


Article

Phosphorus Fractions in the Sediments of Yuecheng Reservoir, China

Chenghua Dang¹, Ming Lu^{1,2,*}, Zheng Mu¹, Yu Li¹, Chenchen Chen¹, Fengxia Zhao¹, Lei Yan¹ and Yao Cheng^{1,*} 

¹ School of Water Conservancy and Hydroelectric Power, Hebei University of Engineering, Handan 056002, China; dangchenghua@sohu.com (C.D.); muzheng1981@hebeu.edu.cn (Z.M.); liyu199607@163.com (Y.L.); ccc2019szy@163.com (C.C.); zhaofx1123@163.com (F.Z.); yanl@whu.edu.cn (L.Y.)

² Department of Earth Sciences, University of the Western Cape, Cape Town 7535, South Africa

* Correspondence: luming_hd@163.com (M.L.); chengyao1108@163.com (Y.C.)

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Abstract: As a result of the inexorable development of the economy and the ever-increasing population, the demand for water in the urban and rural sectors has increased, and this in turn has caused the water quality and eutrophication of the reservoir to become a legitimate concern in the water environment management of river basins. Phosphorus (P) is one of the limiting nutrients in aquatic ecosystems; P in the sediment is a primary factor for eutrophication. Yuecheng Reservoir is located in one of the most productive and intensively cultivated agricultural regions in North China. Detailed knowledge of the sediment is lacking at this regional reservoir. The first study to look into the different P fractions and its diffusion fluxes at the water sediment interface of the Yuecheng Reservoir makes it possible to learn about the internal P loading. According to the results, the concentrations of total phosphorus (TP) ranged from 1576.3 to 2172.6 mg kg and the P fraction concentration sequence is as follows: P associated with calcium (Ca–Pi) > organic P (Po) > P bound to aluminum (Al), ferrum (Fe) and manganese (Mn) oxides and hydroxides (Fe/Al–Pi). The results demonstrated that, although the construction of a large number of water conservancy projects in the upper reaches of the river resulted in the decrease of inflow runoff, the pollutions from terrestrial plants or materials played a key role in the sediment phosphorus fraction, and they should be emphasized on the water environment management of river basin.

Keywords: phosphorus fractions; diffusion fluxes; sediment; Zhangweinan River Basin; Yuecheng Reservoir

1. Introduction

Phosphorus (P) is one of the limiting nutrients in aquatic ecosystems [1,2]. Water trophic status and phytoplankton growth are closely related to P in the water column and surface sediment. P in the sediment is a primary factor for eutrophication [3], which is one of the most serious problems in reservoirs, lakes and rivers. The sediment P adsorption and cycling in different regions of the river basin is important for river environment management [4,5]. There are important influence factors, such as the characteristics of sediments, environmental factors and the concentration of P in the overlying water. All of these factors play a pivotal role in the transfer direction of P at the sediment–water interface [6,7]. As the external loading of P is increased, the sediments will absorb it, while if the external loading is decreased, the sediments release the absorbed P into the water in the long-term [8]. Accordingly, the most common strategy for the restoration of eutrophic reservoirs and lakes is focused on reducing external P loading. This method, however, achieves limited effects because bottom sediments can also release phosphorus to the overlying water, especially when the

P input is reduced [9,10]. Therefore, systematic studies of internal P loading are indispensable to understand the process of P circulation and support effective policies to manage eutrophication [11,12].

The Yuecheng Reservoir is located in the south of Handan, Hebei province, which is the main local water supply of the municipal, industry and agricultural sectors [13]. Today, the precipitation is decreasing and the number of reservoirs and diversion channels is increasing in the upper reaches. As a result, the inflow of the Yuecheng Reservoir is becoming much less than a decade ago. Moreover, the inexorable development of the economy and the ever-increasing population have increased the demand for water in the urban and rural sectors, and these in turn cause the water quality and eutrophication of the reservoir to become a legitimate concern in the water environment management of the river basin.

