Pol. J. Environ. Stud. Vol. 23, No. 6 (2014), 1933-1940
DOI: *10.15244/pjoes/24924*

Original Research

Evaluating the Relationship between Phosphorus Bioavailability and Phosphorus Speciation in Sediments from Rural Rivers in the Taihu Lake Area, China

Luji Bo[1, 2], Dejian Wang[1]*, Gang Zhang[1], Can Wang[1]

[1]Institute of Soil Science, Chinese Academy of Sciences, 71st East Beijing Road, Nanjing, 210008, China
[2]University of Chinese Academy of Sciences, Beijing, 100049, China

Received: 22 January 2014
Accepted: 9 May 2014

Abstract

The concentrations of different phosphorus (P) species and four kinds of bioavailable P (algae-available P, AAP; Olsen P, OLP; water-soluble P, WSP; and readily desorbable P, RDP) were determined to investigate the relationship between phosphorus bioavailability and phosphorus speciation in sediments from rural rivers in Taihu Lake area, China, using a European Commission "Standards Measurements and Testing Program" protocol and four chemical extraction techniques. The total P and inorganic P concentrations in the sediments were 179-2,293 mg/kg and 137-1,857 mg/kg, respectively. The concentrations of the P species in the sediments followed the order NaOH- extractable P > HCl-extractable P > organic P. The bioavailable P concentrations followed the order AAP (340 mg/kg) > OLP (38.7 mg/kg) > WSP (1.8 mg/kg) > RDP (0.3 mg/kg). Regression analyses showed that there were good correlations between the P species concentrations and the bioavailable P concentrations measured using the four different methods, except for between WSP and the HCl-extractable P concentrations. The OLP concentrations had highly significant correlations with AAP, WSP, RDP and the P species concentrations. The OLP method was found to be most suitable for determining the bioavailability of P species under the prevalent geochemical conditions in the rural river sediments that were analyzed.

Keywords: phosphorus speciation, bioavailability, rural rivers, sediment, Taihu Lake area

Introduction

Phosphorus (P) is an essential element for plant growth. To meet our demand for food, we have moved large amounts of P from phosphate rock to fields to feed the Green Revolution, creating a unidirectional flow of P from rocks to farms to rivers, lakes and oceans, and dramatically impairing aquatic ecosystems [1-4].

Sediments are both sinks and sources of P because of the strong interactions between dissolved and sediment-bound PO_4^{3-} through adsorption-desorption and coprecipitation-dissolution processes [5, 6]. P can be released from sediments into the overlying water when the external loading decreases, which can increase the speed at which the trophic status of an aquatic ecosystem can change [7-9]. However, not all P species can be released by sediments into the overlying water [10-13].

In view of the different P species that may be present in sediments, methods that can comprehensively reflect the bioavailability of P species have been explored by many researchers. Four methods for extracting bioavailable P (algae-available P, AAP; Olsen P, OLP; water-soluble P,

*e-mail: djwang@issas.ac.cn

WSP; and readily desorbable P, RDP) have been proposed and have been widely used. For example, these four methods were used to evaluate P bioavailability in sediments in three lakes in China and the UK [14]. Branom et al. also used these methods to study P bioavailability in sediments in a lake used for sludge disposal [15]. However, there has been very little research so far on the relationships between P bioavailability and the different P species.

The Taihu Lake area is one of the most developed regions in China, with the highest population density, and it has a dense river network [16, 17]. Unfortunately, substantial amounts of untreated industrial, domestic, and agricultural wastewater containing high P have been discharged directly into rivers in the Taihu Lake area in recent decades because of overpopulation, rapid industrialization and urbanization, and point and diffuse pollution within the watershed [18]. P from these sources entered rivers and deteriorated water quality, ultimately depositing in the sediments and devastating aquatic ecosystems under certain conditions.

