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Serial parameter: CN 11-2629/X*1989*m*211*en*P*25*2013-4



Speciation of organic phosphorus in a sediment profile of Lake Taihu I: Chemical forms and their transformation

Di Xu¹, Shiming Ding^{1,*}, Bin Li^{1,2}, Xiuling Bai³,
Chengxin Fan¹, Chaosheng Zhang⁴

1. State Key Laboratory of Lake Science and Environment, Nanjing Institute of Geography and Limnology,
Chinese Academy of Sciences, Nanjing 210008, China. E-mail: dxu@niglas.ac.cn

2. College of Forest Resources and Environment, Nanjing Forestry University, Nanjing 210037, China

3. College of Environment and Planning, Henan University, Kaifeng 475000, China

4. GIS Centre, Ryan Institute and School of Geography and Archaeology, National University of Ireland, Galway, Ireland

Received 13 August 2012; revised 28 September 2012; accepted 17 October 2012

Abstract

Organic phosphorus (nonreactive P, NRP) is a major component of P in sediments, but information about its chemical forms and dynamic transformation is limited. The chemical forms and dynamic behaviors of NRP in a sediment profile from Lake Taihu, a freshwater and eutrophic lake in China, were investigated. Five forms of NRP in the sediments were extracted based on a chemical fractionation technique, including easily labile NRP (NaHCO_3 -NRP), reactive metal oxide-bound NRP (HCl-NRP), humic acid-associated NRP ($\text{NaOH-NRP}_{\text{HA}}$), fulvic acid-associated NRP ($\text{NaOH-NRP}_{\text{FA}}$) and residual NRP (Res-TP). There were notable transformations with increasing sediment depth from the labile NaHCO_3 -NRP and NaOH-NRP pools to the recalcitrant HCl-NRP and Res-TP pools, which caused the NRP to become increasingly recalcitrant as the early diagenetic processes proceeded. Further analyses showed that the relative changes in contents of organic matter and reactive Fe oxides in the sediment profile triggered a competition for binding NRP fractions and led to the transformation of NRP. The results highlighted the importance of abiotic processes in regulating the diagenesis of organic P and its stability in sediments.

Key words: organic phosphorus; fractionation; transformation; Lake Taihu; sediment

DOI: 10.1016/S1001-0742(12)60136-3

Introduction

As a major nutrient in aquatic biology, phosphorus (P) has been recognized as the most critical nutrient limiting primary productivity and regulating lake trophic status. Increased concentration of P in the water column, initially from increased external inputs of P, has been the primary factor responsible for accelerated eutrophication (Schelske, 2009). Sediment is usually a major sink for P in lakes, and contains a very large P pool in comparison to that in the water column. Accordingly, sediment plays an important role in the P metabolism in lakes, especially when external inputs are reduced (Søndergaard et al., 2007).

Phosphorus occurs naturally in sediments in both inorganic and organic forms. Compared to inorganic P, which has been studied intensively for a long time, organic P in sediments has received much less attention. Organic

P originates from both allochthonous and autochthonous sources, and may constitute a major proportion of the P settling into the sediment (Reitzel et al., 2007). Previous studies revealed that degraded organic P could be an important source for water column P (Søndergaard et al., 1996; Rydin, 2000; Kaiserli et al., 2002), and could maintain a low magnitude, long-term release of P for most of the year (Spears et al., 2007). The contribution of organic P in a eutrophic lake was found to be greater than that in an oligotrophic lake (Gonsiorczyk et al., 1998). On the other hand, a considerable amount of organic P compounds has been found in deep sediments, despite some of them being easily degraded at the molecular level (Ahlgren et al., 2005; Reitzel et al., 2007). A dynamic mechanism may be involved in the stabilization of organic P in sediments during the early diagenetic process. However, little is known about this mechanism.

Organic P cannot be directly detected by the molybdenum blue method and is traditionally treated as nonreactive

* Corresponding author. E-mail: smding@niglas.ac.cn

P (NRP) in sediments. In sequential extraction schemes concerning primarily inorganic P, organic P in sediments is usually treated as a residue or refractory fraction (Ruttenberg, 1992; Ruban et al., 1999), or its labile fraction is pre-recovered in alkaline solutions (e.g., 0.1 mol/L NaOH) (Lukkari et al., 2007). In addition to simple differentiation and quantification of organic P, conventional characterization of organic P forms is accomplished through chemical fractionation techniques initially developed in soil sciences. In these techniques, organic P in soils is separated into operationally defined fractions based on presumed chemical stability using an acid-alkali sequence of extractions (Bowman and Cole, 1978; Sharpley and Smith, 1985; Ivanoff et al., 1998). Fractionation of organic P in sediments has also been attempted through acid-alkali or chelating compound sequential extractions (De Goot, 1990; Oluyedun et al., 1991; Golterman, 1996). However, systemic applications have been scarce (Zhang et al., 2008; Zhou et al., 2008).

