EnvironmentAsia
The international journal published by the Thai Society of Higher Education Institutes on Environment

Available online at www.tshe.org/ea/index.html
EnvironmentAsia 10(2) (2017) 1-8
DOI

Modeling Spatio-vertical Distribution of Sulfate and Total Sulfide along the Mangrove Intertidal Zone

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Abstract

Given the complexity and heterogeneity of mangrove conservation landscape, research gaps still exists to quantify sulfate and total sulfide and their relationships with sediment properties and environmental covariates. Thirty-two sediment samples in the top layers (0-10 cm) were analyzed to assess biochemical properties, sulfate and total sulfide contents. With an average±SD value of 0.62±0.36 mg/g, the total sulfide content from the study site was high compared to the southern part of Thailand. The distribution of sulfate content exhibited high values in nearby land area which gradually reduced in seaward discharges/runoff, whereas high concentrations of total sulfide were highlighted around the center of the study site and vertically accumulated in the top few centimeters of soil and decreased with depth. The most pronounced factor affecting the amount of sulfate and total sulfide content was organic matter, while pH, organic carbon, potassium, salinity, and sediment-mangrove conditions correlated with sulfate and sulfide at different levels. Total sulfides concentration can be considered as indicator of over nutrient-rich sediments for assessing environmental quality perhaps the die-back of mangroves. Concerns about high total sulfide concentrations across mangrove conservation areas should receive more attention, in particular the reduction of OM from the anthropogenic source.

Keywords: non-parametric model; sediments; sulfur-related compounds; wetlands

1. Introduction

Mangrove forests act as a trapped path for suspended sediments such as organic matter (OM), inorganic matter, and contaminant inputs from runoff and then re-suspended by ocean waves and tides (Furukawa and Wolanski, 1996). When OM breaks down very slowly in the waterlogged environment, sediments not only have high OM but also potentially contain high sulfide contents (Machado *et al*., 2002). In the sulfur cycle, sulfide is produced through dissimilatory sulfate reduction by sulfur-reducing bacteria. The presence of sulfate content is not a main concern among pollution control studies because of its non-toxicity. However, when sulfate concentration is high, the sulfur cycle can become unbalanced and lead to over-abundant sulfide concentrations (Lens *et al*., 1998; Vannucci, 2013). As a consequence, sulfide-rich sediment can damage mangrove aerial roots and cause mangrove dieback due to extremely poor sediment conditions (Fitzpatrick *et al*., 2008). A negative redox potential for hydrogen sulfide in the environment may reduce the bioavailability of dissolved and particulate matters (Postgate, 1984). The binding phase of acid volatile sulfides (AVS) and metals also creates an undesirable soil biota (Ankley *et al*., 1991; Di Toro *et al*., 1992). The distributions of sulfur-related compounds may vary depending on global georeferencing and anthropogenic activities. Wastewater containing sulfur compounds from human activities is likely produced by effluents from food processing, landfills, industrial production, and agricultural and roadway runoff (Lens *et al*., 1998). A further possible source is oxic groundwater passing through soil layers that contain high levels of iron sulfide where more oxygen is needed for chemical oxidation (Brunet and Garcia-Gil, 1996; Molénat *et al*., 2002), thus leading to sulfur immobilization and the release of high concentrations of sulfates (van der Welle *et al*., 2008).

In order to obtain more information about the spatial and vertical dynamics of sulfates and total sulfides and their interrelationships with sediment properties and the intertidal environment, three objectives were carried out inside the mangrove forest in the Chonburi Province of Thailand: 1) assess the spatial distribution of sulfate and total sulfide content across the study area, 2) investigate the vertical variation of sulfate and total sulfide concentrations in selected sites, and 3) characterize factors associated with sulfates and total sulfides.

2. Materials and Methods

2.1 Study site

The study was conducted in a $2.2x10^6$ m^2 mangrove area inside the Nature Education Center for Mangrove Conservation and Ecotourism in the Chonburi Province, located on the eastern coast of Thailand (13°20'37.05"N, 100°56'34.83"E) (Fig. 1). The tree community is dominated by *Avicennia alba*. The obvious absence of any tree communities can be observed in some areas of the study site where plain sediment is present. Located along the east coast of Thailand, Chonburi has a tropical savanna climate. The monsoon season brings heavy rain and somewhat cooler temperatures during the daytime between May and October. Between 1979 and 2006, Chonburi registered an average annual temperature of 27.9°C, evaporation of 1512.5 mm/year, and rainfall of 2085.7 mm/year (Seeboonruang, 2016). The water channels receive effluents from the upstream and drain into the mangrove area before being discharged into the ocean.

