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MERCURY ANALYSIS WITH PRINCIPAL COMPONENT ANALYSIS FOR WATER, SEDIMENT, AND BIOTA SAMPLES IN ACEH, INDONESIA

Khairi Suhud[1], Siti Wahidah[2], Ilham Maulana[1], Rinaldi Idroes[3], Suprayitno[4], Lelifajri[1] and Ahmad Fudholi[5]
[1]Jurusan Kimia, Fakultas Matematika dan Ilmu Pengetahuan Alam (FMIPA), Universitas Syiah Kuala, Banda Aceh, Indonesia
[2]Magister Kimia, Fakultas Matematika dan Ilmu Pengetahuan Alam (FMIPA), Universitas Syiah Kuala, Banda Aceh, Indonesia
[3]Jurusan Farmasi, Fakultas Matematika dan Ilmu Pengetahuan Alam (FMIPA), Universitas Syiah Kuala, Banda Aceh, Indonesia
[4]Jurusan Pendidikan Biologi, Fakultas Keguruan Ilmu Pendidikan (FKIP), Universitas Syiah Kuala, Banda Aceh, Indonesia
[5]Solar Energy Research Institute, Universiti Kebangsaan Malaysia, Malaysia
E-Mail: khairiukm@yahoo.com

ABSTRACT

The analysis of Mercury (Hg) in the water, sediment, and biota collected from the watershed area of Krueng Sabee (KS), Panga (P) and Teunom (T) has been conducted. Hg concentration was determined using Atomic Absorption Spectrophotometer. Meanwhile, the water pH, temperature, and salinity were determined with in-situ method. The in-situ determinations suggest that the qualities of KS, P and T waters are still under the standard threshold. Based on the analysis, the highest Hg concentration was found during the dry season, specifically in the sediment samples from the upstream area of River Krueng Sabee. On contrary, the lowest concentration was found during the rainy season, in the sediment samples from River Teunom. The evaluation of Hg distribution pattern in the water, sediment, and biota was conducted with Principal Component Analysis (PCA). It suggests the strong correlation between Hg concentration in the sediment with the one in the water and biota.

Keywords: sediment, biota, river, heavy metal, Hg, PCA, AAS.

INTRODUCTION

Krueng is a word from Acehnese language referring to river, where it becomes a general guide to put the word in the naming of a river, in Aceh Province. River is one of water sources, which has been utilized since a long time ago by humanity for many life supporting activities. These days, along with the industrial growth, river has been utilized for multiple purposes, along with the development of the civilization and human's culture, either directly or indirectly [1].

There are several rivers connected to the estuary in Aceh Jaya Regency, they are Krueng Sabee (KS), Panga (P) and Teunom (T) watersheds. The three watersheds carry the water mass originated from Mountain Ujeun, located in Panggong Village, Krueng Sabee District, Aceh Jaya Regency. Mountain Ujeun is known by the locals as the gold-rich region, in which it becomes the livelihood of the locals in the small scale with traditional method [2, 3]. One of gold ore processing is amalgamation that uses Hg in the processing [4]. Gold that has been exploited in Mountain Ujeun, transported to the local neighborhood for further processing.

Purnawan *et al*. [5] reported that Hg content in the sediment found in the estuary of KS river was around 0.76 mg/kg, Panga was 0.68 mg/kg and Teunom 1.03 mg/kg. Government Regulation No. 82 Year 2001 on water quality management and water pollution control, explains that the maximum threshold of Hg concentration in the waters is 0.001 mg/L. The toxic Hg is released from the gold processing, flowing and polluting the water and the sediment in the riverbed. The aquatic organism in the area may absorb the Hg, leading to a bioaccumulation [6-8]. Hg use for gold extraction in the small-scale gold mining, especially in Indonesia, has exhibited an indication of a man-made disaster [9].

Heavy metal content in the waters can describe the water origin, because of the different rock types with different heavy metal content. Heavy metals are divided in two groups, essential heavy metals (Zn, Cu, Fe, Co, Mn) and non-essential heavy metals (Cd, Pb, Cr, Hg). Essential heavy metals in a particular amount are needed by the living body, and can be toxic if consumed excessively. As for the non-essential heavy metals, the function in the living bodies are still unknown, even in contrary they can be toxic [10].