The purpose of this study was to investigate the speciation of organic P and its transformation in a sediment profile of Lake Taihu, a large, shallow, eutrophic lake in China. The dynamic transformation of organic P was analyzed by assessing the relationships among those forms. The possible mechanisms involved in the transformation were discussed.

1 Materials and methods

1.1 Description of the site studied

The rapid expansion of industry and agriculture around Lake Taihu has brought large quantities of nutrients to this lake since the 1980s. The concentrations of nutrients have dramatically increased in the water column and sediments. This phenomenon was particularly obvious in Meiliang Bay, which is one of the most eutrophied regions in this lake, largely due to sewage discharges from the Liangxi, Chihugang and Wujingang Rivers. Total nitrogen in the water column of this bay has increased from 1.8 mg/L in 1991 to 4.1 mg/L in 2006, and total P has increased from 0.07 mg/L in 1991 to 0.26 mg/L in 2006 (Zhu, 2008). Meanwhile, P content in the surface sediments was detected at levels as high as 1800 $\mu\text{g/g}$, which was more than three times that in the center of the lake (Bai et al., 2009). The enrichment of nutrients in this bay has caused the onset of a eutrophication phase and associated degradation of its ecological status since the mid-1980s (Chen et al., 2003).

The sampling site was located in the northern part of Meiliang Bay (120.19°E and 31.51°N), which is close to the outlet of the River Liangxi (with a distance of approximately 4 km) (Fig. 1). The depth of the soft sediment which is covered by a 2.2-m water layer, is more than 50 cm. According to monthly monitoring data from the Taihu Laboratory for Lake Ecosystem Research, average

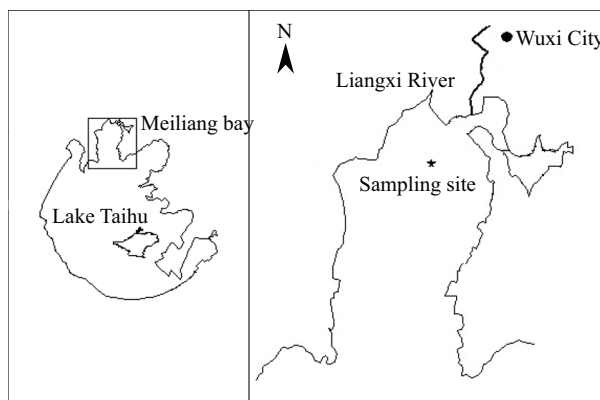


Fig. 1 Location of the sampling site in Meiliang Bay of Lake Taihu, China.

annual concentrations of dissolved O₂, total P, total N and chlorophyll *a* in the water column of this site were 8.95 mg/L, 0.16 mg/L, 5.43 mg/L and 25.1 $\mu\text{g/L}$, respectively, during 2001 and 2006. These parameters are representative of eutrophic and polluted regions in Meiliang Bay.

1.2 Sampling

Six sediment cores were collected using a gravity core sampler in November 2007. Each core was sliced into 0.5 cm sections down to 10 cm, and then into 2.5 cm sections down to 15 cm, under an N₂ atmosphere. The sediments from the six parallel cores were pooled at equal depths and homogenized to obtain a representative sample. The sediments were transported to the laboratory. Some were stored at 4°C, while the majority were lyophilized at -80°C, sieved to pass through a 100-mesh sieve and then stored at 4°C until analysis.

1.3 Sediment property analysis

Sediment pH was determined in a suspension of deionized water using a sediment/water ratio of 1:2.5 (*m/V*) (Lu, 1999). Sediment P, Fe, Al, Mn and Ca were measured using an inductively coupled plasma atomic emission spectroscopy (ICP-AES, Perkin-Elmer DV4300, USA) after fusing 0.05 g (dry weight) sediment with 0.2 g LiBO₂, followed by dissolution with 4% HNO₃ (Lu, 1999). Total NRP was determined by extracting P from the sediments after removal of reactive P (RP) using 1 mol/L HCl extraction, followed by combustion of the sediment at 450°C for 3 hr (Ruban et al., 1999). Concentrations of P in solutions were measured using the molybdenum blue method (Murphy and Riley, 1962). The content of organic matter in the sediment was measured by loss on ignition (LOI) through calcination of the sediment at 550°C for 6 hr. The errors (RSD%) for three duplicate analyses of these parameters were mostly less than 4%.

