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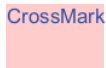
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Magnetic Mineral Concentration and Microcrystalline Characteristics of Fe-Rich Coastal Sand from North Sulawesi

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Abstract. Magnetic mineral concentration, especially from the iron oxide class, is one of the important indicators in determining the potential in sand natural resources. In this study, the magnetic measurement as well as X-ray spectroscopy analysis have been carried out in order to determine the concentration and microcrystalline characteristics of iron sand originating from four locations in the coastal area of North Sulawesi which are Lolan, Lalow, Hais, and Minanga. Magnetic properties study through measurement of saturation isothermal remanent magnetization (SIRM) confirmed by oxide composition obtained from X-ray fluorescence (XRF) observation showed that the iron sand sample from Lolan had the highest concentration of iron oxide minerals followed by samples from Hais, Lalow, and Minanga respectively. The percentage of iron oxide at these sites varied between 60.33 – 76.70 wt.%, implying that it was relatively competitive compared to the samples from other localities in Indonesia. Findings related to variations in crystal size and degree of crystallinity are closely related to the source and mechanism of iron sand formation at each study site.

INTRODUCTION

Indonesia is an archipelagic country with many coastal areas identified as having iron sand potential. In general, the description of characteristics and mineralogy of iron sands in these areas has been studied by various methods such as x-ray spectroscopy [1, 2], scanning microscopy [3] and magnetic methods especially magnetic susceptibility [4]. North Sulawesi is one of these areas that has the prospect of iron sand. The previous study on the characteristics of iron sands in this area has been carried out in several locations, namely Lolan, Lalow, Hais, and Minanga [3]. Granulometric observation and microstructural analysis using a scanning electron microscope (SEM) equipped with energy dispersive X-ray (EDX) provided information about the grain size distribution as well as the morphology and elemental composition of iron-rich mineral grains in samples from that area. However, the concentration of iron oxide minerals has not been studied.

The iron in nature generally binds with oxygen to form iron oxide minerals, the minerals which have magnetic properties and are commonly termed magnetic minerals. Recently, the magnetic method has begun to be widely used for studying the characteristics of magnetic minerals in the iron sand sample. Some of the magnetic properties that are affected by the concentration of magnetic minerals are magnetic susceptibility, saturation isothermal remanent magnetization (SIRM), and anhysteretic remanent magnetization (ARM) [5]. In this study, the magnetic properties of iron sand samples from Lolan, Lalow, Hais, and Minanga was carried out, especially to obtain the concentration of

magnetic minerals. X-ray spectroscopy measurements are also performed to support magnetic studies and to provide an overview of the microcrystalline characteristics of the sample.

MATERIAL AND METHODS

The iron sand samples originate from four locations with coordinates as in Tamuntuan *et al.* [3]. Figure 1 shows the coordinates plot of the sampling locations on a map of North Sulawesi. Two locations are on the west coast and the other two locations are on the east coast.

