Phosphorus release from coastal sediments: Impacts of the oxidation-reduction potential and sulfide

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ABSTRACT

The release of phosphorus (P) from benthic sediments can affect the P content, nutrient status and quality of overlying waters in coastal ecosystem. This study was carried out to investigate the influence of oxidation-reduction potential (ORP) and sulfide on P release from sediments in the coastal estuary of the Yuniao River, China. The results showed that ferric iron-bound P was the main P burial phase in the sediments. The P concentration in overlying water increased with ORP decrease and sulfide increase, displaying a significant linear correlation with the ORP and sulfide concentration. The results indicate that decreased ORP may elevate the zero equilibrium phosphorus concentration, enhancing the capability of P release. And increased sulfide may react or capture reactive iron in sediments, reducing the P adsorption capacity and accelerating P release. Therefore, the control of ORP and sulfide production is important in the sink/source conversion of P in coastal sediments.

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1. Introduction

Phosphorus (P) is an essential nutrient for biological productivity, and therefore plays an important role in oceans and continents. The total input of P into aquatic systems is determined by both external inputs as well as internal recycling from the benthic sediments to the water column (Pan et al., 2013; Kraal et al., 2015). Excessive input of P can lead to eutrophication, which adversely affects coastal zone ecosystems in many areas around the world (Yang et al., 2015). Phosphorus retention and release processes in estuarine and coastal sediments are also significant for the global P cycle because a great portion of P (~25% dissolved P) is trapped in such sediments and thus does not reach the open ocean (Ruttenberg, 1993; Paytan and McLaughlin, 2007).

Under some conditions, benthic sediment may act as an important P source for the overlying water (Sondergaard et al., 1999), and even create N-limited condition (Ferrera et al., 2016). P exchange processes between the sediment and overlying water are known to be affected by physico-chemical conditions and biological processes, which can also influence the existing forms of P (Qian et al., 2011; Pan et al., 2013). Anoxic marine sediments are generally thought to have diminished sedimentary P retention capacity (Ingall et al., 1993). Recent work has provided detailed insight into the relationship between reducing conditions and diminished sedimentary P sequestration (Kraal et al., 2015). Previous studies report that P flux between sediment and overlying water may be strongly influenced by physico-chemical conditions, such as temperature, dissolved oxygen (DO) and pH Smith et al., 2006; Lake et al., 2007; Berbel et al., 2015). In particular, oxidation-reduction potential (ORP) may be an especially crucial factor controlling the release of P to the overlying water from sediments. In this regard, P-sediment adsorption experiments may be useful in examining the ORP-dependent ability of sediment to either remove or release P to the overlying water (Lyons et al., 1998; Pan et al., 2002).

The P cycle in coastal ecosystem can be strongly coupled to sediment iron (Fe) and sulfur (S) transformations (Kraal et al., 2015). Understanding the interaction between Fe, P and S is key to predicting their biogeochemical fates and impact on water quality. In estuarine sediments rich in organic matter, oxygen is quickly depleted with microbial aerobic respiration below the sediment-water interface, thereby creating an anoxic environment. Under these conditions, both microbial iron reduction (Taillefert et al., 2000a) and sulfate reduction will occur (Burton et al., 2011). Iron reduction results in the dissolution of iron oxides to ferrous iron, whereas sulfate reduction results in the generation of sulfide. In the presence of sulfide, the Fe^{2+} ion can be quickly and effectively removed from pore waters by the formation of FeS and FeS_2, which also removes the toxic sulfide in the sediment-water system (Taillefert et al., 2000b). The biogeochemical reactions of Fe and S also affect both availability of sedimentary P to aquatic organisms and mobility of P within the sediments (Rozan et al., 2002).

In light of the global increase in the areal extent of coastal marine systems that experience oxygen depletion and alterations in benthic P cycling in response to eutrophication, it is of considerable interest to clarify the ORP-dependent P release processes (Diaz and Rosenberg,

2008; Middelburg and Levin, 2009; Qian et al., 2011). Therefore, the goals of this study were 1) to investigate the influence of ORP on P release from sediments in a polluted coastal estuary using a combination of P adsorption experiments and selective extractions across an experimentally-controlled range of ORP conditions; and 2) to investigate the influence of sulfide addition on P release from sediments.

