Assessment of phosphorus release from alluvial sediments using single extraction: a case study in Can Gio, Southern Vietnam

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Abstract:

Sediment-water interaction is a complicated process; hence, it is essential to know the potential mobility of an element from sediment to the aquatic environment. In this study, the potential mobility of phosphorus (P) in sediment from Can Gio was investigated. Three single extractions were applied including a mild salt (NH$_4$Cl), an acid (HCl), and a base (NaOH) each representing different environmental factors during sediment resuspension. Other geochemical parameters were also determined. The total amount of P in the sediments varied from 320.41 mg/kg to 668.22 mg/kg in five sampling sites. The general range of potential mobility of P decreased as follows: P-mild (3.70-13.13 mg/kg) < P-base (36.80-78.53 mg/kg) < P-acid (133.85-380.57 mg/kg). Although P-acid was not easily released to the environment, its relatively high concentration can affect the aquatic environment when the environment becomes acidic.

Keywords: **phosphorus release, sediment, sediment resuspension, single extraction.**

Classification number: **5.1**

Introduction

In natural ecosystems, phosphorus (P) is not only a major nutrient controlling eutrophication but it is also a limiting nutrient in many aquatic systems [1, 2]. Besides aquatic plants and algae, sediment is considered as the main contributor of P to the aquatic system from internal sources [3]. Phosphorus can exist in several forms in sediments with different mobilities, however, not all the fractions of P in sediments are likely to be released and become bioavailable in a water environment [4]. Knowledge of the potential release of P from sediment is useful since it can serve as indicators of the potential of P loading contributions to the water environment [3]. Fractionation of P by sequential extractions are often used to evaluate the potential release as well as the origin of P in the sediment [4, 5]. There are several extraction tests for sediments, including single extractions and sequential extractions, each with different purposes and implications. However, sequential extractions, which are laborious and time consuming, will not be applied in the present study. Single extractions with reagents representing different environmental conditions that the sediments might encounter during resuspension is a fast, simple, and relatively cheap way to assess the potential release of elements from sediments to the water environment [6]. Hence, it is recommended as a useful tool for water managers on a routine basis to calculate the releasable P stock in sediment [4].

Can Gio, a coastal district of Ho Chi Minh city, has a large area covered by a mangrove forest that was designated as a Biosphere Reserve by UNESCO [7]. As home to a tropical mangrove ecosystem, Can Gio is a dynamic transitional coastal ecosystem where natural and anthropogenic impacts are mixed [8]. The major sources of nutrients in this area are the decomposition of organic matter by litter mineralization in the mangrove forest, effluents from aquaculture activities, and municipal sewage [9]. As a transitional ecosystem, mangrove is also considered as a barrier retaining nutrients (N and P) [10]. However, mangrove sediments can also act as a source of nutrients for the water environment due to mineralization [11]. Recently, some work has been done to understand the behaviour of nutrients in the Can Gio mangrove

system. However, their research was focused on evaluating the nutrient status of reforested and natural mangrove stands with different site-specific species [12] or the influence of vegetation, tidal cycles, and seasonality to the dissolved inorganic nutrient concentrations in porewater [9].

The objective of this research is to assess the potential release of P during the resuspension of sediments in the Can Gio area. Moreover, instead of simply using the total P (TP), this study applies the single extraction to provide information about the fractions that compose the P load. By using single extraction, this study aims to prove that single extraction can be a valuable tool for environmental management. This kind of study can provide information about the role of the internal release of P from the sediment, which is helpful to local managers in terms of water quality management since most of the water quality protection policy has put the focus on reducing the external source without considering an internal source from sediment.