In the present study, we selected a typical rural river network in Yangyuan Town, which is a microcosm of the river network in the Taihu Lake area. Note that the selected river network is in economically developed area. However, growing industrial, domestic, and agricultural waste discharges, coupled with limited wastewater treatment capacity, have been the principal drivers of water pollution [19]. The aim of this study was to evaluate the relationship between phosphorus bioavailability and phosphorus speciation and identify an extraction method that was suitable for determining the phosphorus status in the sediments by determining the P species and four kinds of bioavailable P in the study area. The conclusions of this study will provide comprehensive insights into the current phosphorus status in sediments in rivers in Taihu Lake area.

Materials and Methods

Study Area and Sample Collection

The river network studied is in Yangyuan Town, in the northeastern part of the Taihu Lake area (120º38′ N, 31º33′ E) (Fig. 1). Several factories (three printing and dying mills, three chemical plants, five garment factories, and three machinery factories), and large numbers of villages and toilets have been built along the rivers in recent decades, and substantial amounts of untreated industrial, agricultural and domestic wastewater containing high P concentrations drain directly into the rivers.

A total of 34 surface sediment samples (0-10 cm) were collected using a core sampler equipped with Perspex tubes (8 cm inner diameter, 30 cm long) in November 2012. The sediment samples were freeze-dried, lightly crushed, passed through a 0.149 mm sieve, and stored in clean polyethylene bags until they were analyzed.

Sample Analysis

The P fractions in the sediment were measured as total P (TP), inorganic P (IP), organic P (OP), NaOH-extractable P (NaOH-P), and HCl-extractable P (HCl-P) using the European Commission "Standards Measurements and Testing Program" (SMT) protocol [20, 21]. TP, consisting of the IP and the OP, was determined by ashing the sample at 450ºC then extracting it in 3.5 mol/L HCl. In a separate extraction, IP, consisting of NaOH-P and HCl-P, was extracted using 1 mol/L HCl, and the solid residue was then ashed at 450ºC to determine the OP. P associated with Al, Fe, and Mn oxides and oxyhydroxides was extracted using 1 mol/L NaOH (to give the NaOH-P concentration). P associated with Ca was extracted using 1 mol/L HCl (to give the

