieve, and then stored in the dark for further analysis. A total of 20 surface sediment sub-samples were analysed for pH, oxidizable matter (OM), TN, TP, and inorganic phosphorus (IP).

Sediment pH was measured by glass electrode in a 1:2.5 (w/v) ratio of sediment suspension to distilled water. The OM represents the oxidizable matter remaining after the treatment of the sample with chromic acid/ H_2SO_4 according to the Walkley-Black method (Tan 1995). The TN contents were determined using the Kjeldahl method. Inorganic P and TP were determined as SRP after extraction of sediments with 1.0 M H_2SO_4 before and after combustion ($550\text{ }^\circ\text{C}$, 2 h), respectively, according to the method of Lu (1999). Extracted Fe was measured with an inductively coupled plasma-mass spectrometer (ICP-MS; model Perkin-Elmer NexION 300X), and extracted P was determined by the molybdenum blue/ascorbic

acid method (APHA et al. 1998), which has a detection limit of around 0.3 $\mu\text{mol/L}$ for P.

Fractionation of Al, Fe and P, and Ca-bound P forms

Sequential extraction was performed on 0.5 g of air-dried sediment to determine the operationally defined fractions of Al, Fe, and P, using the method proposed by Psenner et al. (1988) with slight modifications as suggested by Hupfer et al. (1995). This extraction procedure is used widely to estimate the potential mobility of adsorbed P in the presence of reducing conditions in sediment, and includes five sequential steps: (1) 1 M NH_4Cl to remove ion-exchangeable species (NH_4Cl -extractable); (2) 0.11 M NaHCO_3 and 0.11 M $\text{Na}_2\text{S}_2\text{O}_4$ to remove reducible metal hydroxides, predominantly amorphous $\text{Fe}(\text{OH})_3$, and P associated with these phases (BD-extractable); (3) 1 M NaOH at 25 °C to partially extract organic material, $\text{Al}(\text{OH})_3$ (solid) and associated P (Al-P) (NaOH_{25} -extractable); (4) 0.5 M HCl to extract mineral Al and Fe and P associated with calcite and apatite (HCl-extractable); and (5) 1 M NaOH at 85 °C to extract residual species (NaOH_{85} -extractable) (Lake et al. 2007; Wilson et al. 2008; Norton et al. 2008). Phosphorus bound to calcium compounds was further fractionated according to the method proposed by Jiang and Gu (1989) at Lake NH, which has calcareous soil. Sediment samples were extracted sequentially using 0.25 M NaHCO_3 , 0.11 M NH_4Ac and 0.5 M H_2SO_4 , with focus on the fractions of calcium biphosphate ($\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$, CaP_2), octacalcium phosphate ($\text{Ca}_8\text{H}_2(\text{PO}_4)_6 \cdot 5\text{H}_2\text{O}$, $\text{Ca}_8\text{-P}$), and apatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{HO})_2$, $\text{Ca}_{10}\text{-P}$). The extracted supernatants were filtered (Whatman GF/C, pore size 0.45 μm) prior to analysis. The concentrations of Al and Fe in each extract were measured by ICP-MS, while P was determined as orthophosphate by spectrophotometry as in the above method for sediment analysis. Total extractable P (Ext-TP) is the sum of the P measured in all extracted fractions. All concentrations are presented as mmol/kg of dry sediment weight.

P isothermal adsorption experiments

Isothermal experiments were performed on 20 subsamples to characterise the P sorption capacity of

sediments. Phosphorus-containing solutions were prepared using artificial lake water samples reflecting the local water chemistry, which has a weakly alkaline pH mediated by carbonate-bicarbonate buffers in both the Huainan and Huaibei areas (see Table 1). The P adsorption results presented here for lakes PX, GQ, and XQ were originally published in two studies that investigated the impacts of major ions on P adsorption in the Huainan mining area (Xie et al. 2012; Yi et al. 2013). Isothermal experiments in Lake NH were conducted on the same sediment samples as those subjected to fractionation of P, Fe and Al.

Briefly, an artificial lake water sample was prepared with NaHCO_3 and CaCl_2 dissolved in deionized water, producing a final solution of 4 mmol/L NaHCO_3 and 2 mmol/L CaCl_2 for representing lakes PJ, GQ and XQ, and a solution of 6 mmol/L NaHCO_3 and 3 mmol/L CaCl_2 for Lake NH. Solutions with eight initial P concentrations ranging from 0 to 6.4 $\mu\text{mol P/L}$ were prepared based on these stock solutions. For the isotherm experiments, 0.25 g of sediment was mixed with a 25 mL sample of P-spiked artificial lake water in 50 mL polypropylene centrifuge tubes and shaken at 25 ± 1 °C for 24 h. The suspensions were then centrifuged ($2000 \times g \times 30$ min), and the supernatants passed through 0.45 μm cellulose acetate membrane filters. The phosphorus concentrations in the solutions were determined spectrophotometrically using the molybdenum blue method. The amount of adsorbed P was then calculated based on the difference between the initial amount of P added and the amount in the equilibrium solution.

