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RESEARCH ARTICLE

Phosphorus Fractionation in The Sediment of Kendari Bay, Southeast Sulawesi, Indonesia

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ABSTRACT

Information about the chemical structure of the phosphorus (P) fraction in sediments is very important for identifying the bioavailability of P and eutrophication in marine ecosystems. Sediment as a source of P has an important function in P adsorption in waters. To estimate the phosphorus load in water bodies, it is required to define the fraction of phosphorus compounds. This study purposed to evaluate the composition of phosphorus speciation in sediments of Kendari Bay and evaluate their possible contributions to the eutrophication of the coastal ecosystem. P fractionation was examined by chemical extraction and sequencing methods. Sediment characterization was conducted by gravimetry, hydrometry, and spectrophotometry methods. The study result found the total P concentration in the sediment ranged from 0.30 to 0.41 mg/g. Phosphorus content in exchangeable P (Exc-P), Fe-P, Al-P, Ca-P, and organic P (OP) contributed 1.8%, 12.7%, 1.5%, 63.1%, and 21.5% of total P (TP), respectively. We found significant positive correlations between Exc-P, Fe-P, Al-P, and OP and finer sediment and organic matter. Exc-P and OP are bioavailability (BAP) fractions that potentially release overlying water, contributing 10–35% to TP.

Introduction

Phosphorus (P) is an important macro-nutrient in aquatic ecosystems, playing a direct role in eutrophication. The presence of P in coastal aquatic environments originates from native soil P, atmospheric deposition, and anthropogenic P activities such as soil erosion, excessive use of fertilizer, sewage, and phosphorous-containing detergents. P is transported to the seabed as a sinking particulate containing numerous inorganic minerals and organic compounds [1]. P that sinks in the water column or coastal areas carried from land by currents is transformed at the sediment-water interface and in post-burial sediments, leading to a different composition from sink pools [2]. Phosphate ions (PO_4^{3-}) are an important species of phosphorus. Most plants and organisms in land water directly require this species. Phosphate anions can form compounds that are not easily soluble and are easily immobilized by sediment through the formation of insoluble compounds. Phosphate ions in water interact with suspended particles [3]. Suspended particles can adsorb phosphate ions, which settle to the bottom of the water as sediments [4]. Under anaerobic conditions, adsorbed phosphate is released into dissolved phosphate by chemical and biological processes. Waterborne particles can also act as phosphate adsorbers [5].

Sediments play a significant function in controlling P concentrations in the water column [6]. Overloaded P from external sources can be stored in sediments through dissolved P adsorption [7]. The mobilization and behavior of P in waters are strongly influenced by the P adsorption process in sediments. The characteristics of P adsorption were mainly controlled by the sediment fraction, which may be related to the sediment source. Therefore, grain size is a key factor that affects P adsorption characteristics. In marine sediments, phosphorus is found in the form of organic phosphorus (OP) and inorganic phosphorus (IP). Generally,

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inorganic phosphorus is found in exchangeable P species (Exc-P) and complex compounds of aluminium (Al-P), iron (Fe-P), and calcium (Ca-P) [8]. The P bonds with macro elements in sediments are really influenced by physical, chemical, and biological processes in the waters, therefore having an impact on the movement, degradation, and deposition of P in the marine environment, and finally reduces the of P bioavailability. The P element in the sediment has a reactivity depending on the fraction.

Kendari Bay, an estuary located at the edge of the City of Kendari, Southeast Sulawesi, faces environmental pressure owing to the urban and economic development of the city. One of the main problems encountered in Kendari Bay is a decrease in water quality. The Wanggu watershed with an area of 339.73 km^2 has been the largest contributor to sediment load in Kendari Bay until the last several decades [9]. The transported sediments carry nutrients, including phosphorus (P), which are derived from the runoff of residents and threaten Kendari Bay water quality. Understanding the P fraction in sediments is very important for understanding the impact of anthropogenic influences that can affect water quality in the area and its surroundings. Kendari Bay receives a large amount of sediment from its catchment area, possibly receiving a significant amount of phosphorus along with the load. Studies related to phosphate concentrations in Kendari Bay have been conducted by several researchers [10,11]. Information about the phosphorus geochemical processes in sediments of Kendari Bay is still very limited; therefore, it is crucial to study P in Kendari Bay sediments to advance an understanding of the phosphorus availability in sediments and waters. This study purposed to evaluate the composition of phosphorus speciation in sediments of Kendari Bay and evaluate their possible contributions to the eutrophication of the coastal ecosystem.