2.2 Sampling collection

The grid-based sampling design with a grid size of 80x80 m^2 was outlined across the study site, providing 35 sampling locations. However, 32 available sediment samples were collected for analyses due to the limitations of area accessibility. Sediment samples were taken in October 2015 at soil depths of 0-10 cm using a 15x5 cm sediment core for each site. Of the 32 samples, three selected cores were collected to represent distinctive landscape settings-adjacent community, healthy mangrove, and unhealthy mangrove environments-and were then sliced into 0-1, 1-2, 2-4, 4-6, 6-8, and 8-10 cm sub-layers. All field samples were constantly kept at 4°C before conducting laboratory analyses.

2.3 Laboratory analyses

The pH was determined in 1:2.5 soil-water suspensions using a pH meter (Vetus, pH-099 (III), USA). Electrical conductivity (EC), total dissolved solids (TDS), and salinity were measured by a handheld EC meter (Hach, sensION 156, USA) in 1:2.5 soil-water extracts using a conductivity bridge (Jackson, 1967). The AVS in the study was defined operationally as the reactive forms of total sulfide released from the sediment using acid reaction (Allen *et al*., 1993). The determination of AVS was done after field sampling at the Ang Sila Animal Research Station, which is located about three kilometers from the study site. The solid-phase sulfide was converted to hydrogen sulfide (H_2S) so that values could be measured for the sulfide gas that accumulated in the gas detector tube (Gastec, No201H, Japan) connected to the reactor. The sulfate (SO_4-S) was extracted with $Ca(H_2PO_4)$ in 2 N HOAc. Sulfates in the extracts were precipitated as $BaSO_4$ and measured turbidimetrically (Combs *et al*., 1998). The content of OM was analyzed by using the loss on

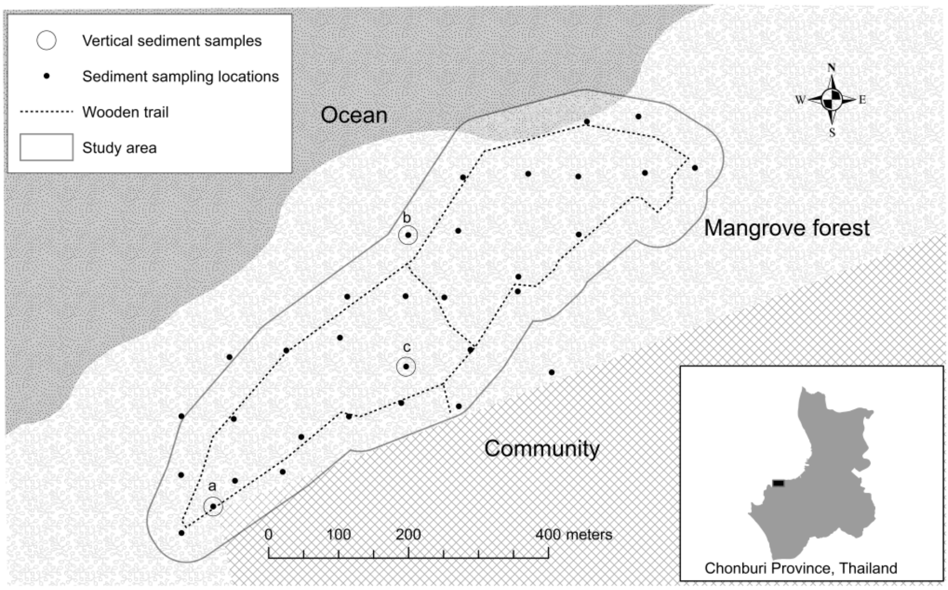


Figure 1. Map showing sediment sampling locations (32 samples) in the mangrove forest within the Nature Education Center for Mangrove Conservation and Ecotourism. Three cores in the circles represent distinguished environmental conditions: (a) adjacent community sediment, (b) healthy mangrove sediment, and (c) unhealthy mangrove sediment.

ignition (LOI) method. Total organic carbon (TOC) was determined by subtracting the concentration of inorganic carbon (IC) from the measurement of total carbon using the Shimadzu gas analyzer (TOC-V CPH). Total nitrogen (TN), available phosphorus (P), and exchangeable potassium (K) were analyzed by Academic Service and Technology Transfer Center, Faculty of Agriculture, Chiangmai University using the Kjeldahl method (Kjeldahl, 1883), Bray II extraction (Bray and Kurtz, 1945), and NH_4OAC pH7 (Chapman, 1965) methods respectively.