Consequently, in this study, the P fractions and the relationships with environmental factors of the reservoirs have been investigated for the first time to enhance the knowledge of P cycling in this high P concentration basin, so that the water quality and eutrophication can be controlled better in the future.

2. Materials and Methods

2.1. Study Area and Sampling Sites

As the main reservoir of flow regulation for the water supply in Zhangweinan Basin [13], Yuecheng Reservoir has a capacity of 1.3 billion m³ and a drainage area of 18,100 km², which provides the water supply for Anyang city and Handan city and irrigation for the two large agricultural areas of Minyou and Zhagnan. The average depth of the reservoir is 20 m and the maximum depth is 37 m. Algae may appear occasionally in some areas of the reservoir between April and September, with a maximum concentration of 40 µg·L⁻¹.

The longitude and latitude of the Zhangweinan River Basin range from 112 to 118° E and from 35 to 39° N, respectively, and it is located in North China. It consists of five main rivers, including the Zhang River, Wei River, Wei Canal, Zhangweixin River and Nan River [14]. This basin is comprised of roughly 25,466 km² mountainous area (i.e., Taihang Mountains and Taiyue Mountain) and about 12,234 km² plain area. Thus, the total area of this basin is approximately 37,700 km². The basin is characterized by semiarid, semi-humid climatic conditions and its average annual precipitation is 608.4 mm, with a mean annual temperature of 14 °C. The distribution of temporal precipitation varies greatly among the four distinct seasons. For example, in July and August, more than 50% of precipitation falls, while in the spring, autumn, and winter seasons, the rainfall occupies 8%–16%, 13%–23%, and 2% of the total for one year in order. As one of the most productive and intensively cultivated agricultural regions in North China, the basin has a population of about 30 million and has 293 × 10³ ha cultivated area. The main cropland features various kinds of cultivated crops, including wheat, maize, cotton, rice, bean, oilseed, and vegetables, and about 75% of the cropland is irrigated, which consumes 70%–80% of the total water resources.

Duplicated samples of superficial sediments (about 0–5 cm deep) and the corresponding overlying water (about 0–4 cm above the interface) were collected using a gravity corer (diameter 6.5 cm and length 60 cm) [15] at five points (YR1–YR5) in Yuecheng Reservoir (Figure 1) in July 2017. Stratified overlying water was injected into PVC bottles at an interval of 2 cm. The sediments were sampled into sections of 2 cm length and packed in polyethylene centrifuge tubes and sealed to avoid sediment oxidation. All samples were collected in triplicate, taken in air-sealed plastic bags and kept at 4 °C until analysis (within 24 h).