2. Methods and materials

2.1. Sampling and handling

The Yuniao River originates from the Muping City (Yantai, China) urban area (0.4 million population), and carries about 60% of the municipal sewage of the city. The river is located in the northeastern Shandong province in northern China on the Bohai Strait and the southern coast of the Northern Yellow Sea (37°23′35″N, 121°33′59″E; Fig. 1). The river is a typical seasonal river, with domestic and industrial wastewater discharge accounting for most of the river water during the dry season (Sun et al., 2016). The tide in the river estuary is a regular semidiurnal tide, and therefore the water quality is variable (Li et al., 2016). Sediment samples were collected from the Yuniaohe River estuary using a Van-Veen grab sampler, and were stored in zip-lock plastic bags on ice in an adiabatic box. Before analysis, the samples were freeze-dried and sieved (80 mesh). All reagents used were analytical grade or above, and deionized water (Milli-Q) was used to prepare the overlying water. Milli-Q water was deoxygenated by purging with high-purity N_2 (>30 min).

2.2. Measurements and methods

Concentrations of P, Fe (digestion prior to determination) and chemical oxygen demand (COD_{Cr}, acid COD_{Mn} and alkaline COD_{Mn}) were measured with the corresponding method specified in the standard methods by APHA (2005). In brief, this involved the dichromate titration method for COD_{Cr}, acid and alkaline permanganate titration method for acid COD_{Mn} and alkaline COD_{Mn} respectively. Dissolved oxygen (DO) and ORP in overlying water were measured using Unisense meters and electrodes. Five solid-phase fractions of P (loosely sorbed P, E&L-P; ferric iron-bound P, Fe-P;

authigenic carbonate fluorapatite + biogenic apatite + $CaCO_3$-associated P, ACa-P; detrital apatite P, De-P; and organic P, Or-P) in the sediment samples were determined by a sequential extraction method (SEDEX) (Ruttenberg, 1992). The concentrations of total nitrogen (TN), total sulfur (TS) and total organic carbon (TOC) in sediments were measured by an elemental analyzer (Vario MACRO cube, Elmentar, Germany). The precision of the measurements was ≤0.5%.

2.3. ORP-dependent P release experiments

A group of experiments were set up to investigate the impact of the ORP on P release. In an anaerobic chamber, freeze-dried sediment was added to 6 beakers (named a, b, c, d, e, f) with anaerobic deionized water to achieve a sediment concentration of 10 g L^{-1} in each beaker. After allowing the sediment-water system to stabilize for 24 h, the DO and ORP were then measured. At the same time, a 5 mL sample of the supernatant water was collected from each beaker without disturbing the sediment-water interface to measure the concentration of P. Then, different dosages of $NaBH_4$ were added to achieve different ORP values in each beaker. 24 h later, DO, ORP and P were again measured.