Mathematical descriptions of the sorption isotherms were created by fitting the datasets with linear functions: $Q = kEPC + b$, where Q is the amount of sorbed P ($\mu\text{mol/kg}$), and EPC is the equilibrium P concentration. The factor k (slope) is the sorption constant (L/kg) and reflects the bonding energy at sorption affinity, and b (intercept, $\mu\text{mol/kg}$) is the native adsorbed P (NAP). The zero equilibrium P concentration (EPC_0) is the solution for EPC when Q equals zero at the y-axis against EPC concentrations at the x-axis. The variable EPC_0 (mg/L) is a measure of the P concentration at which sediment is neither adsorbing nor desorbing P (equilibrium state). When the dissolved phosphorus concentration of ambient sediments is greater than EPC_0 , net adsorption occurs. When it is smaller than EPC_0 , desorption occurs. Higher values of k indicate stronger P sorption ability

Table 1 Chemical properties of surface water and sediments in four lakes selected for study at the lakes NH, PJ, GQ and XQ in the H–H coal mine areas, Anhui

Lake	Surface water						
	pH	DO ($\mu\text{mol/L}$)	TN ($\mu\text{mol/L}$)	NO_3^- -N ($\mu\text{mol/L}$)	TP ($\mu\text{mol/L}$)	Chl-a ($\mu\text{g/L}$)	
NH	8.5 ± 0.2^a	283.1 ± 5.3	19.3 ± 1.4	3.6 ± 0.0	1.2 ± 0.1	3.8 ± 0.8	
PJ	8.5 ± 0.2	264.3 ± 30.3	148.6 ± 15.7	67.9 ± 7.9	2.9 ± 0.3	39.4 ± 6.7	
GQ	8.3 ± 0.1	223.4 ± 7.8	48.6 ± 2.9	10.7 ± 0.0	1.8 ± 0.0	27.5 ± 1.5	
XQ	9.2 ± 0.2	327.8 ± 11.9	151.4 ± 2.9	83.6 ± 0.7	1.3 ± 0.3	53.3 ± 4.9	
Lake	HCO_3^- (meq/L)	Cl^- (meq/L)	SO_4^{2-} (meq/L)	Na^+ (meq/L)	K^+ (meq/L)	Ca^{2+} (meq/L)	Mg^{2+} (meq/L)
NH	4.9 ± 0.2	3.5 ± 1.4	3.1 ± 1.4	5.3 ± 2.1	0.2 ± 0.1	2.0 ± 1.1	2.8 ± 0.5
PJ	3.0 ± 0.1	3.7 ± 0.2	1.9 ± 0.1	4.2 ± 0.1	0.1 ± 0.0	1.7 ± 0.1	1.7 ± 0.1
GQ	3.2 ± 0.0	1.4 ± 0.0	0.9 ± 0.0	2.3 ± 0.0	0.1 ± 0.0	1.9 ± 0.0	1.4 ± 0.0
XQ	3.9 ± 0.1	3.4 ± 0.1	4.0 ± 0.0	5.1 ± 0.1	0.2 ± 0.0	2.5 ± 0.2	2.0 ± 0.0
Lake	Sediments						
	pH	OM (%)	TN (mol/kg)	TP (mmol/kg)	IP (mmol/kg)		
NH	8.6 ± 0.2	2.1 ± 0.6	0.8 ± 0.3	17.8 ± 0.8	15.7 ± 0.6		
PJ	7.8 ± 0.0	4.3 ± 0.5	2.4 ± 0.4	17.6 ± 4.6	14.3 ± 2.3		
GQ	7.5 ± 0.0	2.2 ± 0.2	1.4 ± 0.2	11.3 ± 5.8	10.9 ± 1.2		
XQ	7.8 ± 0.0	1.8 ± 0.3	1.3 ± 0.2	12.2 ± 1.1	9.5 ± 0.8		

^a Mean values and standard errors of all sampling sites in each lake

on sediments; whereas lower values of EPC_0 indicate weaker potential for sediment P release. Linear correlation analyses of adsorption isotherms at all sediment sampling sites were performed by calculating Pearson correlation coefficients at confidence levels of 95 % ($P < 0.05$) and 99 % ($P < 0.01$). Average k and EPC_0 values and their standard errors were calculated, based on measurements across five sediment sampling sites in each lake, in order to describe the general adsorption potential of sediments. Subsequent comparisons of mean k and EPC_0 values among the four lakes were conducted to test for significant differences at a confidence level of 95 % ($P < 0.05$).