Materials and methods

Sampling

The study area is located in Can Gio district, Ho Chi Minh city, Vietnam. Surface sediments (approximately 1 kg each) were collected in the dry season in 2017 (Fig. 1). Sampling sites S1, S2, and S3 were located in a densely populated shrimp farming settlement. On the other hand, sampling sites S4 and S5 were located in the core zone of the mangrove forest. To get a good representation of the system, a sample was composed of 5 sub-samples at each sampling site. After collection, the samples were placed in sealed plastic bags and transported to the University of Science (Ho Chi Minh city, Vietnam) in a cooler box. The samples were air-dried, homogenized in a porcelain mortar, and sieved over a 2 mm mesh sieve.

Fig. 1. Map of the sampling sites in the studied area (map was modified based on the satellite image provided by Google Earth, 2020).

General sample characterization

Organic carbon (OC) was inferred from the organic matter determined by the Walkley and Black manual titration method [13]. Grain size distribution was determined by sieving and hydrometer methods [14]. The pH of each sample was measured in water (1:5 ratio w/w). The total elemental (Al, Ca, Fe, K, Mg, P) content of the samples were determined after digestion by the so-called 3 acids digestion method (HNO_3, $HClO_4$, HF). About 0.5 g of dried sediment sample was digested with an acid mixture containing HCl_{cc} (4 ml), HNO_{3cc} (2 ml), and HF_{cc} (2 ml) in a Teflon beaker on a hot plate. After digestion, the residue was dissolved with HCl 2.5 mol/l. The major elements (Al, Ca, Fe, K, Mg) in the digested solutions were analysed with ICP-OES (Perkin Elmer Optima 7300 DV) while P was determined by the molybdate blue method of Murphy and Riley (1962) [15].

Single extraction

Single extraction was carried out by three independent batches with different reagents and conditions. A mild salt (1 M NH_4Cl), an acid (0.5 M HCl), and a base (0.1 M NaOH) [16] were used to assess the three forms of phosphorus existing in the sediment i.e. P-Ca/Mg (apatite P), P-Al/Fe (non-apatite P), and weak bonding P. The original extraction scheme was a sequential protocol and developed by Hieltjes & Lijklema (1980) [17]. However, it was modified to include different independent steps that are not sequential as in [16]. This single extraction was applied in the present study since it is simple and practical [4]. About 1 g of dried sediment was weighed into a centrifuge tube and then 10 ml of 1 M NH_4Cl was added. The sediment was extracted by shaking for 2 h. All the supernatant was collected and another 10 ml of 1 M NH_4Cl was added to the residue. This mixture was shaken by another 2 h. The solution was centrifuged at 3500 rpm for 10 min. The latter and the first solution were mixed together and measured for weak bonding P (P-NH_4Cl). Another 0.1 g portion of the dried sediment was weighed into a centrifuge tube, then 5 ml of 0.5 M HCl was added and the solution was shaken for 24 h. On the following day, the collected supernatant was measured for apatite P (P-HCl). For the base extraction, 10 ml of 0.1 M NaOH was added to 1 g of sediment and shaken for 17 h. The supernatant was measured for non-apatite P (P-NaOH). All the extractions were carried out at room temperature. After each extraction step, the samples were centrifuged (3000 rpm for 10 min) and the phosphorus in the extracts was determined accordingly. All P forms were expressed as the amount of P in milligrams per kilogram of sediment (mg/kg) taking into account the volume of extraction solvent for each extraction.

Quality assurance and quality control

All reagents were of analytical grade. For each sample, duplicate (total element concentrations and single extractions) or triplicate (pH and organic matter) analyses were performed to ensure the analytical precision and reproducibility. Results are displayed as the averages of the replicates. Duplicate blanks were also inserted into each batch.

Results and discussion

Sediment characteristics

General characteristics (fine fraction, pH, organic matter): the average of the fine fraction (FF) (≤63 µm, silt and clay), OC,

pH, and elemental concentrations in the sediment are presented in Table 1. Grain size analysis revealed the dominance of fine fraction (>90%) and the values were rather uniform between the sampling sites. The pH values in the sediments indicated an acidic to neutral medium (6.30-7.30). The amount of organic carbon in sediments varied from 2.24 to 3.66% and exhibited slightly higher values in the core zone of the mangrove forest (S4-S5). This could be explained by the high biological productivity in sediments associated with mangrove [18].