2.4. Adsorption isotherm experiments

To determine the zero equilibrium phosphate concentration (EPC_0) for sediment samples under different ORP conditions, 0.20 g of freeze-dried sediment was placed in 48 polypropylene centrifuge tubes with 20 mL anaerobic deionized water. The 48 tubes were divided into 6 groups (A–F, with different ORP values), with each group containing 8 tubes. Each group was spiked with KH_2PO_4 to provide a range of initial phosphorus concentrations (0, 0.5, 1.0, 2.0, 6.0, 10.0, 15.0, 20.0 mg L^{-1}). The tubes were placed in a shaking bath (150 rpm, 25 °C) for 24 h. After 24 h, each tube was centrifuged (3000 rpm), then aqueous P was measured after filtration to <0.45-μm. The difference in P between the start and the end of the sorption experiment, along with the sediment dosage were used to calculate the amount of adsorbed P. Sorption curves were obtained by plotting the aqueous equilibrium concentration of P against the amount of adsorbed P per kg sediment.
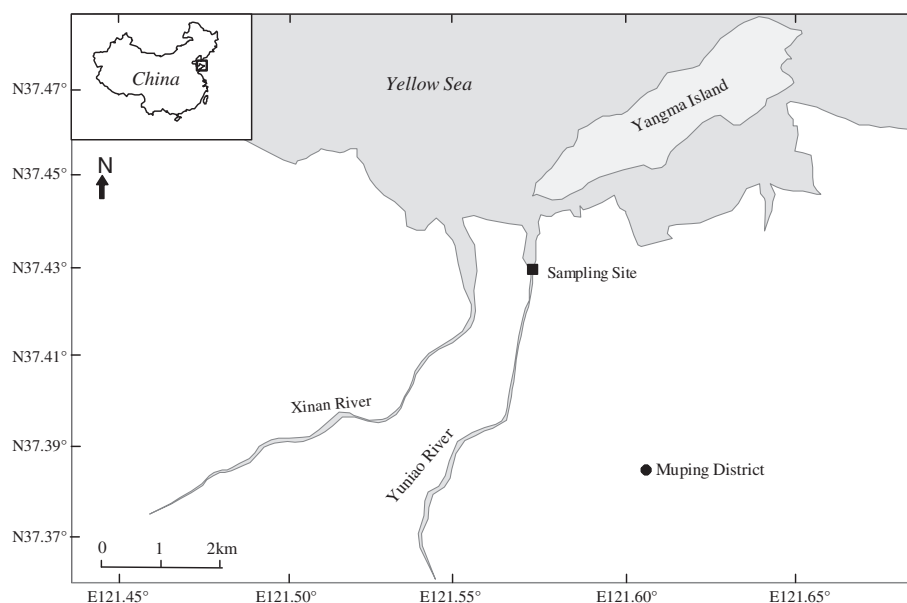

Fig. 1. Detailed location of the sampling site.

Table 1
The average parameters of polluted river water.

| | COD_{Cr} (mg L^{-1}) | COD_{Mn} (mg L^{-1}) | | DO (mg L^{-1}) | Conductivity (ms cm^{-1}) | Salinity (PSU) |
		Acid method	Alkaline method			
Mean value	753.70	266.88	250.24	0.1	3.05	2.51
Standard (V)	40	15	/	2	/	/

2.5. Sulfide addition on P release experiments

A group of experiments were set up to investigate the impact of the sulfide addition on P release. The general experiment design was same as the steps in Section 2.3; freeze-dried sediment was added to 6 beakers (named a, b, c, d, e, f) with anaerobic deionized water to achieve a sediment concentration of 10 g L^{-1} in each beaker. After allowing the sediment-water system to stabilize for 24 h, a $Na_2S \cdot 9H_2O$ stock solution was added to 6 beakers to achieve a sulfide concentration of 0, 5, 10, 15, 20 and 30 mg L^{-1} in each beaker, respectively. After 24 h, the concentrations of sulfide, Fe^{2+} and P in supernatant water of each beaker were then measured.

3. Results and discussion

3.1. Estuary pollution characteristics and in-situ P geochemistry

In the Yuniao River, the average concentrations of COD_{Cr} and acid COD_{Mn} were much higher than the lowest surface water quality level (Chinese Standard, Level V, SEPA, 2002), indicating that the Yuniao River had been heavily polluted with regard to oxygen demand (Table 1).

The average concentrations of TOC, TN and TS in sediments were 7.67 wt%, 1.20 wt% and 1.30 wt%, respectively, which are similar to the Dihe River in northern China (Sheng et al., 2013), but the TOC is higher than the lowest level of Marine Sediment Quality (4×10^{-6} wt%) (AQSIQ, 2001). The results suggest that the sediments in the estuary are rich in organic matter because of long term pollution via sewage inputs. The sediment sample had a high concentration of total Fe (9361 mg kg^{-1}, Table 2) and a correspondingly high total P concentration (1897 mg kg^{-1}), much higher than the average background P concentration for all the bay sediments in Japan is 452 mg kg^{-1} (Fukue et al., 1999). Phosphorus in the surface sediments was predominantly present as Fe-P, with minor contributions from ACa-P, E&L-P, and Or-P (Table 2). Generally, iron hydroxide and iron oxides have the strongest adsorption capacity for P among the various types of natural particles (Berner, 1973). Therefore, in Yuniao River estuary, the low oxygen and high organic matter conditions (Tables 1 and 2) may promote the formation of ACa-P and diminish the preservation of Fe-P. However, in sediment sample examined here, the concentration of Fe-P is much higher than ACa-P, which may be related to sediment's relatively high total Fe concentration. Under oxygen-depleted conditions when organic matter degradation is retarded, Or-P is generally the dominant source of P to the sediment surface and makes up a significant proportion of the sedimentary P reservoir (Ingall et al., 1993; Froelich et al., 1988; Follmi,

