Analysis of Chemical Compound Content and Magnetic Properties of Iron Sand in Rondo Woing Village, East Manggarai

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Received: 16 April 2023; **Accepted**: 12 June 2023; **Published**: 24 June 2023
DOI: http://dx.doi.org/10.29303/jpft.v9i1.4897

Abstract *- The mine sand extraction process used the Methanol Soap Bathed (MSB) method. The results of sample analysis using XRF and VSM (Vibrating Sample Magnetometer) obtained that the most dominant compound content was Silica (SiO2), Iron (Fe2O3), Quicklime (CaO), and Alumina (Al2O3). At the same time, the sample results from the Vibrating Sample Magnetometer (VSM) show that Rondo Woing East Manggarai iron sand has soft magnetic properties. The iron sand sample with code 001 has Hc, Mr, and Ms of 324.83 Oe, 0.31 emu/gram, and 1.61 emu/gram, respectively. The iron sand sample with code 002 has Hc, Mr, and Ms of 319.91 Oe, 0.31 emu/gram, and 1.6 emu/gram, respectively. The results of the analysis show that four dominant elements are the same; namely, Fe is the most dominant element in Fe2O3 compounds, with 32.3%, Al elements in Al2O3 compounds, with levels of 13%, Si in SiO2compounds with levels of 31.1% and Ca in a CaO compound with a content of 17.7% and Ti in a TiO2 compound of around 2%, so that the iron sand at Rondo Woing shows magnetic material properties.*

Keywords: *Iron sand, Iron (Fe), Calcium (Ca), Methanol Soap Bathed (MSB)*

INTRODUCTION

Manggarai Timur Regency is a result of the division of Manggarai Regency, which was established at the end of 2007. Manggarai Timur Regency stretches from the north, bordering the Flores Sea, to the south, bordered by the Sawu Sea. Meanwhile, in the eastern and western parts, it is bordered by Ngada Regency and Manggarai Regency. In 2021, there was a division in three sub-districts of Manggarai Timur Regency: Kota Komba, Sambi Rampas, and Lamba Leda. The additional sub-districts are Kota Komba Utara, Congkar, and Lamba Leda Utara, resulting in 12 sub-districts in Manggarai Timur Regency. The agricultural sector remains the main field of employment that absorbs the most workforce in Manggarai Timur Regency (BPS, 2022)(Ruth & Gustina, 2022).

The topography of East Manggarai Regency is a mountainous region with sandy and clayey soil structures. However, using sand as a building material in that area is still limited. Iron sand from East Manggarai Regency can be utilized as a mixture for building materials because the sand color in that area is dark. Iron sand is a type of sand that contains Iron (magnetite). It is often found along the coast, appearing shiny and black, with darker color indicating a higher iron mineral content. Iron sand is formed through weathering, surface water, and wave transformation from its original rock, which is basaltic to andesitic. Iron sand contains the main mineral magnetite (iron oxide) associated with titanomagnetite, with a small amount of magnetite and hematite, accompanied by impurity minerals such as quartz, pyroxene, biotite, and rutile. Other common impurities found in iron sand are phosphorus and sulfur. It is relatively easy to test whether it is iron sand. Prepare a magnet and bring it close to the sand. If any iron minerals are attracted to the magnet, it is confirmed that the sand is iron sand. Iron sand is formed through the weathering process, surface water, and wave action on the original rock containing iron minerals, and then it accumulates and is washed by ocean waves. Iron sand is used in the cement industry and can be developed as a raw material for steel production according to technology. The prospect of iron sand in Indonesia has been explored and even exploited for utilization. Iron sand mining is widely conducted along the west coast of Sumatra, the south coast of Java and Bali, and the north coast of Papua. The majority of iron sand reserves are scattered in the coastal waters of Indonesia, from the west coast of Sumatra, the south coast of Java and Bali, the coast of Sulawesi, the coast of East Nusa Tenggara (NTT), and the coast of Papua.