1.4 Fractionation of organic phosphorus

The organic P (NRP) in sediments was fractionated based on the scheme developed by Ivanoff et al. (1998)

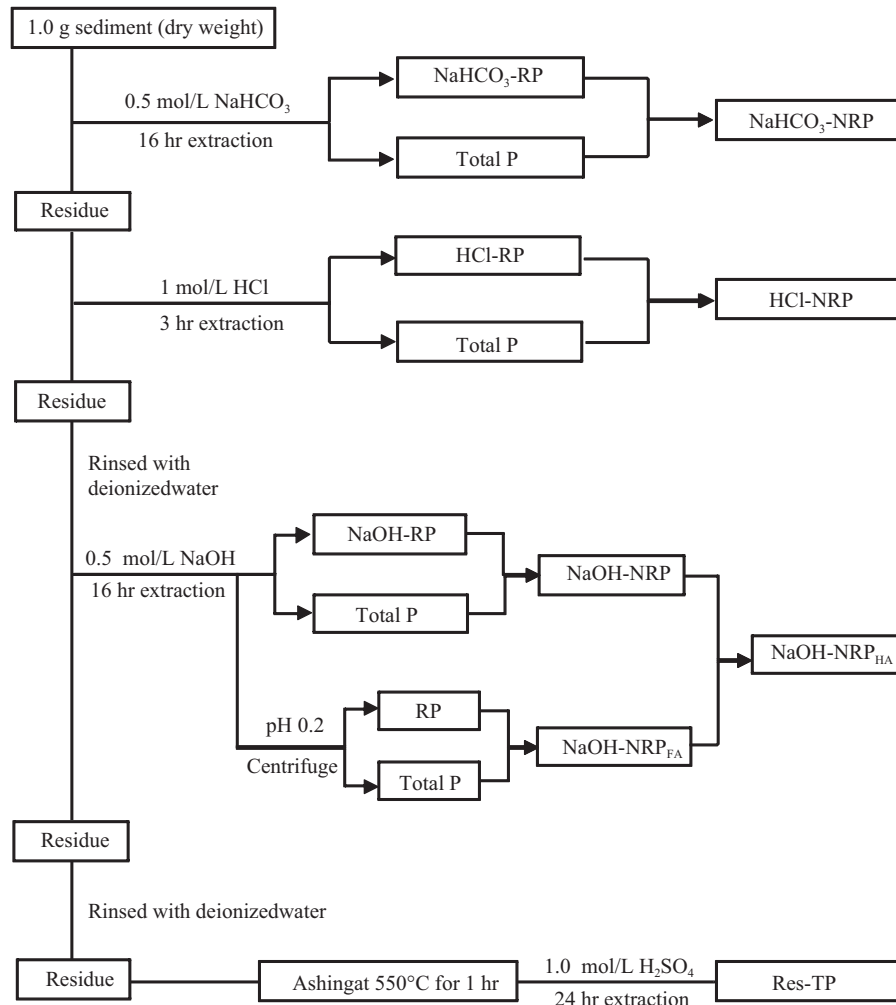


Fig. 2 Scheme used for fractionation of nonreactive P (NRP) in sediments according to Zhang et al. (2008), slightly modified from Ivanoff et al. (1998).