Table 2
The average concentrations of P, Fe, C, N and S in the surface sediments (unit: mg kg^{-1}).

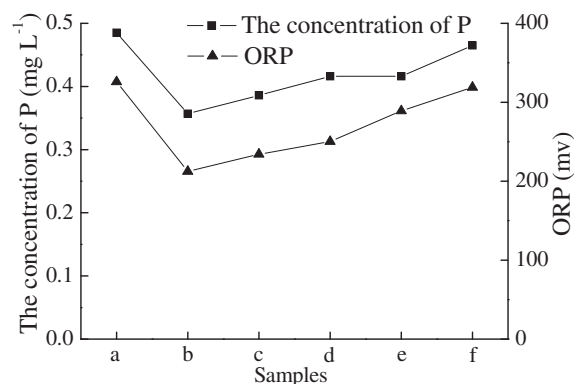|	E&L-P	Fe-P	ACa-P	De-P	Or-P	Total P
Mean value	246.02	1092.29	285.34	95.64	178.20	1897.49
	Fe(III)	Total Fe	TOC (%)	TN (%)	TS (%)	
Mean value	6214.62	9361.97	7.67	1.20	1.3	

Fig. 2. The concentration of P and ORP in different samples (a–f, with the same initial conditions) before $NaBH_4$ addition.

1996; Jilbert et al., 2011). However, Or-P played a minor role in the sedimentary P reservoir in Yuniaohe River sediment examined here. This may be partly relating to the extraction methods used for Or-P. Or-P, measured by SEDEX, merely reflects the refractory organic P. In particular, degradable or soluble organic P may not be included because it would be released to the overlying water through pore water. Given that the refractory organic P is relatively stable, a portion may be flushed from the sediment by tidal currents, allowing transport into the ocean. In addition, mineralization of organic P at the sediment surface would release phosphate to the water column by turbulent mixing (Lee et al., 1977). Thus, Or-P is not the dominant source of P to the sediment surface in this work.

3.2. P variability under different ORP conditions

Prior to our experimental manipulation of the ORP, the average concentration of P in the overlying water was 0.42 mg L^{-1} with average ORP at 272 mv (Fig. 2). In order to investigate the influence of ORP on P release from sediment, the trends of different P concentrations with ORP in overlying water are presented in Fig. 3. It can be seen from Fig. 3, the concentration of P increases with the ORP decrease, and that the concentration of P and ORP has a significant linear correlation (r — 0.8934, $P < 0.05$). These results clearly indicate that P release is strongly influenced by the variation of ORP.

Generally, reducing conditions may lead to diminished Fe(III) (oxyhydr)oxide concentrations. Under such conditions, it is expected that the Fe-P fraction could be released to the overlying water because of the reductive dissolution of Fe(III) (oxyhydr)oxide in sediment (House and Denison, 2000). At the same time, the inhibition of apatite
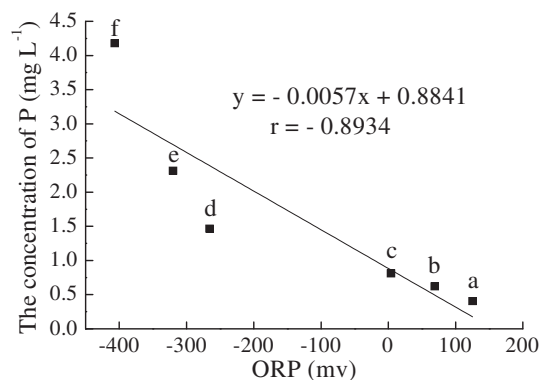