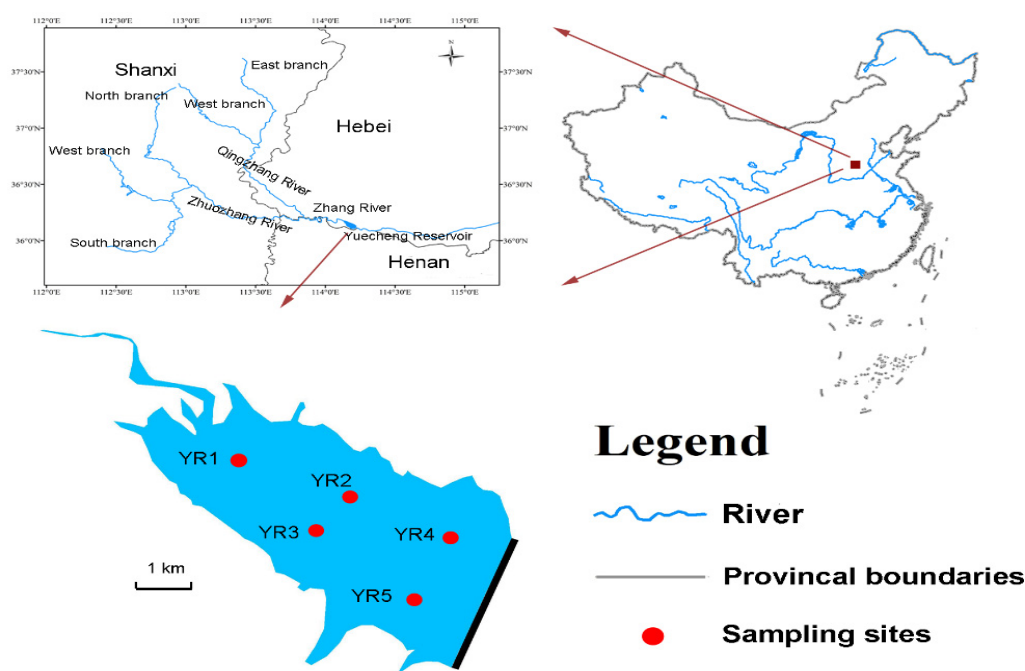


Figure 1. Location of the Yuecheng Reservoir and sampling sites.

2.2. Water Chemistry

The parameters of water, such as the temperature (T), dissolved oxygen (DO), electrical conductivity (EC), oxidation reduction potential (ORP), and pH values, were measured in situ using a YSI (Yellow Springs Instruments Inc., Yellow Springs, OH, USA) EXO2 multisensor sonde. The overlying water and pore water (extracted from sediment by centrifuging at 4000 rpm for 30 min) samples were filtered through 0.45 μm GF/C filter membranes. The concentration of orthophosphate ($\text{PO}_4^{3-}\text{-P}$), the most bioavailable P form, was determined by the molybdenum blue method [16].

2.3. Sediment. Sample Analysis

One sample of sediments was used to calculate the water content by weighing the weight loss after drying the sediments at 105 $^{\circ}\text{C}$. Meanwhile, the water volume of the sediment was approximately regarded as the pore volume, and the porosity of sediment was the ratio between pore volume and total sedimentary volume. The dried samples were homogenized and separated by a laser diffraction particle size analyzer (LS 13 320 MW, Beckman Coulter Company, Erie, PA, USA) into three grain size fractions: the sand fraction (62.5–500 μm), the silt fraction (3.9–62.5 μm), and the clay fraction (0.5–3.9 μm).

Other sediment samples were freeze-dried, homogenized and sieved through a 100-mesh sieve. Total organic carbon (TOC) and total nitrogen (TN) were detected by an elemental analyzer (Vario EL III, Elementar Company, Langenselbold, Germany) after pretreatment in 1 mol L^{-1} hydrochloric acid (HCl) to remove inorganic carbon. P fractions were classified into TP, inorganic P (Pi), organic P (Po), P associated with calcium (Ca) (Ca-Pi) and P bound to aluminum (Al) and ferrum (Fe) oxides and hydroxides (Fe/Al-Pi), and determined by the SMT (Standards Measurements and Testing) protocol [17]. For all samples, triplicates were analyzed and the average or “mean value \pm standard deviation” of data were reported.

2.4. Flux Estimation and Data Analysis

Fick's first law, based on the principle that the concentration gradient initiates the exchange, was used to estimate the theoretical release flux of $\text{PO}_4^{3-}\text{-P}$ at the interface of water and sediment [18].

$$J = \varphi_0 D_s \left. \frac{\partial C}{\partial x} \right|_{x=0} \quad (1)$$

where J is the diffusion flux ($\text{mg m}^{-2} \text{ d}^{-1}$); φ_0 is the porosity of surface sediment; and $\left. \frac{\partial C}{\partial x} \right|_{x=0}$ is the concentration gradient at the water–sediment interface. It takes the slope value of the best fitting line of the distribution profile of $\text{PO}_4^{3-}\text{-P}$ concentrations in the overlying water and sediment. D_s is the effective diffusion coefficient, which is calculated from D_0 by the following equations. D_0 ($\text{cm}^2 \text{ s}^{-1}$) is the theoretical diffusion coefficient of infinite dilution solution [19]. T in Equation (4) is the temperature of the overlying water ($^{\circ}\text{C}$).

