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## The Sediment Profile and Heavy Metal Content of Lead (Pb), Copper (Cu), and Silver (Ag) in The Waters of Saparua Island

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#### Abstrak

This study determined the distribution levels of heavy metals Pb, Cu, and Ag in sediments in the waters of Saparua Island and characterized the sediments in the waters of Saparua Island. Parameters are physical and chemical parameters of waters, determination of particle size, determination of total organic carbon (TOC), digestion of sediment samples for SSA testing, and preparation of calibration curves. The results obtained levels of copper metal contained in the sediments in the waters of Saparua Island ranged from 0.1583–1.9531 mg/L and <4–78 ppm. The level of lead metal contained in the sediments in the waters of Saparua Island ranges <12-26 ppm. The level of silver metal contained in the sediments in the waters of Saparua Island ranges <7 ppm. Metal oxides, especially metal oxides formed from Cu, were found in sediments in the waters of Saparua Island with location points P.8 and P.9, namely CuO 0.0056% and 0.0031% and in sediments in Saparua Island waters with a value of 20 in sediment samples. P.2, P.3, P.6, P.8, P.9, and P.11 have the highest intensity which is the characterization of metal oxide  $2\Theta$ = 27.7464°, 29.4759, 26.2979, 26.6686, 26.7007, and 24.3695 indicate the presence of CaO (calcium oxide) phases at points P.2, P.3, and P.6. the SiO<sub>2 (silica)</sub> phase which is the most abundant metal in sediment samples P.8, P.9, and P.11 based on the XRF test.

Keywords: Metals, sediment characterization, TOC, SSA

#### INTRODUCTION

Heavy metal pollution that enters the aquatic environment will dissolve and accumulate in sediments or biota and may increase over time, depending on the conditions of the aquatic environment. Metal bioaccumulation in a marine organism is the first step before the organism shows its response to contaminants and geochemical cycles. The process of chemical bioaccumulation of heavy metals is a reaction to the formation of complex compounds between heavy metals and the cells of organisms that function as ligands. This process is explained through the Ligand Biotic Model theory, namely, free metal ions or their derivatives are designed to predict how these heavy metals interact with aquatic organisms (Natsir et al., 2019).

Heavy metal pollution is increasing with the process of increasing industrialization. Heavy metal pollution in the environment can pose a health hazard to humans, animals, and the environment. The disturbing effects of heavy metals on human health depend on which part of the heavy metals are bound in the body and the magnitude of the exposure dose. The ability of marine biota (fish, shrimp, and mollusks) to accumulate heavy metals in waters depends on the type of heavy metal, type of biota, length of exposure, and environmental conditions such as pH, temperature, and salinity. The larger the size of aquatic biota, the accumulation of heavy metals will increase. The amount of heavy metals absorbed and distributed in fish depends on the form of compounds and pollutant concentrations, the activity of microorganisms, the texture of sediments, and the types and elements of fish that live in that environment (Emon et al., 2023).

Male's research (2017) on the analysis of cadmium (Cd) and lead (Pb) levels in sediments in the Inner Ambon Bay and Male's research (2019) on the analysis of mineral types and levels of copper (Cu) and lead (Pb) in copper mining areas in Wetar Island shows a pattern of distribution of heavy metal concentrations in the inner Ambon Bay area and Wetar Island (Male et al., 2017; Male et al., 2019). It is possible for the distribution of heavy metals to be found in other areas in Maluku waters, one of which is the waters of Saparua Island. The waters of Saparua Island are waters surrounded by densely populated islands. These waters have become a place of activity for residents as a location for disposal of household, industrial, and sea transportation waste and so on around settlements, so it is necessary to conduct research using several methods.

Densely populated settlements have resulted in sediment pollution along Saparua waters, such as oil spills, household waste, and fishing bombing using explosives. There have been many marine studies on marine pollution, but there has been no research on Saparua waters, so this research was conducted to find out the distribution of heavy metals of Lead (Pb), Copper (Cu), and Silver (Ag) concentrations in sediments in Saparua island waters.

