JPSE (Journal of Physical Science and Engineering), Vol. 6, No. 2, 2021, Page 68–76.
DOI: 10.17977/um024v6i22021p068

JPSE
(Journal of Physical Science and Engineering)

http://journal2.um.ac.id/index.php/jpse
EISSN: 2541-2485

Identification of Elemental Composition and Heavy Metal Content in Maninjau Lake Sediment Using X-Ray Fluorescence (MNJ 18-41B)

Received
28 May 2021

Revised
17 July 2021

Accepted for Publication
29 September 2021

Published
12 October 2021

F Akmal[1]*, H Rifai[1], Syafriani[1], C B De La Maisonneauve[2], J A G Oalmann[2], F Forni[2], S Eisele[2], M Phua[2], and R Putra[3]

1. Department of Physics, Faculty of Mathematics and Natural Sciences, Universitas Negeri Padang, Jl. Prof. Dr. Hamka, Padang, 25173, Indonesia.
2. Asian School of the Environment, Nanyang Technological University, 50 Nanyang Avenue, 639798, Singapore.
3. Bayerisches Geoinstitut, Universität Bayreuth, Universitätsstr 30 Bayreuth, 95440, Germany.

***E-mail:** fajarakmal12@gmail.com

Abstract
Maninjau Lake is a caldera lake located in Agam, West Sumatra. This lake was formed from the volcanic activity of Mount Maninjau Purba about 60,000 years ago. The volcanic material resulting from the eruption is scattered and deposited in various places, one of which is in lake sediments. Volcanic ash contains various types of elements, including heavy metal elements. This study aims to determine the composition and content of heavy metal in the sediments of Maninjau Lake. The sample analyzed was MNJ 18-41B with a core length of 440 mm, focusing on specimens 148 mm and 376 mm. The selection is a specimen based on the magnetic susceptibility value obtained from the measurement meter susceptibility of the MS2E. Specimen 148 has a value susceptibility low of 2.1×10^{-8} m^3/kg, while the 376 specimens have the highest value of susceptibility, the highest 141×10^{-8} m^3/kg. The content of sediment elements was determined using the results of X-Ray Fluorescence (XRF) measurements. The measurement results showed that the mineral-forming elements in the sediments of Lake Maninjau were dominated by Si, Fe, Rh, and Zr. In contrast, the highest heavy metal elements are Mn, Fe, Sr, and Rh. Based on the elemental composition and mineral oxide compounds in the sediments of Lake Maninjau derived from volcanic ash.
Keywords: Sediment, heavy metals, XRF, susceptibility.

1. Introduction

Lake Maninjau is a caldera type lake located in Agam, West Sumatra [1], [2]. The surface area of Maninjau Lake is 97.9 km^2, at an altitude of 459 meters [3]. Based on the bathymetry, Maninjau Lake has an average depth of 100 meters. This lake was formed from the volcanic activity of Mount Maninjau Purba, which erupted about 60,000 years ago. The eruption ejected volcanic material that travelled up to 75 kilometres from the epicentre. Volcanic material settles in various places such as in peatlands, rivers, and seas. Eighty-eight rivers empty into the lake, so many materials are transported, one of which is volcanic material [1]. This is what contributes to the presence of volcanic material in lake sediments.

Sediments are particles resulting from weathering of rocks, biological materials, chemical deposits, dust, plant residue materials, and leaves deposited on a medium [4]. Sediment is formed through a sedimentation process. Sedimentation is a process of deposition of solids in liquids due to the force of gravity [5]. Sediment at the lake's bottom accumulates from various sources, such as river flows [6]. The material at the lake's bottom can also come from volcanic activity, such as volcanic ash carried by the wind, which is then deposited on riverbeds. Volcanic material or tephra undergoes a process of transportation to various places. Four characteristics need to be observed to describe the sediment, i.e. the sediment's colour, structure, texture, and composition [7]. Volcanic eruptions generally contain oxides of several hazardous metal elements such as aluminium (Al), silicon (Si), iron (Fe), calcium (Ca), magnesium (Mg), potassium (K), sodium (Na), sulfur (S), and several dangerous heavy metal elements such as lead (Pb), cadmium (Cd), and arsenic (As) [8]. These elements will combine with other elements and will form minerals in lake sediments.