Method

Data Collection

Collecting data was carried out in May 2022 at five sites located in Kendari Bay, Southeast Sulawesi, Indonesia. Kendari Bay is located between latitudes 3°57'59.37”-3°59'32.39” S and longitudes 122°31'38.07”-122°35'55.93” E (Figure 1). The sampling locations were chosen to represent the inside part of the bay near a large river mouth, including sites of TK-1, TK-2, and TK-3, whereas TK- 4 and TK-5 are located near the mouth of the bay affected by seawater from outside Kendari Bay. Sediment samples were collected using a sediment grab sampler and stored in aluminium wrap at a temperature of 4 °C (cool box or refrigerator) during transportation to the laboratory. Moreover, we used data on the water pH at the sediment-water interface.

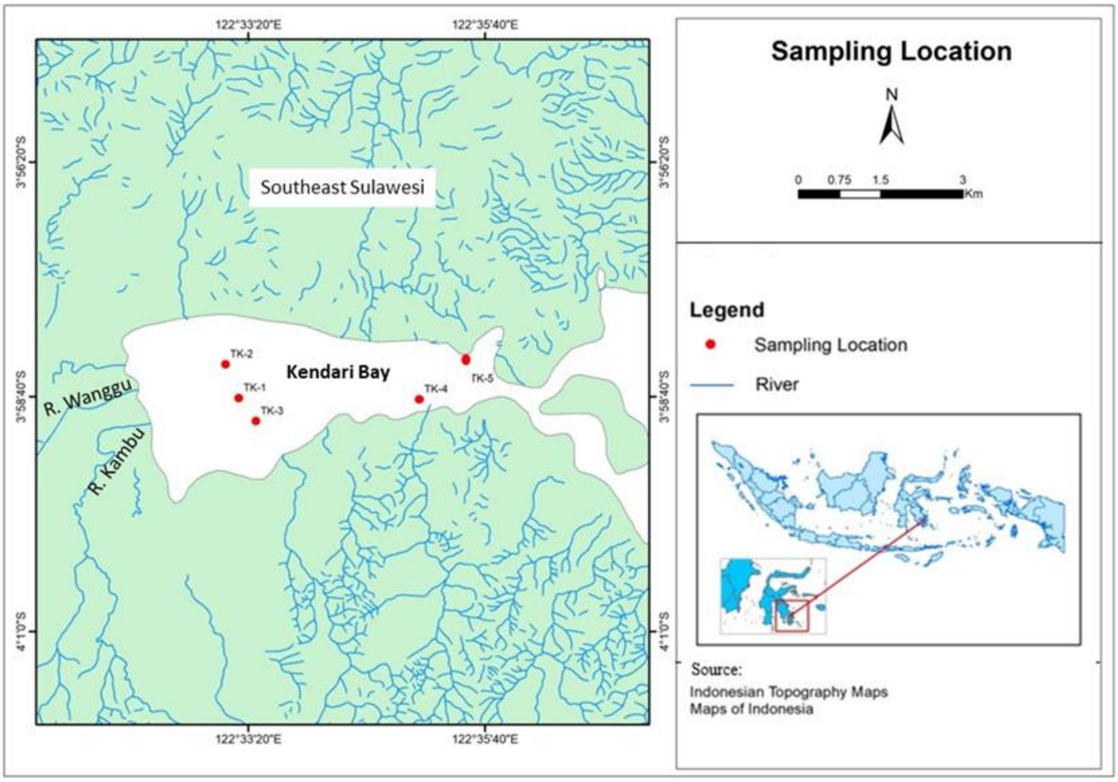

Figure 1. Research location.

Sediment Analysis

Sediment samples were dried at 40 °C for two days in an oven, then ground to obtain samples < 2 µm in size. Fractionation of phosphorus in sediment was carried out using a modified chemical and sequential extraction method (SEDEX) [12,13] (Figure 2). Sediments were extracted sequentially to obtain total phosphorus concentrations, organic P, and inorganic P including exchangeable P (Exc-P), Fe-P, Al-P, and Ca-P. The extracted P species were measured respectively as dissolved reactive phosphate (Soluble Reactive Phosphorous/SRP) using the method described in standard methods [14]. In addition, other sediment characteristics were analyzed, including grain size (sand, silt, and clay), organic matter, and macro element content (Fe, Al, and Ca). The grain size composition was analyzed using the three-fraction method with a hydrometry method based on the procedure described in the soil/sediment analysis technical guide [15]. Organic matter content was determined using gravimetry [16]. The macro element composition of iron (Fe), aluminium (Al), and calcium (Ca) was determined from dry sediment using the Atomic Absorption Spectrophotometer (AAS) Method with wet digestion [17].

