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# Phosphorus fractionation and bio-availability in Taihu Lake(China) sediments

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**Abstract**: Typical sediments from Taihu Lake, a meso-to-hypereutrophic lake, were collected and examined on the basis of P-fractionation by sequential extraction scheme. Sedimentary inorganic phosphorus were fractioned into four forms and the rank order according to the mean concentration of P-fractions in Taihu Lake was NaOH-P > BD-P > HCl-P > $NH_4$Cl-P. The concentrations of BD-P were linearly correlated with the content of active Fe($R^2$ = 0.96). Also, the linear relationship between the sum of BD-P and NaOH-P and the sum of active Fe and active Al content was observed within the six sediments investigated($R^2$ = 0.96). Moreover, the bio-available phosphorus (BAP) content was estimated by the sum of $NH_4$Cl-P, BD-P, and NaOH-P, viz. BAP = $NH_4$Cl-P + NaOH-P + BD-P. In Taihu Lake, the BAP contents are ranging from 0.10 mg/g dw to 1.25 mg/g dw, and average 0.40 mg/g dw for all sediment samples. The relative contributions of BAP to total sedimentary phosphorus(TP) and inorganic sedimentary phosphorus(IP) range from 18.67% to 50.79% (33.61% on average) and from 52.82% to 82.09% (67.81% on average), respectively.

**Keywords**: phosphorus fractionation; bio-availability; sediment; Taihu Lake

## Introduction

Sediment phosphorus has been recognized as the most critical nutrient limiting lake productivity (OECD, 1982). The concentration of phosphorus in the sediment is often 1000-fold higher than that in lake water and its release depends on the concentration and distribution of phosphorus in the sediments, the degree of saturation of exchangeable phosphorus, the intensity of biological processes in the sediment and lake water, and hydrological conditions(Pardo, 1998). The trophic status of lakes are usually dependent on the phosphorus concentration in the water. Also, the trophic status and the trophic development of the system are influenced by the phosphorus content in lake sediments due to its dual roles (sink or source) under certain environmental conditions(Ramm, 1997; Zhou, 2001).

Several methods can be used to determine the bio-availability and mobility of sedimentary phosphorus, such as algae bioassays, chemical fractionation, exchange with anion resin, electrodialysis and isotopic exchange. Among them, the chemical fractionation, which involves sequential extraction procedures, has been widely used. Chang and Jackson (Chang, 1957) proposed the first phosphate fractionation method in soils using chemical extractants. It has been modified by many other authors and these P-fractionations partition sedimentary-P mainly into such different fractions as labile P, reductant P, metal bound P, occluded P and organic P by using various chemical extractants(Williams, 1976; Psenner, 1988; Stone, 1993). In addition, in view of potential P bio-availability, sedimentary P can be considered as nonavailable, potentially available and immediately available(Pettersson, 2001; Zhou, 2001). Among them, immediately available phosphorus in sediments of lakes stands for the most accessible form of phosphorus to algae, which equates to soluble inorganic phosphorus and almost exclusively orthophosphate (Bartsch, 1972).

Taihu Lake lies in the Yangtze River Delta. It is a typical shallow freshwater lake, with an area of 2338 $km^2$ and average depth of 1.9 m. The Taihu Lake region is one of the most densely populated areas in China; it accounts for 0.4% of the total area of China and 2.9% of the nation's population, and it provides more than 14% of the gross domestic production (GDP) (Shen, 2000). The Taihu Lake bed is covered with sediment that averages 0.58 m thick. However, the forms of P in the Taihu Lake sediments have been poorly studied. The knowledge of distribution and bio-availability of P forms of interest will therefore be of significance for further study of mechanism and restoration of eutrophication. The purpose of this study was to investigate the different P-forms present in the sediments of the Taihu Lake and to evaluate their possible contribution to the P-loadings of the system.

## 1 Methodology

### 1.1 Sediment collection and analysis

Six typical sediment samples (T1—T6) were collected from the Taihu Lake related to the trophic status and positional feature in January 2002. Information about all sampling sites is listed in Table 1. In all of these samples, T1, T2 and T4 assume typical trophic status of Taihu Lake. T1 is in the Wuli sublake that is the nearest place to Wuxi City, Jiangsu Province. The ecological system of this area is strongly influenced by industrial production, human activities, and shipping business. The pollution situation of this area is rather serious. T2 is in eutrophic state. T4 is almost located at the center of the lake, and in mesotrophic state. At each sampling site, three cores were used to collect the sediment and the upper 5 cm sediment of each core was sliced and pooled together to obtain relative homogenized materials. Samples were stored wet in sealed Zip-lock bags at 4℃ until used. Back to laboratory the sediment samples were air-dried at room temperature. To remove large debris in the sediments, they were passed through a 60-mesh sieve and subsequently the subsamples were passed through 260-mesh (63 μm) sieve before being used in the adsorption trials.