Industrial activities, human settlement, agriculture, as well as mining, in general, can cause a damage to the environment, such as water pollution, the degradation of natural resources' quality, land crisis, adverse health effect, the decrease of biological natural resources potentials, natural disaster, up to the downstream sedimentation [11]. This heavy metal can also negatively affect the people who use the water and the river organism [12].

As stated before, it is urgent to conduct a research and sampling during the dry and rainy seasons, which has been reported by the previous studies [13]. This study measures the Hg concentration in the waters of River KS, P, and T, which are suspected to be polluted by the Hg from the KS gold mining. Hg content is measured with Atomic Absorption Spectrophotometer (AAS) and the distribution pattern is analyzed with Principal Component Analysis (PCA) with XLSTAT software.

In 1971, Gabriel stated that biplot is a two-dimensional mapping of a principle component analysis (PCA). The information obtained from the biplot is a degree of relative similarities between the observatory objects, correlation between variables, variable value in an object, and a variability. The correlation between variables described by if the angle of two variables $< 90^{o}$, the

correlation is positive; but, if the angle of two variables > 90^o, the correlation is negative; but if the angle is smaller, the correlation is stronger. The object has the variable properties if the object is close to the variable.

The closer object position to the variable, the more dominant character of the variable on the object [14].

Hutagaol [15] had reported a study on the heavy metal content in water, sediment, and green clam with principle component analysis (PCA) procedure. The similar study had also been done on water, sediment, and biotas in the industrial area of Krueng Balee and in the non-industrial area Krueng Reuleung [16]. PCA method is a method to identify the pattern of a data and to present it in a way to determine the similarities and differences of such data [17].

PCA aims to reduce the high data dimension into lower data dimension with a tiny risk of information lost [18]. Therefore, to improve the data efficiency, a method is used to apply PCA on the original set of the data, thus, correlating variables of the original dataset will be changed into possible correlated variables. Before applying PCA, a dataset needs to be normalized priorly, thus each attribute with a bigger domain will not dominate the attribute with a lower domain. This allows the reduction of the dataset through the application of PCA, which will be applied on clustering algorithm [19]. However, metal concentrations or the results of PCA provide little information on environmental behavior and source identification of heavy metals [20].

In this study, Hg analysis with PCA for water, sediment, and biota samples in Aceh were analyzed with aims: (i) to analyze the content and contamination level of Hg in the water, sediment, and biota in KS, P, and T watersheds, Aceh Jaya Regency; (ii) to compare Hg concentration in the biota against the Hg concentration in the water, as well as in the sediment, collected from KS, P, and T watersheds, Aceh Jaya Regency; and (iii) to predict the distribution pattern of Hg in the water, sediment, and biota in KS, P, and T watersheds, in Aceh Jaya Regency, with XLSTAT software through PCA method.

MATERIALS AND METHODS

Description of the Study Area

This study was conducted from February to June 2019. The water sources flowing from Mountain Ujeun, Aceh Jaya Regency, to river KS, P and T were the sampling location. Hg analysis in the water, sediment, and clam (Batissa violacea) were conducted in Research Laboratory of Chemistry Department, Faculty of Mathematics and Natural Sciences, Universitas Syiah Kuala. Samplings were carried-out from the morning until the afternoon (08.00-12.00 AM) [21]. Water, sediment, and clam samplings were conducted in River KS, P, and T decided with purposive sampling. Each river was divided into 3 sampling points, upstream, midstream, and downstream, where the samplings were conducted in the two seasons; dry and raining seasons, as shown in Figure-1 and Table-1.

