Phosphorus fractions and matrix-bound phosphine in coastal surface sediments of the Southwest Yellow Sea

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ARTICLE INFO

ABSTRACT

This paper characterizes the distribution of phosphorus fractions and matrix-bound phosphine (MBP) in coastal surface sediments of the Southwest Yellow Sea from 2006 to 2007. Total phosphorus (TP), inorganic phosphorus (IP) and organic phosphorus (OP) concentrations ($mg\,kg^{-1}$) range from 278 ± 3 to 768 ± 15, 160 ± 1 to 653 ± 27, and 3.42 ± 0.05 to 267 ± 22, respectively. MBP is a small portion of TP with values of 0.69 ± 0.06 to $179 \pm 29\,ng\,kg^{-1}$. Phosphorus fractions and MBP are influenced strongly by riverine input and hydrodynamic conditions. High TP and MBP are found in the old Yellow River mouth and the Yangtze River mouth. OP and MBP are strongly negatively correlated to mean particle size. Significant positive correlations are found between MBP and IP and OM, suggesting that MBP production may be the microbially intermediated transformation of IP.

1. Introduction

Phosphorus plays an important role in primary production in marine systems. Transportation and transformation of phosphorus affects the sustainability of coastal ecosystems, which may lead to significant ecosystem responses in the marine and coastal zone over long periods [1,2]. The coastal zone plays a significant role in the phosphorus biogeochemical cycle because almost all land-derived materials enter this region. Extensive terrestrial inputs of phosphorus to the coastal zone lead to high primary production [3] and harmful algal blooms (HABs). Given the importance of phosphorus cycling to marine ecosystems, more work is necessary to clarify the distribution of phosphorus in the coastal zone.

To date, limited research has been conducted to investigate the fate of phosphine (PH_3) in marine sediments. PH_3 is a natural gaseous carrier of phosphorus in biogeochemical cycling. It is also a highly toxic gas that produces acute lethal effects on humans through inhibition of aerobic respiration [4]. The 4 h LC50 of PH_3 in rats is $\sim 15\,mg\,m^{-3}$ and the time-weighted average exposure standard is $0.4\,mg\,m^{-3}$ [5]. Thus, PH_3 in ocean waters can be acutely toxic to aquatic organisms [6]. PH_3 exists in the environment in two different forms: free gaseous PH_3, and matrix-bound PH_3 (MBP).

Free gaseous PH_3 is defined as the PH_3 presented freely in gas samples, which is found worldwide in the environment. MBP is defined as the amount of PH_3 released from condensed environmental samples (such as sediments and animal manure) during an acidic or alkaline digestion [7].

Although PH_3 is detected universally in the natural environment, the origin of PH_3 is still debated [6]. The solid environmental bio-media used as inocula in experiments to produce free PH_3 include usually abundantly MBP [8]. PH_3 produced during fermentation is prone to adsorption by the condensed matrix, and the actual amount of PH_3 released in free gas form is much less than the total PH_3 formed in sediments [9]. Understanding the behavior of MBP is necessary before that of PH_3 can be determined.

The Yellow Sea, the third largest marginal sea along China's coastline, is a semi-enclosed sea in the northwestern Pacific Ocean with an average depth of 44 m. The Southwest Yellow Sea is a phosphorus-limiting area with a high N/P ratio [10,11]. Although some research on phosphorus distribution in the South Yellow Sea has been conducted [11,12], there has been little research on the distribution of phosphorus fractions, especially MBP in the coastal zone. There is still a lack of information concerning the formation of PH_3 in the marine environment.

In this paper, the Southwest Yellow Sea was chosen to characterize phosphorus fractions and MBP. The aims of this research are: (1) to study the seasonal and spatial distribution of phosphorus fractions and MBP in surface sediments; (2) to investigate the