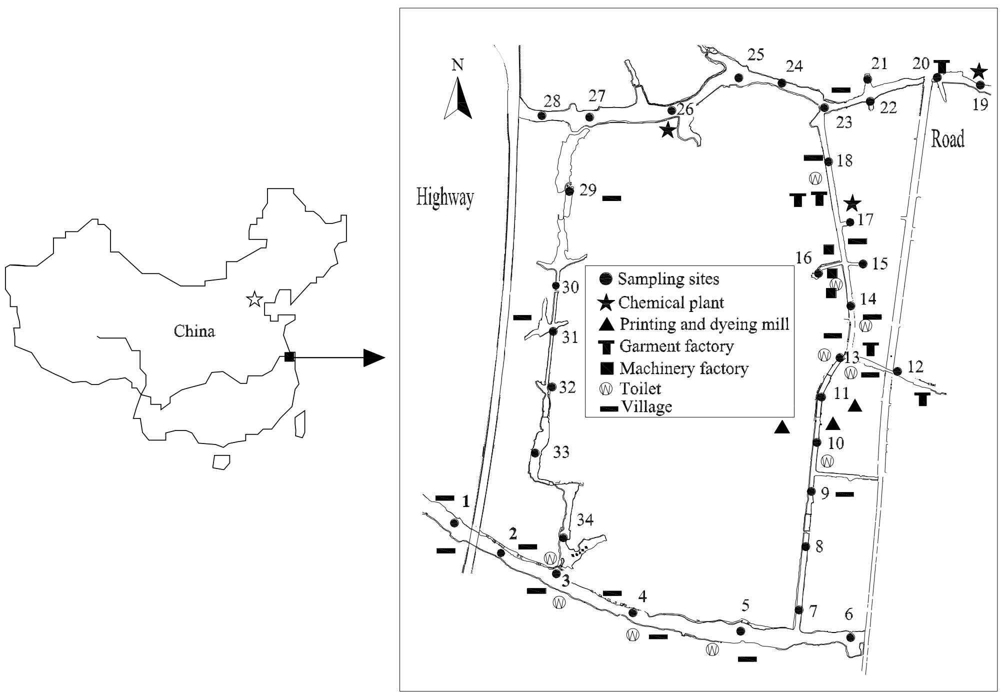

Fig. 1. Map of the sampling area in Yangyuan Town in Taihu Lake area, China, with the sediment sampling sites marked.

Table 1. Statistical summary of the bioavailable P species concentrations (mg/kg) in surface sediments from a river network in the Taihu Lake area, China.

	AAP[a]	OLP	WSP	RDP
Minimum	25.2 (8.7%)[b]	3.8 (1.3%)	0.4 (0.08%)	0.1 (0.01%)
P_{25}[c]	109 (16.4%)	20.4 (3.2%)	0.9 (0.08%)	0.1 (0.01%)
P_{50}	280 (19.0%)	37.2 (3.8%)	1.5 (0.18%)	0.2 (0.03%)
P_{75}	463 (47.5%)	48.2 (4.5%)	2.7 (0.15%)	0.3 (0.02%)
Maximum	1,093 (53.0%)	86.1 (3.8%)	4.3 (0.44%)	0.9 (0.06%)
Mean	340	38.7	1.8	0.3
S.D.[d]	279	20.0	1.1	0.2
CV[e]	0.8	0.5	0.6	0.8

[a] AAP – algae-available P, OLP – Olsen P, WSP – water-soluble P, RDP – readily desorbable P.
[b] the numbers in parentheses are the percentage contribution to the total P (TP) concentration.
[c] P_{25}, P_{50}, and P_{75} are the 25[th], 50[th], and 75[th] percentiles of the distributions.
[d] S.D. – standard deviation.
[e] CV – coefficient of variation.

HCl-P concentration) after NaOH-P extraction. The phosphate concentrations in the supernatants from the extractions were analyzed using the acidic molybdate-ascorbic acid method (AMAA) [22]. For all samples, triplicates were analyzed and the data were reported as the average in this study.

Methods available in the literature were used to determine the bioavailable P concentrations. Different chemical extraction methods were used to identify AAP, OLP WSP, and RDP. The analytical procedures used are given below [14, 15]. Additionally, for all samples, triplicates were carried out and the data were expressed as the average.

AAP: 0.20 g of air dried sediment was extracted with 50 mL of 0.1 mol/L NaOH. The mixture was shaken for 4 h on a shaker at 25ºC, centrifuged at 5,000 rpm for 10 min, and analyzed for AAP using the AMAA method.

OLP: 2.50 g of air-dried sediment was extracted with 50 mL of 0.1 mol/L $NaHCO_3$ (pH 8.5). The mixture was shaken for 0.5 h at 25ºC, centrifuged at 5,000 rpm for 10 min, and analyzed for OLP using the AMAA method.

WSP: 1.00 g of air dried sediment was extracted with 100 mL of deionized water. The mixture was shaken for 2.0 h at 25ºC, filtered through a membrane filter with 0.45 µm pores, and the filtrate was analyzed for WSP using the AMAA method.

RDP: 2.00 g of air dried sediment was extracted with 50 mL of 0.1 mol/L $CaCl_2$. The mixture was shaken for 1.0 h at 25ºC, filtered through a membrane filter with 0.45 µm pores, and the filtrate was analyzed for RDP using the AMAA method.

Statistical Analysis

Descriptive data analysis was performed, the results of which included the minimum, the 25[th], 50[th], and 75[th] percentiles; the maximum, the mean, the standard deviation (S.D.); and the coefficient of variation (CV). Correlation and regression analyses were conducted using SPSS 13.0 to investigate the relationships between the P species and bioavailable P concentrations.