### METHODOLOGY

#### Materials and instrumentals

The materials used in this study included: Sediment samples, label paper, markers, plastic bags, analytical balance (Cyberscan CON 110), Mineral-Free water (Aquabides), concentrated HNO<sub>3</sub> (E.Merck), concentrated HCl (E.Merck), Hydrogen Peroxide (H<sub>2</sub>O<sub>2</sub>) solution (E.Merck), 1000 ppm Cu mother liquor, 100 ppm Cu standard solution, Whatman filter paper No.40. The tools used in this study included: Global Positioning System (GPS), plastic spoons, digital pH meter (Hanna Instrument), thermometer (Philips haris), textile gloves, plastic bags and plastic containers, Styrofoam, a set of glassware (pyrex), 100 Mesh sieve, burette (pyrex) and stative, Hot plate (Cimarec), oven, X-Ray Fluorescence (XRF) instrument, PANalytical X-ray Diffraction instrument X'Pert HighScore (XRD), Atomic Absorption Spectrophotometer (Thermo Scientific Ice 3000 AA05183205 V1.30).

### Methods

# Sample preparation

The tests were conducted for chemical and physical parameters, such as pH, temperature, and TDS, before taking sediment samples. The thermometer used to measure temperature is immersed in the water sample and left for 2–5 minutes until the temperature scale on the thermometer shows a stable number and is then recorded. The same procedure can be carried out for measuring pH using a pH meter, for TDS, and determining the sampling location using GPS. Sediment samples were taken with a plastic spoon to prevent the samples from being contaminated. The sediment was put in a plastic bag, labeled, and then placed in a box that was given ice (*coolbox*), then the sample was taken to the laboratory for further treatment.

#### Physical and chemical parameters of waters

Measurement of physical and chemical parameters of water is carried out in two ways, namely, directly in the field (*in-situ*) and indirectly in the laboratory (*ex-situ*). The physical and chemical parameters of the water measured were pH, temperature, salinity, and the coordinates of the research location. Location and coordinate sampling in the waters of Saparua Island can be seen in Table 1.

Table 1. Location and coordinate sampling in the waters of Saparua Island

No	Sample Code	Location	Coordinate
1	P 1	Paperu	S 03°34'56,22''
1	1.1	ruperu	E 128°39'06,47''
2	Р2	Tiouw	S 03°34'26,46''
2	1.2	1100 w	E 128°39'10,80''
2	D 2	Conomio	S 03°34'23,80''
3	r.3	Saparua	E 128°39'15,14''
4	D 4	Siri Sori	S 03°34'55,74''
4	P.4	Amalatu	E 128°41'08,83''
5	P.5	Siri Sori	S 03°35'44,34''
5		Islam	E 128°42'16,39''
6	P.6	77 1	S 03°34'30,04''
		Kulur	E 128°39'38,03''
7	D 7	D'-	S 03°31'38,07"
/	P./	Pia	E 128°38'42,04''
0	<b>D</b> O		S 03°36'15,04''
8	P.8	Tunana	E 128°41'24,06"
0	DO	T1 1	S 03°30'09,60''
9	P.9	Ihamahu	E 128°41'36,02"
10	<b>D</b> 10	T/ 1	S 03°29'07,38''
10	P.10	Itawaka	E 128°42'32,08''
11	D 11	<b>N</b> 1	S 03°34'07.60''
	P.11	Portho	E.128°36'08,55''
10	P.12	· ·	S 03°35'17,01''
12		Haria	E.128°37'07,32''

#### **Determination of particle grain size**

Sediment samples were dried at 70–80 °C for 24 hours; after drying, they were weighed, and the numbers obtained were recorded as dry weight. The samples were soaked again, which had been weighed, for 5 hours to release the granules. Sieves are arranged according to size from bottom to top; residue section plastic tray 0.032, 0.063, 0.090, 0.250, 0.500, 1.00, 2.00, and 4.00 mm. The soaked sample is placed at the top of the sieve sequence, which is 4.00 mm in size. The sample is then washed with running water while dipping with a brush to remove one grain from

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another, and each sediment fraction left on the sieve is transferred to a 100 mL aluminum *tray*. Each sample fraction was dried in an oven at 70–80 °C for 2 hours until constant. Each sample is weighed according to grain size. The sieving process is correct if the weight figures obtained are the same.