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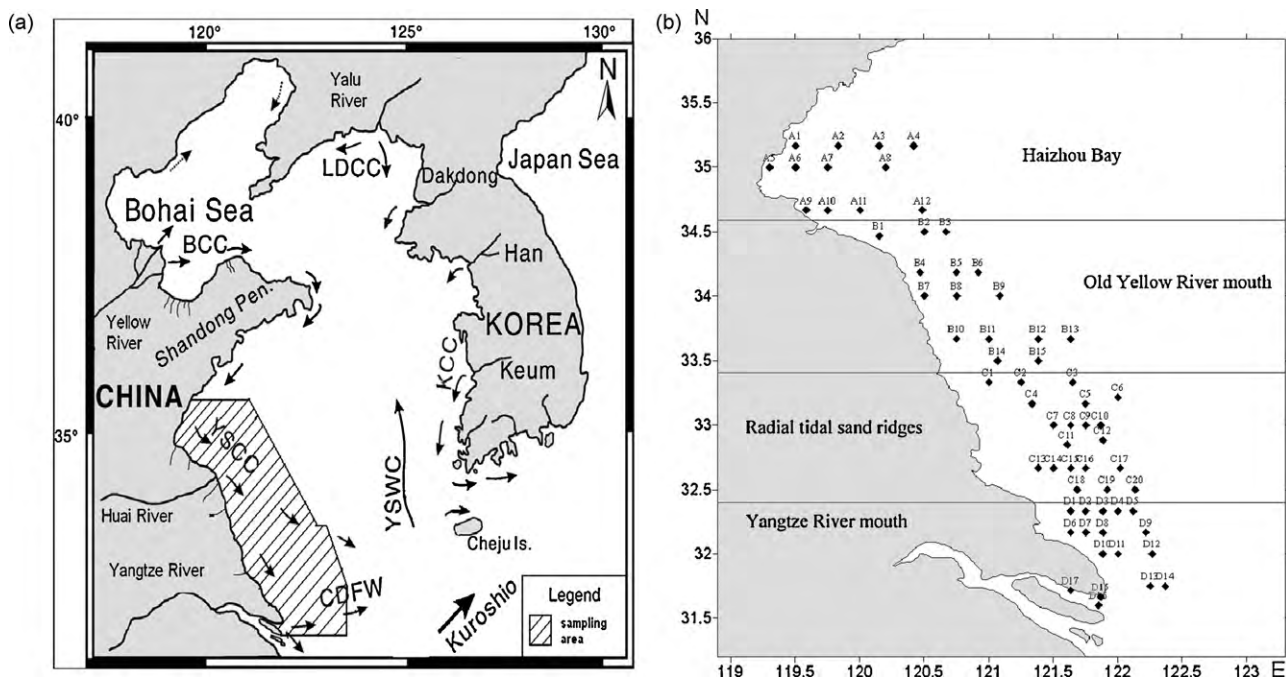
Fig. 1. (a) Sketch map of the Yellow Sea showing sampling areas, and the oceanic circulation pattern [13]. YSCC: Yellow Sea Coastal Current; YSWC: Yellow Sea Warm Current; CDFW: Changjiang Diluted Freshwater. (b) Location of sampling sites from the coastal zone of the Southwest Yellow Sea.

environmental factors affecting phosphorus fractions and MBP; and (3) to identify possible precursors responsible for the production of MBP in marine sediments. The results of this study contribute to further research on the origin of PH_3 and phosphorus biogeochemical cycling in the marine ecosystem.

2. Experimental

2.1. Study area and sampling procedures

Fig. 1a illustrates the marine environments in the sampling area. The Yellow Sea Coastal Current (YSCC, also termed the "Tidal Current") flows from the North Yellow Sea to the Yangtze River mouth and meets the Changjiang Diluted Freshwater (CDFW). The YSCC transports suspended particles from the Yellow River [13,14]. Based on different geographic and hydrodynamic conditions, the sampling areas were divided into four regions (Table 1).

From December 2006 to January 2007 (winter, atmospheric temperature $6.7 \pm 2.6\,°C$), surface sediments at 38 sites in the coastal zone in the Southwest Yellow Sea (from B_{10} to D_{17} in Fig. 1b, except sites C_1, D_4, D_9 and D_{13}) were sampled with a Peterson Sampler. From April to May 2007 (spring, atmospheric temperature $12.9 \pm 4.3\,°C$) and October to November 2007 (fall, atmospheric temperature $21.5 \pm 3.9\,°C$), surface sediments were sampled at all 64 sites. All samples were stored in the dark at $0–4\,°C$.

2.2. Determination of phosphorus fractions

Sediment phosphorus was measured as total phosphorus (TP), inorganic phosphorus (IP) and organic phosphorus (OP) fractions. TP was detected by measuring phosphate using the molybdenum-antimony spectrophotometric method after digestion [15,16], in which all samples were dried at room temperature, then ashed in a muffle furnace at $550\,°C$ for 2 h, followed by extraction with $1\,mol\,L^{-1}$ HCl for 16–18 h at room temperature, including 2 h vibration. IP was analyzed by the same method as TP, excepting the ashing procedure. The difference between TP and IP represents OP.

2.3. Determination of MBP

In this paper, MBP is defined as the quantity of gaseous PH_3 released after treatment of the sediment with sulfuric acid. This includes adsorbed PH_3, metal-PH_3 complexes, and inorganic phosphides. Sediment was digested with H_2SO_4, and the liberated PH_3 gas was determined by GC-NPD. For details see references [17,18].

2.4. Determination of organic matter and particle size

To determine organic matter (OM) content, samples were dried, sieved, acidified with $K_2Cr_2O_7$ solution and 98% H_2SO_4, followed by heating for 90 min at $100\,°C$, then measured using spectrophotometric analysis [19]. Sediment particle size distributions were measured by a Malvern Mastersize 2000 laser granulometer (Malvern Instruments Ltd., United Kingdom).

2.5. Data analysis

Each sample was analyzed at least three times. The results were calculated using mass concentration on a dry weight basis. Correlation analyses were performed using standard statistical methods in SPSS software.

