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Identification of Sediment Formation Based on Magnetic Content and Element Composition of Mud Volcano in Sangiran Sediment using VSM and X-Ray Fluorescence

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Abstract
Based on trace geological history and several studies, the Sangiran mud volcano provides insight into the geology and hydrology of the region, aquifer system in the basin, groundwater flow patterns and characteristics, rock lithology, hydrogeology condition, and saltwater trap mapping. Related to these conditions, studies were conducted on the magnetic content and composition of the major oxide compounds in the Sangiran sediments. Sample analysis was based on geochemical methods. The methods consist of frequency dependent magnetic susceptibility and vibrating sample magnetometer (VSM) analysis. Geochemical analyses using x-ray fluorescence (XRF) analysis have been conducted and various elemental grades have been determined. VSM results confirm that the magnetic content of Sangiran sediments is partly dominated by Fe (17.66%) contained in hematite (Fe_2O_3). At the same time, the samples of Sangiran sediment were enriched by Si, Fe, Al, Ca, Cl, Ti, and K according to XRF measurements. The samples exhibited the highest Si and Fe concentrations in samples T1 (Si = 29.48% and Fe = 13.66%) and T7 (Si = 24.95% and Fe = 12.01%). Meanwhile, in the T4 sample, the highest concentrations were Si and Ca, 23.45% and 13.45%, respectively. Retrieved from the magnetic susceptibility measurement, this paper confirm that Fe content is one of the components of volcanic ash in the Sangiran sediment.
Keywords: Magnetic content, element composition, Sangiran sediment, VSM, XRF.

1. Introduction

Sangiran mud volcano is a geological formation located in Sangiran dome, Central Java, Indonesia. Mud volcanoes are formed when sediments are pushed up by the pressure difference between buoyancy and overpressure and move along with fault structures [1]. Sangiran dome is a geological structure formed around 1.5 Ma due to tectonic activity. The location of the Sangiran dome is on the coordinates of 7°27'5" S and 110°50'15.36" E [2]. Sangiran dome was formed due to the eruption of mud and gas from underground, creating a dome-shaped structure [3]. This dome is composed of layers of volcanic rocks and sedimentary deposits [2]. The Sangiran mud volcano is an important site for scientific research, as it provides insight into the geology and hydrology of the region. That several research are to study aquifer system in the basin [4], to study shallow groundwater flow patterns and characteristics [5], and to identify of rock lithology, hydrogeology condition, and saltwater trap mapping [6].

In addition to geophysical methods, geochemical methods are often chosen to explore the mineral abundance of a sediment. Geochemical methods can provide information about the distribution and composition of elements or compounds such as minerals, rocks, and soils [7]. The geochemical conditions of the crust are always different depending on the geological structure and environment. Some elements and compounds are found together, while others are rarely found together [8]. Geochemistry highly contributes in identifying the presence of unknown minerals [9]. This paper discusses the geochemical method by measuring of magnetic content and element composition form the sediment. Several previous research have also been conducted in various fields to measure magnetic susceptibility. Other various studies have reported that magnetic susceptibility measurements have been applied not only to mud volcano sediment [10], but also to desert [11], rocks [12], sea [13], [14], fluvial [15], coastal [16], [17], river [18]–[21], lake [22]–[24], continental margin [25], mineral [26], and guano [27], [28]. It has also been shown to be useful for other substances such as it was done.

A geochemical method that can be applied to measure or identify the elemental or complex composition of minerals is x-ray fluorescence (XRF). The XRF measurements can identify the magnetic properties of minerals [29]. Measurements by XRF are accurate and non-destructive [30]. The XRF method is widely used for mineralogical and geological measurements [31]–[34], identifying heavy metal elements of lake sediment [35], and for environmental analysis [36]. Meanwhile, magnetic mineral domains can be confirmed from the results of vibrating-sample magnetometer (VSM) analysis [37]. This paper discusses the use of VSM and XRF to identify sediment formation of the mud volcano in the Sangiran area, Central Java, Indonesia based on geochemical properties. Based on these conditions, studies were conducted on the magnetic content and composition of the major oxide compounds in the Sangiran sediments.