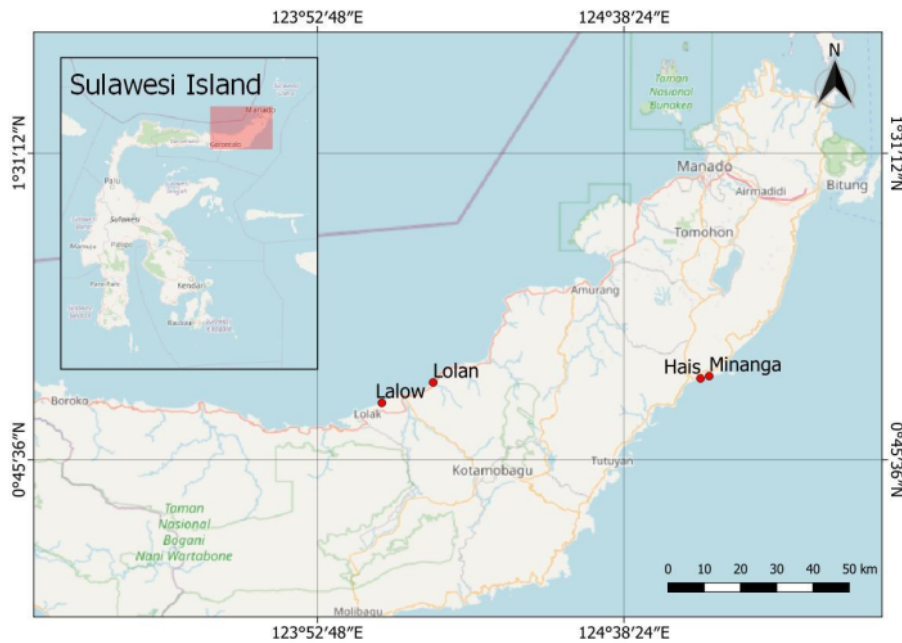


FIGURE 1. The locations of iron sand sampling.

Measurement of magnetic susceptibility (χ_{LF}) and frequency-dependent magnetic susceptibility (χ_{FD}) on the iron sand samples from Lolan, Lalow, Hais, and Minanga were previously conducted and described in Tamuntuan *et al.* [6]. Measurement of magnetic properties in the form of isothermal remanent magnetization (IRM) has been carried out using a vibrating sample magnetometer (VSM) at the Center for Advanced Materials Science and Technology of the National Atomic Agency or BATAN. The type of magnetic mineral is determined based on the *s*-ratio, namely the ratio of IRM of 0.3 T to IRM of 1 T [7]. The relative magnetic mineral concentration was determined based on the value of saturation isothermal remanent magnetization (SIRM). Observation of the composition of elements and major oxides was carried out by measuring X-ray Fluorescence (XRF) at the Central Laboratory of the State University of Malang. For microcrystalline analysis, X-Ray Diffraction measurements have been performed at the Hydrogeology and Hydrogeochemistry Laboratory, Bandung Institute of Technology. Determination of the degree of crystallinity was carried out based on the Soltys equation while the crystallite size was determined using the Debye-Scherrer equation [8]. Analysis of the crystalline and amorphous regions, as well as the full width half maximum (FWHM) of the spectral peaks used in the two aforementioned equations, were conducted using the ORIGIN software.

RESULTS AND DISCUSSION

Figure 2 demonstrates the results of magnetic properties measurements of iron sand samples from four locations in North Sulawesi. In the previous study [6], the magnetic susceptibility (χ_{LF}) values in the four samples varied from

$150 \times 10^{-6} \text{ m}^3\text{kg}^{-1}$ to $350 \times 10^{-6} \text{ m}^3\text{kg}^{-1}$ while the frequency-dependent magnetic susceptibility (χ_{FD}) varied from 0.35% to 2.27% (Fig. 2a). The χ_{LF} values sequentially from the highest to the lowest were Lolan (LOL), Hais (HAIS), Minanga (MNG), and then Lalow (LAL) samples. The higher the magnetic susceptibility value, the higher the concentration of magnetic minerals. However, magnetic susceptibility is a magnetic parameter that depends not only on concentration but also on the type and grains size or domains of the magnetic mineral. On the other hand, it is shown that from the four samples only MNG has an χ_{FD} value of more than 2%. The value of $\chi_{\text{FD}} \leq 2\%$ indicates the sample does not contain superparamagnetic minerals [9].

Figure 2b shows the results of measurements of isothermal remanent magnetization (IRM) at an applied DC field up to 1000 mT. Although the level of magnetization on the IRM acquisition curves looks varied [10] curves were saturated in an applied field less than 300 mT. The S-ratio value ranged from 0.96 – 0.97. This indicates that the magnetic properties of all samples are strongly influenced by ferrimagnetic iron oxide minerals, such as magnetite (Fe_3O_4) and/or maghemite ($\gamma\text{-Fe}_2\text{O}_3$). Ferrimagnetic minerals have low coercivity. Magnetic moments are relatively easy to align which is causing the magnetization will saturate faster when an induced field is applied. Previous studies have shown that samples dominated by ferrimagnetic minerals can even begin to saturate at a given field of 100 mT [10]. On the other hand, in high coercivity magnetic minerals, such as hematite ($\alpha\text{-Fe}_2\text{O}_3$) and goethite (FeOOH), the alignment of magnetic moments is relatively more difficult. The saturation magnetization of these minerals occurs when a DC field induction is equal to or more than 600 mT.