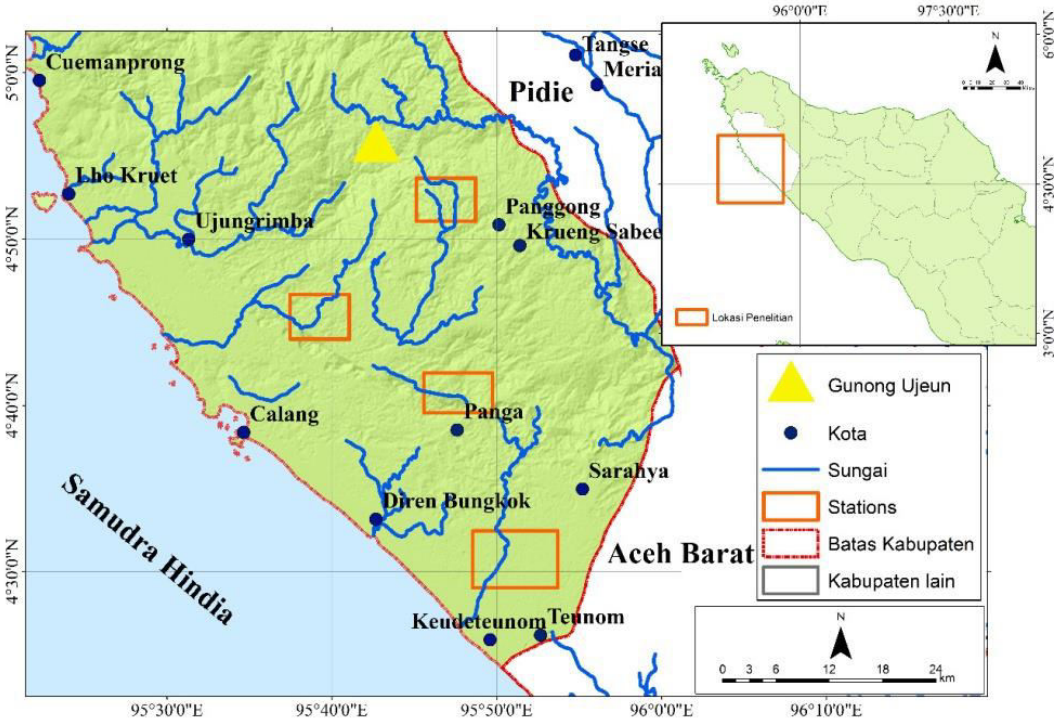

Figure-1. Map of the sampling locations.

Table-1. Coordinate points of water, sediment and *Batissa violacea* samplings.

River location	Sampling point		Coordinate
Krueng Sabee	**Upstream**	**(KS1)**	**4°41′03″ NL and 95°41′01″ EL**
	Midstream	(KS2)	4°38′32″ NL and 95°39′12″ EL
	Downstream	(KS3)	4°36′24″ NL and 95°38′40″ EL
Panga	Hulu	(P1)	4°33′48″ NL and 95°44′27″ EL
	Median	(P2)	4°33′35″ NL and 95°43′38″ EL
	Downstream	(P3)	4°32′33″ LU and 95°42′59″ EL
Teunom	Hulu	(T1)	4°26′29″ NL and 95°54′54″ EL
	Median	(T2)	4°29′33″ NL and 95°52′27″ EL
	Downstream	(T3)	4°32′02″ NL and 95°49′31″ EL

Tool and Material

Tools used include glass equipment, Eickman Grab, titration equipment, sampling bottle 250 mL, pH-meter, global positioning system, thermometer, evaporating dish, water bath, analytical balance, Atomic Absorption Spectroscopy (AAS) (Shimadzu AA 6300). The materials were Hg solution, Pb, concentrated HNO_3, concentrated $H2O_2$ and distilled water.

Water samples were collected on the surface layer with polyethylene bottle, while the sediment samples were taken from the same location with Eickman Grab in a particular depth [21].

Water Sample

River water was taken as much as 50 mL, and dried until the volume reached 15 mL by using water bath. Next, 5 mL concentrated HNO_3 was added into the sample, and the mixture was heated for 15 minutes. Then, the mixture was added with another 5 mL concentrated HNO_3, reheated for another 15 minutes. The sample mixture was then measured with AAS [21].