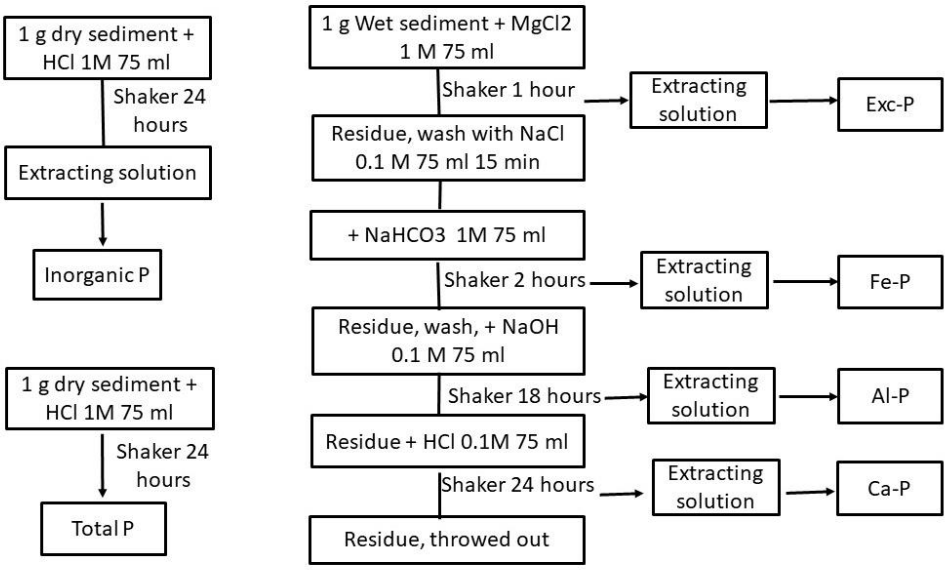

Figure 2. Sequential extraction method (SEDEX modification).

Data analysis

Data analysis was conducted using Ms. Excel 2013 and Multivariate Statistical Package (MVSP) 3.22. To evaluate the correlation between P fraction in sediments and their characteristics, Pearson correlation analysis was carried out. Correlations were considered with a significant level of 5% (α = 0.05). Principal component analysis (PCA) was performed to determine a correlation between environmental variables and the phosphorus fraction.

Results and Discussions

Physicochemical Parameters in The Water Column of Kendari Bay

Kendari Bay is an estuary formed by several rivers in Kendari City, including the two largest rivers, Wanggu and Kambu. The estuary zone is a transitional ecosystem from fresh water to saline water, strongly influenced by tides [18]. Mixing between the river and seawater showed unique physicochemical properties, such as pH, dissolved oxygen (DO), and salinity, in contrast to pure seawater. A study at Genuk Beach in Semarang, Center Java, indicated that sea tides have a major influence on the distribution and concentration of nutrients in the estuary zone [19]. Measurements of physicochemical parameters in Kendari Bay showed that the surface water pH was slightly alkaline, ranging from 8.82 to 9.12. This indicates that the water is a buffer, which is the presence of carbonate salts and bicarbonate. Carbonate salt $CaCO_3$ originates from biogenic apatite and carbonate fluorapatite authigenic (CFA) compounds. These compounds are derived from the bones and teeth of dead marine organisms [20].

The phosphate concentration (SRP) was found to be very low, with a value of < 0.01 mg/L (as shown in Table 1). The high dissolved oxygen content, ranging from to 3.25–4.00 mg/L, indicates that the Kendari Bay surface water is in an aerobic state. Consequently, the collected sediment samples were in an aerobic state. In aerobic conditions, phosphate is known to bond with macro elements such as iron (Fe) and aluminium (Al) [21]. In addition, saline waters with an average salinity concentration ranging from 16.3% to 17.7‰ (16.7 ± 0.56) cause Ca bonds with phosphate ions to become stable and not easily released into the water column [22].

Table 1. The average and standard deviation of the physicochemical characteristic of water at Kendari Bay.