The total reserves for iron ore are 173,810,612 tons, and for metal, 25,412,652.62 tons. However, its utilization is not yet optimal because of PT. Krakatau Steel and PT. Krakatau Posco only produces 24,000 to 36,000 tons of steel plates per year. Meanwhile, the shipbuilding industry requires 900,000 tons of steel plates annually. The need for raw materials for steel plates in the form of sponge iron with Fe $\geq$ 60%, PT. Krakatau Steel still imports from foreign countries. The evidence is that PT. Krakatau Steel imported 3,500,000 tons of iron ore pellets per year from Sweden, Chile, and Brazil before and during the 2000s. This condition hinders the competitiveness of the national steel industry against foreign steel industries due to the import duties imposed on raw materials. There is an opportunity to establish a raw material company for steel production since, currently, there are only two such companies in Indonesia. This situation has prompted sponge iron production with a production capacity adjusted to the installed capacity. Research on the analysis of sponge iron production using Cipatujah iron sand as a raw material has yielded sponge iron with the highest iron content of Fe $\geq$ 60.44%. It can be used for the raw material needs of PT. Krakatau Steel in steel production, as PT. Krakatau Steel has claimed that the local sponge iron product has less than 60% Fe content.

It can drive the self-sufficiency of raw materials for the steel industry, which will impact the self-sufficiency of the defense industry. However, the government should also implement protection measures and prioritize domestic raw materials for national steel production. One approach could be for the government to establish a state-owned national steel industry that fosters a consortium of raw material suppliers (sponge iron) to ensure the quality and continuous supply of sponge iron (Aritonang et al., 2019). With its extensive coastline, Indonesia possesses a complete iron sand resource of 4,280 million tons and reserves of 750 million tons, with a magnetization degree of 65% and a Fe content reaching 45%, as reported by the Ministry of Energy and Mineral Resources in 2018 (Firjatullah et al., 2022).

The utilization of iron sand as a natural resource is still not optimal due to Indonesia's geological position at the convergence of three tectonic plates: the Indo-Australian Plate, the Indo-Eurasian Plate, the Eurasian Plate, and the Pacific Plate. This geological condition creates a complex technical arrangement that inherently supports the potential of rich mineral deposits (Fitri, 2016). Besides its high economic value, iron sand benefits the industry and mining sector. Some of the benefits of iron sand are as follows: Firstly, support for the steel industry: Iron sand, which consists of iron impurities, can be separated to obtain pure Iron. Once the impurity-free Iron is obtained, it can be directly processed into steel. The implementation of appropriate iron sand processing technology is expected to supply raw materials for the national steel industry, thus realizing its self-sufficiency of the national steel industry. Steel is a vital raw material in various industries, and many everyday tools are predominantly steel. Secondly, raw material for cement production: Iron sand mining in coastal areas of Indonesia is carried out to obtain iron sand, which is then processed into cement and concrete. Cement and concrete made from iron sand have better compressive and tensile strength than other materials. Thirdly, the production of antibacterial materials: Another benefit of iron sand is its use as a raw material for antibacterial products. Materials derived from iron sand can be used to create products that offer protection against bacteria, such as antiseptic soap and antibacterial soap. Iron sand in the medical field has significant economic value for industries and society. Fourthly, enhancement of concrete compressive strength and tensile strength: Iron sand is used to enhance concrete's compressive and tensile strength in the concrete industry. Using 80% iron sand by weight of the total sand will provide a maximum compressive strength of 42.65 MPa and increase the compressive strength capability by 28.41%.

Furthermore, additional iron sand can increase the compressive strength by 3.07 MPa and the splitting tensile strength by 4.84%. Concrete plays a crucial role in supporting the loads applied to structures. Its superior compressive strength and ease of procurement make it a continuous solution for infrastructure challenges. However, the large-scale use of materials can deplete natural resources if not properly managed. Iron sand with magnesium (Mg) content can be used as a substitute for fine aggregates in concrete mixtures. This content improves the bond between cement and coarse aggregates and enhances the quality of concrete, such as its compressive strength, tensile strength, and modulus of elasticity (Aji, 2014).