Table 1 Physical and chemical properties of the sediments investigated

Depth (cm)	pH	LOI (%)	Sediment (g/kg)				HCl extracted (g/kg)			
			Al	Fe	Mn	Ca	Al	Fe	Mn	Ca
0–0.5	7.44	5.71	36.9	35.1	0.95	3.34	3.30	11.4	1.08	3.24
0.5–1	7.48	6.47	44.0	36.2	1.01	3.68	3.38	11.7	1.08	3.13
1–1.5	7.56	5.80	45.7	37.0	1.01	3.65	3.37	11.7	1.03	3.23
1.5–2	7.39	5.65	45.0	36.9	0.97	3.91	3.42	11.9	1.01	3.29
2–2.5	7.54	5.75	36.4	37.1	0.92	2.83	3.36	12.0	0.98	3.41
2.5–3	7.46	6.28	37.6	36.2	0.88	3.40	3.38	12.2	0.98	3.60
3–3.5	7.37	5.44	44.1	37.3	0.94	4.04	3.34	11.8	0.93	3.55
3.5–4	7.45	5.47	38.7	34.6	0.84	3.27	3.35	11.5	0.87	3.47
4–4.5	7.39	5.38	35.6	34.0	0.78	3.08	3.36	11.7	0.86	3.40
4.5–5	7.49	5.74	54.9	38.2	0.97	4.91	3.39	12.2	0.82	3.52
5–5.5	7.45	6.01	33.4	35.3	0.74	3.04	3.37	11.9	0.77	3.47
5.5–6	7.43	6.21	33.5	36.0	0.70	2.90	3.34	12.6	0.75	3.50
6–6.5	7.33	6.08	41.5	36.6	0.79	3.56	3.34	12.5	0.71	3.49
6.5–7	7.47	5.87	37.0	35.4	0.67	3.27	3.36	12.7	0.68	3.32
7–7.5	7.35	5.01	37.7	35.5	0.67	3.53	3.31	12.5	0.65	3.22
7.5–8	7.38	5.26	39.3	35.8	0.65	3.42	3.34	12.8	0.62	3.10
8–8.5	7.27	5.15	37.7	34.7	0.62	3.32	3.26	12.4	0.57	3.04
8.5–9	7.21	4.56	36.0	35.6	0.58	3.07	3.32	12.7	0.55	3.09
9–9.5	7.32	5.13	37.9	34.9	0.59	3.02	3.36	12.6	0.54	2.93
9.5–10	7.23	4.13	51.5	36.9	0.67	4.33	3.3	12.8	0.51	2.90
10–12.5	7.19	4.60	31.2	35.6	0.50	2.48	3.31	12.5	0.44	2.57
12.5–15	7.21	3.98	36.8	32.9	0.46	3.21	3.28	12.2	0.40	2.45

with by a slight modification by Zhang et al. (2008). The fractionation scheme is presented in **Fig. 2**. The forms of NRP included 0.5 mol/L NaHCO₃-extracted NRP (NaHCO₃-NRP), 1 mol/L HCl-extracted NRP (HCl-NRP), 0.5 mol/L NaOH-extracted NRP (NaOH-NRP) and residual P (Res-TP), and the forms of inorganic P (RP) were correspondingly defined as NaHCO₃-RP, HCl-RP and NaOH-RP. An aliquot of the NaOH extract was further acidified with concentrated HCl to pH 0.2 and centrifuged at 10,000 r/min. The supernatant was analyzed to determine NaOH-NRP_{FA} (FA refers to fulvic acid), whereas the NaOH-NRP_{HA} (HA refers to humic acid) was estimated by subtracting NaOH-NRP_{FA} from NaOH-NRP. The concentration of the NRP in each extract was determined by subtracting RP from total P, while concentrations of total P were measured using the molybdenum blue method after acid persulfate digestion (Koroleff, 1983). Concentrations of Fe, Al, Ca and Mn in the HCl extracts were simultaneously determined by ICP-AES.

Definitions of organic P forms in the fractionation scheme were mainly based on their binding phases in sediments. The NaHCO₃-NRP, HCl-NRP, NaOH-NRP_{HA} and NaOH-NRP_{FA} were attributed to easily labile NRP, reactive metal oxide-bound NRP, humic acid-associated NRP, and fulvic acid-associated NRP, respectively.

Multiple analyses ($n = 6$) of a sediment sample from the sediment profile selected using the above fractionation technique showed that the analytical errors (RSD%) were mostly within 12% for total P and RP and 25% for NRP during the various steps.

1.5 Statistics

The relationships between organic P forms were investigated using the Pearson correlation coefficient (two-tailed). The statistical analyses were performed using SPSS ver. 11.5.

2 Results and discussion

2.1 Sediment properties

The chemical properties of the sediments varied with sediment depth (**Table 1**). Sediment pH remained near 7.4 in the upper 8 cm, but below this depth it decreased slightly to 7.2. Sediment LOI was above 5.3% and showed no notable changes with depth in the upper 6 cm layers, but below this depth it decreased to 4.0%. Sediment Al, Fe and Ca showed no clear trends with depth, but sediment Mn decreased by 50% from the uppermost layer to the deepest layer. The variations in sediment properties with depth were generally in line with other investigations conducted in Lake Taihu (Zhao et al., 2007; Yin et al., 2008) and in Lake Dongjiu near an estuary of Lake Taihu (Wu et al., 2007).

Extraction with 1 mol/L HCl recovered total Mn 96% ± 8% and most of the Ca 93% ± 12%, but only minor

amounts of Fe 34% ± 2% and Al 9% ± 1%. Fe was dominant in the extracts, accounting for 62% ± 2% of the total metals extracted. It had an overall increase with sediment depth in the deeper 10 cm layer. The concentration of Al extracted remained stable over the whole profile. Extracted Mn followed the same pattern as sediment Mn. The concentration of extracted Ca remained stable in the upper 6.5 cm sediment layers, and below this it showed an evident decrease. These extracted metals were considered active in sediments (Burton et al., 2005). The dominance of reactive Fe implied that it may play an important role in P speciation in sediments.