Sediment Sample

The sediment was weighed for 5 g, dried in the oven to remove the water. Next, the sample was treated with ashing to remove the organic compounds with an oven at 105 ^oC for 12 h. The sample was left cold on the evaporating dish, added with 5 mL concentrated HNO_3, and heated for 15 minutes. HNO_3 as much as 5 mL was re-added, and the sample was reheated for another 15 minutes. Next, the sample is moved to the volumetric flask 25 mL. Lastly, the sample was analyzed with AAS [21].

Clam (*Batissa violacea*) Sample

Batissa violacea sample was placed on the evaporating dish, evaporated in an oven at 105 ^oC for 12 h. After left cold, the sample was grinded until it was homogenous. The sample, as much as 40 g, destructed in a beaker glass with 10 mL concentrated HNO_3. It was heated with a hot plate at 85 ^oC for 8 h. An hour before the process ended, 3 mL H_2O_2 was added to prevent the growth of mollusk. The sample, which was in the liquid phase, was moved to a volumetric flask, in which the volume was made for 200 mL with deionized water. The sample was left overnight for further analysis with AAS [22].

Determination of the Heavy Metal Content

Standard calibration curve to determine Hg heavy metal was obtained from the measurement of standard solution absorbance of the respective element at the optimal condition. The range of the Hg standard solution was 0-8 µg/L. The calibration curve was obtained by plotting the concentration vs. the absorbance of the respective elements. Standard solution and all the prepared samples were measured against the Hg based on the respective wavelengths. The absorbance generated by the sample was used to calculate Hg concentration with the calibration curve.

Descriptive Analysis

To investigate the condition of heavy metal pollution in the water, the Hg analysis in the water will be compared with the quality standard based on the Government Regulation No. 82 Year 2001.

Principle Component Analysis (PCA) Method

The habitat quality data, either the physical or chemical, which had been measured, were presented in the average value and in the charts. The correlation between the physical and chemical qualities of the habitat was observed with an observatory station, thus the physical and chemical qualities change or difference can be identified. The physical and chemical qualities were the y-axis; meanwhile the observatory station was the x axis. The major final results of component analysis were the observable difference of the physical and chemical parameters special distribution of the waters, among multiple observatory points. PCA was constructed with XLSTAT software.

Hg concentration analysis in the area was calculated as the abundance, based on the importance value index and species diversity with Shannon diversity index. The spatial distribution patterns of the samples were analyzed with PCA multivariate analysis approach. The

use of this method aimed to determine the distribution pattern of Hg and the affecting environmental factors.

RESULTS AND DISCUSSIONS

Hg Concentration in the Water and Sediment

Hg concentrations in the water, collected from 3 sampling points, during the dry season, indicate a higher Hg concentration found in River KS, compared to the others (River P and T). It is due to the fact that the sampling location, in River KS, is close to the area with mining activities. For sediment samples, a higher Hg concentration was also found in River KS due to the heavy metal precipitation in a long period of time, thus allowing the increase of the heavy metal concentration.

The heavy metal accumulation in the sediment is easier to occur, allowing the increase concentration of the heavy metal, as opposed in the water [23]. Based on the data in Table-2, the highest Hg concentration was found at the sampling point in the River KS downstream, while the lowest was found in the River P downstream part. In rainy season, the highest concentration was found in the River T downstream, while the lowest was found in the River KS midstream. The highest Hg concentration in the sediment during dry season was found in the River KS upstream, and the lowest was found in the River P downstream. During the rainy season, however, the highest Hg concentration was given by the samples collected from River P midstream, while the lowest was from River T. For clam samples, during the dry season, the highest Hg concentration was detected in the downstream of River KS, meanwhile the lowest was obtained in the downstream of River T. In rainy season, Hg concentration is found to be the highest in River P downstream, and the lowest in River T downstream.

Table-2. Hg concentration (µg/L) during dry and rainy seasons in the water and sediment, in River Krueng Sabee, Panga, and Teunom area.