$$D_s = \varphi_0 D_0 \quad \varphi_0 < 0.7 \quad (2)$$

$$D_s = \varphi_0^2 D_0 \quad \varphi_0 > 0.7 \quad (3)$$

$$D_0 (\times 10^{-6}) = 7.37 + 0.16 \times (T - 25 \text{ } ^{\circ}\text{C}) \quad (4)$$

The mean values and standard deviations were calculated by Microsoft Excel 2013, and correlation statistical analyses were tested with SPSS 22 (Statistical Program for Social Sciences 22).

3. Results and Discussion

3.1. Physicochemical Properties of Overlying Water and Interstitial Water

The physicochemical properties of overlying water and $\text{PO}_4^{3-}\text{-P}$ concentrations of interstitial water are listed in Table 1. The parameters gave pH values that ranged from 7.92 to 8.44. EC, a measure of the ionic strength, ranged from 462.3 to 479.6 $\mu\text{S cm}^{-1}$. The ORP values were found to be 123.3 ± 21.7 mv, and the DO concentrations were 6.3 ± 0.3 mg L^{-1} . The $\text{PO}_4^{3-}\text{-P}$ concentrations (mg L^{-1}) of the overlying water and the interstitial water ranged from 0.14 ± 0.03 to 0.55 ± 0.04 and from 0.34 ± 0.02 to 0.67 ± 0.04 , respectively.

Table 1. Characteristics of overlying water in the Yuecheng reservoir.

Sample	$\text{PO}_4^{3-}\text{-P}$ (mg L^{-1}) ^a	$\text{PO}_4^{3-}\text{-P}$ (mg L^{-1}) ^b	T ($^{\circ}\text{C}$)	DO (mg L^{-1})	Ph	EC ($\mu\text{S cm}^{-1}$)	ORP (mv)
YR1	0.35 ± 0.02	0.44 ± 0.01	18.7	6.1	8.42	479.6	117.9
YR2	0.14 ± 0.03	0.34 ± 0.02	18.5	6.5	7.92	462.3	148.6
YR3	0.24 ± 0.03	0.57 ± 0.02	18.4	5.2	8.07	468.9	124.7
YR4	0.55 ± 0.04	0.67 ± 0.04	18.8	6.7	8.26	464.7	137.2
YR5	0.37 ± 0.01	0.54 ± 0.02	18.7	5.8	8.22	470.4	132.8

^a Represents the mean values of $\text{PO}_4^{3-}\text{-P}$ concentrations of the overlying water (0–4 cm). ^b Represents the mean values of $\text{PO}_4^{3-}\text{-P}$ concentration of interstitial water of the surface sediment (0–4 cm). DO: dissolved oxygen; EC: electrical conductivity; ORP: oxidation reduction potential.

3.2. Characteristics of the Surface Sediment

The physical and chemical properties of sediments are shown in Table 2. The water content of sediment reflects its resuspension ability: the higher the value is, the easier it is to suspend. According to Table 2, the surface sediment contained a large percentage of water ($63.4 \pm 1.9\%$) which facilitates the diffusion of P through its suspension under external disturbance. The average value of the porosity of the surface sediment was $82.2 \pm 1.3\%$. Particle size analysis indicated that most surface sediments

were high in terms of the proportion of clay and silt fractions; on average, at 97.1 %. Higher porosities and smaller sediment particles are more reactive due to the increased surface area [20].

Table 2. Physical and chemical characteristics of the sediments. TP: total phosphorus; TN: total nitrogen; TOC: total organic carbon.