2.2 Total P and organic P in the sediments

The concentration of total P remained relatively high and stable within the upper 2.0 cm of the sediments (RSD = 3%), and below this it decreased (**Table 2**). The concentration of NRP generally followed the same pattern, and the change occurred at a depth of approximately 2.5 cm (RSD = 6% within the upper 2.5 cm). The vertical distributions of total P and NRP in the sediments were similar to those observed in previous investigations in northern Lake Taihu (including Meiliang Bay), which typically exhibited higher concentrations in the upper layers followed by an exponential decrease with increasing depth (Kelderman et al., 2005; Trolle et al., 2009).

Both Kelderman et al. (2005) and Trolle et al. (2009) suggested that the variability of sediment P in Lake Taihu was driven by the differences in external nutrient loading. This was well demonstrated by the horizontal variability of P in the surface sediment, which showed a decreasing trend from the northern region to the center of Lake Taihu, with a decrease in concentration of total P in the water column (Trolle et al., 2009). The high and constant concentrations of total P and NRP in the upper sediment layers observed in this study was thus likely caused by a high and relatively stable external nutrient loading during sedimentation. However, we could not exclude the possibility of P migration from deeper sediment layers, because the enrichment of total P in the surface sediments could occur in the absence of external influences (Søndergaard et al., 1996). A further investigation showed that the concentrations of total P and NRP in the deeper sediment layers (below 5 cm) had significantly positive correlations with sediment LOI ($r = 0.56$, $p = 0.04$ and $r = 0.65$, $p = 0.02$, respectively), suggesting that the decrease in total P and NRP in the deeper sediments likely originated from a decrease in sediment organic matter. This decrease was possibly caused by degradation or decreased external input.

2.3 Chemical forms of organic P

Fractionation of sediment NRP using the sequential extraction technique yielded a recovery of 96% ± 4% for the whole profile, suggesting that the fractionation had

Table 2 Concentrations of total P, NRP and the fractionated RP and NRP forms in the sediments

Depth (cm)	Total P	Total NRP	NaHCO ₃ -NRP (mg/kg)	HCl-NRP (mg/kg)	NaOH-NRP _{HA} (mg/kg)	NaOH-NRP _{FA} (mg/kg)	Res-TP (mg/kg)	Recovery	NaHCO ₃ -RP (mg/kg)	HCl-RP (mg/kg)	NaOH-RP (mg/kg)
0–0.5	1036	382.2	34.22	139.2	72.41	63.94	56.17	95.8%	94.67	372.3	187.6
0.5–1	1004	349.7	34.24	116.5	52.09	83.99	59.27	99.0%	90.60	394.4	176.3
1–1.5	1051	382.6	35.16	130.4	34.03	81.37	81.66	94.8%	96.24	392.7	200.3
1.5–2	1085	377.1	26.01	130.0	36.45	79.78	72.83	91.5%	105.3	387.6	200.0
2–2.5	1061	426.8	30.72	161.4	36.10	86.85	79.09	92.3%	99.21	366.7	196.3
2.5–3	1008	349.9	18.92	138.1	24.02	93.57	69.95	98.5%	99.38	372.7	186.9
3–3.5	994	326.9	15.37	133.2	24.57	86.31	74.39	102.1%	81.28	399.7	177.9
3.5–4	946	344.7	14.61	134.6	30.69	81.35	75.27	97.4%	74.58	391.2	169.3
4–4.5	950	360.2	20.13	152.1	41.01	70.74	66.24	97.2%	72.70	357.3	191.1
4.5–5	968	361.2	11.73	147.1	58.89	56.13	71.94	95.7%	58.61	326.6	185.4
5–5.5	892	355.1	14.74	153.1	70.10	42.26	64.84	97.2%	64.12	326.6	189.0
5.5–6	927	345.9	13.20	151.2	56.17	45.85	61.63	94.8%	55.71	332.2	171.5
6–6.5	844	328.9	10.75	128.7	58.29	58.19	65.98	97.8%	55.62	340.0	175.3
6.5–7	853	302.9	10.87	110.1	71.06	5.16	68.69	98.0%	54.95	358.0	163.7
7–7.5	865	346.6	15.03	159.1	47.40	49.82	63.79	96.7%	51.26	281.6	170.5
7.5–8	850	326.3	5.63	150.3	44.93	50.56	65.27	97.0%	51.84	288.7	177.8
8–8.5	814	310.2	5.79	119.6	49.02	46.24	60.17	90.5%	54.38	310.8	179.2
8.5–9	835	296.3	8.68	120.6	49.93	39.64	63.67	95.4%	49.31	304.2	160.0
9–9.5	802	300.3	11.64	113.1	53.12	50.65	76.53	101.6%	44.89	280.6	162.7
9.5–10	817	305.1	1.28	119.4	38.77	52.26	63.34	90.1%	49.45	288.5	161.5
10–12.5	772	283.6	5.37	146.7	22.12	44.01	67.86	100.9%	41.86	278.3	161.7
12.5–15	792	311.2	1.22	156.3	39.39	36.39	65.59	96.1%	42.80	251.4	161.0
Average	917	339.7	15.7	136.9	45.93	60.68	67.92	96.4%	67.70	336.5	177.5