Results

Chemical properties of surface water and sediments

Water samples from all four lakes were rich in dissolved oxygen, reflecting their eutrophic states affected by the diurnal photosynthetic activity of algae. Overall, these

lakes exhibited weakly alkaline pH values (Table 1), which was mediated primarily by the bicarbonate buffers dominating the local water chemistry. However, the three lakes PJ, GQ and XQ in the Huainan mining area had lower alkalinity and salinity than Lake NH in the Huaibei area, reflecting regional differences in water chemistry. The mean concentrations of Chl-a, TN and TP were in the ranges of 3.8–53.3 $\mu\text{g/L}$, 19.3–151.4 $\mu\text{mol/L}$, and 1.2–2.9 $\mu\text{mol/L}$, respectively, across the four lakes (Table 1). Lake NH in the Huaibei coal mine area displayed characteristics of mesotrophy, whereas the other three lakes in the Huainan coal area exhibited meso-eutrophic states (Qu et al. 2013). The total nitrogen concentrations in the lakes PJ and XQ were significantly higher than that in the other lakes, as a consequence of inputs from local rivers. With respect to inorganic nitrogen forms, only nitrate was present in detectable amounts. Nitrate accounted for approximately 50 % of TN in the lakes PJ and XQ, with average concentrations of 67.9 and 83.7 $\mu\text{mol/L}$, respectively. However, nitrate concentrations in the lakes NH and GQ were much lower, at 3.6 and 10.7 $\mu\text{mol/L}$, respectively. Soluble reactive phosphorus concentrations in all lakes were below detection limits, probably due to algal uptake.

The sediments also had weakly alkaline pH values (Table 1) and exhibited a pattern similar to that for water pH. Sediments at Lake NH in the Huaibei coal mine area exhibited a higher pH than those in the lakes located in the Huainan coal mine area. The mean OM and TN concentrations in sediment were in the ranges of 1.8–4.3 and 1.1–3.3 %, respectively, with the highest values observed at Lake PJ, which receives significant pollutant discharge from the Ni River. Mean sediment TP and IP concentrations at Lakes PJ and NH were higher than at lakes GQ and XQ, ranging from 11 to 18 mmol/kg for TP and 10 to 16 mmol/kg for IP. Inorganic phosphorus comprised the predominant fraction of sediment P, accounting for 77.6–96.4 % of TP.

Fractionation of P, Al, and Fe compounds

The total extractable P (Ext-TP) ranged from 10 to 17 mmol/kg across all four lakes in the H–H coal mine areas (Fig. 2a). At Lake PJ, the largest percentage of Ext-P was in the BD fraction, accounting for 41.7 % of Ext-TP. This fraction also constituted large proportions of Ext-TP at lakes GQ and XQ, with mean values of 21.5 and 27.9 %, respectively. Sediments in Lake NH in the Huaibei coal mine area displayed the smallest mean BD-P percentage, 10.6 %. The contribution of the NaOH₂₅ fraction was similar across the three lakes in the Huainan mining area, ranging from 21.6 to 30.4 % of Ext-TP. The NaOH₂₅-P percentage at Lake NH was lower, accounting for 8.1 % of Ext-TP. The HCl-P fraction was dominant in Lake NH, accounting for 70.0 % of Ext-TP, which was considerably higher than its proportions (21.9–36.5 %) of Ext-TP in lakes PJ, GQ and XQ. Residual P, denoted as the NaOH₈₅ fraction, was consistent across all lakes, in the range of 8.9–13.0 % of Ext-TP. The sum of the NaOH₂₅-P and BD-P fractions was the main contributor to Ext-TP in sediments in the Huainan mining area (accounting for 50.0–65.0 % of Ext-TP), whereas Ca-bound fractions were predominant in sediments from the lake in the Huaibei mining area where NaOH₂₅-P and BD-P together accounted for only 20.0 % of Ext-TP. Apatite (Ca₁₀-P) accounted for 87.1 % of extractable Ca-P in Lake NH, consistent with the characteristics of the calcareous soils typifying this area (Fig. 2b).

The mean total extractable Fe (Ext-Fe) ranged from 126 to 185 mmol/kg for the four lakes (Fig. 3).