Fig. 3. Correlation between ORP and the concentration of P in the overlying water after different amount $NaBH_4$ addition.
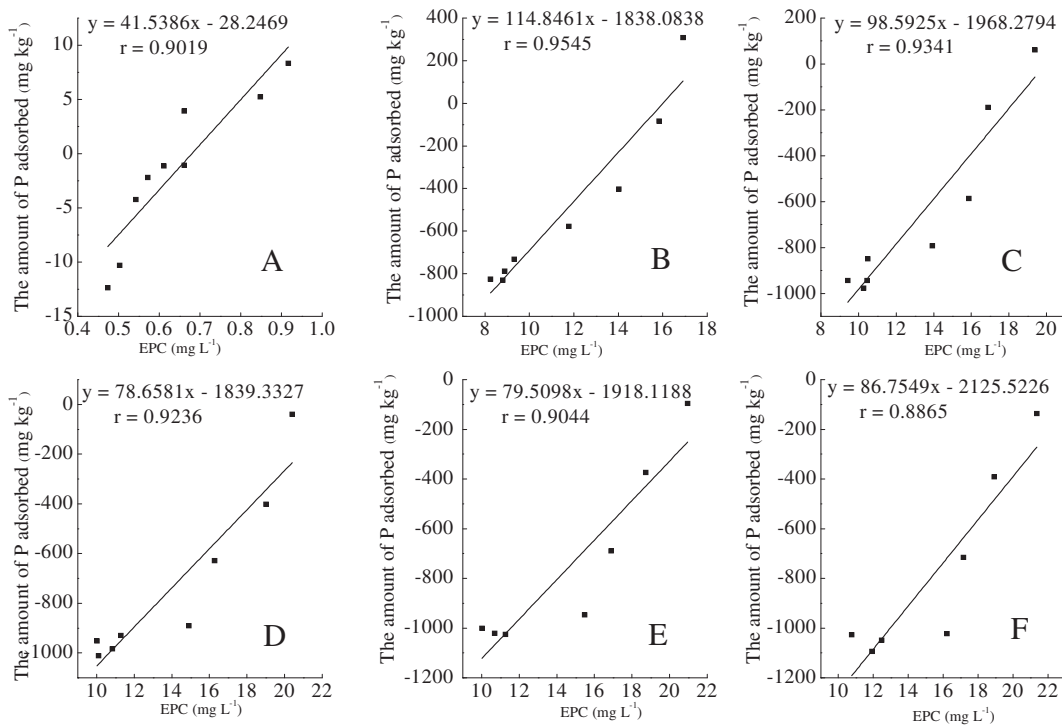
$$y = -0.0057x + 0.8841$$
$$r = -0.8934$$

Fig. 4. Relationship between the EPC and the amount of P adsorbed under different ORP (the average ORP of A–F were 536.59, 415.78, 230.20, 221.43, 182.38, 76.30 mV, respectively).

formation may result in low sorption capacity of reduced sediments and, therefore, the released P may accumulate in the water column (Kraal et al., 2015).

The concentration of P in the beaker *a* (without NaBH$_4$ addition) was comparable to the average P concentration before NaBH$_4$ addition (Fig. 3). This means that there was negligible net adsorption or release of P from sediment from 24 to 48 h. However, after 48 h, the P concentrations in *b–f* beakers were much higher than the average P concentration (0.42 mg L^{-1}) before NaBH$_4$ addition (Fig. 2). This indicates that the experimentally-altered ORP disrupted the original balance between P release and P adsorption. Furthermore, this also hints that ORP may affect the value of EPC$_0$, whereby the EPC$_0$ increases with the ORP decrease.