satisfactory recovery (**Table 2**). Concentrations of the forms of NRP were ranked in the order of HCl-NRP > Res-TP > NaOH-NRP_{FA} > NaOH-NRP_{HA} > NaHCO₃-NRP with average values of 136.9, 67.92, 60.68, 45.93 and 15.70 mg/kg, respectively. Concentrations of the forms of RP were ranked in the order of HCl-RP > NaOH-RP > NaHCO₃-RP with average values of 336.5, 177.5 and 67.70 mg/kg, respectively.

The NaHCO₃-NRP is the part of NRP easily released from metal compounds or organic materials, while Res-TP is composed of highly stable organic materials such as lignin and organometallic complexes (Ivanoff et al., 1998). The HCl-NRP was associated originally with Ca, Fe and Al mineral phases in the sediments, because sediment Ca, Fe and Al were recovered at this extraction step (**Table 1**). It has been found that the surfaces of Fe and Al oxides and clays have a high affinity for adsorption of organic P compounds (Leytem et al., 2002; Berg and Joern, 2006), whereas Ca forms precipitates with inositol hexakisphosphate, a major component of orthophosphate monoesters in natural environments (Celi et al., 2000). A positive correlation was found between LOI and NaOH-NRP in the sediments ($r = 0.89$, $p < 0.001$), demonstrating that both NaOH-NRP_{FA} and NaOH-NRP_{HA} were associated with organic matter in the sediments. Organic matter has been considered to be a major carrier of organic P in soils or sediments, which can retain a range of organic P compounds through physical or chemical incorporation, direct surface adsorption or indirect adsorption via cation bridges (Celi and Barberis, 2005).

The proportions of each form of NRP varied with sediment depth (**Fig. 3**). The proportion of NaHCO₃-NRP remained stable within the upper 1.5 cm of sediments, but it continually decreased below this depth and almost disappeared at the deepest layer. The proportion of HCl-

NRP showed an increasing trend with the increase of depth. The proportion of NaOH-NRP_{HA} showed a “U-shape” change with depth in the upper 5.5 cm of sediments. Below 5.5 cm, NaOH-NRP_{HA} exhibited a decreasing trend. The proportion of NaOH-NRP_{FA} showed an opposite trend to NaOH-NRP_{HA} in the upper 5.5 cm, below which its concentration was fairly uniform with depth. The proportion of Res-TP showed a strong increase with depth in the upper 1.5 cm of sediment, but then remained stable to a depth of 4 cm, below which it slightly increased.

The rapid decline of NaHCO₃-NRP below a depth of 1.5 cm indicated that this form of NRP was likely labile (**Fig. 3**). It has been found to be easily mineralized in soils and could contribute to plant-available P (Ivanoff et al., 1998). Both NaOH-NRP_{FA} and NaOH-NRP_{HA} had no evident change with sediment depth, but their combined concentration (i.e., NaOH-NRP) tended to decrease with sediment depth, reflecting their lability during early diagenetic processes. In contrast, the proportions of HCl-NRP and Res-TP showed overall increases with depth, indicating that the two forms were sinks of NRP during early diagenetic processes. In particular, the sharp increase in Res-TP in the upper 1.5 cm might reflect a rapid transformation of NRP from labile to recalcitrant forms.