Minerals are natural compounds formed through geological processes, while magnetic minerals are a collection of compounds with high magnetic properties [9]. The presence of magnetic minerals can determine the mineral content source in the lake's sediment deposits [10], [11]. Minerals in lake sediments can come from the deposition of volcanic ash from the atmosphere [12], river flow that empties into lakes [13], minerals from human activities [14], and autogenic processes [6]. Magnetic minerals have properties including diamagnetic, paramagnetic, ferromagnetic, and ferrimagnetic [15], [16]. The magnetic properties of a rock depend on the magnetic mineral carrying its magnetic properties, i.e. type, number, shape, and size of the magnetic mineral grains. The magnetic properties of rocks in their application to climate and environmental changes can be seen from these four factors. For example, magnetic minerals in sediment or soil are related to heavy metal elements in environmental studies. Thus, magnetic properties can be used to detect the presence of heavy metal elements in sediments [17].

Heavy metals have a specific gravity of more than 5 grams/cm^3 with atomic numbers 22 to 92 [18]. Elements classified as heavy metals include As, copper (Cu), terbium (Tb), Al, zinc (Zn), nickel (Ni), mercury (Hg), and Cd. Heavy metal contamination is hazardous for the environment because heavy metals cannot be decomposed (non-degradable) by organisms [19]. The presence of heavy metals that exceed the threshold level in waters is hazardous and will affect water quality [20]. One of the impacts of heavy metals on the waters is that they can result in mass fish mortality. The phenomenon of fish mortality that occurred in 2010–2014 was 2,100 tons. In addition, Lake Maninjau has an essential role for the surrounding community, such as hydropower, tourism activities, conservation, and fisheries [21]. So, it is necessary to identify the heavy metal content in the sediments of Lake Maninjau. The X-Ray Fluorescence (XRF) method can be used to identify heavy metal elements in Lake Maninjau sediments. The XRF method is one of the methods used to determine the chemical composition of a mineral, including powder, soil, liquid, or other forms [9], [22], [23].

2. Method

2.1. Sampling

Sampling was conducted in 2018 at coordinate 0˚18'20.20"S and 100˚12'31.36"E in Agam District, West Sumatra, as shown in Figure 1. The samples taken were Lake Maninjau sediments. Sampling was conducted using a gravity core with a length of 1 meter, which was equipped with a deep PVC pipe to collect sediment samples. The gravity core is dropped vertically into the lake sediment so that the sediment enters the PVC pipe. In order to obtain a deeper sediment sample, the gravity core was weighted from the surface so that it could no longer add depth. Following that, the Gravity core is lifted