2. Method

This research was initiated by conducting a field survey with the aim of determining the specific location of the research sampling points. After the map of the sampling location points has been determined, the next step is to sample the mud volcano material. The sample is put into a container made of bottles. The collected samples are taken and stored in the laboratory. Sample preparation is performed to prepare the sample for measurement or laboratory testing.

2.1. A Brief History of Sangiran Sediment

Around 0.9 million years ago, erosion occurred in the southern mountains of Indonesia with material in form limestone fragments and volcanic gravel which were carried to Sangiran to form a hard layer (Grenzbank). In the following period, there was a volcanic eruption around Sangiran which spewed millions of cubic tons of volcanic sand deposited by the river flow. These deposits covered the Grenzbank seam over a period of more than 0.5 Ma and left sand deposits ± 40 m thick fluvio-volcanic called the Kabuh formation deposit, which is a cross of conglomerate rocks, tuff, and tuff sandstone at the top, and calcidurite lenses at the bottom. About 250,000 years ago, volcanic lava that transported gravel and andesite boulders was deposited again about 70,000 years ago. After that, volcanic sand was deposited. Finer sediments exhibit lower energy requirements in sedimentary environments such as clay, silt loam, clay silt, silt, sandy silt, sand, and gravel [38].

Itihara *et al.* [39] conducted field studies for mud volcanoes and some laboratory analysis of mud volcanoes in Sangiran dome. Sangiran dome is part of one of the neogene folds, east-west direction. Itihara *et al.* also made a geological cross-sectional model for the Sangiran mud volcano, which relates to the formation of mud volcanoes with north-south oriented basal faults. This fault served as the center of the outflow mold of lower Miocene argillite climbing over rocks along fault boundary faults that has occurred on the surface along fault planes to become mud volcanoes. Itihara *et al.* also describes the type and size of foreign blocks up to 40 cm consisting of metamorphic rocks, sedimentary rocks, and Eocene-aged Nummulites limestones. The gas contains methane and water, discussing the composition of gas and water limited to the study by Itihara *et al.* [39]. Referring to the Sangiran Zone stratigraphy, the Notopuro Formation overlaps with the Kabuh Formation which consists of the Kalibeng Formation and the Pucangan Formation sequentially from below as shown by Figure 1. The scientists propose to change the name of the formation Kalibeng, Pucangan, Kabuh and Notopuro to Puren, Sangiran, Bapang, and Pohjajar Formation because these four names are found in the Sangiran area [40].