The total contents of metals as Fe, Al, Ca and Mn in the sediments were measured by ICP-AES method after digestion. The ammonium oxalate-oxalic acid extract is considered to yield active iron and aluminum (Heleen, 1993). The sediments were shaken with a solution of oxalic acid and ammonium oxalate at pH = 3. The extraction solution reduces the poorly soluble iron(Ⅲ) ions to the much more soluble iron(Ⅱ) ions. As light influences the reducing action of oxalic acid, the extraction has to be performed in the dark. The total dissolved iron and aluminum in the

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solution, which are extracted as active iron and aluminum, were determined with a spectro analytical instruments ICP type spectroflame with AES. More details were provided by Heleen (Heleen, 1993). Organic matter measurement, expressed as Loss on ignition (LOI), was based on weight losses after combustion at 550℃. TOC represents the oxidizable matter after treatment of the sample with chromic acid/$H_2SO_4$ according to the Walkey-Black method (Tan, 1995).

**Table 1 The characteristics of sampling points**

| Sites | Site position | | Overlying water characteristics | | | | |
|---|---|---|---|---|---|---|---|
| | E | N | Water depth, m | pH | DO, mg/L | Clarity, cm | Trophic status* |
| T1 | 120.22° | 31.54° | 0.9 | 8.36 | 10.88 | 60 | Hypereutrophic |
| T2 | 120.21° | 31.50° | 2.2 | 8.54 | 11.87 | 90 | Eutrophic |
| T3 | 120.06° | 31.44° | 2.0 | 7.97 | 9.47 | 70 | Eutrophic |
| T4 | 120.22° | 31.23° | 2.8 | 8.16 | 10.87 | 20 | Mesotrophic |
| T5 | 119.96° | 31.21° | 2.5 | 8.07 | 10.15 | 30 | Mesotrophic |
| T6 | 120.44° | 31.43° | 1.2 | 8.89 | 10.58 | 120 | Mesotrophic |

Notes: * Factors that influence comprehensive evaluation of trophic state include nutrient concentration, chlorophyll *a* content in algae, clarity of lake water and dissolved oxygen, etc. Here the trophic states in different areas of Taihu Lake were evaluated by the biomass of algae observed and reference of the literature(Jing, 1995)

## 1.2 Phosphorus fractionation

The forms of P were determined by extracting P according to the scheme presented in Fig.1. Silt/clay fraction (< 63 μm) of sediments was subjected to sequential chemical extraction with 1.0 mol/L $NH_4Cl$, 0.11 mol/L $NaHCO_3$/$Na_2S_2O_4$, 0.1 mol/L NaOH, 0.5 mol/L HCl. Extractions were performed in triplicate. After centrifugation, the supernatants were filtered through a 0.45 μm phosphorus-free membrane or polyamide filter for the NaOH extracts. The soluble reactive phosphorus in each extraction was determined by molybdenum blue method as modified by Murphy and Riley(Murphy, 1962). The fractionation scheme yields five chemically defined fractions of phosphorus (Fig.1). In the order of extraction, they are: (1) loosely sorbed P($NH_4$Cl-P); (2) reductant soluble reactive P(BD-P); (3) reactive P sorbe to metal oxides(NaOH-P); (4) calcium bound P(HCl-P); (5) non-reactive organic P(Org-P).

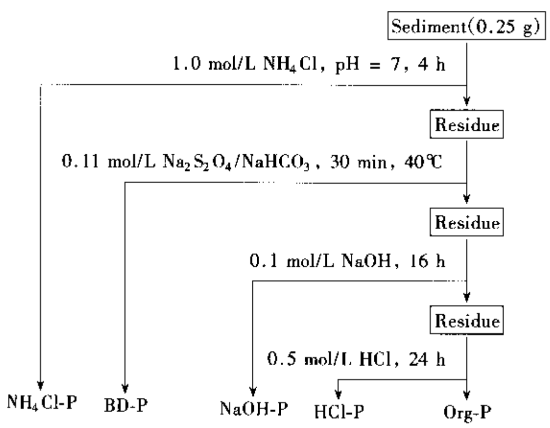


Fig.1 Phosphorus fractionation scheme

In addition, total phosphorus(TP) was determined using the molybdenum-blue complex method(Murphy, 1962) after total destruction with $HClO_4$/$H_2SO_4$ at 370℃. The sum of $NH_4$Cl-P, BD-P, NaOH-P, and HCl-P was taken as sedimentary inorganic phosphorus(IP).