No	Sampling point	Dry season		Rainy season	
		Water	Sediment	Water	Sediment
1	KS1	0.0799	6.2230	0.0000	0.1599
2	KS2	0.0949	0.3758	0.0070	0.0770
3	KS3	0.3328	0.2488	0.0100	0.0720
4	P1	0.2149	0.5227	0.0530	0.0000
5	P2	0.1679	0.2838	0.0420	0.2778
6	P3	0.0176	0.1409	0.0450	0.0200
7	T1	0.2189	0.1749	0.0450	0.0000
8	T2	0.1269	0.2039	0.0390	0.0000
9	T3	0.0979	0.3898	0.0560	0.0000

The results explain that the water and sediment samples give the highest Hg concentration were collected from River KS area. This is due to the fact that the other two rivers, P and T, are located far away from the mining sources. River P area has a slower river flow than the ones in River KS and T. If the water volume decreases, the Hg concentration in the sediment increases. In the same condition, the Hg concentration in the biota also increases. The results of Hg concentration found in the biota indicate the increase of Hg concentration in the sediment leading to the increase of Hg concentration in the biota samples during the rainy season. This explains the results obtained for River P and T downstream, which is comparable with the study by Kassegne *et al*. [24], where during rainy season, higher Hg concentration was found in the sediment and biota samples collected from the reservoir, hospital, and urban area, than during the dry season.

Hg analysis with AAS gave the results suggesting that River KS is polluted with Hg released from the tailing of gold process, done by the local. The highest pollution was found at the sediment collected from the river upstream. Syarifah *et al*. [25] reported the same Hg analysis in the KS area, where the highest Hg concentration was also found in the upstream. Based on the Hg concentration from the water and sediment samples, the statistical test suggests that $F_{calculated} < F_{theoretical}$, which means the H0 is accepted and the H1 is rejected, thus can be concluded that there is no significant difference of Hg concentration of each analyzed sample collected from the KS, P and T rivers area, with the data shown on F treated as shown in Table-3.

Table-3. Statistical test of average comparison of Hg in the water and sediment during dry and rainy seasons in KS, P and T rivers area.

Variation source	DB	JK	KT	$F_{calculated}$	$F_{theoretical}$ (0.05)
Treatment	3	2.5484	1.2742	1.9213	2.85
Error	36	35.1502	0.6632		
Total	39	37.6986			

Hg Concentration in Biota (*Batissa violacea*)

In general, heavy metals get in the biota tissue cannot be released and tend to be accumulated. The biotas are exposed with the heavy metals continuously in the waters, thus increasing the concentration and causing the bioaccumulation on the aquatic biotas [26]. Analysis results of the biota sample (Table-4) suggest that the Hg concentration was only found in the downstream of the three rivers. In dry season, Hg concentration was found to be the highest in River KS and the lowest in River T. Furthermore, in rainy season, the highest concentration was obtained in River P downstream, meanwhile the lowest was in River T downstream. Hg concentration was not detected during the dry season in River P, meanwhile during the rainy season, the concentration was not detected in River KS downstream.

Table-4. Hg concentration (µg/L) in *Batissa violacea* in River KS, P and T during the dry and rainy seasons.

No	Sampling points	Dry season	Rainy season
1	KS3	0.0070	0.0000
2	P3	0.0000	0.2149
3	T3	0.0050	0.0469

Difference of concentration is stem from the biota samples collected in the river downstream, and the Hg in the biota samples was accumulated, along with the accumulation in the sediment, which is the habitat of the biota. The high metal concentration in the sediment also affects the metal concentration consumed by the biota. In the river downstream, biota samples live in the mud and grow in the environment, where the pH is similar with the waters condition near the sea estuary. Nevertheless, salinity also affects the metal concentration in the biota samples. Where in that condition, the Hg absorbance on the biota tends to be reduced, due to the higher salinity in the downstream. And so, the otherwise, if the salinity of the waters decreases, the metal absorbance on the biota will increase. It is in line with the report by Bryan [27], that one of the factors determining the degree of Hg absorbance in the biota samples is salinity. Hg absorbance tends to decrease as the water salinity increases.

Iwandikasyah *et al.* [28] reported that the accumulation of Hg content in River KS downstream is higher than in the upstream and in the midstream of the river. Hg is suspected to be present in the riverbed and absorbed by *Batissa violacea*. The river flowing to the estuary is located in the coasts, where the coast area is less contaminated by the pollutant. However, the location in the coast can receive the effect from the tide. Therefore, the heavy metal content in that area is suspected to be originated from the sediment absorbing the heavy metal. Nurfadillah *et al.* [29] also reported that Hg in *Batissa violacea* collected from River KS, P and T are still edible, and in accordance with the food standard [21]. It allows the Hg content in the Bivalvia, mollusk, and clam under 1 mg/kg to be consumed.