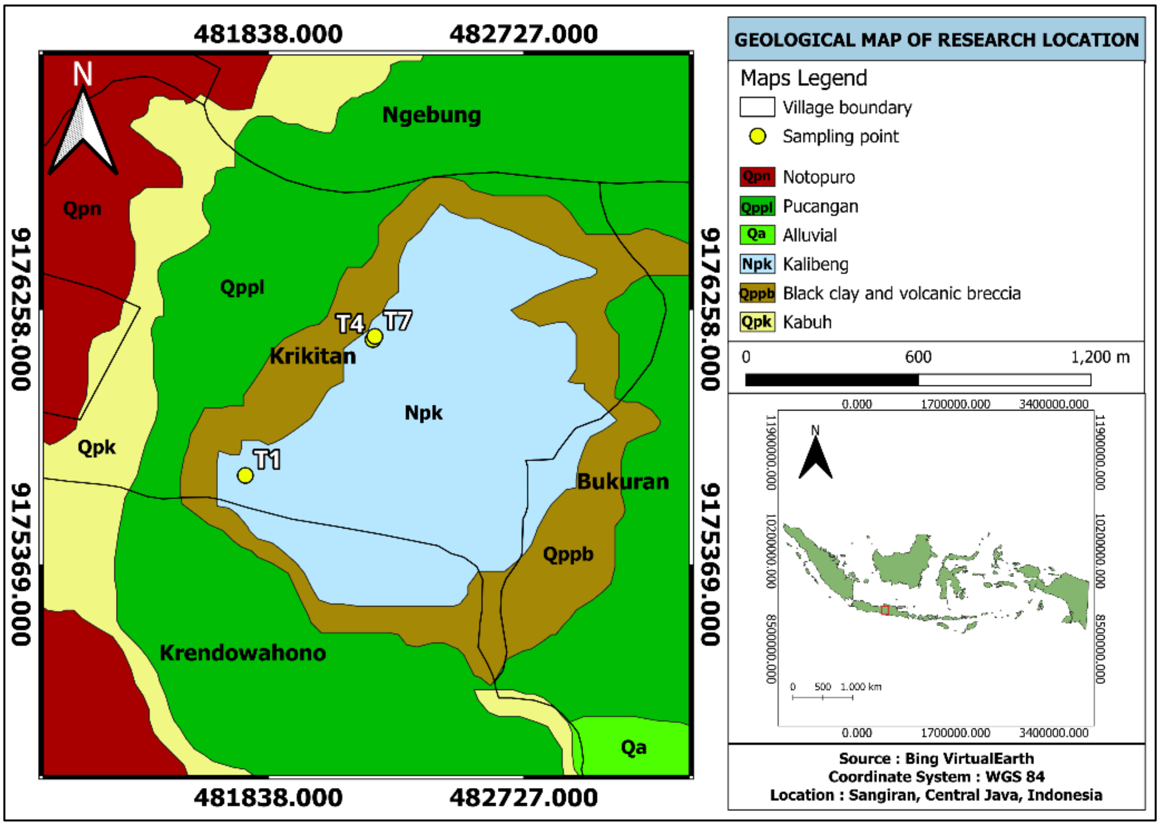


Figure 1. Simplified structural geological map of Sangiran sediment.

2.2. Sampling

Sampling was conducted in 2022 which is separately into two areas located at northern and southern areas of Sangiran sediments, Sragen, Central Java, Indonesia. The coordinate at the northern area is 7°27'26.26" S and 110°50'04.05" E, while at the southern part is 7°27'10.28" S and 110°50'19.34" E as shown by Figure 2. At the southern area, sampling at one point with the name T1. Meanwhile, at the northern part, 2 sampling points were taken with the names T4 and T7. The selection of these three locations was based on the manifestation of the mud volcano which was dominated by saltwater bubbles as shown by Figure 3. This place has several other sources of mud volcanoes such as an old mud volcano that is no longer active. Therefore, at sample points T1, T4, and T7, the only indication of mud volcanoes is salt water. The manifestation of the saltwater occurs continuously all year round. These mud volcanoes occur naturally, but on a small scale. The bottom Sangiran soil layer is about 2.4 million years old, blue clay, sediments from the marine environment in the form of Kalibeng layers [2]. Samples in the form of mud volcano material were taken using a scoop and then put in an airtight bottle container. Each bottle is named according to the sampling point.