Results

Phosphorus Speciation

The TP concentration was 179-2,293 mg/kg (Fig. 2). The IP and OP concentrations were 137-1,857 mg/kg (66.4-91.2% of TP concentration) and 44.5-465 mg/kg (8.8-33.6% of TP concentration), respectively (Figs. 2 and 3). IP was the major P fraction in the sediment samples (Fig. 3) and this fraction consists of NaOH-P and HCl-P. The NaOH-P and HCl-P concentrations were 54.0-1,473 mg/kg and 61.2-712 mg/kg, respectively, accounting for 12.2-65.9% and 21.9-77.7% of the TP concentrations, respectively (Figs. 2 and 3). Overall, the P species concentrations measured were in the order NaOH-P > HCl-P > OP.

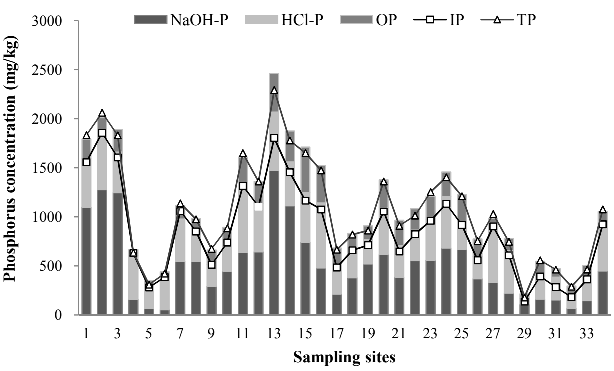

Fig. 2. NaOH-extractable P (NaOH-P), HCl-extractable P (HCl-P), organic P (OP), inorganic P (IP), and total P (TP) concentrations found in the sediment samples from the rural river network.

Table 2. Stepwise regression results for the bioavailable P concentrations measured using four different methods and the NaOH-extractable P (NaOH-P), HCl-extractable P (HCl-P), and organic P (OP) concentrations in rural river sediments from Taihu Lake area, China.

Variable	n	Multiple linear regression equation	Model r^2
AAP[a]	34	AAP=94.714+0.808[NaOH-P]−0.890[OP]	0.791***
OLP	34	OLP=−0.603+0.104[OP]+0.021[NaOH-P]+0.024[HCl-P]	0.910***
WSP	34	WSP=0.772+0.005[OP]	0.241**
RDP	34	RDP=−0.037+0.002[OP]−0.0002[NaOH-P]	0.679***

[a] AAP, algae available P, OLP – Olsen P, WSP – water-soluble P, RDP – readily desorbable P.
** and *** show the significance at $p < 0.01$ and $p<0.001$, respectively.

Bioavailable Phosphorus

The bioavailable P concentrations varied widely, the AAP concentrations being 25.2-1,093 mg/kg, the OLP concentrations being 3.8-86.1 mg/kg, the WSP concentrations being 0.4-4.3 mg/kg, and the RDP concentrations being 0.1-0.9 mg/kg, accounting for 8.7-53.0%, 1.3-3.8%, 0.08-0.44%, and 0.01-0.06% of the TP concentration, respectively (Table 1). The mean concentrations of the four bioavailable P measures followed the order AAP (340 mg/kg) > OLP (38.7 mg/kg) > WSP (1.8 mg/kg) > RDP (0.3 mg/kg).

Relationship between Phosphorus Speciation and Bioavailable Phosphorus

We used regression analysis to identify relationships between the P species concentrations and the bioavailable P concentrations (Fig. 4). Highly significant relationships were found between the TP concentration and the concentrations of bioavailable P measured using all four methods, and the equations describing the relationships were:

AAP=0.408TP–93.11 (R^2=0.626, $P<0.01$)
OLP=0.033TP+3.021 (R^2=0.827, $P<0.01$)
WSP=0.0009TP+0.796 (R^2=0.202, $P<0.05$)
RDP=0.0002TP+0.077 (R^2=0.265, $P<0.05$)

The P species (IP, NaOH-P, HCl-P, and OP) concentrations had very significant or significant relationships with the bioavailable P concentrations, except that the HCl-P concentrations did not have a significant relationship with the WSP concentrations (p>0.05). Of the four bioavailable P concentrations that were measured, the OLP concentrations had the strongest relationships with the different P species concentrations.