#### **Determination of Total Organic Carbon (TOC)**

The 5 g sediment sample was put into a 50 mL beaker and added to an  $H_2O_2$  solution until bubbles appeared from the sediment or until it looked boiling (the aim is to oxidize organic matter). After that, it was heated on a hotplate at 90 °C until it was clear. The clear was dried in an oven at 150 °C, then cooled in a desiccator, and weighed with an analytical balance to obtain a constant weight.

# Sample preparation for SSA, XRF and XRD testing

The sediment samples were evenly placed in the styrofoam cup to completely dry in the oven at 150 °C for 6 hours. The dried sediment samples were crushed until smooth and then sieved with a 100 mesh sieve, after which each sediment sample was separated for SSA, XRF, and XRD testing.

# Destruction process of sediment samples for SSA testing

The sediment sample was weighed 2 g into a glass beaker. After that, 2 mL of concentrated HNO<sub>3</sub> and 6 mL of concentrated HCl (1:3) were added, then allowed to stand for 1 hour, and heated slowly until dry over an electric heater. The distilled water 14 mL was added until it boiled, then filtered and put into a 100 mL measuring flask, and distilled water up to the mark was added for further testing with AAS.

#### Creation of a Cu calibration curve

The solution of 100 ppm copper standard was prepared by taking 10 mL of 1000 ppm standard solution into a 100 mL measuring flask, then adding 10 mL concentrated HNO<sub>3</sub> after adding distilled water up to the mark. The 10 ppm of Cu standard solution was prepared by taking 10 mL of a 100 ppm standard solution, then diluting it in a 100 mL measuring flask, after which distilled water was added up to the mark. The series of Cu standard solutions were taken as much as 2, 4, 6, and 8 mL of 10 ppm standard solution, respectively, then diluted in a 100 mL measuring flask and added distilled water up to the mark to obtain a concentration of 0.2, 0.4, 0.6, and 0.8 ppm. The absorbance of the solution was measured with AAS at 324 nm, then a standard concentration vs absorbance curve was made, and the linear regression equation was obtained.

### **RESULTS AND DISCUSSION**

#### **Chemical and Physical Parameters**

The measured parameters are pH (chemical parameter), temperature, and salinity (physical parameter) for points 1 to 12. The map of sediment sampling points in the waters of Saparua Island can be seen in Figure 1.



Figure 1. Map of sediment sampling point of Saparua island waters

#### Acidity Level (pH)

The results of pH measurements can be seen in Figure 2.



Figure 2. Histogram of pH of Saparua island

Measurement of the pH of seawater is relatively the same from point 1 to point 12; that is, at a pH of 8.1–8.5, the seawater is in an alkaline state. Research conducted by Liu et al. (2019) shows that pH in alkaline conditions can reduce the solubility of several types of metals. In research using mass spectrometry analysis techniques to measure the solubility of metals at different pH levels, results show that metals become less soluble in water as the pH increases.

#### Temperature

Changes in water temperature will affect the physical and chemical processes of the waters. Changes in water temperature will affect the activity of microorganisms and oxygen consumption in the water (Tan et al., 2021). The results of temperature measurements can be seen in Figure 3.



island waters

Seawater temperature measurements are carried out during the day. The results of temperature measurements at locations P.1 to P.12 ranged from 25-38 °C. Figure 2 shows that location P.1 has a temperature of 38 °C because this location is not blocked by trees or mangroves, so the temperature at this location is higher than other locations. The temperature at locations P.2 to P.12 ranges from 25–28 °C. The highest temperature is at P.1, which is 38 °C. This is because the intensity of sunlight directly penetrates the water body, causing the amount of dissolved  $O_2$  in the water to decrease. The lowest temperature at P.6 is around 25 °C due to the fact that around the river, there are tall mangrove trees that block the intensity of sunlight.