# 2 Results and discussion

## 2.1 Sediment characteristics and elemental content

Table 2 presents the chemical component concentrations measured in Taihu Lake sediments. Organic matter, expressed as loss on ignition (LOI), exhibits concentrations ranging from 4.51% dw in T6 to 10.20% dw in T1. Samples of T1—T3 from northern Taihu Lake, in more serious eutrophic situation, have relative larger LOI values than the samples from the rest sites (T4—T6). Chemically digestible organic matter, represented by TOC, exhibits lower concentrations than LOI ranging from 0.66% dw in T4 to 1.97% dw in T1.

Total metal concentration of Fe, Al, Ca and Mn were also listed in Table 2. Among them, total Fe and Al concentrations ranging from 12.96 mg/g dw in T6 to 38.57 mg/g dw in T1, and from 17.85 mg/g dw in T6 to 38.62 mg/g dw in T1, respectively, are much higher than that of total Ca(from 2.10 mg/g dw in T6 to 5.95 mg/g dw in T1) and Mn(from 0.22 mg/g dw in T6 to 1.81 mg/g dw in T3) in the six sediment samples collected. The metal Fe and Al content has been considered to be the main factors that determine phosphorus adsorption capacity on sediments, because of the high specific surface of the iron/aluminium (hydr) oxides(Lijklema, 1980; Lucotte, 1988; Brinkman, 1993). Therefore, further analyses of active Fe and Al were carried out(Table 2). It is obvious from Table 2 that T1 has the absolutely highest content of active Fe and Al and T6 has relative lower active Fe and Al content than that in others. The percentage of active Fe in total Fe ranges from 25.5% in T4 (collected from the central area of the lake and with relative lower trophic state) to 78.5% in T1 (collected from the area in hypereutrophic state), and averages 43.7% for all samples. Similarly, active Al accounts for 3.7% in T4 to 23.3% in T1, 8.3% on average, of total Al. The respective ratios of active Fe and active Al in their total contents are probably related to organic matter, trophic state, and water chemistry in sampling point. Because active Fe and Al are the main factors to retain P dissolved in solution or mineralized from organic matter (e.g. algal debris), the sediment of T1 has the best ability to trap dissociative P and at the same time establishes the largest pool of available P that can be released when environmental conditions permit. Therefore this mechanism of temporary retention rather than permanent burying of P will certainly go against the restoration of eutrophication.

## 2.2 Fractional composition of phosphorus in sediments

The concentrations of different phosphorus fractions determined in the sediments from Taihu Lake system are showed in Fig.2a. The relative contribution of each fraction to the total sedimentary inorganic phosphorus(i.e. the sum of the four extracted phosphorus fractions ) is presented in

**Table 2 Sediment elemental contents**

| Sites | Metals total contents, mg/g dw | | | | Active Fe and Al, mg/g dw | | | | LOI, % dw | TOC, % dw |
|---|---|---|---|---|---|---|---|---|---|---|
| | $Fe_T$ | $Al_T$ | $Ca_T$ | $Mn_T$ | $Fe_{act}$ | $Al_{act}$ | $Fe_{act}/Fe_T$ | $Al_{act}/Al_T$ | | |
| T1 | 38.57 | 38.62 | 5.95 | 0.46 | 30.28 | 8.98 | 0.785 | 0.233 | 10.20 | 1.97 |
| T2 | 30.39 | 37.69 | 3.24 | 0.58 | 16.74 | 3.49 | 0.552 | 0.093 | 6.95 | 1.43 |
| T3 | 32.86 | 42.54 | 3.56 | 1.81 | 11.60 | 2.14 | 0.353 | 0.050 | 7.84 | 1.72 |
| T4 | 21.61 | 36.84 | 3.26 | 0.45 | 5.50 | 1.38 | 0.255 | 0.037 | 4.58 | 0.66 |
| T5 | 25.33 | 38.79 | 2.40 | 0.34 | 8.23 | 1.55 | 0.325 | 0.040 | 5.08 | 0.73 |
| T6 | 12.96 | 17.85 | 2.10 | 0.22 | 4.61 | 0.81 | 0.356 | 0.045 | 4.51 | 1.22 |

Fig.2b. The mean concentrations of P-fractions for the whole lake sediment were calculated from concentrations of P-fractions in six sediment samples. The rank order according to the mean concentration of P-fractions in Taihu Lake was NaOH-P > BD-P > HCl-P > $NH_4Cl$-P. A separate discussion of each P-fraction is given below.