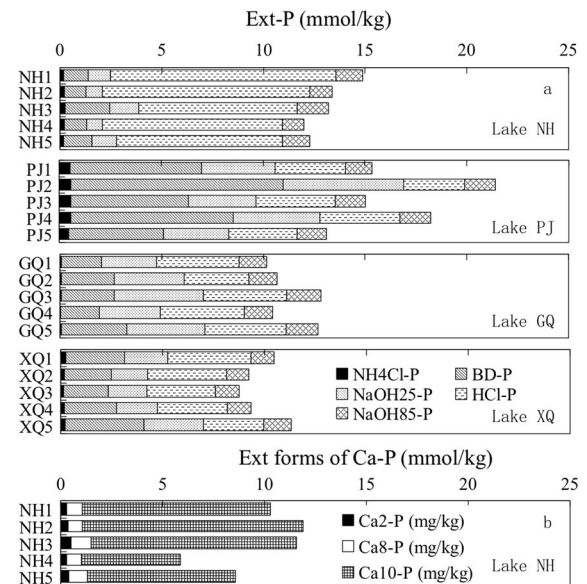


Fig. 2 Fractions of extractable P (NH₄Cl-P, BD-P, NaOH₂₅-P, HCl-P and NaOH₈₅-P) in five sediment samples at lakes NH, PJ, GQ and XQ (a), and extractable calcium-bound P (Ca-P₂, Ca₈-P, and Ca₁₀-P) at Site NH (b), in the H–H coal mine areas, Anhui

Sediment in Lake XQ displayed the lowest extractable Fe. Regardless of regional differences, the majority of the extractable sediment Fe appeared in the BD- and HCl-extractable fractions, ranging from 15.6 to 24.4 % for BD-Fe and 61.4 to 76.2 % for HCl-Fe, both of which contributed approximately 85.5–91.8 % of Ext-Fe. Less than 2 % of Fe was present in the NaOH₂₅-Fe fraction because the BD solution extracted most of the reducible Fe, including Fe(OH)₃.

The total extractable sediment Al ranged from 336 to 624 mmol/kg (Fig. 4). The surface sediments in Lake NH indicated the lowest concentration of Al in comparison to the other three lakes (approximately half of that in Lake PJ). Sediment Al concentrations were generally comparable among lakes PJ, GQ, and XQ, likely a function of similar soil properties. An inverse pattern to that observed for the Fe fractions was observed for the NaOH₂₅-Al fraction (10.2–13.7 % of total Ext-Al), which was significantly higher than BD-Al (2.2–4.5 %) for all lakes, because Al(OH)₃ is stable under reducible conditions. The most abundant Al forms in the sediments were NaOH₈₅-Al, in the range of 37.7–52.9 % of Ext-Al, followed by HCl-Al, in the range of 29.5–44.4 % of Ext-Al. Overall, the characteristics of the sediment P, Fe and Al fractions

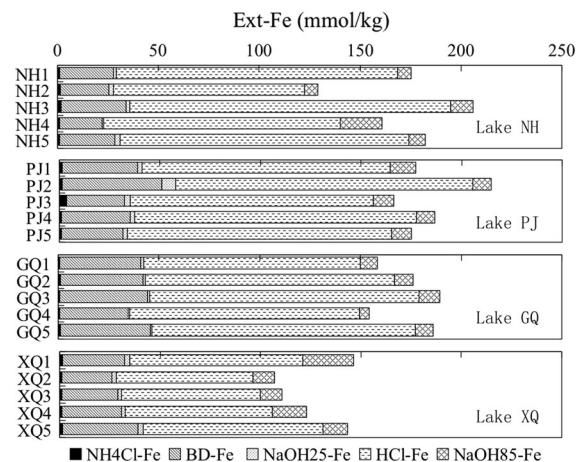


Fig. 3 Fractions of extractable Fe ($\text{NH}_4\text{Cl-Fe}$, BD-Fe , $\text{NaOH}_{25}\text{-Fe}$, HCl-Fe and $\text{NaOH}_{85}\text{-Fe}$) in five sediment samples at lakes N, P, G and X in the H–H coal mine areas, Anhui

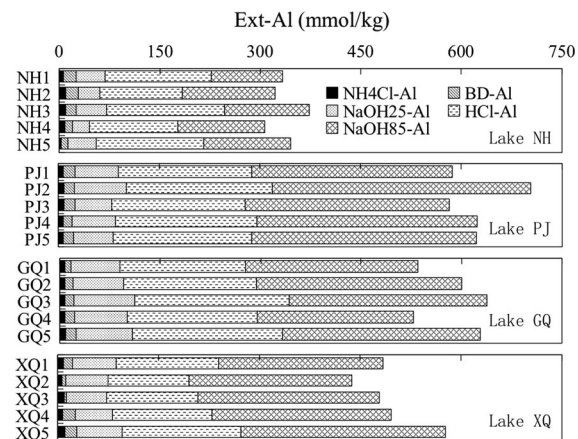


Fig. 4 Fractions of extractable Al ($\text{NH}_4\text{Cl-Al}$, BD-Al , $\text{NaOH}_{25}\text{-Al}$, HCl-Al and $\text{NaOH}_{85}\text{-Al}$) in five sediment samples in each lake at lakes NH, PJ, GQ and XQ in the H–H coal mine areas, Anhui

were primarily differentiated by regional soil properties, although there was spatial heterogeneity between sampling sites within each lake.