Table 1. The average of the fine fraction (FF) (≤63 µm, silt and clay), OC, pH, and elemental concentrations in the sediment.

Parameter	Unit	Site						
		S1	*S2*	*S3*	*S4*	*S5*	*Average S1-S3*	*Average S4-S5*
pH		6.30	6.50	6.37	7.30	7.29	6.39	7.29
OC	%	2.24	2.90	3.30	3.66	3.04	2.81	3.35
FF	%	99.62	98.92	97.90	90.18	97.93	98.81	94.06
Al	g/kg	77.2	79.5	81.2	61.3	50.9	79.3	56.1
Fe	g/kg	43.4	44.8	42.0	39.3	44.9	43.4	42.1
Ca	g/kg	3.8	2.4	2.5	3.5	1.9	2.9	2.7
Mg	g/kg	13.3	11.7	10.9	11.1	8.1	11.9	9.6
TP	mg/kg	668	575	431	645	320	558	483
OC:TP		34	50	77	57	95	53	76

Major element and total phosphorus: among the sampling sites, the concentration of Fe in the sediments did not change much from 39.3 to 44.9 g/kg (Table 1), while the Al concentration varied over a much wider range from 50.9 to 81.2 g/kg. The sediment at sites S1-S3 had a higher content of fine fraction and Al in comparison to S4-S5, which suggests higher content of clay minerals in S1-S3 since Al is considered representative for clay minerals [19]. Data of Al in Can Gio area is rather limited making it difficult to compare. However, Al content was slightly higher than those reported in other studies in the mangrove forests of other countries [1]. Iron content was in the same range of those in other studies conducted in particulate sediments in Can Gio [20] as well as other mangrove forest sediments [1]. Generally, Ca concentrations were very low with a minimum of 1.9 and a maximum of 3.8 g/kg. The concentration of TP in the sediments were moderately different from each other and ranged from 320 to 668 mg/kg (Table 1). Total phosphorus in the sediments was higher than those observed in the previous study carried out in the Can Gio area (87-306 mg/kg) [21] but more or less in a similar range to another study in a mangrove forest in India (360-550 mg/kg) [18]. The higher content of TP in the present study might be attributed to the completed digestion of the samples by using a mixture of HCl_{cc}, HNO_{3cc}, and HF_{cc}. The use of HF is believed to digest minerals associated with a silicate matrix, which is difficult to be digested using other acids and thus results in higher TP [22].

Single extractions

The results from the single extractions are presented in Fig. 2. Apatite P was the main form of P in all the investigated sediments,

which ranged from 133.85 to 380.57 mg/kg and thus represented 31 to 62% of their total P pools. Generally, acid-soluble P is considered as apatite P (Ca- and Mg-bound P) [23]. However, a small amount of P fraction that is absorbed into amorphous Fe oxides, FeS, and Fe phyllosilicates is also dissolved during this extraction [24]. The second most important form of P was non-apatite inorganic P. This form ranged from 36.80 to 78.53 mg/kg, thus representing 7 to 14% of the total P pools. Non-apatite P includes P bound to Al, Fe, and Mn (hydr)oxides [3]. Samples having a relatively higher apatite P amounts are expected to have higher pH values while samples having a relatively higher non-apatite P amount are expected to have lower pH values. This is due to the fact that elevated adsorption of P into the Al and Fe (hydr) oxides are observed at lower pH values, whereas precipitation of P with CaCO3 compounds occurs at higher pH values [25]. However, this assumption was not observed in the present study. The lowest form of P was weak bonding P, which only contributed to 1-2% of the total P pool (ranging from 3.70 to 13.13 mg/ kg). This fraction of P is considered as loosely bound, labile, or exchangeable P because NH_4Cl is used to extract P adsorbed by exchange sites [17]. However, small amounts of P associated with Al and Fe compounds are also dissolved during this extraction [4].
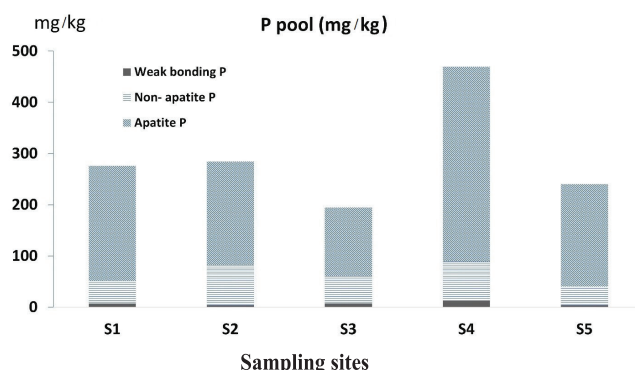