Stepwise regression analysis between the four types of bioavailable P concentrations and the NaOH-P, HCl-P, and OP concentrations were performed separately (Table 2). The AAP and RDP concentrations were related to the combined NaOH-P and OP concentrations. The OLP concentration was associated with the OP, NaOH-P, and HCl-P concentrations, and the WSP concentration was related to the OP concentration.

Discussion

Phosphorus Speciation

The TP concentrations in the river sediments, resulting from runoff losses and discharges in untreated rural domestic and industrial wastewater [23], had great potential for supplying P to the overlying water, which would threaten

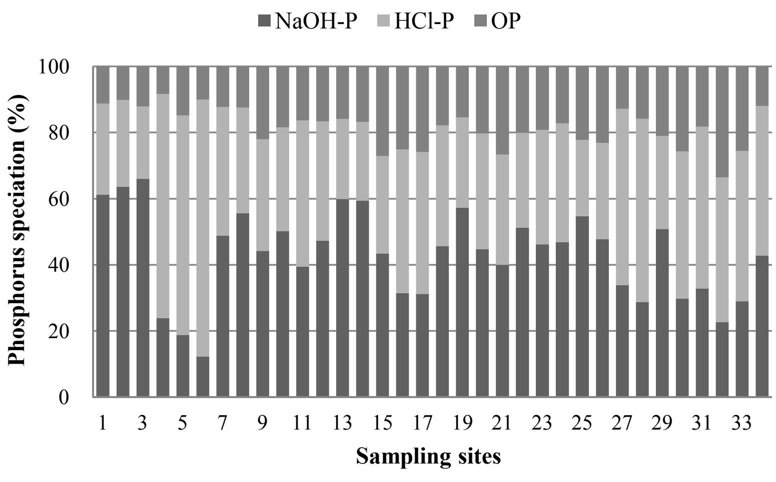

Fig. 3. Contributions (in percentages) of each P species to the total P concentration in the sediment samples from the rural river network.

Table 3. Correlations between the concentrations of the different P species and the bioavailable P concentrations measured using four methods in rural river sediments from the Taihu Lake area, China (n=34).

	HCl-P	OP	IP	TP	OLP	WSP	RDP
NaOH-P[a]	0.580**	0.665**	0.954**	0.949**			
HCl-P		0.604**	0.780**	0.788**			
OP			0.672**	0.777**			
IP				0.986**			
AAP[b]					0.580**	0.262	0.196
OLP						0.586**	0.673**
WSP							0.623**

[a] NaOH-P – NaOH-extractable P, HCl-P – HCl-extractable P, OP – organic P, IP – inorganic P, TP, total P.
[b] AAP – algae available P, OLP – Olsen P, WSP – water-soluble P, RDP – readily desorbable P.
**shows the significance at $p < 0.01$.

the benthos [24]. According to Canadian sediment quality guidelines [25], the TP concentrations at 82.4% of the sampling sites exceeded the lower sediment quality value/guideline (550 mg/kg), and the TP concentrations at sites 2 and 13 reached the higher sediment quality value/guideline (2,000 mg/kg) (Fig. 2), indicating that there was a high level of ecological toxicity to sediment-dwelling organisms from P at sites 2 and 13.