Model of Al:Fe and Al:P ratios

The empirical approach of Kopacek et al. (2005) predicts a low P flux if the molar ratio of Al:Fe > 3 or Al:P > 25 , under anoxic conditions in sediment. The molar ratios of Al:Fe versus $[\text{NH}_4\text{Cl-P} + \text{BD-P}]$ in sediment (Fig. 5) were graphed for all four lakes using the model proposed by Kopáček et al. (2005). All of

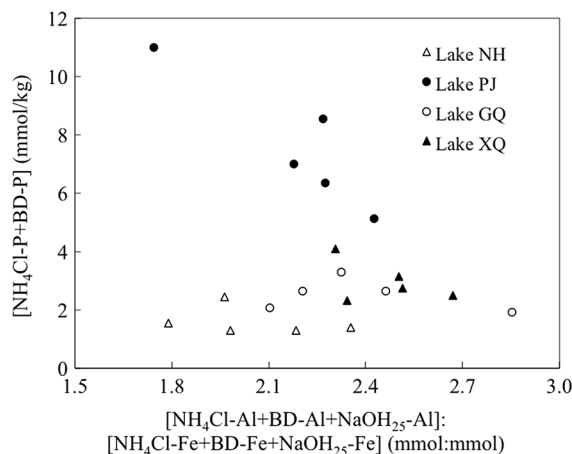


Fig. 5 Concentrations of reactive P in NH_4Cl and BD extraction solutions versus the Al:Fe ratio as measured in NH_4Cl , BD , and NaOH_{25} solutions from sediments at lakes NH, PJ, GQ and XQ in the H–H coal mine areas, Anhui

the Al:Fe ratios were less than 3:1, suggesting the potential for sediment P release under anoxic conditions. Lake PJ exhibited $[\text{NH}_4\text{Cl-P} + \text{BD-P}]$ of 5–11 mmol/kg, which was higher than observed in the other lakes, where the mean $[\text{NH}_4\text{Cl-P} + \text{BD-P}]$ was less than 3 mmol/kg P.

To further validate the model, the molar ratios of Al:P were calculated for sediment samples from each lake (Fig. 6). At Lake GQ, the Al:P ratios were >25 , whereas Lake PJ displayed values <25 , consistent with the Fe-based model predicting a high potential for sediment P release in Lake PJ and a low potential at Lake GQ. The mean Al:P ratios at lakes NH and XQ were approximately 24 and 21, respectively. If the threshold of the Al:P ratio is relaxed from >25 to >20 , the lakes NH and XQ become suitable for application of the model. The threshold of the Al:P ratio <25 is a generalized empirical mean value based on statistical results obtained from many different types of lakes, permitting minor adjustments to the model for specific sites.

Phosphorus adsorption isotherms

The phosphorus sorption of sediments from the four lakes was tested by performing isotherm adsorption measurements. Isotherm plots (Q vs. EPC) were fit to a linear adsorption isotherm model by adjusting average values of k , EPC_0 and b on a site-specific basis (Fig. 7). Most isotherms were highly linear ($P < 0.01$)

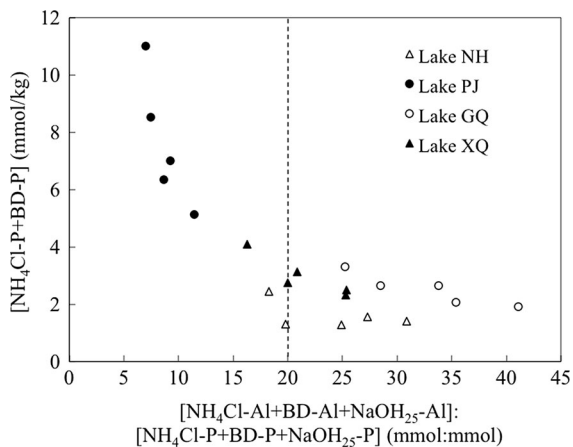


Fig. 6 Concentrations of reactive P in NH_4Cl and BD extraction solutions versus the Al:P ratio as measured in NH_4Cl , BD, and NaOH_{25} solutions from sediments at lakes NH, PJ, GQ and XQ in the H–H coal mine areas, Anhui. The vertical solid line denotes an Al:Fe ratio of 20:1 in terms of molarity

except at some sediment sampling sites, due to the spatial heterogeneity of sediment properties. In particular, samples from Lake NH exhibited regression slopes that were almost vertical, which may have been caused by the considerable P sorption potential of calcium-rich soils.