Research conducted by Hilman (2014) states that the Sungai Opak sand contains a mixture of non-metallic particles such as quartz, calcite, feldspar, amphibole, pyroxene, biotite, and tourmaline, which are elements found in iron sand. The main iron content in the sand deposit consists of tetanomagnetic minerals, which consist of fine sand grains with diameters between 0.074 - 0.075 mm (fine grains) and 3-5 mm (coarse grains). Iron sand also contains Fe_2O_3, SiO_2, MgO, which have characteristics that make them suitable as a substitute for fine aggregate in producing high-quality concrete (Aji, 2014). The Iron (Fe) content in iron sand is widely used as a raw material for steel production. Additionally, iron sand contains magnetic minerals such as magnetite (Fe_3O_4), hematite (α-Fe_2O_3), and maghemite (γFe_2O_3), which can be applied in various fields (Juharni, 2016); (Losa, 2013); (Widianto & Fauji, 2018). Magnetite (Fe_3O_4) can be applied as a magnetic recording media, high-density digital recording disk, magnetic fluids, data storage, MRI, drug delivery system, biosensor SPR, microwave device, and magnetic sensing (Ghandoor et al., 2012). Magnetite (Fe_3O_4) nanoparticles can be obtained through synthetic or natural materials. The consideration for using natural materials such as Iron or black sands is due to the abundance of existing natural resources that have not been utilized and the lower production costs (Indrayana, 2019). The exploration of iron sand as nanoparticles is still limited compared to the exploration of iron sand as a raw material. It poses a challenge for research in the field (Widianto & Fauji, 2018).

Iron sand contains the main mineral, magnetite (iron oxide), associated with titanomagnetite with small amounts of magnetite and hematite, accompanied by impurity minerals such as quartz, pyroxene, biotite, and rutile. Other impurities commonly found in iron sand are phosphorus and sulfur. Iron sand is grey to black, very fine-grained, with sizes ranging from 75 to 150 microns, a density of 2-5 gr/cm3, specific gravity (SG) of 2.99 - 4.23 gr/cm3, and magnetization degree (MD) of 6.4 - 27.16%. These minerals have a Mohs hardness value of 5 6.5 (Adi, 2018). Iron sand containing the main magnetite mineral is characterized by magnetite grains that are always bonded to other magnetite grains, forming a chain-like structure. The mineral grains have an isometric crystal system, causing iron sand (magnetite) to tend to be rounded or subrounded in shape. This research aims to analyze the chemical composition of iron sand and investigate its magnetic properties.

RESEARCH METHODS

The research was conducted at the Laboratory of Science, Nusa Cendana University in Kupang. The samples were further tested at the Laboratory of Mineral and Advanced Materials, Faculty of Mathematics and Natural Sciences, State University of Malang, and the Advanced Characterization Laboratory in Serpong, LIPI. The equipment used in this research includes a scoop, measuring glass, sand pestle, 80-mesh sieve, digital scale (Ohaus), hot plate, mortar and pestle, oven, permanent magnet, spatula, pipette, watch glass, volumetric flask, X-Ray Fluorescence (XRF) instrument, and Vibrating Sample Magnetometer (VSM) instrument. The materials used in this research include CH_3OH, HCl, soap powder (detergent), and sand collected from Rondo Woing Village, East Manggarai.

The variables in this research are as follows: the independent variable is the sand sample, the control variables are the extraction methods (Methanol Soap Bathed and Hydrochloric Acid), and the dependent variables are the chemical compound content and magnetic properties. The research sample analysis, the sand taken from Rondo Woing, involved cleaning the sand with water to separate it from impurities like soil (Sari & Manurung, 2019). The purification process using the Methanol Soap Bathed (MSB) method aims to increase the purity level of the obtained sand. It involves preparing a container filled with sand and water mixed with detergent, stirring until soap bubbles appear, and then adding methanol to the solution. The iron sand is then extracted using a permanent magnet.

In this research, the sample preparation of East Manggarai Iron Sand at Rondo Woing involves grinding the sample with a mortar and sieving it with an 80-mesh sieve. The sample is then extracted using the Methanol Soap Bathed (MSB) method to separate impurities from the Rondo Woing sand and obtain pure sand. Afterward, the sample is washed with hydrochloric acid (HCl). The extracted sample is soaked in hydrochloric acid solution for 2 hours to remove salt impurities. It is then washed with distilled water until the pH reaches 7, as the sample is acidic. The sample is dried, and the analysis process begins. The washed sample, previously treated with hydrochloric acid, is dried at 100°C to remove moisture content. The next step involves the separation (extraction) of magnetic materials from the sand using a permanent magnet. The magnetic sand is analyzed using X-Ray Fluorescence (XRF) equipment to determine the mineral content of the Rondo Woing iron sand. The magnetic analysis is conducted using the VSM method to determine its magnetic properties, and the characterization results from VSM will yield a hysteresis curve.