IP has been found to be an important source of bioavailable P in eutrophic sediments [21]. NaOH-P, which mainly includes P bound to Al and Fe oxides and hydroxides [3, 21], can be released from sediment and lead to the growth of phytoplankton under the anoxic conditions that prevail at the sediment–water interface. NaOH-P is, therefore, regarded as a good parameter for describing pollution [23, 26]. In our study, the NaOH-P concentration was very variable (Fig. 2) because of the uneven inputs of external P in the study area. The relative contribution of NaOH-P to the IP concentrations in the sediment samples ranged from 13.6% to 75.1% (Fig. 3). Similar results for NaOH-P existed in the sediments of Volvi Lake and Koronia Lake in Greece [11], and Chaohu Lake, Jiulong River, and Haihe River in China [3, 27, 28].

HCl-P, which includes P associated with Ca, has been found to be a relatively stable P fraction in sediments [11, 21]. Similar to the NaOH-P concentrations, the HCl-P concentrations also varied widely, and the relative contribution of HCl-P to the IP concentrations in the sediment samples ranged from 24.9% to 86.4% (Fig. 3). Moreover, the TP concentration was lower when HCl-P provided a higher proportion of the IP concentration (Fig. 2), which may be explained by HCl-P dominating the IP concentration in sediments that suffered no or only minor pollution.

OP, which can be exploited by organisms, is derived from unwashed organic P and autochthonous sources, such as agriculture [21, 23]. OP was also variable, as were the other P fractions (Figs. 2 and 3). However, the range of OP concentrations was relatively low compared with the ranges of the NaOH-P and HCl-P concentrations. Our results were opposite the results for Chaohu Lake in China because of the decomposition of death cyanobacteria [27].

It can be seen that NaOH-P was the main form of P in areas that were heavily polluted with P while HCl-P was the main form of P in areas that were only slightly polluted with P (Figs. 2 and 3), which was similar to the pattern found by Jin et al. in Xuanwu Lake in China [29]. Changes in the P speciation were strongly associated with the contamination status of the sediments, which was, inevitably, reflected in their intercorrelations (Table 3). There was a highly significant relationship between the TP concentrations and the individual P species concentrations. The correlation coefficient for the relationship between the TP and IP concentrations (0.986, $p<0.01$) was higher than that between the TP and OP concentrations (0.777, $p<0.01$). The correlation coefficient for the relationship between the IP and NaOH-P concentrations (0.954, $p<0.01$) was higher than that between the IP and HCl-P concentrations (0.780, $p<0.01$), suggesting that NaOH-P was the main contributor to the IP concentration. In addition, the highly significant relationships between the NaOH-P, HCl-P, and OP concentrations indicated that something caused changes to occur in the P speciation. Our results demonstrate that the input of external P not only affected the TP concentration, but also influenced the concentrations of different P species and changed the speciation balance, altering the bioavailability of P in the sediments, which will be discussed in the following sections.

Relationship between Phosphorus Speciation and Bioavailable Phosphorus

P in sediments can be classified as non-available, potentially available, and immediately available P [30]. The WSP concentration has been found to be the best estimate of immediately available P [14, 30]. The results obtained after extracting our samples with deionized water suggest that the WSP concentrations were low in the river sediments in our study area, accounting for no more than 0.5% of the TP concentrations. Linear regression analysis showed that there were significant or very significant correlations between the WSP concentrations and the TP, IP, NaOH-P, and OP concentrations ($p<0.05$), but that there was no correlation between the WSP and HCl-P concentrations (Fig. 4e), indicating that the WSP concentrations were related to the NaOH-P and OP concentrations. Stepwise regression analysis clearly demonstrated that the WSP concentrations were mainly affected by the OP concentrations (Table 2).

Some P in sediments can easily be desorbed and are exchangeable, particularly when the P concentration in the water column is depleted [14]. RDP, measured by extracting sediment with a neutral salt solution, was introduced to express the available P concentration. RDP provided no more than 0.01% of the TP concentrations, which was much lower than the proportion provided by WSP. Linear regression analysis showed that there were significant or very significant correlations (i.e., at $p<0.05$ or $p<0.01$) between the RDP concentrations and the TP, IP, NaOH-P, HCl-P, and OP concentrations (Fig. 4), and stepwise regression analysis showed that the RDP concentrations had a positive relationship with the OP concentrations and a negative relationship with the NaOH-P concentrations (Table 2), indicating that RDP was mainly provided by the OP in our samples.