3.3. The effects of ORP on EPC$_0$ and the distribution P fractions

In order to investigate the P release mechanism from sediment, the relationship between the equilibrium P concentration (EPC) and the amount of P adsorbed under different ORP (Fig. 4), correlation between ORP and EPC$_0$ (Fig. 5) and the distribution of P fractions in the surface
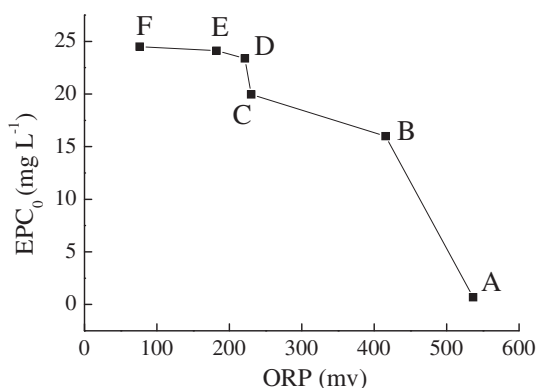
sediments after NaBH$_4$ addition (Fig. 6) were plotted. From Fig. 5, it can be seen that the EPC$_0$ decreases significantly as the ORP increases. Generally, low EPC$_0$ indicates a high affinity of sediments to sorb P, and vice versa, the sediments with higher EPC$_0$ have a higher potential to release P (Agudelo et al., 2011). After adding NaBH$_4$, the decreased ORP may result in the EPC$_0$ increase. This leads to the EPC$_0$ being greater than the P concentration in the overlying water, thereby causing the sediment to act as a P source to the aqueous-phase (Kunishi et al., 1972). As a result, the concentration of P in the overlying water increased significantly with the ORP decrease.

In Table 3, NAP represents native adsorbed P at the start of the experiment, Q is the amount of P adsorbed during the experiment, K is a sorption constant reflecting the sorption affinity of the sediment for P, α is an empirical constant. EPC$_0$ was calculated as the concentration of P at Q = 0 mg kg^{-1} (Kraal et al., 2015).

Fig. 6 shows that, after NaBH$_4$ addition, P was still predominantly present as Fe-P, with minor contributions from ACa-P, E&L-P, and Or-P. This suggests that the change of ORP may have caused insufficient bulk P release to cause a quantifiable change in the solid-phase P
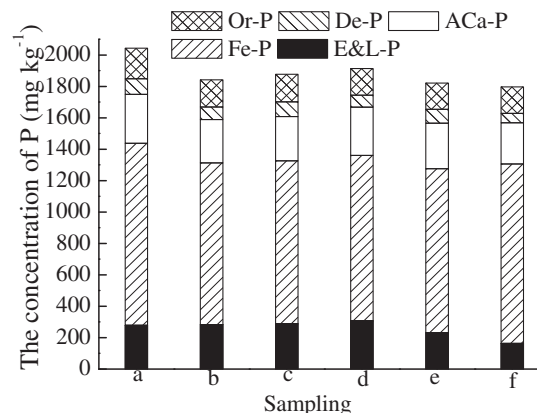

Fig. 5. Correlation between ORP and EPC$_0$.

Fig. 6. The distribution of P fractions in the surface sediments after NaBH$_4$ addition.

Table 3
The P adsorption equilibrium concentrations and parameters of its adsorption isotherms by linear equation on sediments from the Yuniaohe River estuary.

	Equation	EPC_0 (mg L^{-1})	r	NAP (mg kg^{-1})	K (L kg^{-1})	α
A	y = 41.5386 x - 28.2469	0.680	0.9019	28.2469	41.5386	1
B	y = 114.8461 x - 1838.0838	16.005	0.9545	1838.0838	114.8461	1
C	y = 98.5925 x - 1968.2794	19.964	0.9341	1968.2794	98.5925	1
D	y = 78.6581 x - 1839.3327	23.384	0.9236	1839.3327	78.6581	1
E	y = 79.5098 x - 1918.1188	24.124	0.9044	1918.1188	79.5098	1
F	y = 86.7549 x - 2125.5226	24.500	0.8865	2125.5226	86.7549	1

forms in sediments. Furthermore, the content of E&L-P, Fe-P, ACa-P, De-P, and Or-P in sediments decreased with the ORP decrease, resulting in P increase in the overlying water.