2.4 Organic phosphorus transformation

The relationships of different forms of NRP were investigated, which may provide valuable information on their transformations. Taking into account the differences in vertical changes of NaOH-NRP_{HA} and NaOH-NRP_{FA} between the upper 5 cm and the layers below, correlations between each form of NRP were investigated separately in the two layers (**Table 3**). In the upper 5 cm, NaHCO₃-NRP was found to be negatively correlated with HCl-NRP and Res-TP, likely indicating transformation of NRP from

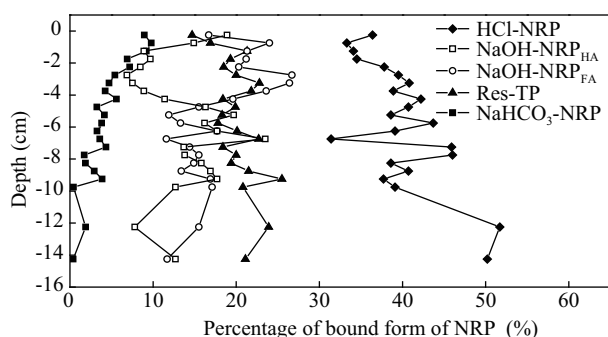


Fig. 3 Speciation of chemical forms of NRP in the sediment profile.

NaHCO₃-NRP to HCl-NRP and then Res-TP. Negative correlations were also found for NaOH-NRP_{HA} with both NaOH-NRP_{FA} and Res-TP. The sharp decrease in NaOH-NRP_{HA} in the upper 3 cm reflected a transformation of NRP from NaOH-NRP_{HA} to NaOH-NRP_{FA} and Res-TP. An opposite and rapid transformation should then occur from NaOH-NRP_{FA} to NaOH-NRP_{HA} between 3 and 5 cm, reflected by the sharp increase of NaOH-NRP_{HA} and decrease of NaOH-NRP_{FA} in this section (Fig. 3). In the layers below 5 cm, the only correlation found was a negative correlation between NaOH-NRP_{HA} and HCl-NRP, indicating a transformation from NaOH-NRP_{HA} to HCl-NRP.

For the whole sediment profile, the increase in recalcitrant HCl-NRP was attributed to the transformation from NaOH-NRP_{HA} (with 8% of the total organic P in sediments involved) and NaHCO₃-NRP (6%), and the increase in recalcitrant Res-TP was from NaOH-NRP_{HA} (5%) and NaHCO₃-NRP (2%). A consistent transformation of NRP thus occurred from the labile (i.e., NaHCO₃-NRP and NaOH-NRP_{HA}) to the recalcitrant forms (i.e., HCl-NRP and Res-TP). As a result, organic P in the sediments became increasing recalcitrant as the early diagenetic processes proceeded.

2.5 Dynamic mechanisms for organic P transformation

The dynamics of NRP in the environment are controlled by abiotic degradation and stabilization, enzymatic hydrolysis and microbial turnover, etc. Previous studies have revealed

that the binding phases can play a dominant role in regulating RP behaviors in sediments (Christophoridis and Fytianos, 2006). Similar situations may appear during early diagenesis of NRP in sediments. In this study, the ratio of HCl-extracted Fe to LOI showed a strong increase from 0.20 in the uppermost sediment layer to 0.30 in the deepest sediment layer (Fig. 4), likely implying a corresponding increase in the capacity of reactive Fe oxides to bind NRP fractions. A positive correlation was further found between the ratios of HCl-extracted Fe to LOI and HCl-NRP to NaOH-NRP (Fig. 4). This demonstrated that the relative changes in contents of reactive Fe oxides and organic matter had resulted in a competition for binding NRP and caused the observed transformation from NaOH-NRP to HCl-NRP.

The adsorption of NRP fractions onto reactive Fe oxides has been emphasized in the literature partly due to their abilities of controlling adsorption and mobility of phosphates in sediments (Mortimer, 1941; Böstrom

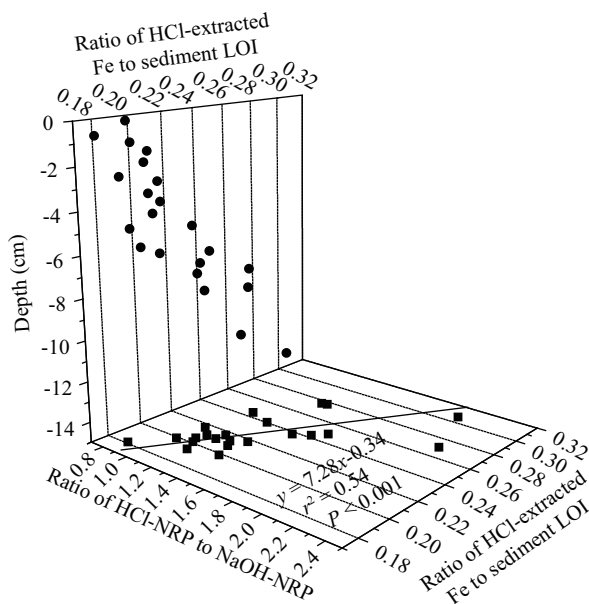


Fig. 4 Relationship of the ratio of HCl-extracted Fe to sediment LOI with sediment depth and the ratio of HCl-NRP to NaOH-NRP.