Traditionally, OLP has been seen as the best indicator of P available to plants, and it has often been used as a quantitative index of P available to algae [14]. The OLP concentrations varied widely in our samples, the highest OLP concentration, at site 13, being more than 22 times higher than the lowest concentration, at site 32. More than 25% of the samples had OLP concentrations of more than 46.0 mg/kg, indicating that these samples each had a high nutrient status, according to the index system used by the UK Agricultural Development and Advisory Service [31]. Regression analysis showed that the OLP concentrations were derived from the OP, NaOH-P, and HCl-P concentrations (Fig. 3 and Table 2).

It has been recommended that AAP should be used to estimate both short-term and long-term available P in sediments [32]. The AAP concentrations varied more than the OLP concentrations in our samples. The AAP concentrations

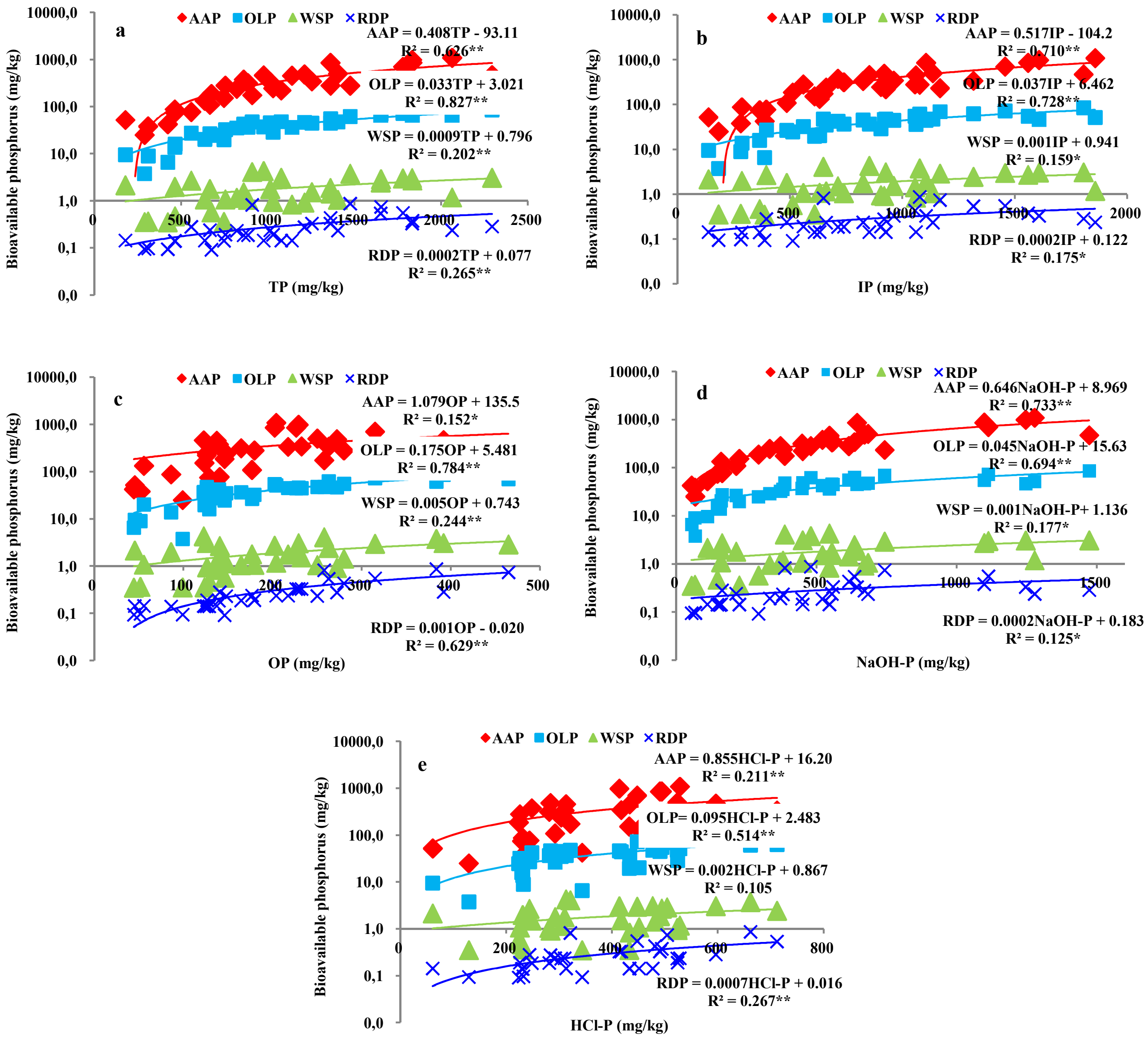

Fig. 4. Comparison of the P species concentrations with the four bioavailable P concentrations measured in the sediment samples from the rural river network.

accounted for more than 50% of the TP concentrations at sites 2 and 3 (Table 1), indicating that there was a strong potential for the sediment to supply P to the overlying water at these sites. The AAP concentrations also had a very significant relationship with the TP concentrations (Fig. 4a), which was similar to the relationship found by Zhou et al. [14] but opposite to that found by Branom et al. [15]. Stepwise regression analysis of the AAP concentrations and the NaOH-P, HCl-P, and OP concentrations (which gave the relationship AAP=94.714+0.808[NaOH-P]−0.890[OP]) in the sediment samples clearly demonstrated that NaOH-P was the main contributor to the AAP concentrations (Table 2).

There was a great deal of spatial variability in the AAP, OLP, WSP, and RDP concentrations in the sediment samples (Fig. 4). However, the rank order of the four different P extract concentrations was generally similar in the samples from all of the sampling sites, the order being AAP > OLP > WSP > RDP, agreeing with the bioavailable P concentrations that have been found in lakes in China and the UK [14, 33]. The four measures of bioavailable P were all related to the NaOH-P and OP concentrations (Fig. 4 and Table 2), indicating that the NaOH-P and OP concentrations are important contributors to the bioavailable P concentrations in the sediments.