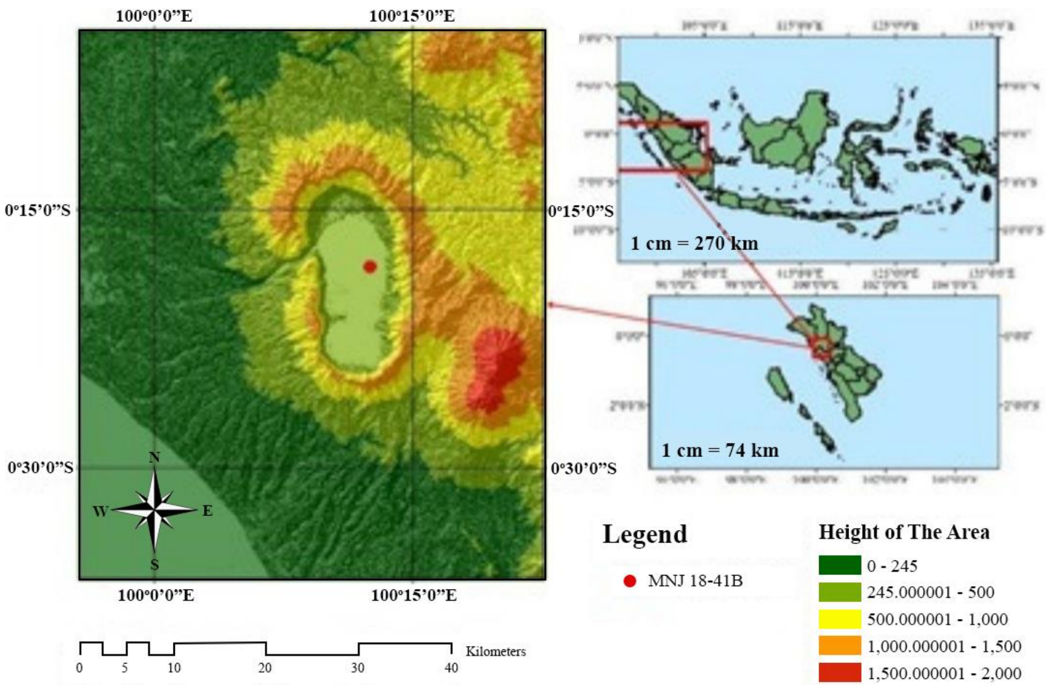

Figure 1. Sampling location in Lake Maninjau.

and closes the top and bottom of the PVC pipe, preventing the sample from escaping. Researchers gave PVC pipe containing sediment the name and position mark of the sample. The sample must be vertical because if it changes, it will damage the structure and position of the sediment in the PVC pipe. After the sampling process is complete, the sample is stored in a cupboard or cold room to maintain the structure and position of the sample. The sample will shrink and dry out if it is left in an open room. The samples were stored until the preparation was carried out.

2.2. Sample Preparation

Preparation begins with the activity of splitting the PVC pipe into two parts using a splitting machine. The results of the splitting of the sample are shown in Figure 2. The sample used is MNJ 18-41B. After splitting, one part of the sample is stored back in the cold room and another part will be measured. Before measuring the sample's surface, it must be levelled first because the measurement stage uses a sensor that is sensitive to the sample surface.

2.3. Sample Measurement

The measurements made consisted of two stages. The first stage is the measurement of magnetic susceptibility using the susceptibility meter type MS2E. The results of magnetic susceptibility measurements are used for the selection of specimens to be identified. The second stage measures the concentration of elements in the sample using XRF with the type Avaatech XRF Scanner. The XRF measurement results obtained the element concentration per 2 mm of the total sample length of 440 mm. In this study, two specimens were selected to be identified based on the highest and lowest susceptibility values. The identified specimens were at a depth of 148 mm and 376 mm. Specimens 148 mm have the lowest susceptibility value of 2.1×10^{-8} m^3/kg, while the 376 mm specimens have the highest susceptibility value of 141×10^{-8} m^3/kg.