Fig. 2. Concentrations of different P-forms from single extractions.

Environmental implications

Pollution of sediments based on the TP can be classified as no P pollution (TP<500 mg/kg), medium pollution (500 mg/kg<TP<1000 mg/kg), and pollution (TP>1000 mg/kg) [5]. The results indicated that the status of the investigated sediment is from no P pollution to medium pollution.

Based on the relationship between TP and other geochemical characteristics (organic matter or major elements), preliminary information about the potential release and the origination of P in the sediments may be predicted. Reddy and Delaune (2008) [26] concluded that mineralization of organic phosphorus and OC:TP ratio is inversely related. Hence, the lower OC:TP ratio results in the higher potential release of P from organic matter [27]. Among the studied sediments, S1 had the lowest OC:TP and this site is predicted to have the highest mineralization and subsequently highest release of P from organic matter. However,

organic phosphorus is known to be very stable and thus requires a long time to be degraded. The OC:TP ratios (by weight) of S2 to S5 (50-95) were higher than the Redfield ratio [28], which indicates that the organic matter is not enriched with phosphorus [27]. Hence, it can be inferred that most of the P in the investigated sediments (except S1) are associated with inorganic compounds. It is reported by Jensen, et al. (1992) [29] that the ratio of total iron to total phosphorus (Fe:TP ratio) in the sediment is an indicator for the release of P into the water environment. Sediments with Fe:TP over 15 (by weight) release relatively less P in anoxic conditions whereas Fe:TP ratios below 10 seem to be unable to retain P [29]. This is due to the fact that the formation of minerals of Fe (II) and P is favoured when there is A high availability of Fe in sediment [30]. The ratio of total iron to total phosphorus (Fe:TP ratio) of the sediments in Can Gio was higher than 15, suggesting a low release of P from iron minerals.

Although the total concentration of P can provide information on the current pollution status and the ratio of TP to other elements in sediments may help to predict P-leaching, they do not allow the assessment of the mobility of P into the water environment. Single extractions may give some information about the mobility of P under changing environmental conditions. To assess the potential release of P as an internal load from sediment to the water environment, two P-pools were characterized as mobile P and non-mobile P. The mobile P was determined by the sum of the weak bonding P, apatite P, and non-apatite P form, which is the P that can be released when external environmental conditions change. The difference between TP and the mobile P represents the non-mobile sediment P pool (Fig. 3). The mobile P forms ranged from 41 to 49% (S1-S3) and 73 to 75% (S4-S5). Hence, it seems likely that mangrove forest sediments are more sensitive to P release. As mentioned above, sediments in S1-S3 had a higher content of clay materials, Fe, and Al, and thus had a better ability to adsorb and retain P than the sediments in S4-S5. Different forms of P are of different significance in terms of environmental meaning [4]. The potential mobility of P increased as follows: weak bonding P < non-apatite P < apatite P. This order agreed well with the findings of other studies [2, 14]. Phosphorus extracted by mild extraction with NH4Cl is known as the most labile P among investigated forms of P. Hence, weak bonding P can be represented by the immediately available P when sediment is resuspended in water. The result obtained from NH4Cl extraction in this study was quite low (1-2%) and similar to Kapanen (2008) [16] who found a low extraction yield of P (1% of the TP) in the sediment samples. This indicated that sediments in Can Gio have a poor leachability of P during resuspension in water.