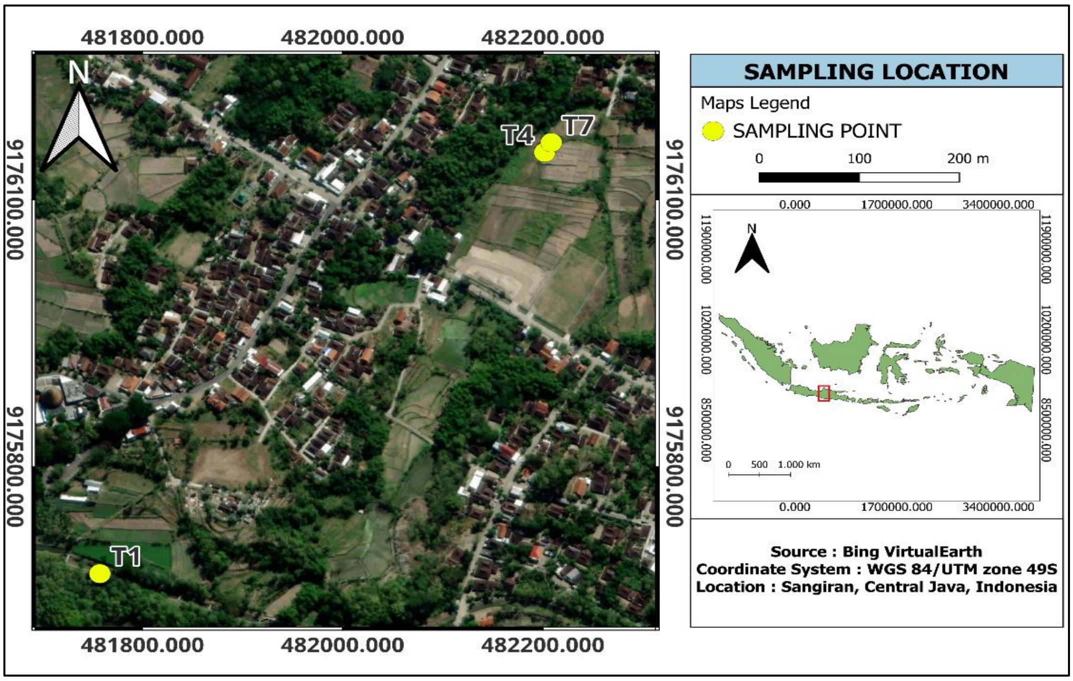


Figure 2. Location of Sampling at Sangiran, Central Java, Indonesia.

(a) (b) (c)

Figure 3. Field condition of Sampling point (a) T1, (b) T4, and (c) T7 at Sangiran sediment.

2.3. Sample Preparation

Sediment samples were collected from the Sangiran deposit in Central Java, Indonesia. There were three sediment sampling sites in this study. The exact locations of the sampling points are shown in Figure 3. A four-liter sediment sample was obtained from any sampling point and bottled. The samples were laboratory filtered over a 2 mm mesh screen to eliminate gravel and several contaminants. The samples were settled on plastic trays and allowed to dry at room temperature. At the current process, a large amount of sediment samples were produced. In addition, 10 g of dry sediment sample was placed in 100 ml of Aquavidest in a beaker and subjected to extraction of magnetic. Manually rotated a powerful bar magnet was operated in the beaker to engage the magnetic particles composed in the samples. Samples were allowed to dry at room temperature. The collected samples were produced at this stage procedures.

Measurement of magnetic susceptibility were performed on bulk samples by operating an instrument of Bartington MS2 including an MS2B sensor operating at two kind of frequencies, 470 Hz and 4.7 kHz. Metal content was determined by XRF analysis. Bulk samples were prepared by operating a pellet mill. The XRF analysis was characterized by on an instrument namely PANalytical AXIOS XRF. During that time, collected sediment samples were investigated by operating two categories of tests. Parameter of hysteresis tests were performed using an instrument of 1.2 H/CF/HT VSM to determine domains of magnetic minerals.

2.4. *Sample Measurement*

Sample analysis was based on geochemical methods. The methods consist of frequency dependent magnetic susceptibility and VSM analysis. Geochemical analyses using XRF analysis have been conducted and various elemental grades have been determined. The results of this analysis are run through a data interpretation process. The location of the mud volcano to be investigated is the Sangiran mud volcano. Not much research has been conducted on mud volcanoes. The very high magnitude of subsidence velocity in fine-grained sediments compared to other formations leads to the formation of overpressure zones, which are components of the transport subsystem.

The values of magnetic susceptibility can be measured at two frequencies, that are low frequency (χLF) and high frequency (χHF). The parameter χLF may be an indicator of the concentration of magnetic minerals in the sample. The result of magnetic susceptibility shown by Table 1. The magnetic susceptibility parameter is then found to depend on the frequency χFD. The χFD indicates the presence of superparamagnetic particles [41], which the formula is expressed by [42],

$$\chi\text{FD}\ (\%) = \left(\frac{\chi\text{LF} + \chi\text{HF}}{\chi\text{LF}}\right) \times 100\% \qquad (1)$$