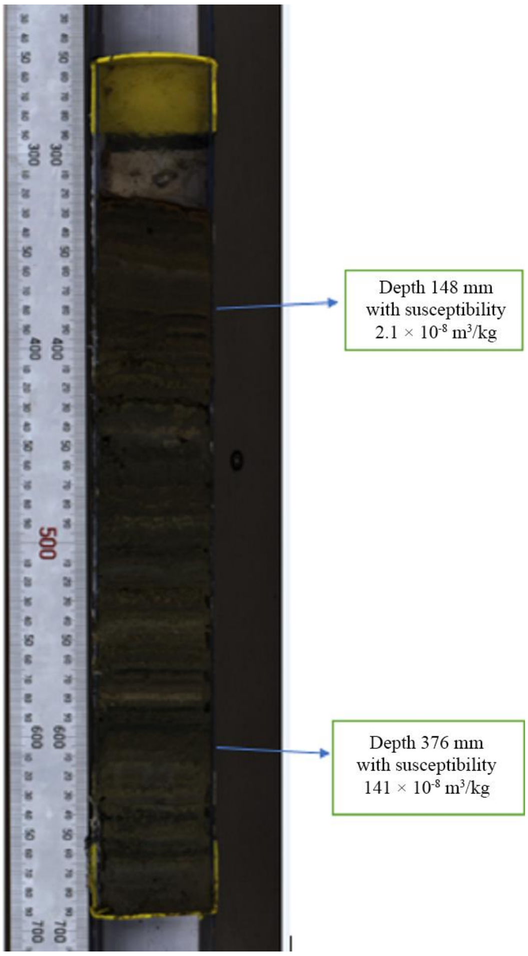

Figure 2. Lake Maninjau sediment (MNJ 18-41B).

Table 1. Composition elements and oxides in depth 148 mm (2.1 × 10^{-8} m^3/kg).

Elemental	Area (c/s)	Concentration (%)	Oxide	Concentration (%)
Aluminum (Al)	598	0.255	Al_2O_3	0.667
Silicone (Si)	14,869	6.342	SiO_2	18.122
Phosphor (P)	4	0.002	P_2O_5	0.006
Sulfur (S)	722	0.308	SO_2	0.827
Chlorine (Cl)	6,009	2.563	Cl_2O	4.182
Potassium (K)	1,912	0.815	K_2O	1.320
Calcium (Ca)	5,120	2.184	CaO	4.070
Titanium (Ti)	3,306	1.410	TiO_2	3.134
Chrome (Cr)	1,007	0.429	Cr_2O_3	0.837
Manganese (Mn)	1,779	0.759	MnO	1.307
Iron (Fe)	43,201	18.426	FeO	31.596
Rhodium (Rh)	51,746	22.070	-	-
Vanadium (V)	464	0.198	V_2O_5	0.471
Cobalt (Co)	7,695	3.282	CoO	5.561
Nickel (Ni)	1,488	0.635	Nio	1.068
Copper (Cu)	4,597	1.961	CuO	3.267
Zinc (Zn)	9,523	4.062	ZnO	6.747
Gallium (Ga)	453	0.193	-	-
Bromine (Br)	10,523	4.488	-	-
Rubidium (Rb)	13,278	5.663	-	-
Strontium (Sr)	17,996	7.675	SrO	12.095
Litrium (Y)	10,644	4.540	-	-
Zircon (Zr)	19,866	8.473	-	-
Lead (Pb)	6,377	2.720	PbO	3.907
Bismuth (Bi)	1,285	0.548	Bi_2O_3	0.817
Total		**100.000**		**100.000**

Table 2. Composition elements and oxides in depth 376 mm (141 × 10^{-8} m^3/kg).

Elemental	Area (c/s)	Concentration (%)	Oxide	Concentration (%)
Aluminum (Al)	803	0.375	Al_2O_3	0.793
Silicone (Si)	18,781	8.762	SiO_2	21.269
Phosphor (P)	78	0.036	P_2O_5	0.102
Sulfur (S)	1,096	0.511	SO_2	1.156
Chlorine (Cl)	4,476	2.088	Cl_2O	2.901
Potassium (K)	2,225	1.038	K_2O	1.416
Calcium (Ca)	5,114	2.386	CaO	3.792
Titanium (Ti)	5,153	2.404	TiO_2	4.533
Chrome (Cr)	1,060	0.495	Cr_2O_3	0.811
Manganese (Mn)	11,390	5.314	MnO	7.767
Iron (Fe)	47,046	21.948	FeO	31.977
Rhodium (Rh)	46,212	21.559	-	-
Vanadium (V)	321	0.150	V_2O_5	0.304
Cobalt (Co)	5,609	2.617	CoO	3.773
Nickel (Ni)	1,583	0.739	NiO	1.065
Copper (Cu)	4,432	2.068	CuO	2.935
Zinc (Zn)	4,920	2.295	ZnO	3248
Gallium (Ga)	540	0.252	-	-
Bromine (Br)	4,839	2.258	-	-
Rubidium (Rb)	10,753	5.017	-	-
Strontium (Sr)	12,877	6.007	SrO	8.048
Litrium (Y)	6,815	3.179	-	-
Zircon (Zr)	11,049	5.155	-	-
Lead (Pb)	6,058	2.826	PbO	3.455
Bismuth (Bi)	1,122	0.523	Bi_2O_3	0.656
Total		**100.000**		**100.000**