To characterize spatial distribution of phosphorus fractions, Surfer 8 (Golden Software Inc.) was used to draw isoline charts. These are based on variable values, latitude and longitude of sampling sites, and the base map covering the Southwest Yellow Sea.

3. Results and discussion

3.1. Distribution of TP, IP, OP and MBP

#### 3.1.1. TP in coastal surface sediments of the Southwest Yellow Sea

TP concentrations range from $278 \pm 3\,mg\,kg^{-1}$ at A1# (Haizhou Bay, fall, 2007) to $768 \pm 15\,mg\,kg^{-1}$ at C3# (RTSRs, winter, 2006) (Fig. 2). Average TP levels decrease in the order: Yangtze River mouth > Old Yellow River mouth > RTSRs > Haizhou Bay (Table 2).

Table 1
Sampling sites within four sea regions.

Sea region	Haizhou Bay	Old Yellow River mouth	Radial tidal sand ridges (RTSRs)	Yangtze River mouth
Sampling sites	A$_1$–A$_{12}$	B$_1$–B$_{15}$	C$_1$–C$_{20}$	D$_1$–D$_{17}$
Latitude and longitude	34°40′–35°10′N	33°30′–34°30′N	32°30′–33°20′N	31°36′–32°20′N
	119°18′–120°28′E	120°9′–121°38′E	121°–122°8′E	122°–121°22′E

High TP concentrations are observed in the Yangtze River mouth and Old Yellow River mouth, both of which are influenced by the CDFW and YSWC. The distribution of TP changes little between seasons (Table 2).