Sample	Moisture (%)	Porosity (%)	Clay (%)	Silt (%)	Sand (%)	TP (mg·kg ⁻¹)	TN (g·kg ⁻¹)	TOC (g·kg ⁻¹)
YR1	61.1	80.3	29.5	66.7	3.8	2172.6	8.2	109.6
YR2	62.2	81.5	43.5	53.2	3.3	1576.3	7.0	75.4
YR3	64.7	83.1	40.3	57.4	2.3	1972.8	8.1	108.6
YR4	65.7	83.3	42.3	55.2	2.5	1627.3	7.7	95.6
YR5	63.1	82.7	44.3	52.8	2.9	1844.7	7.4	97.5

As presented in Table 2, TN varied from 7.0 to 8.2 g·kg⁻¹ and TOC varied from 75.4 to 109.6 g·kg⁻¹. However, TP ranged from 1576.3 to 2172.6 mg·kg⁻¹. The C/N (TOC/TN) ratios ranged from 12.6 to 15.7 with an average of 14.8. The C/N ratio varied from 2.6 to 4.3 in bacteria, and 7.7 to 10.1 in aquatic plants, whereas this ratio of terrestrial plants or materials was higher than 20 [21]. Almost all C/N ratios in the sampling sediments of Yuecheng Reservoir were nearly 15, which illustrated that both endogenous sources and terrestrial plants or materials had a pivotal role in sedimentary P.

3.3. Phosphorus Fraction Composition

As presented in Figure 2, the fractions of different P values varied greatly. For five sampling sites, the rank order of P fractions concentrations was Ca-Pi > Po > Fe/Al-Pi.

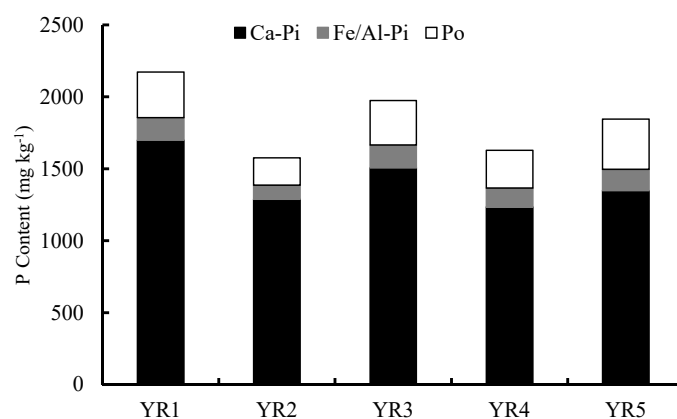


Figure 2. Concentrations of different P forms in surface sediments in Yuecheng Reservoir.

Po showed the P fraction bound to organic matters and its stability were relative to the different Po structures [12]. Po contents in the surface sediments varied from 188.6 to 358.8 mg kg⁻¹, and this contributed 12.1%–19.2% to the TP (Figure 2), which mainly resulted from the difference of TP contents. Fe/Al-Pi, which was considered as algae-available, could be easily affected by pH and ORP, and consequently was released into pore water under reductive conditions [22]. About 6.1%–8.9% of the sedimentary TP was Fe/Al-Pi, which ranged from 96.4 to 178.8 mg kg⁻¹. Ca-Pi was assumed to mainly consist of apatite P (natural and detritus) [22]. The Ca-Pi contents ranged from 1192.5 to 1703.6 mg kg⁻¹, and the relative contribution ranged between 72.4%–81.9% of TP. This inorganic fraction was a subject of debate because it had long been considered to have little mobilization. However, much research has shown that this fraction could be mobilized with decreased pH and specific biochemical effects [12,22].

3.4. Estimated Diffusion Fluxes of $\text{PO}_4^{3-}\text{-P}$

Figure 3 showed the typical distribution profiles of $\text{PO}_4^{3-}\text{-P}$ concentrations in the overlying water and sediments of five sampling sites. The $\text{PO}_4^{3-}\text{-P}$ in sediment is higher than overlying water, which indicates that the sediment was releasing P in all sections of the reservoir. The variations of $\text{PO}_4^{3-}\text{-P}$ diffusion fluxes across the water–sediment interface in the sampling sites are presented in Figure 4. The fluxes ranged from 0.10 to 0.31 $\text{mg m}^{-2} \text{d}^{-1}$. Ca–Pi is more stable in the sediment and less easily released into overlying water compared with Po and Fe/Al–Pi. Although the TP was high at YR3, the flux was not the highest.