Differences among the four lakes were reflected in the values of the isotherm coefficients k and EPC_0 . The k value at Lake PJ was 186.4 ± 29.7 L/kg (Fig. 7b), which was significantly lower than the values observed at lakes GQ and XQ, whose mean k values were 613.2 ± 279.2 (Fig. 7c) and 567.2 ± 231.9 L/kg, respectively (Fig. 7d). The EPC_0 of 1.9 ± 0.4 $\mu\text{mol/L}$ at Lake PJ was significantly higher than that at the lakes GQ (1.0 ± 0.7 $\mu\text{mol/L}$) and XQ (1.4 ± 0.5 $\mu\text{mol/L}$). The sediment samples from Lake NH exhibited a moderate k value of 368.2 ± 149.3 L/kg and a very low EPC_0 value of 0.5 ± 0.3 $\mu\text{mol/L}$. However, these were not significantly different from the corresponding values observed in sediments from lakes PJ, GQ and XQ due to considerable variability in these two parameters in NH isotherms. Sediment samples from Lake PJ, with the lowest k values and largest EPC_0 values, had the weakest P adsorption ability and greatest potential for sediment P release among the four lakes.

Based on the above results, the sediment adsorption results are consistent with predictions by the model proposed by Kopáček et al. (2005), indicating the highest potential for sediment P release at Lake PJ with an Al: Fe ratio < 3 and Al:P ratio < 25 and the

lowest potential for sediment P release at the lakes NH, GQ, and XQ with Al: P ratios > 20 .

Discussion

Characterisation of Al, Fe and P in the sedimentary environment of soil inundation

The mean extractable-TP concentrations of the four subsidence lakes were less than 17 mmol/kg, which is lower than the range of 20–100 mmol/kg reported for eutrophic lakes (Kopáček et al. 2005; Wilson et al. 2008). Some studies have indicated that, qualitatively, soils in wetlands have a significant P sorption potential related to their amorphous Fe and Al content (Dunne et al. 2005; Lai and Lam 2009; SanClements et al. 2009). Quantitative relationships (ratios) describing the nature and abundance of P, Fe and Al in lake sediments could be adapted in a predictive capacity to environments characterized by soil inundation. Surface P in the sedimentary environment of soil inundation maintains the characteristics of local agricultural soils because of the relatively short time that has typically elapsed since lake formation. Sediments at the lakes GQ, XQ and NH were primarily derived from inundated soils, while the sediments in Lake PJ were derived mostly from lake sediment, on the basis of the texture and appearance of sediment samples. The behaviour of P in the sediments of the lakes studied here was primarily determined by the local soil properties displayed fundamental differences between Lake NH in the Huaibei coal mine area and lakes PJ, GQ, XQ in the Huainan coal mine area. Fractionation of Al, Fe, and P in sediments collected from lakes PJ, GQ, and XQ revealed similar compositions across these lakes, characterised by high ratios of Fe- or Al-bound P to TP, albeit with some heterogeneity. However, the sediment samples from Lake NH were characterised by a high ratio of Ca-bound P to TP and a high Al: P ratio, in spite of a low concentration of Ext-Al.

Further fractionation of Ca-P confirmed that the sediments in Lake NH are dominated by apatite, an inert, insoluble form of P (Fig. 2b). The behaviour of P is generally related to the presence of calcium carbonate in high-pH lakes or soil solutions, where P can be adsorbed or co-precipitated with calcium compounds, resulting in P immobilisation (Tunesi