3. Result and Discussion

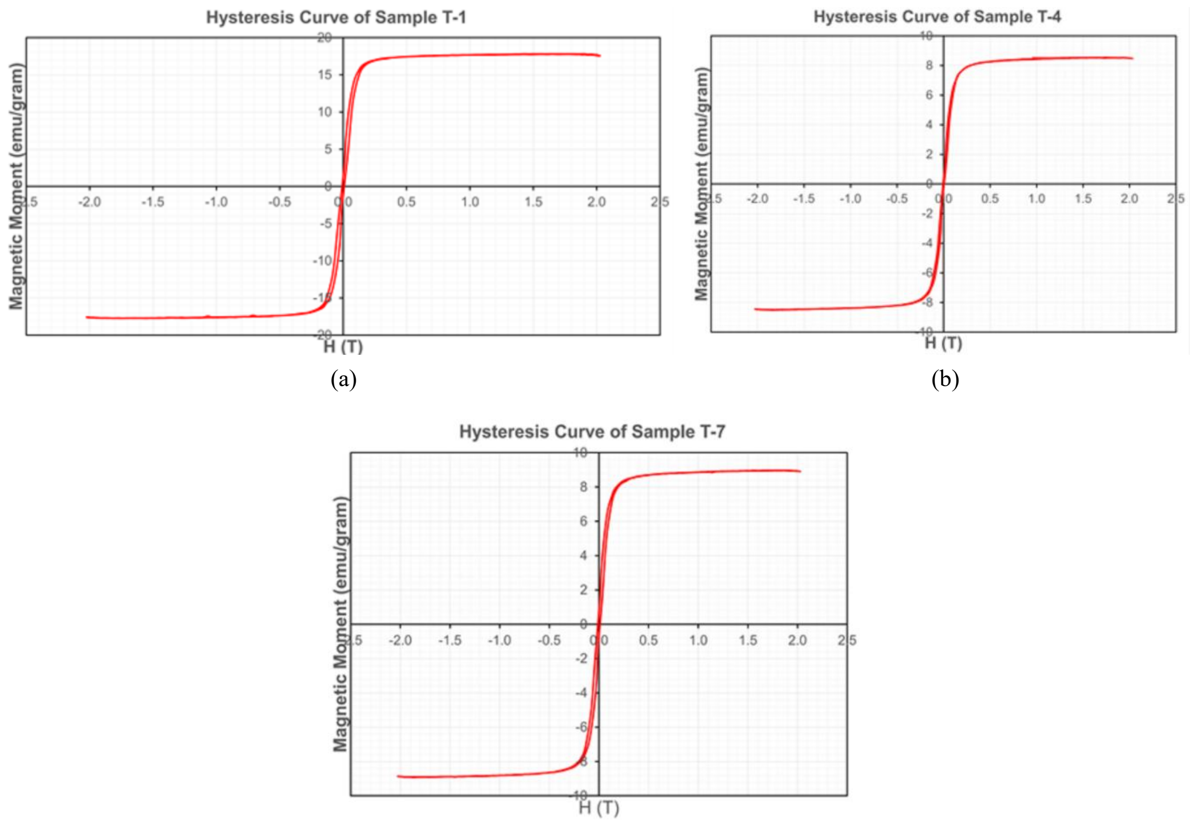
This measurement applies VSM and XRF from a sample of Sangiran sediment and provides information on the magnetic content, oxide compound, and element concentration of the sample.

3.1. *Magnetic Content*

The magnetic susceptibility measurement results are presented in Table 1. Sample susceptibility was measured using two frequencies, namely low frequency (χLF) that uses 31 kHz and high frequency (χHF) that uses 464 kHz. This measurement is intended to generate the frequency dependence magnetic susceptibility χFD (%). This method is also applied to measure and analyze the magnetic susceptibility of rocks [43] and estuary rivers in volcanic regions [44]. The average mass specific magnetic susceptibility (χLF) of the sediment sample T1, T4, and T7 respectively are 465.5 × 10^{-8} m^3kg^{-1}, 235.5 × 10^{-8} m^3kg^{-1}, and 897 × 10^{-8} m^3kg^{-1}. The frequency dependent magnetic susceptibility value (χLF) of the sediment sample T1, T4, and T7 respectively are 464.2 × 10^{-8} m^3kg^{-1}, 232 × 10^{-8} m^3kg^{-1}, and 891.1 × 10^{-8} m^3kg^{-1}. The value of dependent magnetic susceptibility (χFD) of these samples respectively are 0.28%, 1.44%, and 0.66%.