2.4 Geostatistical and statistical analyses

Descriptive statistics were implemented to describe the overall characteristics of sediment properties. The spatial distributions of sulfate and total sulfide content were interpolated using the ordinary kriging method in ArcGIS^{TM} v.10.3. Multivariate analysis and regression tree analysis were computed using R (3.2.0) statistical computing software to elucidate the non-parametric relationships of sulfates and sulfides with and related factors. Organic matter, organic carbon (OC), total nitrogen (TN), available phosphorus (P), exchangeable potassium (K), pH, salinity, electrical conductivity (EC), total dissolved solids (TDS) and sediment-mangrove conditions were the explanatory variables that predicted sulfate and total sulfide content in this study. The sediment-mangrove conditions were categorical data that included three levels of factors: sediments with no mangroves surrounded, sediments with scattered mangroves surrounded, and sediments with high density of mangroves surrounded.

3. Results

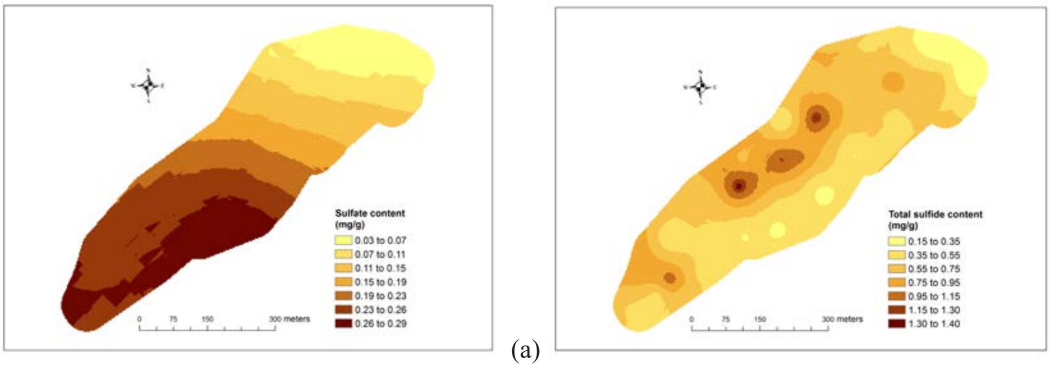
3.1 Descriptive statistics of parameters and spatial distributions of sulfate and total sulfide

Statistical descriptions of parameters from 32 sediment samples are presented in Table 1. The overall distribution of sulfates exhibited a gradual reduction from southwest to northeast, and high values were accented in adjacent land areas. A stable model was used to fit the experimental semivariogram of sulfates (Fig. 2(a)). The model provided a nugget variance of 0.002, a sill variance of 0.022, and a range of 0.001 m. The strength of spatial dependency as indicated by the nugget:sill ratio was very strong for sulfate distribution at a value of 9%. The total sulfide variable was log transformed to stabilize variances and comply with stationarity assumptions (Webster and Oliver, 2001). The concentrated total sulfide hotspots were found in the central study area. The semivariogram of total sulfide was fitted by an exponential model (Fig. 2(b)). The model exhibited a nugget variance of 0.02, a sill variance of 0.40, and a range of 0.001 m. Spatial autocorrelation of total sulfides was longer with a nugget: sill value of 50% compared to a spatial pattern of sulfates.