RESULTS AND DISCUSSION

Iron sand is a material formed from metal mines through the transportation and sedimentation process of sand materials containing Iron (Tellu et al., 2020). The collected samples underwent treatment by filtering using an 80-mesh sieve to ensure uniform particle size. Each sample was weighed at 100 grams in separate containers. The Methanol Soap Bathed (MSB) method was used to clean the sand from dust and other deposits. The MSB method is a faster and cost-effective extraction method that maintains the purity of magnetic minerals without damaging the magnetic grains (Rifai et al., 2010). Each sample was washed using 4 grams of soap powder and stirred until clean. After that, it was left for 5-10 minutes, and the water was drained. Next, 200 ml of methanol with concentrations of 2M and 4M was added to each sample, stirred with a spatula, and dried in an oven at 100°C for 2 hours to reduce the moisture content.

Table 1. Analysis of XRF Elements and Oxidation of Sample 1

Element	Al	Si	K	Ca	Ti	Fe	Others Element
Wt %	13	31,1	1,2	17,7	2,05	32,3	2,18
Oxidation	Al2O3	SiO2		CaO	TiO2	Fe2O3	Other Oxide
Wt %	17	42,8		13,8	1,79	22,2	2,177

In the next step, the samples were washed using 200 ml of HCl in three containers with concentrations of 2M, and magnetic stirring was performed for 2 hours. This washing process made the samples acidic. Therefore, the samples were washed with distilled water until reaching a pH scale of 6-7. The samples were then dried in an oven at 100°C for 2 hours. Each sample was weighed at 7 grams for characterization using XRF and VSM. The results of the Iron Sand Rondo Woing Magnetic Property Test using XRF. Analysis of Rondo Woing iron sand using XRF, as shown in Table 1 and Table 2, reveals that the iron sand contains Al, Si, K, Ca, Ti, V, Cr, Fe, Cu, Zn, Sr, Eu, and Re. For each analyzed sample, four dominant elements are consistent: Fe is the most dominant element in the form of Fe2O3 compound, with a concentration of 32.3%; Al in the form of Al2O3 compound, with a concentration of 13%; Si in the form of SiO2 compound, with a concentration of 31.1%; and Ca in the form of CaO compound, with a concentration of 17.7%. Additionally, Ti is present as a TiO_2 compound, with a concentration of approximately 2%. Therefore, the iron sand from Rondo Woing exhibits magnetic properties, is black, and can be attracted by a magnet.