Table 1. Measured magnetic susceptibility data for samples of the sediment. Parameter of χLF is the mass-specific low-frequency susceptibility, χHF is the mass-specific high-frequency susceptibility, and χFD is the frequency-dependent susceptibility using VSM.

No.	Sample/ Station	MH (gram)	MH+MS	χLF (10^{-8} m^3/kg)	χHF (10^{-8} m^3/kg)	Avg. χLF	Avg. χHF	χFD (%)
				465.5	464.2			
1	T1	4.05	12.30	465.5	464.2	465.5	464.2	0.28
				465.5	464.2			
				235.5	232.0			
2	T4	4.05	12.50	235.5	232.0	235.5	232.0	1.49
				235.5	232.0			
				897.0	891.1			
3	T7	4.05	12.50	897.0	891.1	897.0	891.1	0.66
				897.0	891.1			



(a) (b)

(c)

Figure 4. Hysteresis curves of representative samples of the extracted sediments (a) T1, (b) T4, and (c) T7.

The VSM result shows that the magnetic content of Sangiran sediments is dominated by Fe, which is contained in the hematite (Fe_2O_3). This result is consistent with previous studies on magnetic content in mud volcano areas [37], [45]–[48]. A VSM measurement produces a hysteresis curve for a collected sample of sediment. This curve includes an downward and upward curve. The rising curve is the result of VSM measurements with increasing auxiliary magnetic field or magnetic field intensity (**H**) and the falling curve is reversed. Figure 4 shows the hysteresis curve from the result of VSM measurement. Magnetic susceptibility is the comparison between the magnitude of magnetization and the magnetic field intensity [24]. Based on the value of magnetic susceptibility, the results of this study confirm that Fe content is one of the components of volcanic ash in the Sangiran sediment.

The susceptibility results can provide information on the ability of the Sangiran sediment to be magnetized. It was also the most prominent parameter indicating possible heavy metal contamination [49]. As shown by Table 1, the value of χFD (%) is a parameter of mineral content that is superparamagnetic (SP) in size. If the value of χFD (%) is less than 2%, it indicates that the magnetic minerals composed in the sample of Sangiran sediment have the characteristics of a superparamagnetic material. Several characteristics of minerals, the properties of surface sediments, and the existence of superparamagnetic minerals [50]. Reviews have shown that materials sand, ash, lava, gravel indicate higher magnetic susceptibility content from volcanic environments [51], [52]. Based on the value of magnetic susceptibility can characterize volcanoes [53].

3.2. Element Composition

According to the XRF measurements, the Sangiran sediment samples are dominated by Si, Fe, Al, Ca, Cl, Ti, and K. The samples exhibited the highest Si and Fe concentrations in samples T1 (Si = 29.48% and Fe = 13.66%) and T7 (Si = 24.95% and Fe = 12.01%). Meanwhile, in the T4 sample, the highest concentrations were Si and Ca, 23.45% and 13.45%, respectively, as shown in Figure 5. This result also shows that these samples are composed of heavy metal content, namely Fe. Magnetic minerals include