Table 1. Statistical description of sediment characteristics at a depth of 0-10 cm (*n=32*)

Parameters	Unit	Min	Max	Mean	Median	SD	Skewness
Sulfate	mg/g	0.04	0.33	0.20	0.23	0.09	-0.46
Total sulfide	mg/g	0.14	1.49	0.62	0.50	0.36	0.91
Organic matter (OM)	%	6.14	17.34	11.20	11.23	2.14	0.50
Organic carbon (OC)	%	0.05	9.8	5.07	5.48	2.68	-0.19
Total nitrogen (TN)	%	0.12	0.21	0.16	0.16	0.02	0.23
Available phosphorus (P)	mg/g	0.33	0.60	0.48	0.50	0.08	-0.40
Exchangeable potassium (K)	mg/g	1.15	2.93	1.91	1.87	0.48	0.47
pH	$-\log[H^+]$	7.07	8.78	-	-	-	-
Electrical conductivity (EC)	mS/cm	2.29	5.70	3.48	3.34	0.70	1.03
Total dissolved solids (TDS)	g/L	1.14	2.85	1.74	1.66	0.35	1.04
Salinity	ppt	1.24	3.28	1.93	1.84	0.42	1.09

Note: mg/g is measured based on the sediment dried weight; SD is standard deviation.



(a) (b)

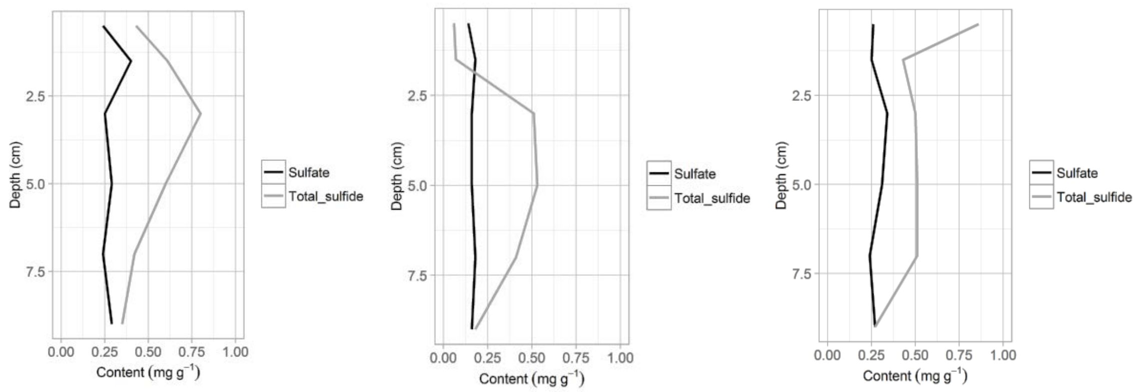
Figure 2. Sulfate and total sulfide content estimates at 10 cm depth derived by ordinary kriging: (a) sulfate content (mg/g) and (b) total sulfide content (mg/g).

3.2 Vertical distribution of sulfates and total sulfides

Overall sulfate and total sulfide content along the profiles were similar in the adjacent community and unhealthy mangrove sites with mean sulfate values of 0.29 and 0.28 mg/g and mean total sulfide values of 0.54 and 0.51 mg/g, respectively. Healthy mangrove sites, on the other hand, contained lower sulfate and total sulfide content with mean values of 0.16 mg/g and 0.27 mg/g, respectively. Sulfate content at all the selected sites had relatively homogeneous distributions from the surface down to the 10 cm horizon (Fig. 3). Total sulfide concentrations in the adjacent community site sediment peaked at the 2-4 cm horizon (0.80 mg/g) and in the healthy mangrove sediment peaked at the 4-6 cm horizon (0.53 mg/g) before decreasing with greater depths in both sediments (Fig. 3(a), 3(b). For unhealthy mangrove sediment, total sulfide concentrations peaked at the near-surface interval (0-1 cm) with an average of 0.86 mg/g before decreasing at greater soil depths (Fig. 3(c)).

3.3 Relationships of sulfates and total sulfides with sediment and environmental factors

The Scatterplot of the matrix indicated pronounced nonlinearity of sulfates and total sulfides with OM, OC, TN, P, K, pH, salinity, EC, and TDS. The same parameters with additional categorical data (sediment-mangrove conditions) were applied for further analysis in a regression tree model. The summary of the predictive model of sulfate content showed a deviance of 0.0857, or a root mean square of 0.29, which indicates that our model strongly performed given its simplicity. The summary of the regression tree model of total sulfide content showed a lower deviance with a value of 0.0032, or a root mean square of 0.06, which indicates a very powerful prediction model. At the bottom of the terminal branches, the numbers illustrated the mean of sulfate and total sulfide content derived from a regression tree model.