3.1.2. IP in coastal surface sediments of the Southwest Yellow Sea

IP concentrations vary from 160 ± 1 mg kg^{-1} at A1# (Haizhou Bay, fall, 2007) to 653 ± 27 mg kg^{-1} at C3# (RTSRs, winter, 2006) (Fig. 3). IP is the most important mass fraction of TP and repre-
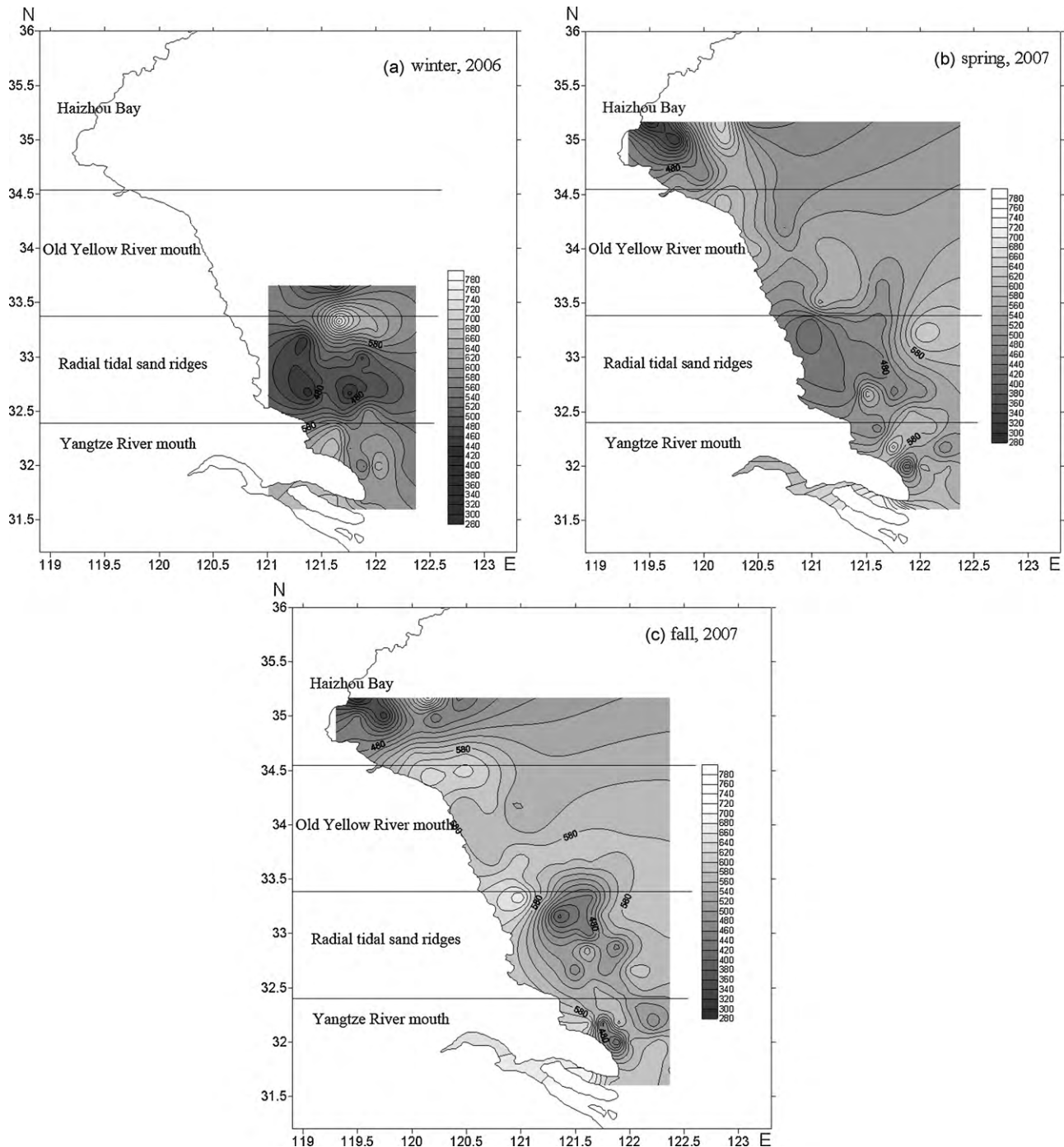
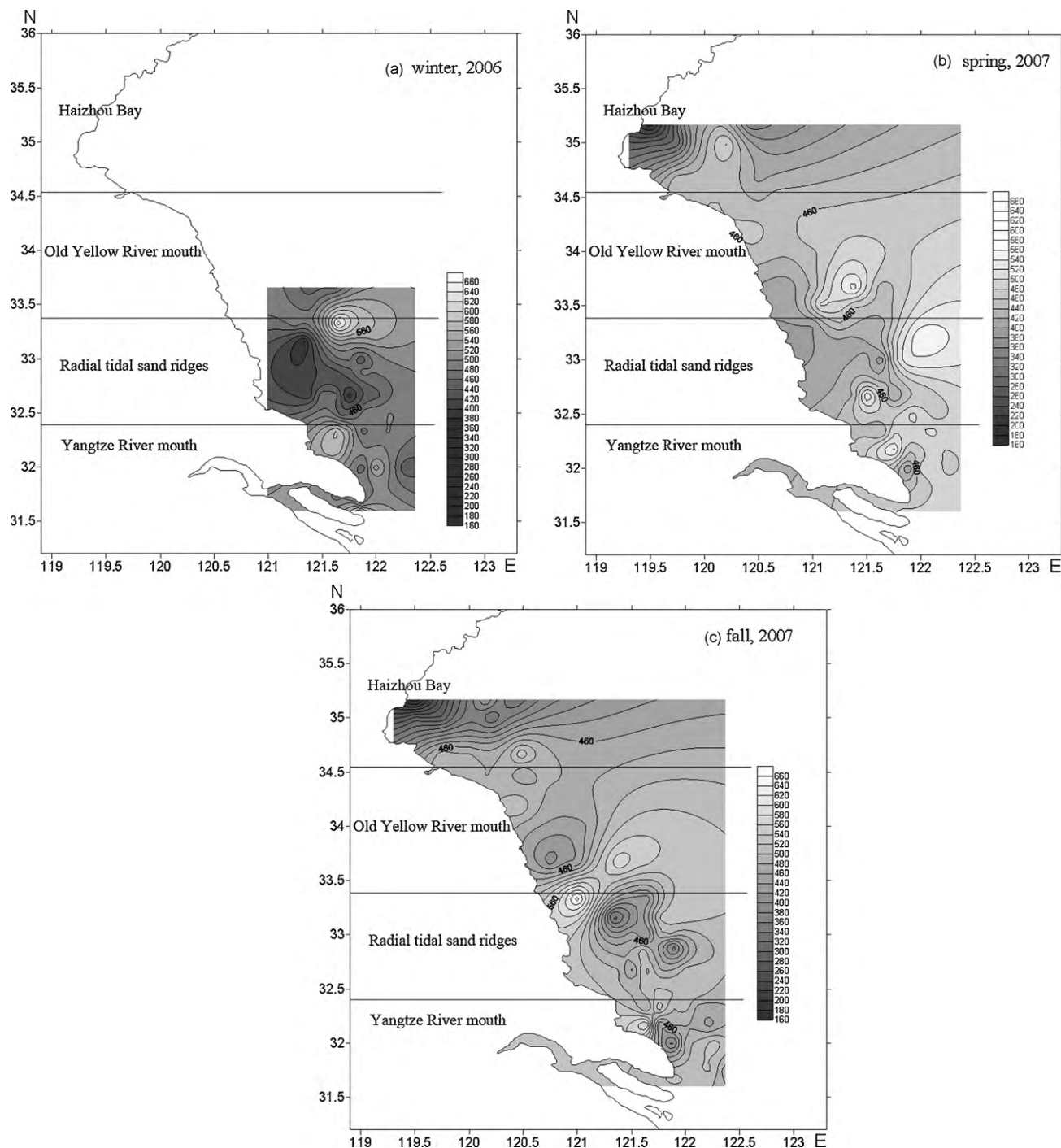**Fig. 2.** Spatial distribution of total phosphorus (mg kg^{-1}) in surface sediments.

Table 2
Average TP concentrations in four different sea regions (mg kg^{-1}).