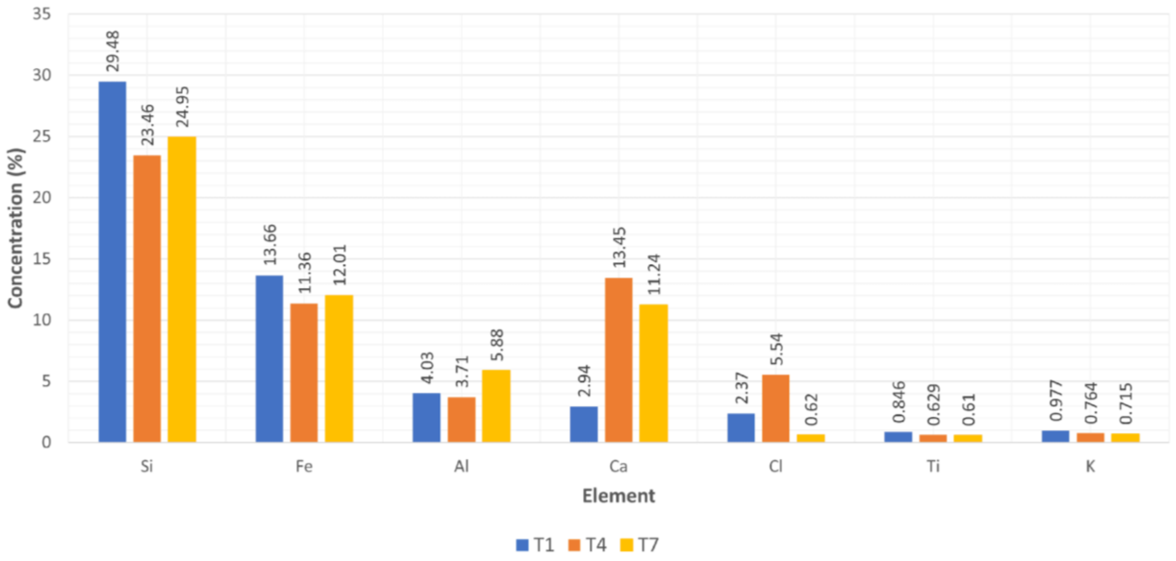


Figure 5. Comparison of the element concentration of sample T1, T4, and T7.

an essential composition of the elements of the fourth subgroup (Sc, Ti, V, Cr, Mn, Fe, Cn, Bi, Cu, Zn) [28]. The average Fe concentration of the three samples is 12.34%. Fe is a ferromagnetic metal containing the highest magnetic susceptibility [37]. As suggested by a previous study, Fe is an element of ferromagnetic that strongly influences the magnetic susceptibility values of sediment samples. An increase in the concentration of elemental Fe induces a gain in the magnetic minerals concentration, which affects the increase in magnetic susceptibility values. However, caution is required in interpreting the magnetic characteristic of the sediments in the volcanic environment [37].

Based on the element composition, the magnetic mineral-forming elements affected the magnetic susceptibility values. This condition is presumably due to samples with high magnetic susceptibility values that interprets higher magnetic mineral content. In general, the mineral composition greatly influences the chemical and physical properties of sediments [47]. However, the magnetic minerals abundance of sediments in volcanic area is suggested to be fully considered [41]. Due to the accumulation of various trace elements (Si, Fe, Al, Ca, Cl, Fi, K, *etc.*), the sedimentation activity of the Sangiran mud volcanoes can also affect surface and soil water geochemistry (sub-) surface sediments, at least regionally [46]. Fe in sediments is presumably highly contained by Fe from geologically weathered sediments [54]. High concentrations of heavy metals in sampling locations deposited by mud volcano conditions. Environmental conditions also play a role to deposit the heavy metal content such as sediment transport from agricultural and residential areas.

3.3. Content of Oxide

Oxides are compounds consisting of one or more oxygen atoms and one other element [53], [55]. A mineral is a chemical compound with a composition within a certain range that can be represented by a formula. Mineral formulations can be complicated or simpler. This condition is determined by the element number and the ratio of their combinations. An oxide compound is one category of mineral. Some of the most important oxide compounds in this research are SiO_2, Fe_2O_3, Al_2O_3, CaO, TiO_2, K_2O and ZrO_2.

The comparison of oxide concentrations for the three samples is presented in Figure 6. Results of XRF measurements imply Sangiran sediments contain ash elements of Si, Al, Si, Ca, Fe, K, and Zr. The average oxide compound concentration of the samples T1, T4, and T7 from the highest to the lowest are as follows: SiO_2 (55.54%), Fe_2O_3 (17.66%), Al_2O_3 (8.58%), CaO (12.89%), TiO_2 (1.16%), K_2O (0.97%), and ZrO_2 (0.05%). The elements obtained from this measurement correspond to Si and Fe, which are components of volcanic sediments formation. The combined composition of SiO_2, Al_2O_3, and Fe_2O_3 above 70% indicates that the samples of the Sangiran mud volcano can be classified as pozzolanic materials, which is compatible with substitute materials for portland cement [56].