The results shown in Table-4, suggest that the Hg content is relatively higher in during the rainy season compared to the Hg content found in the dry season. This is due to the fact that the salinity in the rainy season decreased, thus allowing the increase of the Hg absorbance on the biota. Table-4 is also in line with the results of the Hg concentration in the water, suggesting that Hg is accumulated in the biota (*Batissia violacea*) as reported by Wanna *et al.* [30] and Nurfadillah *et al.* [29].

Hg concentrations obtained from the biota in the downstream of the three rivers suggest that the highest concentration was found in River P downstream, thus the result of the statistical test obtained is $F_{calculated} < F_{theoretical}$, which means H_0 is accepted and H_1 is rejected. It means that there was no significant difference of Hg concentration found in each analyzed sample collected from the river downstream of KS, P, and T, with the data in the treatment $F_{theoretical}$ shown in Table-5.

From the Hg concentrations contributing to the classification of the analyzed sample, it is found that the river water has a big influence against the distribution condition of Hg metal. It also affects the condition of water, the aquatic biodiversity, and the humans living near the river, especially the one close to the wastewater disposal of the mining that use Hg.

Table-5. Statistical test of Hg concentration in the biota (*Batissa violacea*) in River KS, P, and T.

Variance Source	DB	JK	KT	$F_{calculated}$	$F_{theoretical}$ (0.05)
Treatment	1	0.0104	0.0104	1.630	6.61
Error	4	0.0256	0.0064		
Total	5	0.0360			

Hg Distribution Patterns in Dry and Rainy Seasons in River KS, P, and T

The distribution pattern of Hg concentration in the water, sediment and *Batissa violacea*, during dry and rainy season, in River KS, P and T was analyzed with PCA. The PCA employs a variable approach (Hg water, Hg sediment, Hg *Batissa violacea*, during the dry and rainy seasons) and variable (KS 1-3, P 1-3 and T 1-3). To investigate whether the correlation between the variables is positive or negative, it can be observed in Figure-2.

Cumulative percentage in the dry and rainy seasons with eigenvalue and total variance is obtained at 6 initial PCs, they are: PC1 36.060%; PC2 26.512%; PC3 17.521%; PC4 14.396%; PC5 5.397% and PC6 0.114%. Based on the stated information, PC1 and PC2 are chosen because they have more variance in the data. Data variance can be explained in Figures 4.3, that PC1 36.06 % and PC2 26.51 % contribute to 62.57 % (combination of PC1 and PC2) (Figure-2 and Table-6).