Location	TK.1	TK.2	TK.3	TK.4	TK.5
Temperature (°C)	30.1 ± 0.1	30.9 ± 0.1	30.2 ± 0.1	30.1 ± 0.1	30.0 ± 0.1
pH	8.9 ± 0.1	8.8 ± 0.1	8.9 ± 0.1	9.0 ± 0.1	9.1 ± 0.1
Salinity (‰)	16.3 ± 1.5	17.7 ± 1.9	16.5 ± 2.0	16.6 ± 2.0	16.5 ± 2.4
DO (mg/L)	4.0 ± 0.8	3.8 ± 1.0	3.3 ± 0.9	3.6 ± 0.7	2.6 ± 0.6
SRP (mgP/L)	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01

Note: SRP= Soluble Reactive Phosphorus; ± = deviation standard.

Sediment Characteristics

Sedimentation and organic material transfer also affect estuaries. The transit, resuspension, and deposition processes in estuaries cause sediment dynamics. In sediments, phosphorus is directly related to physicochemical environmental parameters, such as particle size of sediment, macro elements, and organic matter. The sediment in Kendari Bay is composed of sand, silt, and clay, with silt being the dominant particle type, ranging from 74.8 to 79.25% (Table 2). The complex interaction between hydrodynamic and geomorphological processes influences variability in sediment particle size in each region [23]. The study sites have a shallow depth of 5 to 12 meters. A study reported low-energy water flow in the Kendari Bay area [24]. This study revealed that the water flow inside the bay (5.26 cm/sec) tended to be lower than that in the middle and mouth of the bay (17.31 cm/sec).

Table 2. The average and standard deviation of physicochemical characteristics of surface sediments at Kendari Bay.

Sediment parameters	TK.1	TK.2	TK.3	TK.4	TK.5
Sand (%)	3.8 ± 1.9	2.8 ± 1.7	2.9 ± 1.5	7.8 ± 2.7	4.5 ± 2.3
Silt (%)	76.3 ± 1.8	77.7 ± 1.8	75.8 ± 1.7	79.3 ± 2.0	74.8 ± 1.5
Clay (%)	20.0 ± 3.4	19.4 ± 3.2	21.3 ± 3.9	12.9 ± 2.6	20.7 ± 3.7
Organic matter (%)	68.4 ± 21.8	62.6 ± 17.5	64.4 ± 20.8	25.2 ± 77.5	57.9 ± 19.9
Al (mg/g)	0.27 ± 0.12	0.13 ± 0.06	0.21 ± 0.07	0.09 ± 0.02	0.23 ± 0.10
Ca (mg/g)	2.41 ± 0.98	1.74 ± 0.31	2.42 ± 1.21	2.18 ± 0.52	4.10 ± 1.37
Fe (mg/g)	9.08 ± 2.50	5.50 ± 1.90	5.57 ± 2.21	4.52 ± 1.18	5.10 ± 1.41

± is Deviation standard; TK is site codes

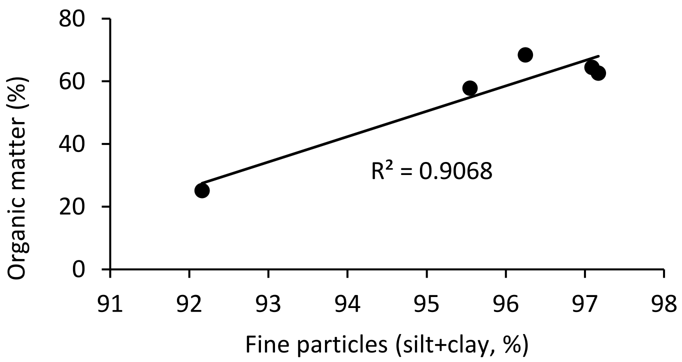

Figure 3. Relationship between fine sediment fraction (silt + clay) and organic matter content.

The organic matter content in the sediment ranged from 25.2 to 68.4%. Organic materials with concentrations greater than 35% are considered extremely high [25]. Sediment organic matter is derived from bacteria, detritus of plants and animals, or plankton, as well as natural and anthropogenic sources in catchments. This study revealed a correlation between organic content and sediment grain size, which was dominated by fine particles (silt and clay) with an R^2 value of 0.9068 (Figure 3). A previous study reported that sediments with high proportions of finer grains tended to have high organic material content [25].