Generally, Fe-P is relatively sensitive to alterations in ORP as well as pH. Under reducing conditions, Fe oxides will undergo reductive dissolution, causing release of Fe(II) and a loss of P sorption sites (House and Denison, 2000). However, the results of this study show that there is no obvious relationship between ORP and Fe-P. This may be related to the high concentration of Fe(III) (~6200 mg kg^{-1}, Table 2) in sediment. In addition, the interaction of P with sediments can occur via a reversible two-step sorption process: 1) adsorption/desorption on surfaces with fast kinetics (minutes-hours); 2) solid-state diffusion of adsorbed phosphate from the surface into the interior of particles with slow kinetics (days-months) (Froelich, 1988). Reactive iron oxide minerals have a high capacity for absorbing phosphate in the second step, possibly resulting in similar concentrations of Fe-P during the ORP changing process.

3.4. The effects of sulfide addition on P release

In order to investigate the influence of sulfide addition on P release from sediment, the relationship between P concentrations and sulfide addition and the variations of ORP and Fe^{2+} concentration with sulfide addition in overlying water are plotted in Fig. 7. The 24 h equilibrated conditions reached without added sulfide were average concentrations of P and Fe^{2+} equaling 0.66 and 0.38 mg L^{-1}, respectively (Fig. 6, left). It can be seen from Fig. 7 (left) that the concentration of P increased with sulfide, and that the concentration of P and sulfide therefore have a significant linear correlation (r 0.9916, $P < 0.001$). This clearly indicate that the addition (or in-situ generation) of sulfide enhances P release from sediments. At the same time, the concentration of Fe^{2+} increases with the ORP decrease (Fig. 7, right). Furthermore, a significant

linear correlation also exists between P concentrations and Fe^{2+} concentrations in overlying water.

The results in these tests support the hypothesis that low ORP enhances P release from sediment. In this work, when the sulfide dosage increased, ORP would be decreased thereby driving the observed P release along with the release of Fe^{2+} via reductive dissolution of Fe(III) oxides. During this process, increased sulfide may react or capture reactive iron (with different forms) in sediments as Fe sulfide phases, reducing the P adsorption capacity and enhancing the P release.

4. Conclusions

Fe-P is the main P burial phase in coastal sediments of the Yuniao River estuary, China. The P concentration in overlying water increased with ORP decrease and a significant linear correlation was evident between ORP and the concentration of P. This result indicates that decreased ORP may elevate the value of EPC$_0$, enhancing the capability of P release from sediments. The results also indicate that increased sulfide may react with reactive iron (with different forms) in sediments, reducing the P adsorption capacity and accelerating P release. Therefore, the control of ORP and sulfide production is important in the sink/source conversion of P in coastal sediments.
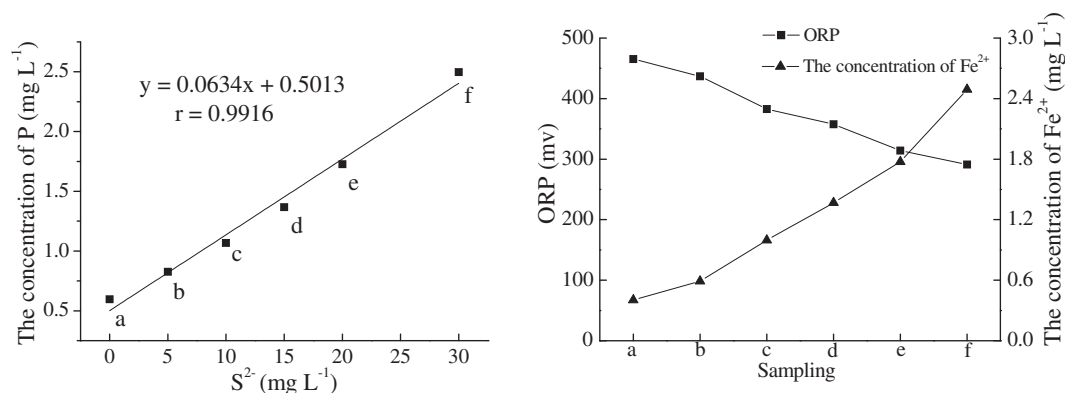
Fig. 7. The relationship between P and sulfide concentrations (left) and variations of ORP and Fe^{2+} after sulfide addition (right).

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