#### Salinity

Based on measurements of the salinity value in the waters of Saparua Island, it ranges from 20 ppm to 30 ppm. The salinity value in the waters of Saparua Island is below the quality standard for marine biota, which ranges from 33 ppm to 34 ppm (KMNLH, 2004). The results of salinity measurements can be seen in Figure 4.



island waters

The spatial distribution of salinity is shown in Figure 4. It can be seen that the salinity value tends to be low from the offshore direction towards the middle of the waters. Seen at points P.1, P.2, P3, P.4, P.5, P.6, P.8, P.9, P.10, P.11 have salinity values that tend to be higher than Point P.6 and P.12. The two points are in an open area and are directly facing the land area with the neighboring island as well as the land area on Saparua Island which has rivers or small streams, causing a mixture of seawater and river water so that the salinity value of the waters becomes low.

#### **Sediment Grain Size Analysis**

Determination of sediment size aims to determine the relationship between sediment particle size and organic matter, which affects the percentage of heavy metal content in sediments in the waters of Saparua Island. In accordance with test results data from the Geological Physics Laboratory of the National Research and Innovation Agency (BRIN) Ambon, the classification of sediments is shown in Table 2.

Table 2. Sediment classification

Sample	Gravel	Sand	Sludge	Classification
code	(%)	(%)	(%)	Classification
P.1	36.56	51.89	3.30	Sandy gravel
P.2	20.07	79.01	4.14	Sandy gravel
P.3	53.04	65.58	5.46	Gravelly sand
P.4	9.59	90.31	0.68	Gravelly sand
P.5	3.31	96.59	0.25	Gravelly sand
P.6	8.21	90.59	14.17	Muddy sand
P.7	4.25	94.96	16.64	Muddy sand
P.8	29.42	66.78	11.58	Muddy sand
P.9	40.62	52.57	14.56	Muddy sand
P.10	75.88	44.48	0.65	Mud gravel
P.11	3.36	96.58	1.00	Mud gravel
P.12	25.37	74.11	1.39	Mud gravel

Based on the data in Table 2, the grain size of the sediment in the waters of Saparua Island is dominated by muddy sand, and this can be seen by the presentation of muddy sand at 4 locations ranging from 11.58–16.64%. Sediment grain size distribution is influenced by several factors, such as the type of transport agent, local winds, and currents, which also play a role in sediment distribution. The deposition of silt sediments is followed by the accumulation of organic matter at the bottom of the waters, while coarse sediments have lower organic content because the fine particles do not settle (Male et al., 2017).

#### **Total Organic Carbon (TOC) Analysis**

Total organic carbon in sediments is the amount of carbon bound in organic compounds originating

from decaying animals or plants which then sinks to the bottom of the water and mixes with silt, this parameter is often used as an indicator of a body of water. The yield of total organic carbon is shown in Table 3.

Table 3. Total Organic Carbon data of sediment

		samples	
No	Sample	Total Organic	Sadimont Tuna
	Code	Carbon (%)	Sediment Type
1	P.1	53.78	Sandy gravel
2	P.2	67.32	Sandy gravel
3	P.3	43.58	Gravelly sand
4	P.4	65.77	Gravelly sand
5	P.5	64.77	Gravelly sand
6	P.6	44.63	Muddy sand
7	P.7	34.86	Muddy sand
8	P.8	34.26	Muddy sand
9	P.9	22.40	Muddy sand
10	P.10	62.16	Mud gravel
11	P.11	67.00	Mud gravel
12	P.12	51.82	Mud gravel

From the test results, we can get a very high TOC value. Namely, P.2 of 67.32%, and the lowest is P.9 of 22.40%. This is due to the coarse grain size of the sediment, so the TOC absorption is smaller when compared to the finer grain size of the sediment. Around the waters of Saparua Island, many plants are found which are thought to be able to add to the total organic carbon concentration. Aquatic plants can reduce sediment resuspension and increase TOC concentrations in the waters. This is due to the role of aquatic plants in reducing the speed of water flow and stabilizing sediments at the bottom of the waters, thereby reducing the occurrence of sediment resuspension, which can interfere with water quality. (Fen et al., 2017).