Table 3 Correlation matrix for chemical forms of NRP from the upper 5 cm and deeper 10 cm sediments

	NaHCO ₃ -NRP	HCl-NRP	NaOH-NRP _{HA}	NaOH-NRP _{FA}	Res-TP
Upper 5 cm					
NaHCO ₃ -NRP	1.000	-0.833**	0.266	-0.040	-0.581*
HCl-NRP		1.000	-0.166	-0.026	0.328
NaOH-NRP _{HA}			1.000	-0.723**	-0.739**
NaOH-NRP _{FA}				1.000	0.505
Res-TP					1.000
Deeper 5 cm					
NaHCO ₃ -NRP	1.000	-0.388	0.585*	-0.153	-0.144
HCl-NRP		1.000	-0.867**	0.006	-0.075
NaOH-NRP _{HA}			1.000	-0.348	-0.069
NaOH-NRP _{FA}				1.000	0.254
Res-TP					1.000

* and ** show the significance at $p < 0.05$ and $p < 0.01$ levels, respectively.

and Pettersson, 1982; Amirbahman et al., 2003). Several studies reported the adsorption of organic P compounds onto pure reactive Fe oxides (Celi et al., 2005; Ruttenberg and Sulak, 2011), but relevant studies in aquatic systems are limited (Huang and Zhang, 2010).

In this study, the sediments of Lake Taihu contained high levels of Fe (**Table 1**). Reactive Fe was not only the dominant reactive metal but also the only one exhibiting an increasing trend in concentration over a broad depth range (in the upper 10 cm). These features might have enabled reactive Fe oxides to play a vital role in stabilization of NRP in sediments. Although the adsorption by reactive Fe oxides may decrease in deep sediment layers due to their crystallization or reduction (Martynova, 2010; Ruttenberg and Sulak, 2011), they can still absorb more organic matter because of a rapid decrease in organic matter with depth. Furthermore, degradation of organic matter during early diagenesis processes may lead to the release of bound NRP fractions and facilitate the binding of NRP by reactive Fe oxides.

Other transformations of NRP, such as from NaHCO_3 -NRP to HCl-NRP and Res-TP, could be caused by similar dynamic processes related to changes in binding phases. However, this remains to be verified because the solid binding phases of NaHCO_3 -NRP and Res-TP are unknown. As the combined concentrations of NaOH-NRP and HCl-NRP accounted for more than 70% of the total NRP in the sediments (**Fig. 3**), the relative changes in the binding phases should be a major dynamic mechanism triggering the redistribution of NRP in sediments. This result highlighted the importance of physical-chemical adsorption or binding in regulating the diagenesis of NRP fractions.

3 Conclusions

Five forms of NRP in the sediments were extracted, including NaHCO_3 -NRP, HCl-NRP, $\text{NaOH-NRP}_{\text{HA}}$, $\text{NaOH-NRP}_{\text{FA}}$ and Res-TP. Concentrations of the forms of NRP were ranked in the order of HCl-NRP > Res-TP > $\text{NaOH-NRP}_{\text{FA}}$ > $\text{NaOH-NRP}_{\text{HA}}$ > NaHCO_3 -NRP, with average values of 135, 68, 61, 47 and 16 mg/kg, respectively. The declines in proportions of NaHCO_3 -NRP and NaOH-NRP with sediment depth reflected their lability. In contrast, the increases in the proportions of HCl-NRP and Res-TP with the increase of depth indicated that the two forms were sinks of NRP during the early diagenetic processes. A consistent transformation of NRP occurred from the labile NaHCO_3 -NRP and NaOH-NRP pools to the recalcitrant HCl-NRP and Res-TP pools. As a result, organic P in the sediments became increasingly recalcitrant as the early diagenetic processes proceeded. The relative changes in organic matter and reactive Fe oxides in the sediment profile triggered a competition for binding NRP fractions and causing the transformation of

organic P forms.

Acknowledgments

This work was supported by the National Natural Scientific Foundation of China (No. 40871220, 40730528), the Natural Scientific Foundation of Jiangsu Province, China (No. BK2010606) and the Nanjing Institute of Geography and Limnology, Chinese Academy of Sciences (No. NIGLAS2010KXJ01).

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Vol. 25 No. 4 2013

Supervised by	Chinese Academy of Sciences	Published by	Science Press, Beijing, China
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ISSN 1001-0742