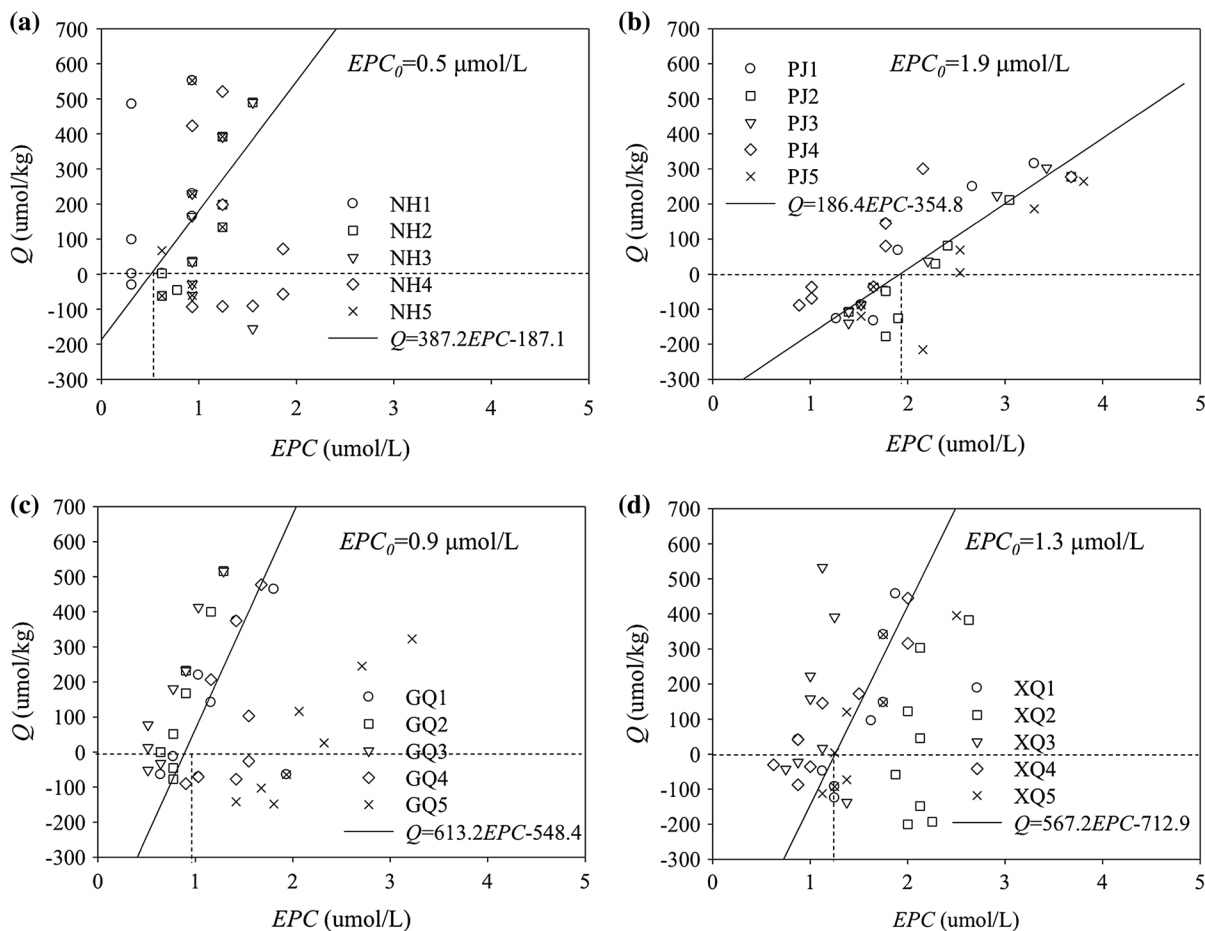


Fig. 7 Comparison of P adsorption isotherms for surface sediment samples from lakes NH (a), PJ (b), GQ (c), and XQ (d) sites in the H–H coal mine subsidence areas, Anhui, China

et al. 1999; Dittrich et al. 2011). This may be another important factor (in addition to having an Al: P > 20) favouring P sorption on sediments in Lake NH.

Different processes, related to regional soil properties, are responsible for the differing mechanisms of P transformations in the sediments of lakes in the Huainan and Huaibei mine areas. Phosphorus in the fertilised soils of the Huainan area is typically associated with Fe or Al compounds due to high Fe-oxide reactivity (Fig. 3) and high Al concentrations (Fig. 4); whereas in the Huaibei area, P tends to be associated with calcium compounds.

In the Huainan mining area, the sediment samples from Lake PJ exhibited greater potential for sediment P release than those from lakes GQ and XQ. There may be two explanations for this phenomenon. First, Lake PJ receives a heavy pollutant load from the Ni

River, whose surface sediments develop through sedimentation (not inundation) and which have a darker colour and more OM content. This external input could provide enriched levels of Fe-bound P, leading to an increased BD-P content and a higher ratio of $\text{NH}_4\text{Cl-P}$ and BD-P to TP, favouring P-release. Some studies demonstrated competition between OM and phosphate for available adsorption sites on Al hydroxides, which could reduce the capability of Al to re-trap P (Tipping 1981; Canfield et al. 1993; Kopáček et al. 2005). High sediment OM concentrations in Lake PJ likely decrease P re-adsorption on $\text{Al}(\text{OH})_3$. Secondly, OM degradation, which supplies electron donors in redox reactions, is considered the primary process that enhances biological reactions or OM cycling and, indirectly, P flux from sediments (Lovley 1997; Kristensen 2000; Katsev et al. 2006). This may

explain why a higher potential for sediment P-release would be expected in Lake PJ under conditions of anoxia. This hypothesis was verified in previous research on the simulation of soil inundation with the addition of external OM (Yi et al. 2014b).