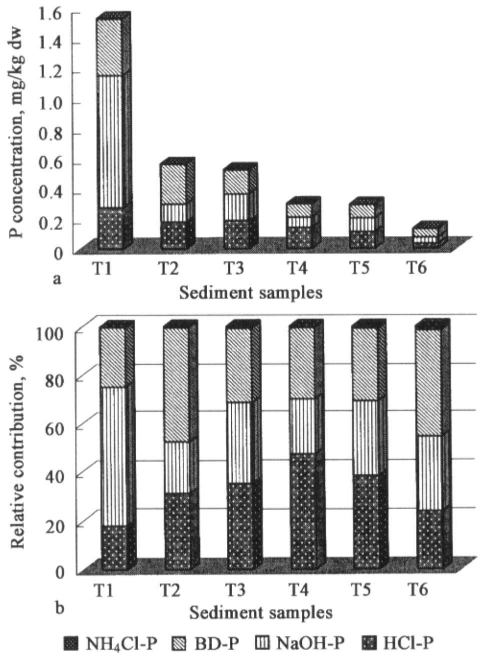


Fig.2 a. Phosphorus fractionation in fine(silt/clay) sized lake sediments; b. relative contribution of each P-fractions to the sedimentary inorganic phosphorus

### 2.2.1 Loosely sorbed phosphorus($NH_4Cl$-P)

This fraction represents the loosely sorbed P that comprises porewater P, P released from $CaCO_3$-associated P in hardwater lakes or leached P from decaying cells of bacterial biomass in deposited phytodetrital aggregates (Rydin, 2000; Pettersson, 2001). The $NH_4Cl$-P is a seasonally variable pool of phosphorus compounds dissolved in the interstitial water.

In Taihu Lake the concentrations of $NH_4Cl$-P in the six sediments range from 1.74 μg/g dw to 4.53 μg/g dw, and average 3.07 μg/g dw for all sediment samples. The highest $NH_4Cl$-P concentration is exhibited in sediment T1 where the highest sedimentary inorganic P concentration is also found (1.53 mg/g dw). This fraction of P accounts for < 1% of the sedimentary inorganic P in Taihu Lake system(Fig.2b).

### 2.2.2 Reductant soluble phosphorus(BD-P)

This fraction of P was extracted by use of a bicarbonate buffered solution of sodium-dithionite (BD-reagent) and is sensitive to low redox potential. The BD-reagent reduces the oxidized species of iron and manganese and thereby liberates the phosphorus adsorbed onto (hydro) oxides of the two metals. It is found that iron bound P measured by BD-extraction provides the best estimate of the internal P loading in stratified lakes with anoxic bottom water when compared to iron bound phosphorus measured by NaOH extraction (Nürnberg, 1988). BD-P represents the redox-sensitive P forms that is considered as potentially mobile pool of P and is algal available.

The BD-P concentrations in Taihu Lake exhibit high variability ranging from 0.058 mg/g dw in T6 to 0.37 mg/g dw in T1. The BD-P concentration is almost six times higher in sediment T1 from northern lake (Wuli sublake) than in sediment T6 from eastern lake. This difference might be due mainly to the trophic status of the both lake zones and sediment composition (Gonsiorczyk, 1998). The relative contributions of BD-P to the sedimentary inorganic P range from 24% in T1 to 44% in T6, and average 34% (Fig.2b). The content of reductant P in sediments has been related to DO depletion occurring in sediments of hypertrophic lakes, and/or to high pH values or bacterial activity that might enhance P-release from this fraction (Perkins, 2001; Kaiserli, 2002).