The analysis results of the macro element showed that Fe had the highest concentration compared to the other elements, with values between 4.52 and 9.08 mg/g (Table 2). This was consistent with a previous study that reported research at the same location [26]. The high content of Fe in Kendari Bay sediment is influenced by the rivers entering the bay, which carry anthropogenic pollutants, especially those from untreated mining and industrial waste. The ability to bind with phosphate ions through the adsorption process, the presence of iron, aluminium, and calcium elements in the phosphorus cycle is very important. In this study, phosphate concentrations were very low, suggesting that some iron oxyhydroxides bind to phosphate ions, which is supported by aerobic DO concentrations.

Phosphorus Fraction

Phosphorus deposition in sediment may result in nutrient fractionation, affecting its availability and environmental impact. A study revealed that the different fractions composition of P affects the mobility of sedimentary phosphorus [27]. The fractionation of phosphorus compounds in sediments is a significant parameter for estimating the internal load of phosphorus compounds in water bodies. By examining the various species of phosphorus in a sample, we can determine their sources and pathways in the ecosystem and pinpoint excess phosphorus that may lead to eutrophication. The results of phosphorus fractionation in Kendari Bay sediments are shown in Figure 4.

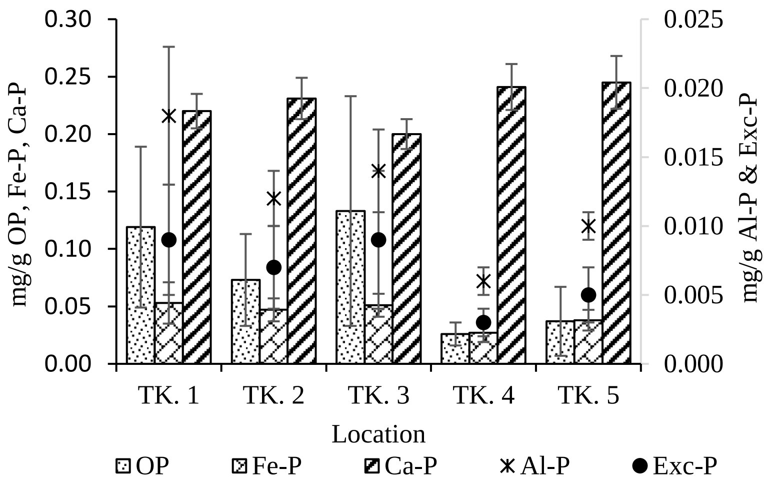

Figure 4. Distribution of P fractions in Kendari Bay sediments (the vertical line on the bar is the standard deviation).

Table 3. The average concentration of P species in surface sediment measured in this study compared with measurements from other estuaries and coastal areas (μg/g).

Locations	Exc-P	Fe-P	Ca-P	OP	TP	References
Kendari Bay	6.6	43.2	227.4	77.6	360.2	This study
Jobokuto Bay, Jepara	8.7	71.1	157	48.9	291.4	[28]
Daya Bay	15.7	33.2	57.9	100.7	395.3	[31]
Laizhou Bay	10.7	40.7	60.2	62.1	493.7	[20]
Changjiang Estuary	14.0	13.7	29.4	183.4	542.9	[35]
Rushan Bay	3.62	49.0	40.5	53.7	408.2	[29]
Bengal Bay, India	26.3	5.8	588.6	137.4	824.7	[30]
Guapimirim estuary	9.1	37.7	40.3	nd	159.8	[23]

Note: nd = no data; some of the data are originally in μmol/g.

The total phosphorus concentration in Kendari Bay ranged from 0.328 to 0.410 mg/g with an average of 0.366 ± 0.038 mg/g. When compared to the organic phosphorus (OP) percentage, the inorganic P percentage contributed more to the total P content. Organic phosphorus (OP) concentration was highest at locations near the bay. In this study, the TP concentration was higher compared to research in Jobokuto Bay in Jepara [28], but lower than Laizhou Bay, Rushan Bay, and Daya Bay in China, as well as Bengal Bay in India (Table 3) [20,29–31].