Implications of ecological rehabilitation from the perspective of sedimentary environments

Decisions regarding nutrient management and water utilization in lakes should consider regional geography, climate and water availability. The Huaibei mine area has a cold and dry climate, alkaline water chemistry and calcareous soils, favouring conditions of P or N deficiency in the water column. This leads to low primary productivity and decreased fishery potential, but it improves source protection of the local drinking water supply, compensating for the water shortages experienced in Huaibei City.

The lake nutrient level is determined by mass balance controlled by four factors, namely, external load, sedimentation or volatilization (for nitrogen), sediment release and lake effluent. The potential for sediment P release is naturally lower in the Huainan mining area due to the abundance of Al and its capacity to enhance P adsorption, even though the Al: Fe ratio is <3 . However, the sediment Al: P ratio was close to the threshold of 20:1, which may be easily reduced by external factors such as high OM and enriched P loads in the lake sediments. The likelihood of P release from Fe-oxides is significantly enhanced under conditions of anoxia and increased P loading. Additionally, coal mine subsidence lakes are usually small, and thus, water quality parameters may respond quickly to changes in nutrient loading. For example, Lake PJ is rich in Fe-oxide associated P. Moreover, the water column TP concentrations are considerably greater than those in the lakes GQ and XQ. The lakes GQ and NH presented the lowest nutrient levels, a consequence of loading of nonpoint source pollutants and low potential for sediment release. Concentrations of both N and P in these two lakes therefore meet Grade III of Environmental Quality Standards for surface water in China (GB338-2002), at 1.0 mg/L for TN (71.4 $\mu\text{mol N/L}$) and 0.05 mg/L for TP (1.6 $\mu\text{mol P/L}$). Previous research confirmed that the primary production in these two lakes displayed patterns of N limitation and/or N-P co-limitation due to a deficiency of inorganic nitrogen (Wang et al. 2013; Yi et al. 2014a).

Agricultural areas usually exhibit high N: P ratios in runoff. In the research area loading of pollutants to lake XQ and Lake PJ greatly increased N concentrations at the relative expense of P. Meanwhile, the potential for sediment P release was still limited by high Al: P ratios, resulting in P deficiency. Furthermore, P is easily removed through sedimentation while N loss through denitrification may be inhibited under the relatively oxic environment in lakes without thermal stratification. In addition, a smaller proportion of nitrate is likely assimilated (relative to P uptake) by algae. Consequently, higher ambient nitrate concentrations and P limitation on phytoplankton production occurred in these two lakes (Yi et al., 2014a). Additionally, when nitrate accounts for a large proportion of TN, the reduction and dissolution of ferric hydroxides as electron acceptors for OM microbial decomposition is inhibited, especially when oxygen at the sediment interface is also depleted (Tirén and Pettersson 1985; Hansen et al. 2003; Wauer et al. 2005).

Therefore, nutrient management for ecological conservation should focus on P because it is the nutrient that limits primary production in subsidence lakes. Furthermore, it is important to control OM load and to maintain oxidative conditions at the sediment–water interface.

Conclusions

Characterisation of sediment behaviour based on speciation of Al, Fe and P in the sedimentary environments of soil inundation is strategically important for regional ecological rehabilitation and conservation in various aquatic zones. The subsidence lakes that have formed in the Huainan and Huaibei coal mine areas provide an opportunity to address the issue of eutrophication. The main conclusions of this study are as follows:

- (1) Control of sediment P release by Al and Fe compounds in inundated sedimentary environments is primarily a function of regional soil properties. The potential for sediment P release in lakes located in the Huainan coal mine area is governed primarily by Fe and Al compounds; whereas that in the studied lakes located in the Huaibei coal mine area is controlled by the

calcareous properties of local soils. In this study, P concentrations in the subsidence lakes displayed a clear relationship with the features of sedimentary environments, as characterised by fractionation of P, Al and Fe. This observation was further supported by the isotherm adsorption measurements of surface sediments.

- (2) The results observed fit the model proposed by Kopáček et al. (2005) and verified its success in predicting the potential for sediment P release by using the threshold of an Al: P ratio > 25 when the Al: Fe ratio is <3. This threshold has some flexibility across sites, with its lower limit approaching 20:1, making it applicable for the three lakes in the Huainan coal mine area. Higher loading of OM and/or enriched Fe-oxide-bound P could lead to increased potential for sediment P release in an anoxic environment.
- (3) Because coal mine subsidence lakes are small and have little assimilative capacity for nutrients, controlling inputs from local rivers is particularly important for ecological conservation. Nutrient management should focus on P because it is the nutrient that limits primary production in such lakes.

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