Linear regression analysis indicated that active Fe and BD-P, observed in six sediment samples from Taihu Lake, are significantly correlated by the equation showed in Fig.3a ($R^2$ = 0.96). This relationship demonstrates that active Fe extracted by oxalate-oxalic acid plays a significant role in P sorption and retention on natural sediments, and hence the element is a major variable in the eutrophication process.

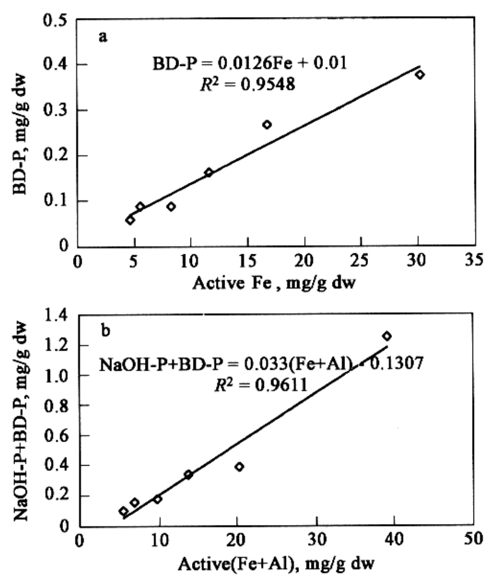


Fig.3 a. Linear regression analysis for BD-P and active Fe; b. linear regression analysis for (NaOH-P + BD-P) and active (Fe + Al)

#### 2.2.3 Metal oxide bound phosphorus(NaOH-P)

This fraction of sedimentary P refers to P bound to metal (hydr) oxides, mainly of Fe and Al, which is exchangeable again with $OH^-$, anion of organic ligands and inorganic P compounds soluble in bases(Stone, 1993; Rydin, 2000). NaOH extractable phosphorus could be released for the growth of phytoplankton when anoxic conditions prevail at the sediment-water interface (Ting, 1996). The NaOH-P concentrations in the six sediment samples range from 0.04 mg/g dw in T6 to 0.88 mg/g dw in T1, and average 0.23 mg/g dw. The highest active Fe and Al are also found in sediment T1. The relative contribution of NaOH-P to sedimentary inorganic P is between 21% (in T2) and 57% (in T1), and averages 33% for all six samples(Fig.2b).

Active Fe and Al have been believed to be the main sorbents for adsorbing P in natural sediments (Heleen, 1993). BD-P and NaOH-P are mainly binding on Fe and Al (hydr) oxides, especially on their amorphous and active forms. The linear relationship between the sum of BD-P and NaOH-P and the sum of active Fe and active Al content is indicated in Fig.3b($R^2 = 0.96$).

#### 2.2.4 Calcium bound phosphorus(HCl-P)

The HCl-P refers to P forms sensitive to low pH, assumed to consist mainly of apatite P, which is natural and detrital, P bound to carbonates and traces of hydrolysable organic P. Calcium bound P is a relatively stable fraction of sedimentary P and contributes to a permanent burial P in sediments (Kaiserli, 2002). The concentrations of this fraction of P in Taihu Lake sediment range from 0.03 mg/g dw in site T6 to 0.27 mg/g dw in T1, and account for from 18% in T1 to 47% in T4 of sedimentary inorganic P(Fig. 2b).

### 2.3 Bio-available phosphorus(BAP) in the sediments

Bio-available P has been defined as the sum of immediately available P and the P that can be transformed into an available form by naturally occurring processes. Previous studies have used sequential chemical extraction methods to estimate the bio-availability of sedimentary phosphorus(Boström, 1988). The BAP has been estimated by the sum of $NH_4Cl$-P, BD-P and NaOH-P and is referred to as non-apatite inorganic P(Stone, 1993). Among them, NaOH-P is in most studies the most algal-available P fraction. Also, de Jonge *et al*. (de Jonge, 1993) assumed that the bio-available fraction of phosphorus usually consists of the adsorbed and metal-associated P fractions, but a part of the organic phosphorus or the calcium associated fraction may be available to micro-organisms, although organic phosphorus does not play an important role in fuelling local primary production, at least not in the short term.