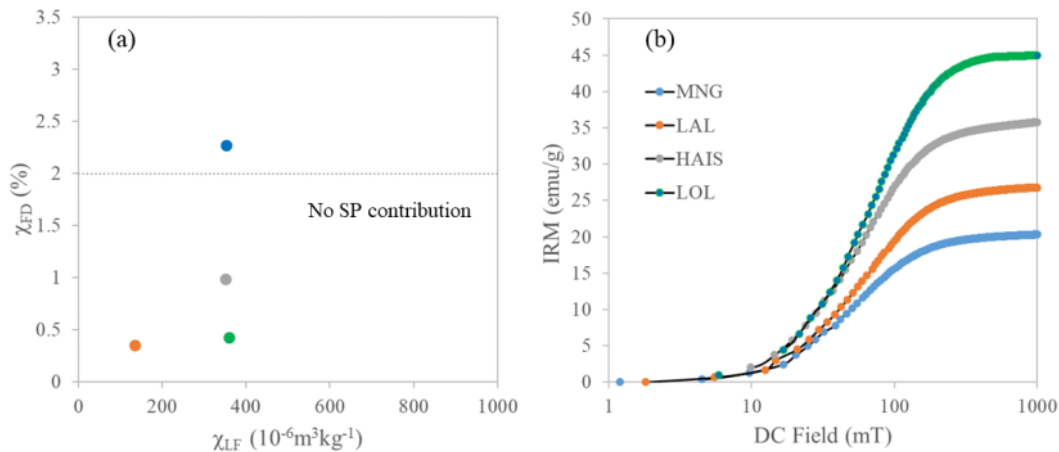
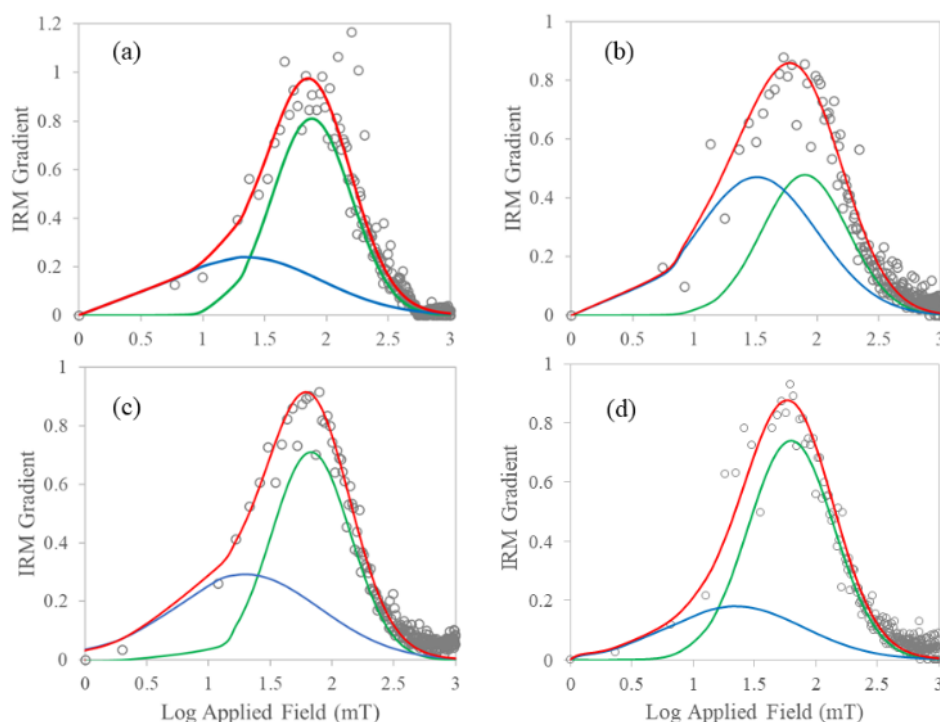