The concentration of the non-apatite P representing P bound to metals is also used to estimate the available algal P [31]. This form of P is sensitive to changing redox conditions [3]. When the sediment becomes anaerobic, Fe(III) binds lightly with P and is reduced to Fe(II), which is more soluble and can be more readily exchanged with the solution [21]. Therefore, non-apatite P can act as a source of P under anoxic conditions. The concentration

of non-apatite P present in the sediment in Can Gio (44-79 mg/kg) was higher than the concentration found in normal sediments in Estonia (11.95-39.43 mg/kg) [16], but in the lower range of sediments in a eutrophic area of Brazil (119-279 mg/kg [3]) and Norway (480-710 mg/kg [30]).
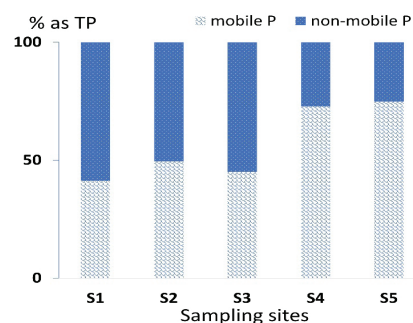

Fig. 3. The relative contributions of mobile and non-mobile forms to the total P pool.

Among the mobile forms, the dominant form of P in Can Gio sediments was apatite P (Fig. 3). This form corresponds to P bound to calcium, including apatite-P and P bound to carbonates [4]. The amounts of apatite P present in the Can Gio sediments (134-381 mg/kg) were similar to the values obtained in other studies (154.23-570.90 mg/kg [16], 260-350 mg/kg [30], 46-366 mg/kg [3]). Apatite-P is observed to be the main form of P in mangrove sediment based on its stability under the redox variations [27]. This form is considered to be non-bioavailable and difficult to release, however, under weakly acidic conditions, it can be partly released [32]. Therefore, the result from the P-HCl extraction is useful to assess the potential mobility of P when the external pH decreases. Results from single extraction indicated that acidification has a more profound impact on P-leaching of sediments in Can Gio compared to changing redox condition because apatite P is much higher than non-apatite P. However, non-apatite P can be mobilized and turn into an internal P source in anoxic conditions [3]. Hence, non-apatite P is also an important pool in terms of the potential release of P from Can Gio sediments due to the fact that most of the sediments in Can Gio are affected by periodic tidal forces and inundation [9]. Therefore, reducing the loading of P by improving the quality of wastewater being discharged into public water ways, as well as avoiding drastic acidification of the sediment, would be helpful in preventing pollution from P release.

Conclusions

The sediments investigated in Can Gio are characterized by low-to-medium P concentrations, which reflects a sediment status ranging from no pollution to medium pollution of P. Single extractions were useful to assess the potential release of P when the external environment changes. Results from the single extractions indicated that the mobile form of P was dominant in the core zone of the mangroves (71-75%), which suggests that these mangrove sediments have the potential to act as a source of phosphorus in the aquatic environment. The mobile-P consisted mainly of apatite P (31-62%), while non-apatite P and weak bonding P only contributed

a minor part (1-14%) to the studied sediments. Although apatite P is considered to be non-bioavailable and difficult to release, it may attribute to the permanent pool of P in sediments. Therefore, these fractions are considered as a source for internal P-loading to the water environment.

ACKNOWLEDGEMENTS

COMPETING INTERESTS

The authors declare that there is no conflict of interest regarding the publication of this article.

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