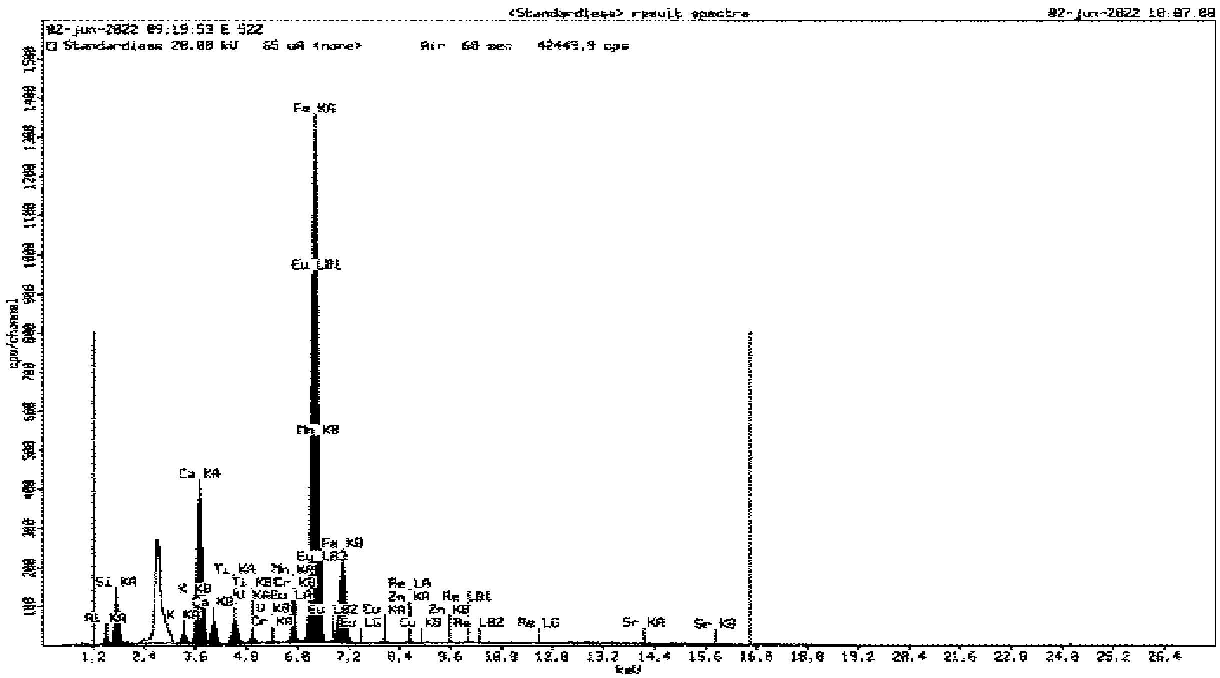

Figure 1. Analysis of XRF Spectrum for Sample 1 Results of VSM testing for Sample 1. (CH_3OH 1M)

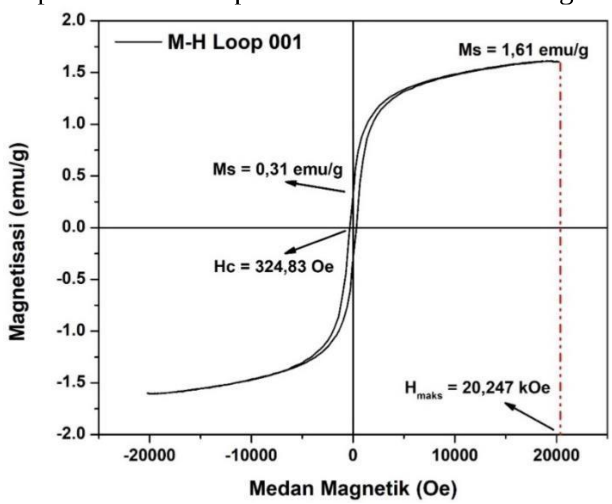

Figure 2. Hysteresis Curve for Sample 1. XRF Results for the second sample. (CH_3OH 4M)

Table 2. XRF Analysis Results of Elements and Oxidation for Sample 2

Element	Al	Si	K	Ca	Ti	Fe	Others Element
Wt %	13	31,2	1,3	17,4	2,07	32,7	2,165
Oxidation	Al_2O_3	SiO_2		CaO	TiO_2	Fe_2O_3	Other Oxide
Wt %	17	43,0		13,6	1,82	22,6	2,19