Sulfate range: 0.24-0.40 mg/g
Total Sulfide range: 0.35-0.80 mg/g
(a)

Sulfate range: 0.14-0.18 mg/g
Total sulfide range: 0.07-0.53 mg/g
(b)

Sulfate range: 0.24-0.34 mg/g
Total sulfide range: 0.27-0.86 mg/g
(c)

Figure 3. The vertical distribution of mean sulfate and total sulfide content (mg/g) with 10-cm depth for: (a) adjacent community site sediment, (b) healthy mangrove sediment, and (c) unhealthy mangrove sediment

Our regression tree model suggested that OM was by far the most important factor driving the presence of sulfates and total sulfides in the sediments. Besides OM, the model revealed that K, pH, and TOC were correlated with sulfates (Fig. 4(a)). For example, the right branch showed that when OM was ≥ 11.03%, pH was ≥ 7.54, and OC was ≥ 4.68%, sediments contained about 0.26 mg/g sulfate content. Important factors associated with total sulfide content were OM, mangrove conditions, salinity, K, and OC. Interestingly, the mangrove condition factor was the second most important covariate explaining the amount of total sulfides (Fig. 4(b)). The tree can be interpreted such that, for example, sediments that contained OM <13.08% and surrounded by some mangroves and high density of mangroves with K ≥ 1.82 mg/g were correlated with a 0.49 mg/g average total sulfide content.

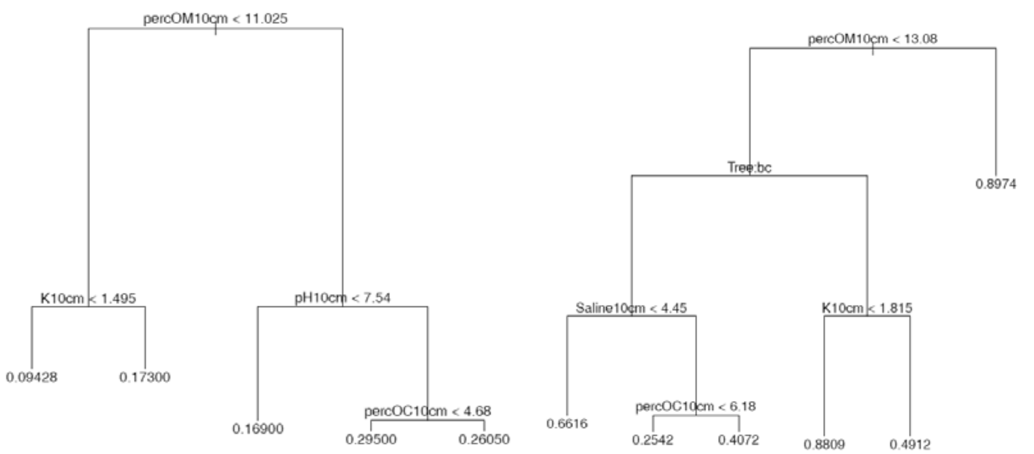
4. Discussion

4.1 Sedimentary organic matter, sulfate, and total sulfide concentrations

Our OM and AVS values were high when compared to findings from Khaodon *et al*. (2011) who found OM ranged from 0.75 to 15.81% and AVS ranged from 0.0001 to 0.1998 mg/g dry weight on the eastern coast of the Gulf of Thailand. Organic matter in the range of 0.6-4.0%, with the AVS ranged from 0.0004 to 0.1620 mg/g were observed in the inner Gulf of Thailand (Mingkwan *et al*., 2012). While in the south of Thailand, observed OM ranged from 1.03 to 7.51% and AVS ranged from 0.02 to 0.40 mg/g in the U-Taphao Canal (Nusong *et al*., 2014). Overall, our findings showed high total sulfide values compared to other locations in Thailand and the concentration of total sulfides was about three times the concentration of sulfates, implying that sulfate reducers used OM to stabilize one-third of the sulfate content to produce the total sulfides in the intertidal environment. In general, OM should be positively correlated with AVS; however, in some cases the amount of OM did not directly indicate the amount of total sulfides in the same way because more than 40% of the OM content cannot be used by sulfate-reducing bacteria (Meksampan, 2002). In addition, up to 50% of the OM flux to sediments was estimated to be mineralized by sulfate reducers and up to 80% of total sulfides produced by microbial sulfate reduction is reoxidized which adds a complex interrelationship in the natural sulfur cycle (Jørgensen, 1982).