3. Result and Discussion

The measurement uses X-Ray Fluorescence (XRF) of samples Lake Maninjau sediment MNJ 18-41B yields information about the concentration of elements in the sample. The measurement results are shown in Table 1 and Table 2.

3.1. Elements Composition

From Table 1 and Table 2, it can be seen that in the 148 mm specimen the element with the highest concentration was the Rh element, while in the 376 mm specimen it was the element Fe. To be more apparent, the comparison of the concentrations of each element at the two depths shown in Figure 3.

Based on Figure 3, the ratio of the element concentration of each specimen has different susceptibility values. The blue graph shows a 148 mm specimen with low susceptibility (2.6 SI), while the yellow one shows a 376 mm specimen with high susceptibility (141 SI). The dominant elements in Lake Maninjau sediment with a susceptibility value of 2.1 SI (low susceptibility) are Si, Fe, Rh, Br, Rb, Sr, Y, and Zr, with the highest concentration element is Rhodium (Rh) 22.07%. In 376 mm specimen with 141 SI susceptibility value (high susceptibility), the dominant elements are Si, Mn, Fe, Rh, Rb, Sr, and Zr, with the highest concentration element is Iron (Fe) 21.56%. Magnetic mineral-forming elements influenced the susceptibility value because specimens with high susceptibility values are indicated to have high magnetic mineral content [24], [25].

Magnetic minerals have the essential constituent elements of the fourth group transition group elements (Sc, Ti, V, Cr, Mn, Fe, Cn, Bi, Cu, and Zn) [11], [25]. The magnetic mineral-forming elements found in this sample are Ti, V, Cr, Bi, Cu, Zn, Fe, and Mn. Examples of magnetic minerals of this element are TiO_2, V_2O_5, Cr_2O_5, CuO, ZnO, and Fe_3O_4. The elements forming magnetic minerals (Ti, V, Cr, Cu, Zn, Fe, Mn) have a higher concentration at a depth of 376 mm compared to a depth of 148 mm. Meanwhile, the Bi element is higher at a depth of 148 mm, but the difference is not very significant. These elements cause different susceptibility values, especially Fe [26]. Fe element has the highest magnetic susceptibility value and the magnetic property of the material is ferromagnetic. The relationship between the element Fe and its susceptibility is comparable. Because the 376 mm specimen contains a higher concentration of magnetic elements than the 148 mm specimen, it has a higher susceptibility value.