#### Atomic Absorption Spectrophotometer (AAS) Analysis

The absorbance measurement results of the Cu standard solution are plotted against the concentration of the Cu standard solution, as shown in Figure 5.





The level of Cu metal that accumulated into the waters of Saparua Island was very different because it could be seen from the point of view of the size of the sediment grains, and the total organic height at each sediment sample point was different. At 12 sampling points, the metal content was below the threshold value, according to *the Canadian Council of Ministers for the Environment* (CCME) in Setiawan & Subiandono (2015), which was 18.7 mg/kg. The results of measurements of Cu levels are shown in Table 4.

Table 4. The result of Cu metal content and percent (%) sludge

ŊŢ	Sample_ Code	Cu co	ontent	Sludge	Total Organic
No		(mg/L)*	(ppm)**	(%)	Carbon (%)
1	P.1	0.2010	<4	3.30	53.78
2	P.2	0.3104	4	4.14	67.32
3	P.3	0.7863	11	5.46	43.58
4	P.4	0.2029	5	0.68	65.77
5	P.5	0.2194	6	0.25	64.77
6	P.6	0.2246	4	14.17	44.63
7	P.7	0.5938	6	16.64	34.86
8	P.8	1.9531	78	11.58	34.26
9	P.9	0.8604	21	14.56	22.40
10	P.10	0.1790	5	0.65	62.16
11	P.11	0.1863	10	1.00	67.00
12	P.12	0.1583	5	1.39	51.82

Note:

\*testing at the Ambon Industry Standards and Services Center \*\*testing at the Geological Survey Central Laboratory

The lowest Cu content was at P.12 when compared to other points. Test results for Cu levels at the Ambon Industrial Standards and Services Center and obtained P.12 were very low, namely 0.1583 mg/L. The analysis results for Cu levels analyzed by the Geological Survey Center Laboratory for Cu levels were very low at point P.1 with levels <4 ppm. The total organic matter obtained was low at 51.82% and 53.78%, percent (%) silt was 1.39% and 3.30%. This is due to the presence of river water, causing most of the metals not to be deposited in the sediments around the coast.

The results of testing for Cu levels at the two test sites showed that the same point in the sediments of the waters of Saparua Island at P.8 was 1.9531 mg/L and 78 ppm, sludge was 11.58%, and the total organic carbon content was 34.26 higher from another point, the high concentration of Cu at point P.8 can be sourced from the inclusion of Cu metal elements in the natural and non-natural environment. Data from

research on Pb levels in sediments in the waters of Saparua Island are shown in Table 5.

No	Sample Code	Pb Content (ppm)	Sludge (%)	Total Organic Carbon (%)
1	P.1	<12	3.30	53.78
2	P.2	13	4.14	67.32
3	P.3	20	5.46	43.58
4	P.4	24	0.68	65.77
5	P.5	13	0.25	64.77
6	P.6	<12	14.17	44.63
7	P.7	<12	16.64	34.86
8	P.8	26	11.58	34.26
9	P.9	<12	14.56	22.40
10	P.10	<12	0.65	62.16
11	P.11	31	1.00	67.00
12	P.12	<12	1.39	51.82

Table 5. The result of Pb content

Based on Table 5, at 12 sediment sampling locations in the waters of Saparua Island, the levels of Pb metal obtained due to the calculation results were still below the threshold set by the Swedish Environmental Protection Agency (SEPA) 2000, with a threshold range of 30–35 mg/kg.

The Pb level at location P.11 had the highest Pb level, namely 31 ppm. Several factors caused the metal content to be high at this location, such as depth, calm or not fast river flow, and high total organic content (Indrawan & Putra, 2021). Pb levels at locations P.1, P2, P.3, P.4, P.5, P.6, P.7, P.8, P.9, P.10, and P.12 have Pb levels sequentially -respectively are <12, 13, 20, 20, 24, 13, <12, <12, 26, <12, <12, and <12 below the threshold value. This can also be seen from the shallow depth, strong water currents, and the ability to absorb heavy metals is very small (Arinda & Wardhani, 2018). Data from research on Ag levels in sediments in the waters of Saparua Island are presented in Table 6.