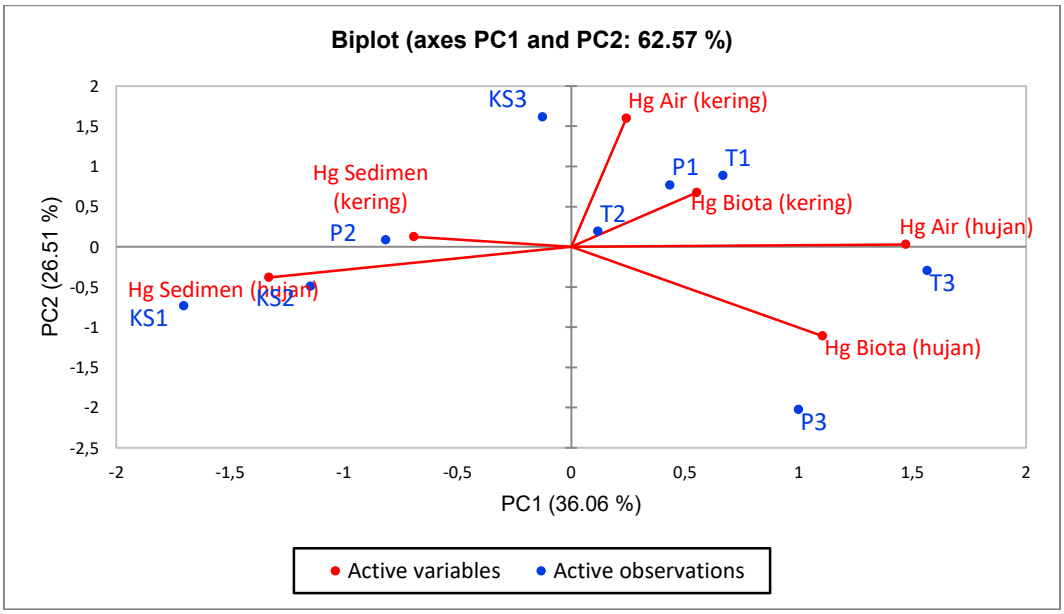

Figure-2. PCA of Hg found in dry and rainy seasons

Table-6. Principle component analysis of Hg concentration distribution during the dry and rainy seasons.

	PC1	PC2	PC3	PC4	PC5	PC6
Eigenvalue	**2.164**	**1.591**	**1.051**	**0.864**	**0.324**	**0.007**
Variability (%)	36.060	26.512	17.521	14.396	5.397	0.114
Cumulative %	36.060	62.572	80.093	94.489	99.886	100.000

Based on Figure-2, Hg in the sediment collected from KS3 and P2 during dry season, has a negative correlation because of the varibles are at the < 90 ^{o}C and the sediment variables during the dry season have smaller angle, thus the correlation with the Hg sediment in the rainy season variables is stronger. On the other hand, Hg *Batissa violacea* during the dry and rainy seasons shows a negative correlation. It means, if Hg in the biota at P1 (dry) increases, the Hg biota at the P3 and T3 (rainy) uncertainly increases, which is in line with Table-5.

On the Hg concentration of the sediment in the dry season, point P2 has a negative correlation against KS2. Meanwhile for the sediment in the rainy season, the correlation is positive between KS2 and KS1. If the Hg concentration at KS2 undergoes an increase, then the Hg concentration at KS1 automatically will increase along with the river condition, where they are in the same

quadrant (Figure-2). Next, Hg concentrations in the water during the dry and rainy season give a result to a positive correlation at point T2 and T2. That means, if the Hg concentration at T1 increases, then the Hg concentration against T2 will also increase.

Hg concentration in biota (*Batissa violacea*) at P3 and T3 during the rainy season gives a positive correlation. It means, if Hg concentration in the biota at P3 increases, the Hg concentration at T3 increases as well, with the assumption that in sampling location is in the river downstream. On the contrary, Hg water at T1 during dry season and Hg sediment at KS3 during the dry season give a negative correlation, with different quadrants. It means, if Hg T1 increases, the Hg sediment KS3 uncertainly increases. Hg concentration in the sediment at KS1 during rainy season and Hg biota (*Batissa violacea*) at P3 in the rainy season also give a negative correlation, which means the increase of Hg concentration in the sediment at KS1 does not guarantee the increase of Hg biota at P3.

CONCLUSIONS AND RECOMMENDATIONS

The followings are the conclusions, based on the results obtained in this study:

a) The waters quality in the River KS, P, and T areas is still in line with the quality standard set by the Indonesian Government.

b) The Hg concentration in the water and sediment increased during the dry season and decreased during the rainy season.

c) The Hg concentration in Batissa violacea decreased in the dry season and increased in the rainy season.

d) The results of PCA exhibit a strong correlation between the Hg concentration in the dry season and the Hg concentration in the rainy season.

e) High concentration of a metal in a sample leads to another high metal concentration, if the sample is in the same river and quadrant in PCA chart.

The followings are the recomenmendations, based on the results obtained in this study:

a) The monitoring for water quality, especially in regard of heavy metal, in River KS, P, and T, which can be done once a year minimally.

b) Further study for the water quality parameters affecting the heavy metal concentration in the sediment and Batissa violacea as well as the length of mining duration.

c) Serious attention of the local communities, regent government, and regional drinking water companies on the water and biota use or consumption in the River KS, P, and T areas.

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