4.2 Vertical distributions

Phuong *et al*. (2005) observed the concentration of AVS was higher in the inner bay area than in the central area, while both areas performed alike in vertical distributions with high concentrations at the 3 cm soil depth. Likewise, higher AVS content in sediment layers at 2-4 cm depths within the mangrove forests and lower AVS content on the bay side of the mangrove forests was observed (Simpson *et al*., 2004). Similar to our study, the high accumulation of total sulfide content stored in the top few centimeters of sediments at all the sites, which then decreased at greater soil depths. This can be explained by an anoxic environment. Dominant sulfate reduction and reaction of Fe oxides with dissolved H_2S can be assumed at soil



(a) (b)

Figure 4. Regression tree of sulfates and total sulfides at 0-10 cm, illustrating (a) prediction model of sulfate concentrations (mg/g) and (b) prediction model of total sulfide concentrations (mg/g)

depths of a few centimeters (Peiffer *et al*., 1992) because the sediment-water interface allows dissolved oxygen to penetrate only the top sediment surface. Interestingly, sediments with unhealthy mangrove environments at a soil depth of 0-2 cm contained the highest total sulfide content compared to the other sediment conditions. High total sulfides may be responsible for the die-back or poor mangrove growth rates which already explained seagrass mortality in other regions (Carlson and Forrest, 1982; Borum *et al*., 2005).

4.3 Factors affecting sulfate and total sulfide in the sediments

Organic matter was strongly correlated with the amount of sulfates and total sulfides since OM showed up at the top of all the branches in the regression tree models. In natural, during the sulfate reduction process, sulfate-reducing bacteria coupled with the oxidation of OM in sulfate ion as an electron acceptor to the production of sulphide (Heijs *et al*., 1999). Apart from the natural sulfide source, the OM-rich circumstances within our study site probably came from anthropogenic activities. When excessive OM is profound under limited oxygen conditions, it sometimes constrains the ability of microbes to degrade organic materials. Because OM contained approximately 58% OC as suggested by Sprengel (1827) or 50% as suggested by Pribyl (2010), OC would be a relatively important factor to explain the persistence of sulfate and sulfide content. However, it is unclear about the relationships between K and sulfates and total sulfides in the sediments.

Although low pH has been accounted for in the high production of sulfides, for example, at pH<5, almost the entire product is H_2S inhibitory (Icgen and Harrison, 2006). In the present study, pH did not appear to be a powerful explanatory variable for total sulfides. Instead, pH value split at 7.54, informing the influence of sulfates at this cutting number. Low pH directly inhibits the sulfate reduction rate because protonation of volatile fatty acids can be substituted for sulfate-reducing bacteria (Koschorreck, 2008). Several studies affirmed that salinity and the conditions of mangrove sediments were associated with total sulfides. It was found that high salinity can affect the ability of plant growth. The colonization of *Phragmites* expansion rates in marshes and found more extensive plants living at lower, rather than higher, salinity concentrations (Rice *et al*., 2000). In addition, plants may die when salinity is greater than 35% (Chambers *et al*., 1998). In our study, salinity is a subtle variable describing the presence of total sulfides compared to sediments in different environmental conditions.

5. Conclusions

There is great concern about high amounts of total sulfides across the sampled mangrove conservation area compared to other areas of the Gulf of Thailand and the southern region of Thailand. Spatial distribution analysis showed a decreasing trend of sulfate concentrations with increasing distance from land, whereas hotspots of total sulfide concentrations were found in the center part of the area. The vertical distribution of sulfates was fairly constant along the depth profile, while high concentrations of total sulfides were contained in a few centimeters of soil depth and decreased with soil layers at greater depths. Of the ten variables, the regression tree model depicted four variables involved with sulfates: OM, K, pH, and OC. The five variables involved with total sulfides were OM, sediment in different mangrove conditions, salinity, K, and OC. The outcome guided us to focus on OM, the major influencing factor for environmental management. When OM was over 13.08%, total sulfide content was positively associated with OM; otherwise, many factors were engaged in the limitation of OM. Total sulfides in a form of AVS concentration can be considered an indicator of over nutrient-rich sediments for assessing environmental quality and perhaps the die-back of mangroves in the sampled conservation area.

Acknowledgements

This work has been partially supported by the Sci Super III Fund (Ratchadaphiseksomphot Endowment

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Received 7 December 2016
Accepted 18 January 2017

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