Based on Table 6, at 12 locations for sampling sediments for measuring Ag levels, the metal content of Ag was above a predetermined threshold, was <7 ppm. According to RI Government Regulation No. 85 of 1999 concerning the TCLP (*Toxicity Characteristic Leaching Procedure*) quality standard, the maximum

permissible limit for silver content is 5.0 mg/L or 0.1 - 1.0 ppm.

No Sample Ag Content Sludge Total Organi	C 26 ( )
Code (ppm) (%) Carbon (	70)
1 P.1 <7 3.30 53.78	
2 P.2 <7 4.14 67.32	
3 P.3 <7 5.46 43.58	
4 P.4 <7 0.68 65.77	
5 P.5 <7 0.25 64.77	
6 P.6 <7 14.17 44.63	
7 P.7 <7 16.64 34.86	
8 P.8 <7 11.58 34.26	
9 P.9 <7 14.56 22.40	
10 P.10 <7 0.65 62.16	
11 P.11 <7 1.00 67.00	
12 P.12 <7 1.39 51.82	

The reason is that there is a high content of silver metal in the waters of Saparua Island because silver chloride dissolves to form a complex salt of silver thiosulphate. Dissolved silver in the form of complex silver salts is what is usually called fixer liquid waste. This stage is known as the fixing process or photo fixation, and the solution is referred to as Hadikawuryan fixer, (2005) in (Kurniasih et. al., 2020).

# Test the Composition of Chemical Compounds in Sediments

Based on data analysis of Pb, Cu, and Ag metals from the sediments of Saparua Island waters using the XRF method, the chemical compounds found in the sediments are in the form of oxides and in a free state such as CaO, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> Na<sub>2</sub>O Fe<sub>2</sub>O<sub>3</sub> MgO, K<sub>2</sub>O, SO<sub>3</sub>, TiO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>, ZnO, MnO, Cr<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, ZrO<sub>2</sub>, Rb<sub>2</sub>O, SrO, La<sub>2</sub>O<sub>3</sub>, CuO, NiO, Y<sub>2</sub>O<sub>3</sub>, Based on the results of the XFR test that has been carried out, it is shown that at points P.2, P.3, P.6, P.8, P.9, and P.10, there are no metal elements Pb and Ag because they are estimated to have concentrations small so that the detection tool does not read the concentration of the metal. The results of the XRF test for this study indicated the presence of Cu metal at points P.8 and P.9 with a percent concentration of 0.0056 and 0.0031. The results of metal oxide analysis are shown in Table 7.

	Compund	-		w/w %	of Compund		
INO	Name	P.2	P.3	P.6	P.8	P.9	P.11
1	CaO	39.55	35.53	43.49	2.12	9.48	6.16
2	SiO <sub>2</sub>	15.55	17.35	1.06	47.89	30.11	57.46
3	$Al_2O_3$	3.09	3.84	0.400	12.44	8.77	13.99
4	Na <sub>2</sub> O	1.93	2.46	2.12	4.88	5.60	3.75
5	Fe <sub>2</sub> O <sub>3</sub>	0.586	0.854	0.108	4.46	2.95	1.17
7	MgO	1.80	2.05	1.79	2.03	2.26	0.919
8	$K_2O$	0.781	0.843	0.0970	1.80	1.26	3.35
9	SO <sub>3</sub>	0.392	0.641	0.480	1.61	1.54	0.252
10	TiO <sub>2</sub>	0.0280	0.0431	0.0140	0.472	0.361	0.0440
11	$P_2O_5$	0.106	0.150	0.115	0.310	0.159	0.0584
12	ZnO	-	0.0079	-	0.0292	0.0074	0.0049
13	MnO	0.0113	0.0152	0.0047	0.0273	0.0153	0.0563
14	$Cr_2O_3$	-	-	-	0.0259	0.0192	0.0041
15	$V_2O_5$	-	-	-	0.0160	0.0080	-
16	$ZrO_2$	-	-	-	0.0153	-	-
17	Rb <sub>2</sub> O	0.0040	0.0053	-	0.0099	0.0072	0.0196
18	SrO	0.471	0.399	0.669	0.0079	0.120	0.0483
19	$La_2O_3$	-	-	-	0.0060	-	-
20	CuO	-	-	-	0.0056	0.0031	-
21	NiO	-	-	-	0.0053	0.0038	-
22	$Y_2O_3$	-	-	-	0.0032	-	-