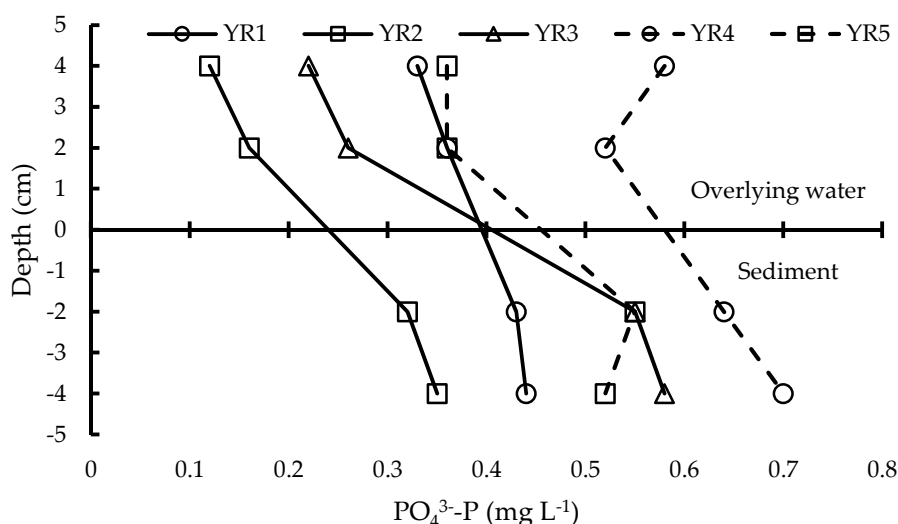


Figure 3. $\text{PO}_4^{3-}\text{-P}$ concentrations of pore water and the corresponding overlying water.

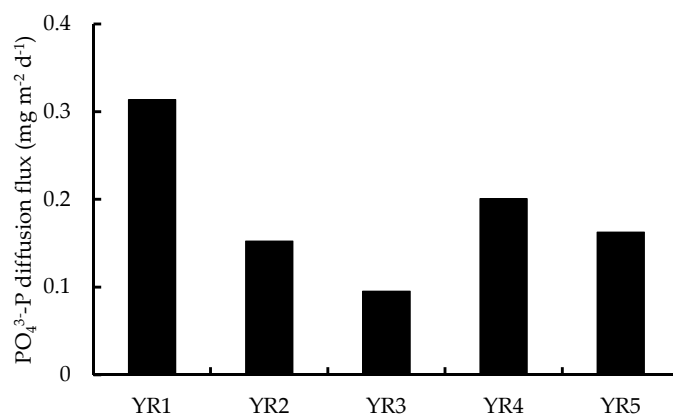


Figure 4. Variations of $\text{PO}_4^{3-}\text{-P}$ diffusion fluxes at the water–sediment interface.

The threshold ratio of Fe to TP in the sediment is 15 for P retention under aerobic conditions. If this ratio holds true across the reservoir, the internal P loading could be controlled by keeping the surficial sediments oxidized. However, the ratios of Fe to TP of the sediment (6.1–8.9) were below that threshold, which indicated that the sediments could not adsorb more $\text{PO}_4^{3-}\text{-P}$ of the overlying water. The microbial degradation of Ca–Pi probably contributes to the $\text{PO}_4^{3-}\text{-P}$ diffusion due to their acclimation of living environment under this high Ca–Pi concentration background. Perez [23] detected that a total of 130 heterotrophic bacteria showed different degrees of mineral tri-calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$)-solubilizing activities.