IP is a bioavailable source of P in sediment that can be released into the water column to be consumed by phytoplankton [32]. A study reported that the phosphorus released to the sediment surface was derived from inorganic phosphorus compounds [23]. The concentration of inorganic P fraction is the sum of the Exc-P, Fe-P, and Ca-P fractions. Exc-P in sediment is P-reactive and can be directly consumed by phytoplankton [33]. The concentration in this study was low, ranging from 0.003 to 0.009 mg/g (average 0.007 ± 0.003 mg/g), contributing approximately 1.8% of TP (Figure 5). This condition is thought to be related to the high content of organic and silty sediment. Particle size of sediment and organic matter are significant parameters controlling the Exc-P concentration. Finer sediment has a large surface area, providing more area to absorb and bind phosphorus [34,35]. Exc-P species are the decomposition product of organic matter [36]. Environmental factors including temperature, pH, water movement, redox activity, and chemical components have an effect on the release of Exc-P into the water-sediment interface. The average Exc-P concentration in Kendari Bay was relatively higher than that in Rushan Bay [29] and lower than those in Laizhou Bay, Daya Bay, and Bengal Bay [20,30,31].

The analysis results of Fe-P and Al-P species in sediment ranged between 0.027 to 0.053 mg/g, and 0.003 to 0.009 mg/g respectively, contributing 12.7 and 1.5% to TP, respectively. A higher concentration of Fe-P was found inside Kendari Bay (TK-1, TK-2, and TK-3), which has a relatively lower salinity than outside the bay (TK-4 and TK-5). The Fe-P concentration decreases offshore. Many studies have shown that phosphorus trapped by Fe hydroxide/oxide in brackish water sediments has an effect on reducing Fe-P in saline water sediment. [33,34]. There was a positive correlation between Fe-P and Fe concentration. Phosphorus in the sediment is bonded to Fe and Al through chemisorption and these compounds are used to determine the availability of P for algae growth in the water [37].

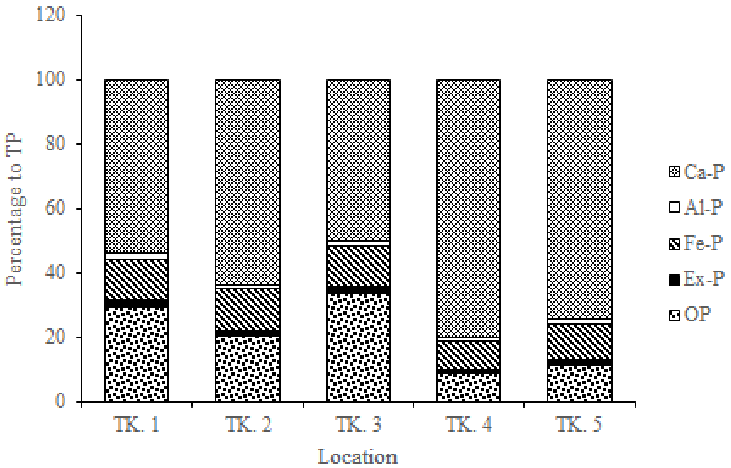

Figure 5. Percentage contribution of P fraction (Ca-P, Al-P, Fe-P, Exc-P, and OP) to Total P.

Information on the existence of Fe-P species in sediment is important to control the availability of P in the water column triggering eutrophication. It can be a significant indicator of phosphorus and Fe mobility [38] and also an indicator of sediment quality and pollution levels [39]. This species is formed by the co-precipitation of phosphate with iron oxide and iron hydroxide [40] and is mainly derived from human activities, such as domestic and industrial waste [33]. Under anaerobic conditions, Fe (III) is converted to Fe (II). Generally, Fe-P is associated with Fe-oxide, which is an important adsorbent for dissolved IP in aquatic environments. However, Fe-P is also a P species that is vulnerable to redox activity because P can be released from Fe-P bonds into the water column under anaerobic conditions [41]. The average Fe-P concentration in Kendari Bay was higher than that in Daya Bay [31] and lower than those in Rushan Bay and Jobokuto Bay [28,29].

Ca-P is a P species derived from the biogenic apatite compound, that is a component of bones and teeth of marine vertebrates [42], and the CFA compound [34], which may crystallize in interstitial water [20]. Calcite is formed through precipitation reactions and biological activity under high salinity conditions [30]. In Kendari Bay, the Ca-P concentration ranged from 0.200 to 0.245 mg/g, representing 63% of the TP. The high concentration of Ca-P is assumed to be an accumulation of calcium in Kendari Bay that is influenced by high salinity and pH. A study revealed that salinity can increase Ca^{2+} concentration, which can affect in immobilization of P through co-precipitation with Ca^{2+} in calcium carbonate [24].