Tabel 7. Result of compound analysis

The type of sediment obtained from the results of sediment observations at the sampling location was muddy sand. Sediment particle size plays an important role in the absorption capacity of heavy metal accumulation. The smaller the particle size, the greater the heavy metal content. Fine sediment particles have a larger surface area and a more stable ion density to bind metals than larger sediment particles (Sahara, 2009). The test results for the elemental composition of sediments are shown in Table 8.

#### X-Ray Diffraction (XRD) Analysis

X-ray diffraction analysis was carried out with a scan angle range ( $2\Theta = 10-65$  °C), and a speed of 2°/min. The sediment at the characteristic yield location in the form of a diffractogram is shown in Figure 6 to Figure 11.



sample P.2

The diffractogram shows the presence of several reflection peaks. The three peaks with the highest intensity are at  $2\Theta = 27.7464$ ; 29.4388; and 26.2490°. The reflection peak with a value of  $2\Theta$  of 27.7464° has the highest intensity and is relatively sharper than the other reflection peaks. This  $2\Theta$  angle indicates the presence of minerals CaO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> which are the highest content in the sediment based on the results of characterization with XRF, namely 39.55; 15.55; and 3.09. The characterization results using XRD and a diffractogram are shown in Figure 7.

NT	Nama	ma m/m % Unsur					
No	Unsur	P.2	P.3	P.6	P.8	P.9	P.11
1	Si	7.27	-	-	22.39	14.07	26.86
2	Al	1.64	2.03	0.212	6.58	4.64	7.41
3	Na	1.43	1.82	1.57	3.62	4.15	2.78
4	Cl	1.46	2.16	2.09	4.72	5.83	1.84
5	Fe	0.410	0.598	0.0759	3.12	2.06	0.820
6	Ca	28.28	25.40	31.09	1.52	6.78	4.41
7	Mg	1.09	1.24	1.08	1.23	1.36	0.555
8	Κ	0.648	0.700	0.0805	1.50	1.04	2.78
9	Sx	0.157	0.257	0.192	0.643	0.616	0.101
10	Ti	0.0168	0.0258	0.0084	0.283	0.216	0.0255
11	Px	0.0464	0.0654	0.0504	0.135	0.0693	-
12	Zn	-	0.0063	-	0.0235	0.0059	-
13	Mn	0.0088	0.0118	0.0036	0.0211	0.0118	0.0436
14	Cr	-	-	-	0.0177	0.0131	0.0028
15	V	-	-	-	0.0090	0.0045	-
16	Zr	-	-	-	0.0113	-	-
17	Rb	0.0037	0.0048	-	0.0091	0.0066	0.0179
18	Sr	0.398	0.338	0.566	0.0067	0.101	0.0408
19	La	-	-	-	0.0051	-	-
20	Cu	-	-	-	0.0045	0.0025	-
21	Ni	-	-	-	0.0042	0.0030	-
22	Y	-	-	-	0.0025	-	-
23	Si	-	8.11	0.494	22.39	-	-
24	Al	-	-	-	6.58	-	-

Tabel 8. Elemental compotition test in sediments



The diffractogram shows the presence of several reflection peaks. The three peaks with the highest intensity are at  $2\Theta = 29.4759^{\circ}$ ;  $26.2335^{\circ}$ ; and  $45.8731^{\circ}$ . The reflection peak with a value of  $2\Theta$  of  $29.4759^{\circ}$  has the highest intensity and is relatively sharper than the other reflection peaks. This  $2\Theta$  angle indicates the presence of minerals CaO, SiO<sub>2</sub>, and

Al<sub>2</sub>O<sub>3</sub>, which are the highest content in the sediment



Figure 8. Diffractogram of the sediments of sample P.6

based on the results of characterization with XRF, namely 35.53; 17.35; and 3.84. The characterization results using XRD and a diffractogram are shown in Figure 8.