Sampling season	Sea region				
	Haizhou Bay	Old Yellow River mouth	RTSRs	Yangtze River mouth	Seasonal average
Winter, 2006	–	531 ± 49	527 ± 80	614 ± 43	560 ± 76
Spring, 2007	484 ± 114	550 ± 38	516 ± 55	582 ± 73	534 ± 78
Fall, 2007	467 ± 107	581 ± 445	527 ± 66	557 ± 70	537 ± 79
Spatial average	476 ± 108	561 ± 39	523 ± 67	582 ± 67	–

sents over 85% of TP in coastal surface sediments of the Southwest Yellow Sea. Average IP levels decrease in the order: Yangtze River mouth > RTSRs ≥ Old Yellow River mouth > Haizhou Bay. IP and TP have similar spatial and temporal patterns (Table 3).

3.1.3. OP in coastal surface sediments of the Southwest Yellow Sea

OP concentrations vary from 3.42 ± 0.05 mg kg^{-1} at A7# (Haizhou Bay, spring, 2007) to 267 ± 22 mg kg^{-1} at D17# (Yangtze River mouth, spring, 2007). Average OP levels decrease in the

Fig. 3. Spatial distribution of inorganic phosphorus (mg kg^{-1}) in surface sediments.

Table 3
Average IP concentrations in four different sea regions (mg kg^{-1}).

Sampling season	Sea region				
	Haizhou Bay	Old Yellow River mouth	RTSRs	Yangtze River mouth	Seasonal average
Winter, 2006		472 ± 20	474 ± 62	517 ± 48	490 ± 58
Spring, 2007	370 ± 88	460 ± 37	465 ± 48	483 ± 33	451 ± 66
Fall, 2007	369 ± 110	488 ± 50	489 ± 65.2	496 ± 51	465 ± 84
Spatial average	370 ± 97	474 ± 39	476 ± 59	497 ± 46	

Table 4
Average OP concentrations at 4 different sea regions (mg kg^{-1}).

Sampling season	Sea region				
	Haizhou Bay	Old Yellow River mouth	RTSRs	Yangtze River mouth	Seasonal average
Winter, 2006	–	58.9 ± 32.3	52.8 ± 29.1	97.0 ± 36.9	70.6 ± 38.6
Spring, 2007	114 ± 61.9	89.7 ± 33.9	51.1 ± 22.8	98.8 ± 61.5	98.8 ± 61.5
Fall, 2007	98.2 ± 57.5	93.5 ± 53.0	38.6 ± 21.0	61.7 ± 45.2	61.7 ± 45.2
Spatial average	106 ± 59	86.9 ± 45.2	47.4 ± 24.9	85.1 ± 51.7	–

order: Haizhou Bay > Old Yellow River mouth ≥ Yangtze River mouth > RTSRs (Table 4). The coastal zone of Haizhou Bay has been utilized for fish-farming, and use of excess fertilizer may lead to increased OP concentrations in sediments [20]. Because of the strong hydrodynamic turbulence in Radial Tidal Sand Ridges (RTSRs) zone, the sedimentation environment is poor for phosphorus particles and organic residue, and OP concentration is quite low in this sea region (Fig. 4). The seasonal distribution of OP differs from TP and OP, where higher OP concentration is observed in spring than in winter and fall (Table 4).

3.1.4. MBP in coastal surface sediments of the Southwest Yellow Sea

The lowest concentration of MBP of 0.69 ± 0.06 ng kg^{-1} is observed at A1# (Haizhou Bay, fall, 2007), while the highest of 179 ± 29 ng kg^{-1} is detected at A11# (Haizhou Bay, fall, 2007) (Fig. 5). MBP concentrations in sediments decline in the following order: Old Yellow River mouth > Haizhou Bay ≈ Yangtze River mouth > RTSRs (Table 5). Higher seasonal MBP concentrations in sediments are found in fall than in spring or winter. Compared with other phosphorus fractions MBP is a small fraction of TP, although it is an important intermediate carrier in the phosphorus biogeochemical cycle [7,18].

3.2. Effects of riverine input and hydrodynamic conditions on phosphorus distribution

Riverine input and hydrodynamic conditions may be the important factors in changing potential phosphorus levels in the coastal zone of the Southwest Yellow Sea. High TP and MBP are found in the old Yellow River mouth and the Yangtze River mouth. Old Yellow River mouth is the confluence of CDFW and YSWC, a branch of Kuroshino Current, which brought rich nutrient (Fig. 1a) [21].

Phosphorus is a limiting factor for marine biological productivity in the Southwest Yellow Sea. HABs frequently occur in the Yangtze River mouth, where phosphorus-rich matter is input by large rivers [21,22]. Considering the increased utilization the Southwest Yellow

Sea's coastal zone, increasing anthropogenic phosphorus input may lead to HAB risk in the old Yellow River mouth. Phosphorus load should be controlled in the old Yellow River mouth, where TP is relatively high.