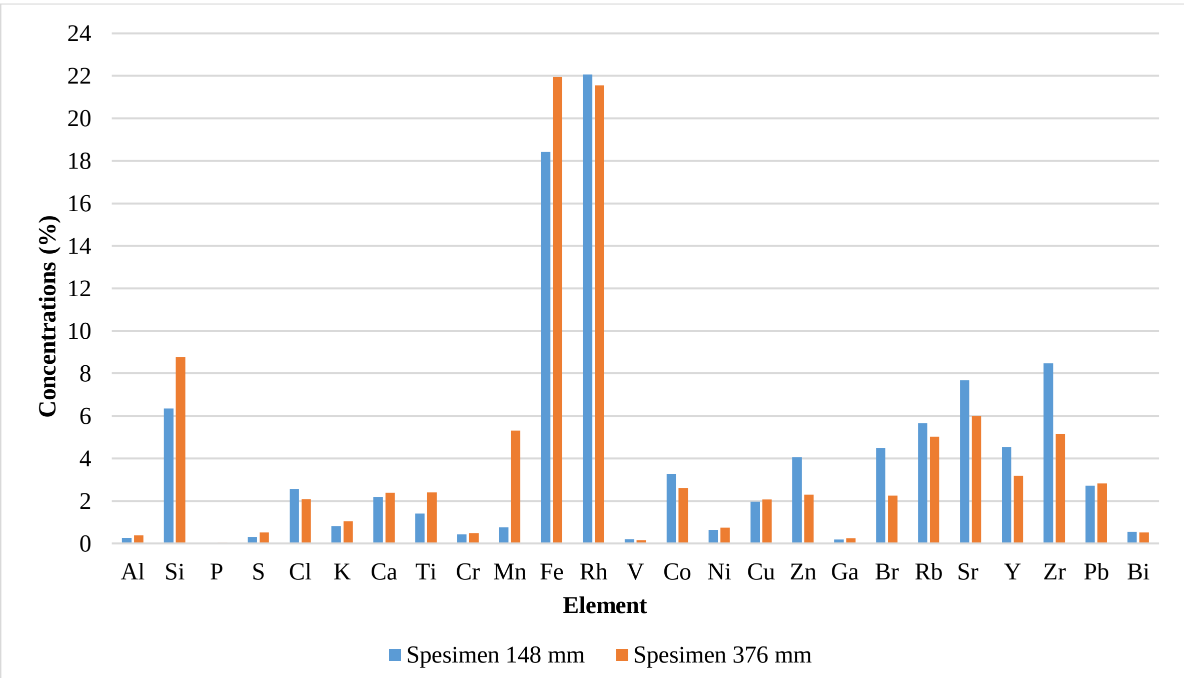

Figure 3. Comparison of the element concentration of MNJ 18-41B for 148 mm and 376 mm specimens.