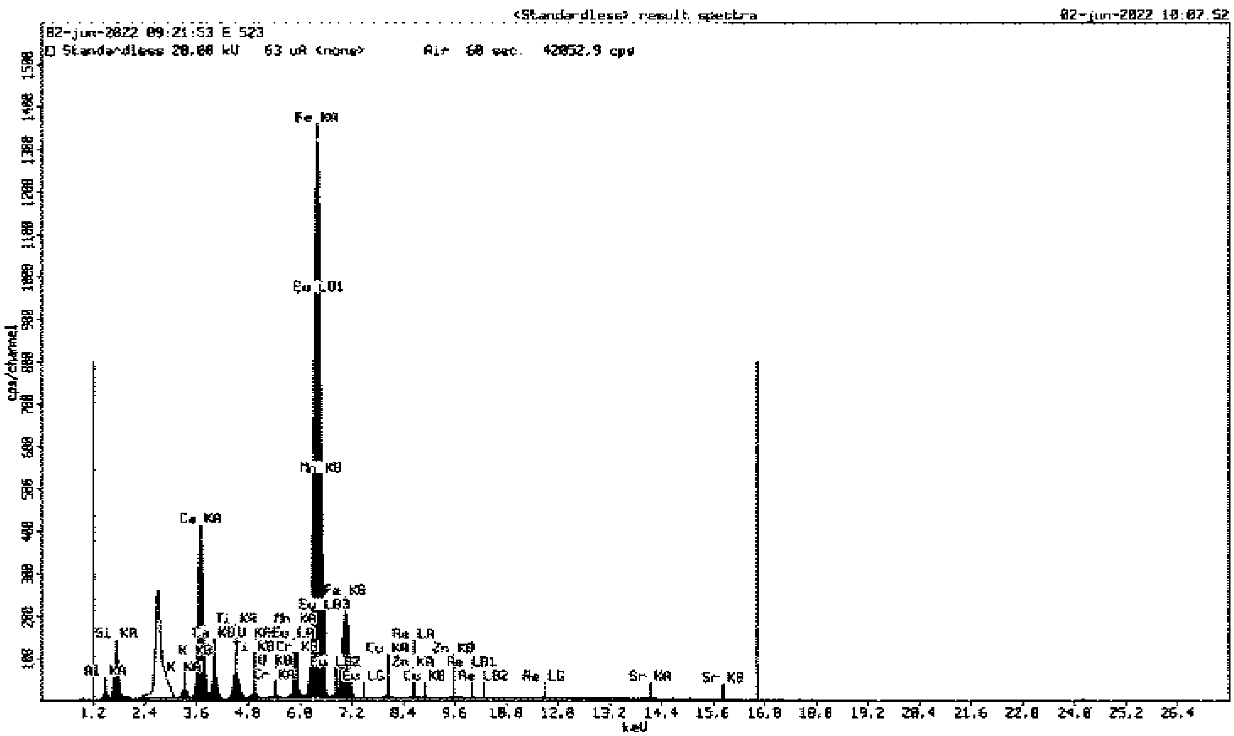

Figure. 3 XRF Spectrum for Sample 2 VSM Results for Sample 2 (CH_3OH 4M)

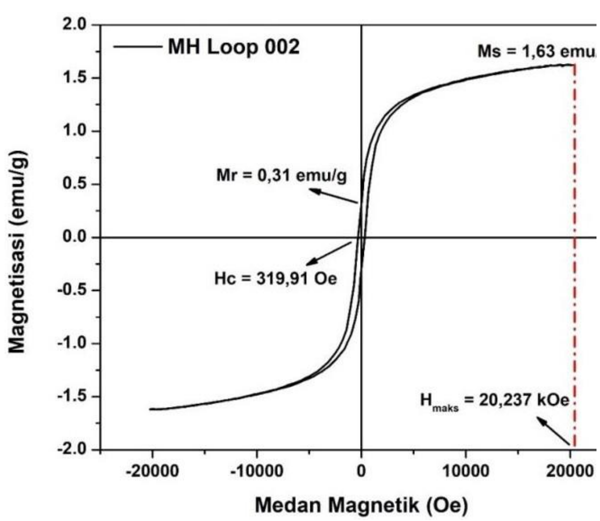

Figure 4. Hysteresis Curve for Sample 2

In general, the composition of the iron sand in Rondo Woing shows economic value in the cement industry due to its Fe content of approximately 32.3%. Based on a study conducted by Aji (2014) on the mining and processing of iron sand in the Kulonprogo Regency area, it is a mining project with a large investment, ranging from IDR 5.4 to 6 trillion, covering a Contract of the Work area of 2,987.79 hectares. The total iron sand resources in Kulonprogo amount to 605 million tons, with a Fe content of approximately 10.8%, requiring a workforce of 2,100 people. It is also supported by research findings (Junursyah & Rahmat, 2019) the composition of economically valuable iron sand in the cement and steel industries consists of minerals such as magnetite (Fe_3O_4), ilmenite ($FeTiO_3$), hematite (Fe_2O_3), and limonite (Moon et al., 2006; Dipatunggoro, 2012), mixed with non-metallic mineral grains such as quartz, calcite, feldspar, amphibole, pyroxene, biotite, and tourmaline (Aji, 2014). The washing process with Methanol Soap Bhated (MSB) and the particle size of the sand grains affect the composition of the iron sand. The factors influencing this are the sample preparation with varying sizes, specifically 80 mesh, and the washing with MSB, as this extraction method is faster, cost-effective, and can maintain the purity of magnetic minerals without damaging the existing magnetic grains. The chemical compounds Fe2O3 and SiO2 have higher percentage concentrations than other components. It is due to the influence of the concentration of methanol used during the extraction process. The purpose of using methanol in this research is to break the bond between magnetic and non-magnetic materials. To determine the magnetic properties of Rondo Woing iron sand, a Vibrating Sample Magnetometer (VSM) is needed. VSM is one of the instruments used to understand and study the magnetic properties of materials. Magnetic properties in materials occur due to temperature changes and magnetic properties as a function of the measurement angle or the anisotropic conditions of the material (Tebriani, 2019).