FIGURE 2. (a) Plot of χ_{FD} versus χ_{LF} based on data from Tamuntuan *et al.* [6] for the samples from Lolan (LOL), Lalow (orange), Hais (grey), and Minanga (blue). SP is superparamagnetic mineral. (b) IRM acquisition curves in the applied DC field up to 1000 mT

The value of saturation IRM (SIRM) depends on the concentration of magnetic minerals in the sample [11]. The higher the concentration of magnetic minerals, the higher the SIRM value, and vice versa. SIRM values of the measured samples ranged from 20 – 45 emu/g. Samples from Lolan had the highest SIRM value of 44.98 emu/g, followed by samples from Hais with a value of 35.77 emu/g, Lalow with a value of 26.80, and the lowest was from Minanga at 20.3 emu/g. In contrast to the magnetic susceptibility parameter, the SIRM parameter is not affected by the concentration of superparamagnetic minerals due these minerals do not have magnetic remanence [12]. Therefore, it can be interpreted that iron sand sample from Lolan has the highest concentration of magnetic minerals, followed by samples from Hais, Lalow, and finally Minanga. This also confirms that the magnetic susceptibility value of the Minanga sample which is higher than the Lalow sample is due to the influence of the contribution of superparamagnetic minerals.

The IRM acquisition curve can also be used to identify the component of magnetic coercivity through cumulative log-Gaussian analysis [13, 14]. Figure 3 shows the IRM gradient acquisition plot for the four samples used. In general, a curve model (red curve) as an approximation to the distribution of IRM data can be constructed based on the

cumulative of two log-Gaussian which indicate that the ferrimagnetic mineral population in the sample is distributed at two different mean coercivity values.



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FIGURE 3. Gradient acquisition curves of samples from (a) Lolan, (b) Lalow, (c) Hais, and (d) Minanga.

Table 1 shows the selected major oxides as a result of XRF measurements at the four sites. The iron oxides are represented as Fe_2O_3 and FeO^* , the iron (II) oxide is calculated according to the method described in Yang *et al.* [15]. All sample show a relatively low content of glass material, which is between 13-20 wt.%, but were rich in iron. This indicates that iron oxide as well as titanium oxide in iron sands from Lolan, Lalow, Minanga, and Hais are relatively competitive when compared to other regions in Indonesia [4, 16].

TABLE 1. Selected major oxides on iron sand samples.

Site	Oxides (wt.%)					
	Al_2O_3	SiO_2	TiO_2	CaO	Fe_2O_3	FeO^*
Lalow	6.10	18.00	5.23	4.31	64.72	58.18
Lolan	5.00	14.60	7.83	0.93	76.7	68.95
Hais	5.80	13.00	6.64	3.94	67.39	60.58
Minanga	4.50	20.00	7.36	3.88	60.33	54.24

Figure 4 shows the relationship between SIRM and the selected major oxide. In contrast to TiO_2 and Al_2O_3 , FeO^* and SiO_2 have a strong correlation with SIRM. SiO_2 is negatively correlated with SIRM, while FeO^* as a representation of magnetic minerals is positively correlated with SIRM. This indicates that SIRM can be a good indicator in determining the relative difference in the concentration of magnetic minerals in the sample, where the higher the SIRM value, the higher the concentration of magnetic minerals.