Compared with other studies (Table 3), the TP concentration in the sediments was much smaller than that of almost all typical eutrophic lakes. Compared to Dianchi and Taihu, a greater proportion (more than 70%, Figure 2) of TP in the sediments was made up of Ca–Pi, which was considered to have

little mobilization under physical–chemical conditions compared to Fe/Al–Pi [12,22]. Moreover, it is speculated that the behavior of the microbial degradation of Ca–Pi is driven by the microorganism to absorb reactive P nutrients from the environment for its own use. Thus, it may be controlled within a certain range by some related enzymes [23,24].

Table 3. Comparisons of TP and P diffusion fluxes between the reservoir and other areas.

Site	TP (mg kg ^{−1})	PO ₄ ^{3−} –P Diffusion Fluxes (mg m ^{−2} d ^{−1})	Reference
Hongze Lake, China	76.6–932.2	0.172–0.793	[25]
Dianchi Lake, China	1537–4695	1.00–4.36	[26]
Taihu Lake, China	213.7–724.4	0.76–4.57	[27,28]
Three Gorges Reservoir, China	415.5–1047.9	−0.003–0.013	[24,29]
Yuecheng Reservoir, China	1576.3–2172.6	0.10–0.31	This study

3.5. Relationship of Sediment Characteristics and P Fractions

The multiple regression analysis of characteristics and different P forms of the surface sediments are listed in Table 4. A non-parametric test was performed since environmental data do not usually follow a good normal distribution. The pairs of Ca–Pi with TP, with Fe/Al–Pi, and with Po clearly showed an association and implied an external Ca–Pi input to the reservoir (Table 4) [30]. The correlation coefficient of Ca–Pi and TP was 0.95, also indicating that the variance of TP concentrations was mainly related to the Ca–Pi contents. In addition, there was a significant correlation among TN, TOC, Ca–Pi and TP, which reflected that the sediment probability mainly came from the same origin [17].

Table 4. Physical and chemical characteristics of the sediments.

Sample	TP	TN	TOC	C/N	Po	Fe/Al–Pi	Ca–Pi
TP	1						
TN	0.77 **	1					
TOC	0.82 **	0.84 *	1				
C/N	0.64 *	0.49	0.88 **	1			
Po	0.72 *	0.61	0.79 **	0.76 *	1		
Fe/Al–Pi	0.75 *	0.64 *	0.86 **	0.85 **	0.82 **	1	
Ca–Pi	0.95 **	0.71 *	0.68 *	0.46	0.49	0.56	1

** Correlation is significant at the 0.01 level (2-tailed). * Correlation is significant at the 0.05 level (2-tailed).

4. Conclusions

This study showed that different P-forms and PO₄^{3−}–P diffusion fluxes of the surface sediments of the Yuecheng Reservoir, which is located in the productive and intensively cultivated agricultural region of North China. The rank order of P-fraction concentrations obtained from sample sites was Ca–Pi > Po > Fe/Al–Pi. Ca–Pi, varying from 72.4% to 81.9% of TP, was the primary driver of variation in TP. The C/N ratios ranged from 12.6 to 15.7, which suggested that Po mainly originated from endogenous sources and terrestrial plants or materials. The analysis of environmental factors indicated that there was an association among P-forms. Although the Yuecheng Reservoir is located in one of the most productive and intensively cultivated agricultural regions in North China, Po is not a major part of the sediment, which is simply because the construction of the cascade reservoirs in the upper stream blocked a large number of terrestrial plants in the basin. Thus, for water environment management in this basin, although the construction of a large number of water conservancy projects in the upper reaches of the river has resulted in the decrease of inflow runoff, the pollution from terrestrial plants or materials played a key role in the sediment phosphorus fraction, and this should be emphasized in the

water environment management of the river basin. Future work on the biological characterization of the sediments would provide more information on the status of this reservoir.

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Conflicts of Interest: The authors declare no conflict of interest.

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