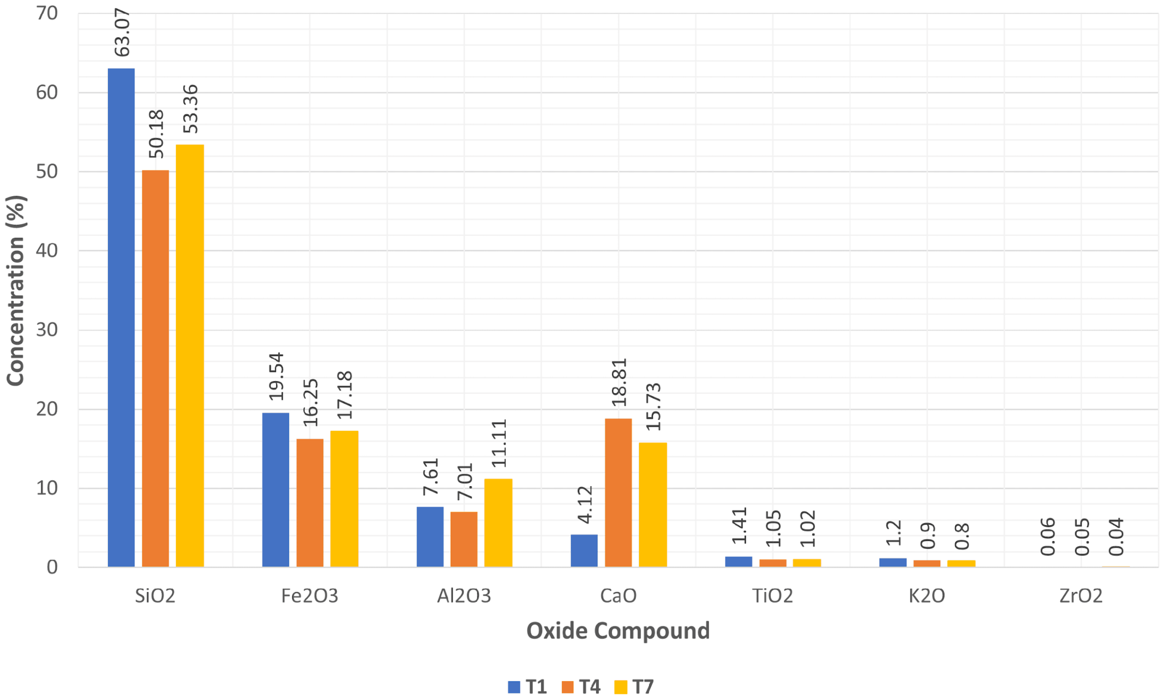


Figure 6. Comparison of the oxide concentration of sample T1, T4, and T7.

4. Conclusion

Sangiran mud volcano is a geological formation located in Sangiran dome, which is formed due to tectonic activity. The mineralized elements in the Sangiran sediments, including the sample of T1, T4, and T7, are formed and dominated by Si, Fe, Al, Ca, Cl, Ti, and K. The content of oxide in the samples are dominated by SiO_2, Fe_2O_3, Al_2O_3, CaO, TiO_2, K_2O, and ZrO_2. This compound was evenly distributed in all three samples. Based on the generated magnetic content, through further testing on VSM, the magnetic content is found to be superparamagnetic. This is evidenced by the hysteresis curves of the three samples, which tend to form thin curves. The magnetism stored in the sample is dominated by Fe contained in the Fe_2O_3 (hematite) oxide compound. Fe is an element of ferromagnetic that strongly influences the magnetic susceptibility values of the sediment samples. Increased concentrations of elemental iron led to increased concentrations of magnetic minerals. Retrieved from the result of magnetic susceptibility, the outcomes of this study confirm that Fe content is one of the components of volcanic ash in the Sangiran sediment.

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