3.3. Effects of mean particle size and OM on phosphorus distribution

Negative relationships exist between mean particle size and TP, OP, suggesting that the distribution of TP and OP may be affected by particle characteristics (Table 6). This may be explained by the capacity, efficiency and rate of phosphate sorption by sediments increasing as particle size decreases [23]. The correlation between OP and particle size is higher than that between TP and particle size, indicating that particle size may impact mainly on OP transportation.

A significant negative correlation between MBP concentrations and mean particle size is also observed (Table 6), suggesting that finer particles contain higher MBP concentrations. In general, finer particle size means larger specific surface area, which can increase MBP adsorption to particles [24–26]. On the other hand, coarse particles are porous and potentially aerated, which enhances the release and oxidation of PH$_3$ [27]. Furthermore, the occurrence of finer particles indicates lower disturbance intensity in the marine environment, which decreases MBP release from sediments [9].

The strong positive correlation between OM and OP may be linked to organism activities. A higher correlation coefficient is observed between OP and OM than between OP and TP, indicating that OM plays a more important role in OP distribution in sediments.

3.4. Potential precursors for MBP production

The origin of PH$_3$ in nature is still under debate. PH$_3$ evolution has been reported from microbial habitats [6]. It is hypothesized that anaerobic bacteria reduce organic phosphorus compounds or inorganic phosphate to PH$_3$. However, no correlation between

Table 5
Average MBP concentrations in four different sea regions (ng kg^{-1}).

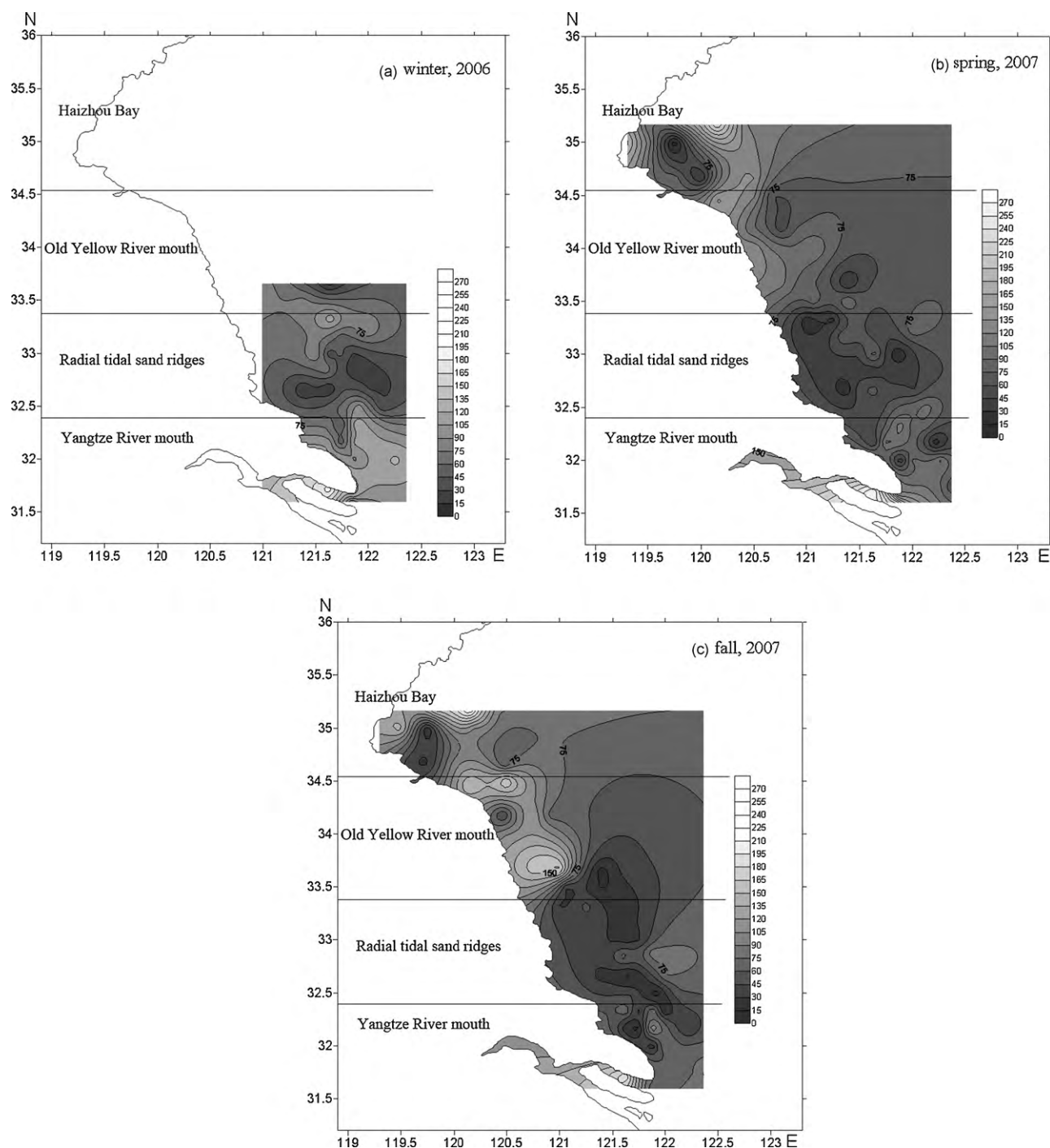| Sampling season	Sea region				
	Haizhou Bay	Old Yellow River mouth	RTSRs	Yangtze River mouth	Seasonal average
Winter, 2006	–	15.0 ± 11.2	12.2 ± 8.2	20.8 ± 11.5	15.7 ± 10.4
Spring, 2007	25.2 ± 31.5	44.6 ± 30.2	17.7 ± 13.2	29.3 ± 15.2	28.0 ± 23.9
Fall, 2007	41.7 ± 63.2	53.7 ± 55.3	28.2 ± 33.2	47.6 ± 29.5	42.8 ± 44.0
Spatial average	33.4 ± 49.6	44.3 ± 39.7	19.5 ± 22.0	33.3 ± 23.2	–