In other studies, Ca-P is known to be a highly stable mineral in alkaline environments [43], which is comparable with the conditions in Kendari Bay with a pH value > 8. The strong mineral fluorapatite can attach to other chemicals, including phosphorus, and accumulate in sediments [44]. In this study, Ca-P distribution increased from the inside to the mouth of the bay. Study conducted in Jobokuto Bay, Jepara found that Ca-P is the most representative P species in marine sediments [28]. Averaged of Ca-P concentration value in Kendari Bay was relatively higher than in Jobokuto Bay, Laizhou Bay, Rushan Bay, and Daya Bay [20,28–30], but lower than in Bengal Bay India [30] (Table 3).

The OP concentration ranged from 0.062 to 0.092 mg/g, with a mean of 0.227 ± 0.003 mg/g. The OP contribution was 21.5% of the total phosphorus, suggesting the presence of phosphate minerals containing organic matter at the sediment surface [30]. Sources of organic phosphorus mostly come from biological processes in terrestrial area and marine primary production [20]. The content of organic and phosphate in the Kendari Bay water body can be derived from high anthropogenic activities in the water catchment area. The average OP concentration in Kendari Bay was relatively higher than those in Laizhou Bay, Rushan Bay, and Jobokuto Bay [20,30,31], but lower than those in Bengal Bay and Daya Bay [30,31].

The results of the Pearson correlation analysis found a significant positive correlation between Fe-P, Al-P, Exc-P, and OP species with clay and organic matter (Table 4). This phenomenom indicated that finer sediment grain size and organic matter are environmental factors that are very effective in controlling phosphorus concentrations. A similar phenomenon was also reported in some coastal waters in which the high organic matter content and finer sediment fractions tended to increase the organic phosphorus concentration [23,28,33,44,45]. They revealed that, in a large specific surface area, sediments with finer particles have a stronger adsorption capacity.

Table 4. Pearson correlation between phosphorus species and sediment characteristics.

	OP	Exc-P	Fe-P	Al-P	Ca-P	Sand	Silt	Clay	Org. matter	Al	Ca	Fe
OP	1											
Exc-P	0.97	1										
Fe-P	0.91	0.99	1									
Al-P	0.64	0.74	0.76	1								
Ca-P	–0.94	–0.84	–0.75	–0.36	1							
Sand	–0.72	–0.85	–0.91	–0.52	0.59	1						
Silt	–0.39	–0.50	–0.52	–0.45	0.27	0.61	1					
Clay	0.63	0.77	0.81	0.54	–0.49	–0.92	–0.88	1				
Org. matter	0.74	0.88	0.94	0.73	–0.54	–0.95	–0.73	0.94	1			
Al	0.58	0.65	0.66	0.80	–0.38	–0.53	–0.86	0.76	0.75	1		
Ca	–0.31	–0.23	–0.20	0.02	0.37	0.05	–0.72	0.34	0.10	0.52	1	
Fe	0.64	0.69	0.70	0.98	–0.38	–0.39	–0.28	0.37	0.60	0.70	–0.13	1

Significance level (P) = 0.05

Based on the eigenvalues in the PCA analysis results, two principle components were obtained. The percentage of variance that can be explained is 83.0% (Figure 6). The 65.0% value of 83.0%, represents PC1 which reflects significant positive charges of OP, Exc-P, Fe-P, Al-P, Fe, clay, and organic materials, for a while on sand was negative charges. The high of P species was associated with fine sediments (especially clay) and the high organic content. While PC2 explained 18.0% of the total variance of PC1 reflecting significant positive charges on Ca-P and Ca, for a while on Ca was negative charges. Some phosphorus fractionation results at several study locations are listed in Table 3.