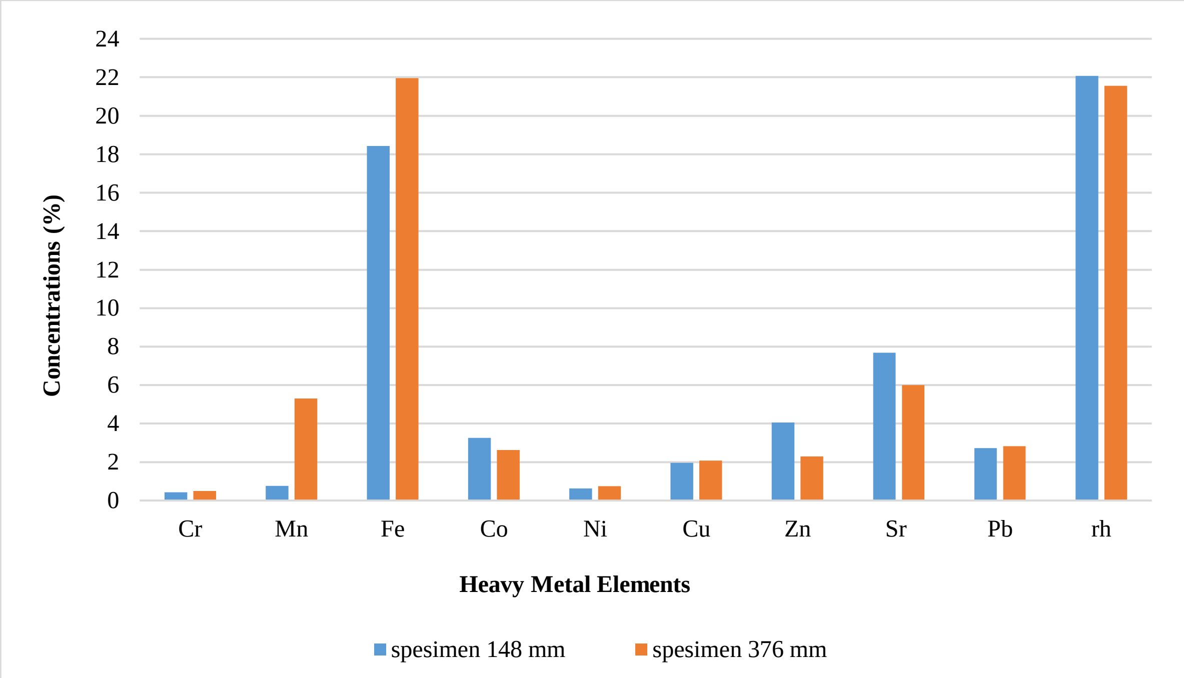

Figure 4. Comparison of the heavy metal element concentration of MNJ 18-41B for 148 mm and 376 mm specimens.

3.2. Content of Heavy Metal Elements

Heavy metal elements also affect magnetic susceptibility values. So, it is necessary to identify the content of heavy metal elements. The comparison of heavy metal concentrations in each specimen is shown in Figure 4. Both specimens have the same elemental content but different concentrations value. The highest metal element in the 148 mm specimen was the Rh element, with a concentration of 22.07%. Meanwhile, in the 378 mm specimen was the Fe element, with a concentration of 21.95%.

Heavy metal elements contained in this sample are Cr, Mn, Fe, Cu, Zn, Ni, Sr, and Pb. The concentration of heavy metals at each depth is different. At a depth of 148 mm specimen, the concentration of heavy metal elements Co, Sr, Zn, and Rh were higher than at a depth of 378 mm specimen. The element Co (3.2%) is ferromagnetic, Sr (7.68%) and Rh (22.07%) are paramagnetic, while Zn (4.06%) is diamagnetic. The highest heavy metal at a depth of 148 mm is Rhodium (Rh), which is paramagnetic. As a result, the susceptibility value of the specimen is low with many paramagnetic elements, whereas it is high with elements with low ferromagnetic properties. For a depth of 378 mm specimen, heavy metal elements are Cr, Cu, Mn, Ni, Fe, and Pb have a higher concentration than the 148 mm specimen. The element Cr (0.49%), Ni (0.74%), and Fe (21.95%, which is the highest element in this specimen) are ferromagnetic, Cu (2.07%) and Pb (2.83%) are diamagnetic, while Mn (5.31%) is paramagnetic [7].

Several factors influence the susceptibility value based on heavy metal element analysis. The first is the elemental content in the sample. Samples that contain high heavy metal elements will have a high sufficiency value. The second is the depth of the sample. The deeper the sample is from the surface, the higher the susceptibility value. This is because sediment is formed during the deposition process, so the surface sediment contains more impurities than the depth sediment. The variation in depth from the surface is directly proportional to the value of magnetic susceptibility [27]. These factors cause the susceptibility value of 376 mm specimen to be higher than 148 mm specimen.

3.3. Content of Oxide

Minerals are chemical compounds with composition within certain limits, which a formula can express. Mineral formulas can be simple or complex, depending on the number of elements present and the proportions of their combinations [28]. One type of mineral is an oxide compound. Oxides are chemical compounds that contain at least one oxygen atom and one other element [29].