Fig. 4. Spatial distribution of organic phosphorus (mg kg^{-1}) in surface sediments.

Table 6
Pearson correlation coefficients between different parameters.

	TP	IP	OP	MBP	OM	Mean particle size
TP	1					
IP	0.794**	1				
OP	0.441**	−0.196*	1			
MBP	0.253**	0.218**	0.086	1		
OM	0.292**	−0.010	0.485**	0.374**	1	
Mean particle size	−0.156*	0.037	−0.307**	−0.327**	−0.620**	1

* Correlation is significant at the 0.05 level (2-tailed), $n = 166$ ($n = 164$ for Pearson correlation between mean particle size and other parameters).

** Correlation is significant at the 0.01 level (2-tailed), $n = 166$ ($n = 164$ for Pearson correlation between mean particle size and other parameters).
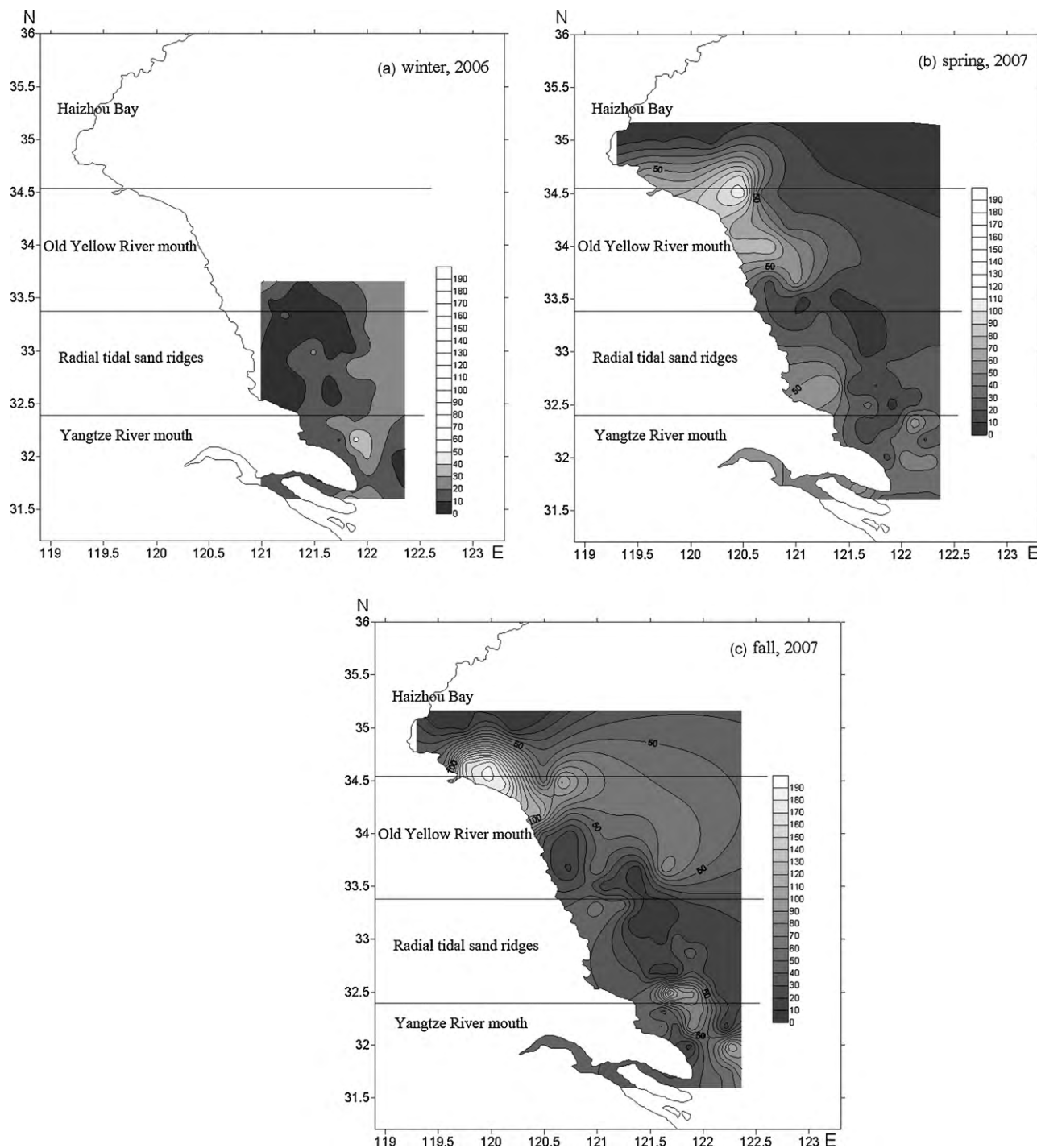
Fig. 5. Spatial distribution of MBP (in ng kg^{-1}) in surface sediments.