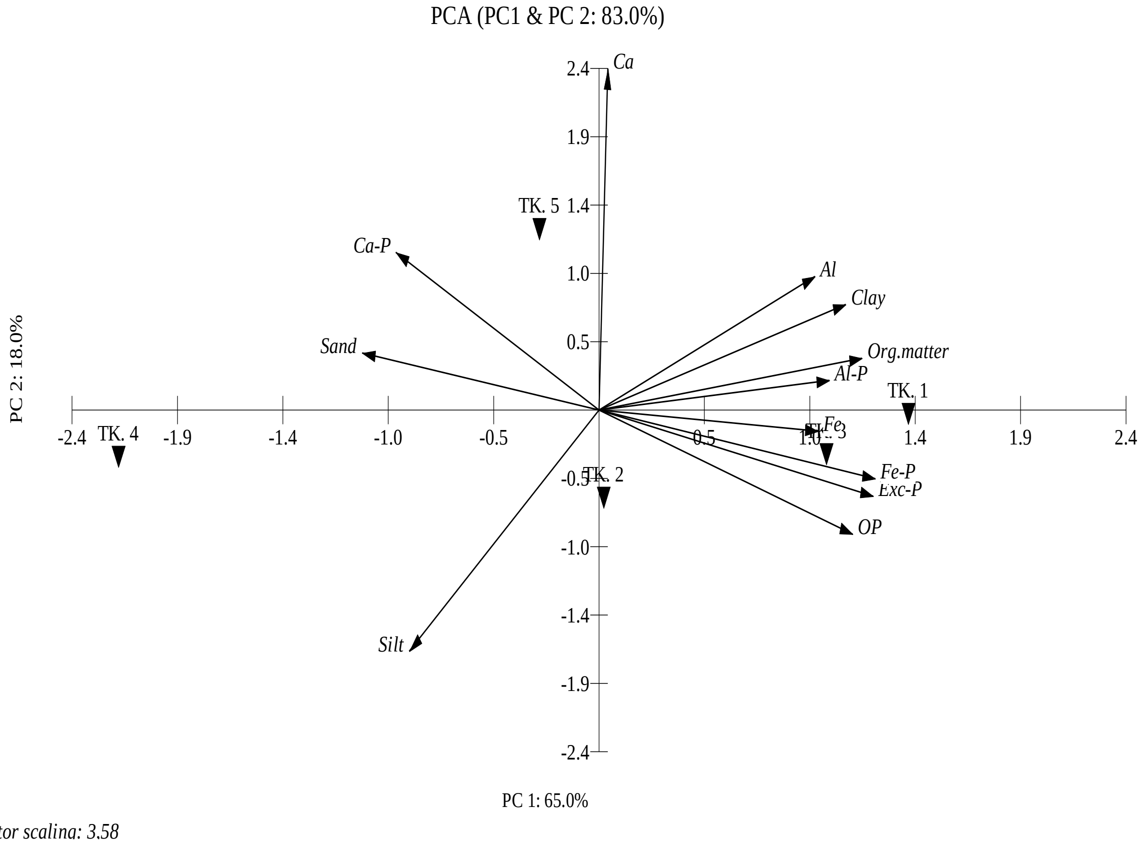

Figure 6. The Principal Component Analysis (PCA) of all parameters and locations.

Evaluation of Potential Bioavailable P in Sediments

Many studies have attempted to classify sediment quality based on P concentrations in sediments. A concentration of total P in sediment exceeding 0.7 mg/g indicated a significant anthropogenic impact on the aquatic environment [46]. Other criteria set classifies sediment as moderately polluted if total P ranges from 0.495 to 1.3 mg/g (16 to 42 mol/g) and heavily polluted if it exceeds 1.3 mg/g (>42 mol/g) [47]. Based on these references, the impact of human activity on Kendari Bay may be relatively small.

Exc-P species will be released slowly when the phosphate concentration in the water column is lower than in pore water, and OP can be available through the demineralization process of phosphate-containing organic matter. However, the phosphorus content in the surface sediments of Kendari Bay is dominated with Ca-P species. This condition shows that phosphorus species in Kendari Bay waters are relatively stable and less bioavailable. In addition, phosphorus in the iron/aluminium-phosphorus (Fe/Al-P) species cannot be released under aerobic conditions. Kendari Bay is shallow water and transportation activities encourage turbulence and mixing; Ca-P and Fe/Al-P will be stable under normal conditions. They potentially reduce their contribution to eutrophication and water quality issues. In this study, only Exc-P and OP had the potential to encourage eutrophication, with a contribution of 10–35%.

Conclusion

This research revealed various forms of phosphorus in Kendari Bay and their distribution. The contribution of inorganic P is very dominant to total P in Kendari Bay sediments, mainly composed of Ca-P (63%). The low Exc-P concentration was presumed to be the cause of the low phosphate ion concentration in the Kendari Bay water column. Because the bioavailable P is relatively small, ranging from 10% to 35% of the total P content, the potential for eutrophication in Kendari Bay is low as long as conditions of the sediment-water interface column are still aerobic. Nevertheless, the estimated loads of P, both internal and external, are still required to prevent eutrophication in the bay and adjacent coastal ecosystems.

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