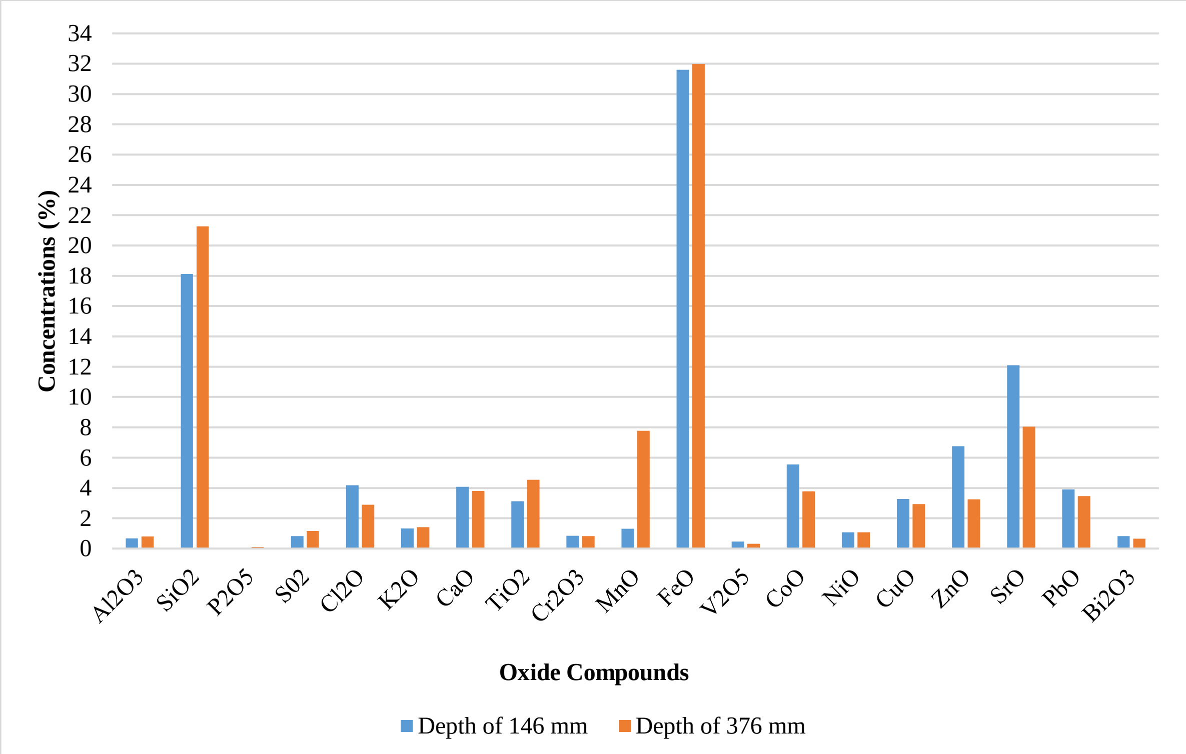

Figure 5. Comparison of the oxie compounds concentration of MNJ 18-41B for 148 mm and 376 mm specimens.

Some of the dominant oxide compounds in this study are SiO_2, FeO, SrO, and MnO. However, several elements cannot bind to oxygen from the elements obtained, namely Rh, Ga, Br, Rb, Y, and Zr. One of the reasons that some elements cannot be bonded is that they are precious metals that are difficult to oxidize [30]. The ratio of oxide concentrations in the two specimens can is shown in Figure 5. Based on the results of XRF measurements, Lake Maninjau sediment contains volcanic ash elements in Al, Si, Ca, Fe, K, Mn, P, S, and Ti. Because volcanic ash contains various major elements (Al, Si, Ca, and Fe) and minor elements (K, Mg, Mn, Na, P, S, and Ti), so it can be said that nothing in the sediments of Lake Maninjau comes from various sources, one of which is volcanic activity.

4. Conclusion

Based on the results and discussion, it can be concluded that the mineral-forming elements in the sediments of Maninjau Lake with MNJ 18-41B samples are dominated by Si, Fe, Rh, and Zr. The heavy metal elements present in the sample are Cr, Mn, Fe, Co, Ni, Cu, Zn, Sr, and Pb. The highest concentration of heavy metal elements is the Iron (Fe). The higher the content of heavy metal elements, the higher the susceptibility value. Apart from heavy metals, susceptibility is influenced by depth. The relationship between susceptibility and depth is directly proportional. Along with heavy metal elements, volcanic ash is discovered. The elements of volcanic ash found in the sample are Al, Si, Ca, Fe, K, Mn, P, S, and Ti.

Acknowledgment

The authors acknowledge financial support from PNBP Universitas Negeri Padang with contract number 449/UN35.13/LT/2019.

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