characterized microorganisms and PH$_3$ production has been established. PH$_3$ is also supposed to be produced by corrosion of phosphorus-containing metal or mechanochemical reactions of apatite-bound phosphate. Possible precursors and origins for PH$_3$ are summarized in Table 7.

The correlation coefficients in Table 6 show that IP ($r = 0.218$, $p = 0.005$, $n = 166$) rather than OP ($r = 0.086$, $p = 0.268$, $n = 166$), has a significant positive relationship with MBP, suggesting that IP is a more likely precursor for the production of MBP in marine sediments. Other laboratory simulation experiments have found that

adding additional inorganic phosphate enhances the release of PH$_3$ [28–30]. However, it remains unclear how inorganic phosphorus is transformed into PH$_3$.

A significant positive correlation between MBP and OM ($r = 0.374$, $p < 0.005$, $n = 166$) is found, indicating that active microbial metabolism is correlated to MBP production. In coastal sediments, OM is a potential source of carbon and energy for microorganism metabolism. High OM concentrations reflect active microbiological processes correlated to PH$_3$ production [25,39]. It is reasonable to hypothesize that MBP in marine sediments is formed

Table 7
Possible precursors and origins for PH_3.

Possible precursors	Possible origins	Experimental proofs	Reference
IP			
$Na_2HPO_4 \cdot 2H_2O$, K_2HPO_4	Biological reduction	PH_3 gas detected in anaerobic bacterium culture grown on inorganic P-compounds	[28]
Phosphate	Biological reduction	PH_3 production increased significantly in marsh soil by adding additional phosphate and peptone	[29]
K_2HPO_4	Biological reduction	PH_3 accumulation was stimulated by increasing phosphate concentration of the medium	[30]
K_2HPO_4	Biological reduction	Pure cultures of bacteria generated PH_3 gas	[4]
Phosphate	Corrosion	Phosphate in the yeast extract was reduced by *Desulfovibrio desulfuricans* to PH_3 during corrosion experiment	[31]
Iron phosphide	Corrosion	Iron phosphide present in iron hydrolyzed to PH_3 during corrosion	[32]
P in steel	Corrosion	Conditions favoring iron corrosion induced higher emissions of PH_3	[8]
Apatite-phosphate	Mechanochemical reduction	Rubbing apatite-phosphate led to detection of more than $70\,000\ ng\,kg^{-1}$ PH_3	[33]
OP			
Chicken manure, bone powder, lectin	Biological reduction	PH_3 emissions from sediments increased after addition of chicken manure, bone powder and lectin under laboratory conditions	[34]
Phosphonoacetic acid (P(O)(OH)2CH2COOH)	Biological reduction	PH_3 emissions from paddy soils increased after addition of phosphonoacetic acid	[35]
Organic phosphorus	Biological reduction	MBP was correlated significantly to OP in marine sediment	[25,36]
Uncertain phosphorus			
Human feces	Biological reduction	Large increase in PH_3 content was observed after incubating PH_3-free medium, inoculated with human feces	[37]
Manure	Biological reduction	PH_3 release from maize fields was stimulated by additions of manure, glucose, formate, pyrogallol and sulfide under laboratory conditions	[38]
Apatite-bound phosphate, OP	Mechanochemical and biological reduction	MBP was correlated significantly with apatite-bound phosphate and labile OP in intertidal sediments	[27]

by microbial IP reduction. Further studies should evaluate the origin and precursors responsible for PH_3 production in the study area.

4. Conclusions

In the surface sediments of the coastal zone of the Southwest Yellow Sea, concentrations $(mg\,kg^{-1})$ of TP, IP and OP ranged from 278 ± 3 to 768 ± 15, 160 ± 1 to 653 ± 27, and 3.42 ± 0.05 to 267 ± 22, respectively. IP is the most important mass fraction of TP, while MBP is a small fraction of TP with values of 0.69 ± 0.06 to $179 \pm 29\ ng\,kg^{-1}$. Seasonal distributions of phosphorus fractions change little. High TP and MBP occur in regions influenced by riverine input and marine currents. Finer particles contain higher OP and MBP concentrations. MBP may be produced from microbial reduction of inorganic phosphorus.

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