This study will discuss the analysis results of Rondo Woing iron sand using the Vibrating Sample Magnetometer (VSM). The VSM analysis produces a curve known as the hysteresis curve, as shown in Figures 1 and 2. In determining the magnetic properties based on the hysteresis curve, several important parameters need to be considered, including magnetic saturation (Ms), coercivity (Hc), and remanent magnetization (Mr). The value of magnetic saturation, also known as saturation magnetization, indicates the ability of nanoparticles to maintain their magnetic domain alignment when subjected to an external magnetic field (Tebriani, 2019). From Figures 1 and 2, the measured sample at a magnetic field of 20.247 KOe with code 001 (2M) has a maximum saturation magnetization (Ms) value of 1.61 emu/g, a coercivity (Hc) value of 324.83 Oe, and a remanent magnetization (Mr) value of 0.31 emu/g. For the measured sample at a magnetic field of 20.237 KOe with code 002 (4M), it has a maximum saturation magnetization (Ms) value of 1.63 emu/g, a coercivity (Hc) value of 319.91 Oe, and a remanent magnetization (Mr) value of 0.31 emu/g.

Based on the shape of the magnetic moment change with the applied external field, the analyzed magnetite with methanol exhibits paramagnetic properties, showing symmetric hysteresis curves when subjected to an external magnetic field, resulting in a very narrow hysteresis curve width. A narrow hysteresis curve width indicates that the sand material is easily magnetized or requires low energy, which falls into the category of soft magnetic materials and exhibits ferromagnetic properties. From the graph, it can be seen that the iron sand from Rondo Woing exhibits ferromagnetic properties.

The magnetic properties were tested on three samples with different treatments, namely using a 2M Methanol Soap Bhated solution for sample 1 and a 4M Methanol Soap Bhated solution for sample 2. The magnetic domains of the iron sand initially have zero values. When a current is applied, a magnetic field appears as the magnetic domains within the iron sand become aligned. This process is called magnetization until all magnetic domains of the iron sand align in the same direction as the external magnetic field, called the saturation point (Ms).

Therefore, the higher the value of Ms, the higher the magnetic properties of the iron sand. It also applies to the value of Mr. When the electric current is turned off, the magnetic domains in the iron sand do not automatically become zero; there will be residual magnetism known as Mr or permanent magnet. The larger the value of Mr, the higher the magnetic properties of the iron sand. The same applies to the coercivity value (Hc). A demagnetization process is carried out to demagnetize the iron sand and return it to a non-permanent state (zero value). This process involves applying an opposite electric current to the zero point to obtain the coercivity value.

The results of this research are expected to provide information about the mineral resources in the form of iron sand in the Rondowoing area. It can impact the economic value and empowerment of the community in processing natural resources. However, the impacts of iron sand mining should also be considered. There are various responses from the local community regarding iron sand mining, ranging from pro-mining to anti-mining. For example, iron sand mining in the Binangun District has caused conflicts among the community due to differing arguments regarding this mining activity. Mining proponents argue that iron sand mining can improve the economic conditions of the community and the local government. On the other hand, opponents argue that iron sand mining has more negative impacts than positive ones, such as environmental damage and displacement of agricultural land belonging to the community.

CONCLUSION

In the Rondo Woing Manggarai Timur sand sample, the Fe element content increases with methanol at 2M and 4M concentrations. The analysis of the iron sand reveals four dominant elements: Fe, which is the most dominant element in the form of Fe2O3 with a concentration of 32.3%; Al, in the form of Al2O3 with a concentration of 13%; Si, in the form of SiO2 with a concentration of 31.1%; and Ca in the form of CaO with a concentration of 17.7%. Additionally, Ti is present in TiO2 with a concentration of approximately 2%. It indicates that the iron sand in Rondo Woing exhibits magnetic properties and holds economic value.

The analysis using Vibrating Sample Magnetometer (VSM) and the hysteresis curve shows that the Rondo Woing sand sample possesses soft magnetic characteristics, known as ferromagnetic.

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