Figure 5 shows the degree of crystallinity and crystal size of the iron sand samples based on XRD observations. The sample from Hais had the highest degree of crystallinity (77.02%), followed by Minanga (70.67%), then Lolan

(66.87%), and the lowest was from Lalow (62.68%). Crystalline analysis using the Debye-Scherer equation demonstrated that the average crystal size varied from 12.38 nm to 18.60 nm. Samples from Hais had an average crystal size of 18.60 nm, while those for Minanga, Lolan, and Lalow were 14.55 nm, 13.89 nm, and 12.38 nm, respectively. Samples from Lolan and Hais have a relatively wider spectrum of crystal size, while the crystal size spectrum of samples from Minanga is relatively narrow which indicates that the distribution of crystal sizes tends to be homogeneous.

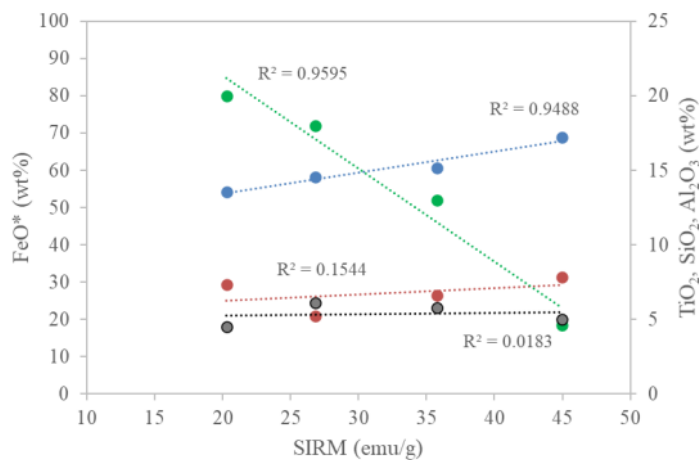


FIGURE 4. The relationship between SIRM and concentration of several major oxides in iron sand samples, namely FeO* (blue circle), SiO₂ (green circle), TiO₂ (red circle), dan Al₂O₃ (dark grey circle).

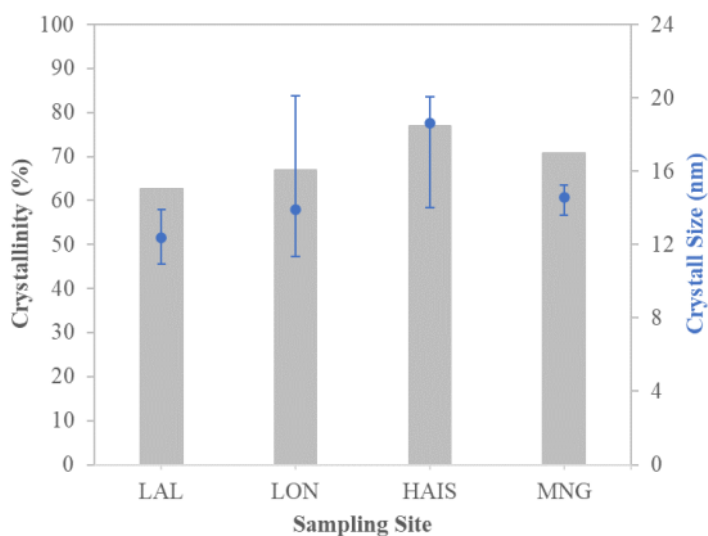


FIGURE 5. The degree of crystallinity and crystal size in iron sand samples.

CONCLUSION

Magnetic parameters, especially SIRM, are very prospective in the study of the potential of iron sands. Based on SIRM data analysis, magnetic minerals in iron sands from four different localities in North Sulawesi show varying levels of concentration. The highest concentration was found in the iron sand sample from Lolan and was followed by samples from Hais, Lalow, and Minanga, respectively. Microcrystalline characterization also shows the various level of crystallinity and crystal size. The relatively high degree of crystallinity indicates a high amount of iron oxide crystallization compared to volcanic glass. Comparatively, the iron oxide content in the study sites is very competitive with other locations in Indonesia and has promising potential for future development. Further study about subsurface geophysical exploration to estimate iron sand thickness is needed in order to get a comprehensive overview of the iron sand potential in that study area.

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