Of the four methods used to extract bioavailable P, the WSP and RDP methods extracted the least P and gave the poorest correlations with both the TP and IP concentrations. The AAP method extracted the most P, probably overestimating the bioavailable fraction, which was suggested because of the lack of correlations between the AAP concentrations and the WSP and RDP concentrations (Table 3). The OLP concentrations, which had highly significant correlations with the rest of the bioavailable P concentrations and the P species concentrations, and has been used as a criterion in the UK Agricultural Development and Advisory Service index system, was found to be suitable for determining P bioavailability under the specific geochemical conditions present in the rural rivers we studied. Our results were in agreement with the findings of a previous study of a lake used for sludge disposal [15].

Conclusions

There is a P contamination problem in the rural river network in the Taihu Lake area. The P species concentrations varied widely in sediment samples and their rank order was NaOH-P > HCl-P > OP. The bioavailable P concentrations determined using four methods followed the order AAP > OLP > WSP > RDP. The AAP method extracted the most P and the WSP and RDP methods extracted the least P, and all four methods gave bioavailable P concentrations that were related to the OP concentrations. The OLP concentrations had highly significant correlations with AAP, WSP, RDP and the P species concentrations. The OLP method was found to be the most suitable for determining P bioavailability in sediments in the rural river network studied and could be used to indicate bioavailable P concentrations in other similar areas in the Taihu Lake area.

Acknowledgements

This work was supported by the National Key Technology R&D Program of the Ministry of Science and Technology (No. 2012BAJ24B06).

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