In present study, the estimate scheme proposed by Stone and English(Stone, 1993) is adopted to reckon BAP content in Taihu Lake, viz. BAP = $NH_4Cl$-P + NaOH-P + BD-P. As indicated in Fig.4, the BAP contents in Taihu sediments are ranging from 0.10 mg/g dw in T6 to 1.25 mg/g dw in T1, and average 0.40 mg/g dw for all samples. Potentially the bio-available phosphorus can contribute substantially to the local primary production when this fraction reaches the water column during one growing season. This can be resulted from resuspension or due to bioturbation. Sediment T1 has the highest BAP content and meanwhile this area has the most serious trophic status observed among the six sampling sites (Table 1). Fig.5 shows the relative contributions of BAP to total sedimentary phosphorus(TP) and inorganic sedimentary phosphorus(IP) and relative contributions of bio-unavailable phosphorus(BUAP) to TP and IP. In Taihu Lake, from 19% to 51%, on average 34%, of TP is bio-available(Fig.5a). Moreover, the BAP account for 53% to 82%, on average 68%, of IP in Taihu Lake sediments(Fig.5b). Namely, more than half of inorganic sedimentary P is conditionally available in Taihu Lake.

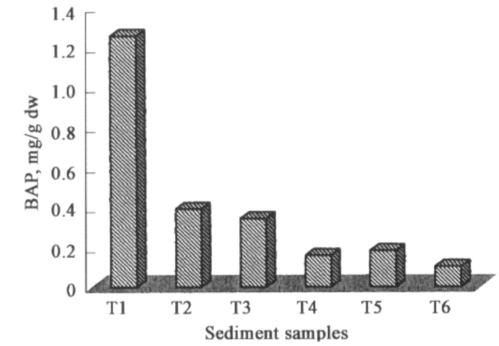


Fig.4 The bio-available phosphorus (BAP) contents in Taihu sediments

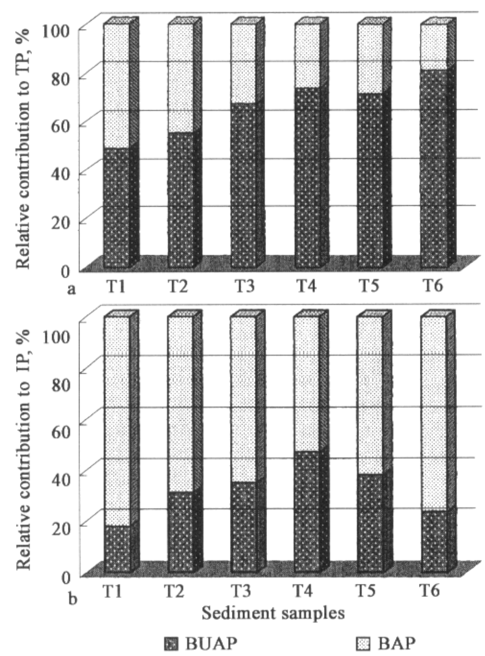


Fig.5 a. Relative contributions of BAP and BUAP to the total sedimentary phosphorus (TP); b. relative contributions of BAP and BUAP to the sedimentary inorganic phosphorus (IP)

The estimate of BAP may differ due to different extraction methods and definitions of BAP. Immediately available phosphorus in sediments has been considered as the best estimate of BAP(Hesse, 1971). However, Dorich *et al*. (Dorich, 1984) found that 0.1 mol/L NaOH extractable phosphorus was significantly correlated with 2 d and 14 d available phosphorus for an alga. They then suggested that NaOH-P could be used to estimate both short-term and long-term available phosphorus in sediments. Potentially available phosphorus forms, BD-P and NaOH-P for examples, can easily be available when conditions in water system permit and therefore should be counted up for BAP evaluation in natural sediments.

## 3 Conclusions

Sedimentary inorganic phosphorus in present study consists of four fractions (NaOH-P, BD-P, HCl-P, and $NH_4Cl$-P). Inorganic phosphorus was the main content of phosphorus in Taihu Lake sediment as observed in the six sediments. The linear relationship between active Fe and BD-P indicates that active Fe extracted by oxalate-oxalic acid may play a major role in P sorption and retention on natural sediments. Similar relationship between the sum of BD-P and NaOH-P and the sum of active Fe and active Al content is also observed, which holds the assumption that active Fe and Al are the main sorbents for adsorbing P in natural sediments, BD-P and NaOH-P are mainly binding on Fe and Al(hydr) oxides, especially on their amorphous and active forms. BAP are the sum of immediately available P and potentially available P. In Taihu Lake, BAP content ranges from 0.10 mg/g dw to 1.25 mg/g dw, and averages 0.40 mg/g dw for all the six samples. More than half of sedimentary inorganic phosphorus (mean 68%) were bio-available and can be conditionally released into water column.

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