The diffractogram shows the presence of several reflection peaks. The three peaks with the highest intensity are at  $2\Theta = 26.2979^{\circ}$ ,  $33.2083^{\circ}$ , and  $27.2879^{\circ}$ . The reflection peak with a value of  $2\Theta$  of  $26.2979^{\circ}$  has the highest intensity and is relatively sharper than the other reflection peaks. This  $2\Theta$  angle indicates the presence of CaO, Na<sub>2</sub>O, and MgO



Figure 9. Diffractogram of the sediments of sample P.8

minerals, which are the highest content in the sediment based on the results of characterization with XRF. namely 43.49, 2.12, 1.79. The and using characterization XRD results and а diffractogram are shown in Figure 9.

The diffractogram shows the presence of several reflection peaks. The three peaks with the highest intensity are at  $2\Theta = 26.6686^{\circ}$ ,  $20.8993^{\circ}$ , and  $50.1719^{\circ}$ . The reflection peak with a value of  $2\Theta$  of  $26.6686^{\circ}$  has the highest intensity and is relatively sharper than the other reflection peaks. This  $2\Theta$  angle indicates the presence of minerals SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Na<sub>2</sub>O, which are the highest content in the sediment based on the results of characterization with XRF, namely 47.89, 12.44, and 4.88. The characterization results using XRD and a diffractogram are shown in Figure 10.



The diffractogram shows the presence of several reflection peaks. The three peaks with the highest intensity are at  $2\Theta = 26.7007^\circ$ ,  $20.9219^\circ$ , and  $50.2104^{\circ}$ . The reflection peak with a value of 2 $\Theta$  of 26.7007° has the highest intensity and is relatively sharper than the other reflection peaks. This 2O angle indicates the presence of SiO<sub>2</sub>, CaO, and Al<sub>2</sub>O<sub>3</sub> minerals, which are the highest content in the sediment based on the results of characterization with namely and 8.77. XRF, 30.11, 9.48, The characterization using XRD results and а diffractogram are shown in Figure 11.

The diffractogram shows the presence of several reflection peaks. The three peaks with the highest intensity are at  $2\Theta = 24.3695^{\circ}$ ,  $45.6226^{\circ}$ , and  $28.4183^{\circ}$ . The reflection peak with a value of  $2\Theta$  of  $24.9322^{\circ}$  has the highest intensity and is relatively



sharper than the other reflection peaks. This  $2\Theta$  angle indicates the presence of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and CaO minerals, which are the highest content in the sediment based on the results of characterization with XRF, namely 57.46, 13.99, and 6,16.

According to (Saniah et al., 2015), the reflection intensity of X-ray diffraction identifies concentration, crystalline perfection, and the density of the arrangement of atoms in a crystal so that the sharper the reflection intensity of a material, the better its crystalline properties with the denser arrangement of atoms. Meanwhile stated that the reflected X-ray diffraction intensity identified the crystalline phase in the material by determining the parameters of the lattice structure of the test sample (Gobah et al., 2016; Sekewael et al., 2018).

#### CONCLUSION

The levels of copper metal contained in sediments in the waters of Saparua Island range 0.1583-1.9531 mg/L and <4-78 ppm. The level of lead metal contained in the sediments in the waters of Saparua Island ranges from <12 - 26 ppm. The level of silver metal contained in the sediments in the waters of Saparua Island ranges from <7 ppm. Metal oxides, especially metal oxides formed from Cu, were found in sediments in the waters of Saparua Island with location points P.8 and P.9, namely CuO 0.0056% and 0.0031% and in sediments in Saparua Island waters with a value of  $2\Theta$  in sediment samples. P.2, P.3, P.6, P.8, P.9 and P.11 have the highest intensity which is the characterization of metal oxide 2⊖= 27.7464°, 29.4759, 26.2979, 26.6686, 26.7007 and 24.3695 indicate the presence of CaO (calcium oxide) phases at points P.2, P.3 and P.6. the siO2 phase (silica) which is the most abundant metal in sediment samples P.8, P.9 and P.11 based on the XRF test.

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