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### Grain size distribution, morphology, and elemental composition of iron sand from North Sulawesi

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Abstract. The coast of North Sulawesi has very potential natural resources. Coastal sand with relatively high iron content or commonly known as iron sand was detected in several locations. Iron sand is a source of economical minerals, namely iron (Fe), which is easier to treat compared to sources in stone form. However, scientific studies on iron sand minerals, especially those in the coastal areas of North Sulawesi, are still poor. Therefore, a study is needed to find out how the mineralogy, morphology and characteristics of iron sand particles in North Sulawesi. The results show that distribution of sand deposit range fine to coarse in size. However, fine-sized grains were dominant in samples from Lalow, Lolan, as well as Hais regions, whereas medium-sized grains were dominant in sample from Minanga. It probably indicates that Minanga area is relatively close to rock as a source of sand formation than other sampling location. In general, SEM observations show grains with relatively rounded in their morphology as well as have a smooth surface which is indicating a long range transport of these grains. EDX results show that the major elements in representative sand grains are Fe, Ti, and O, while the minor elements are Si, Al, Mg, Na, Co, Cl, and Ca. Fe content range from 45.18 to 61.48 wt%, whereas Ti content range from 4.35 wt% to 7.1 wt%.

#### 1. Introduction

Iron sand is one of the potential mineral resources. Various industries such as steel industry, concrete [1], as well as permanent magnets [2], ink powder and coloring agent in a number of applications [3] utilize raw materials from iron sand. Some important minerals contained in iron sand deposits are magnetite (Fe<sub>3</sub>O<sub>4</sub>), hematite (α-Fe<sub>2</sub>O<sub>3</sub>) [4], and titanium [5]. These minerals have different concentrations at each location depending on the source rock or geological conditions of an area.

North Sulawesi is an area with geological conditions that are dominated by volcanic processes [6]. Weathering of high Fe-containing rocks is then carried away by the river flow and deposited in the lower regions, including on the coast [7]. Several coastal areas in North Sulawesi were identified as containing iron sand deposits. These areas are spread from the east and west coasts of the Minahasa area as well as several areas in the Bolaang Mongondow region. However, information about the potential and characteristics of iron sand in these areas is still poor.

The characteristics of iron sand can be studied using physical or chemical methods [8]. One of these methods is a microstructure scanning techniq using Scanning Electron Microscope (SEM). Several previous studies have utilized SEM which is equipped with energy dispersive X-ray (EDX) to analyze the morphology and mineralogy of materials due to various needs. Morphology and behavior of elemental composition can differ in different grain size classifications. Therefore, to enrich

information about the characteristics of iron sand deposits in North Sulawesi, it is recessary to carry out granulometric studies as well as SEM-EDX observations for analysis the morphology and elemental composition of iron sand from several coastal areas in North Sulawesi.

#### 2. Material and Methods

Sample in this study was iron sand obtained from four different locations on the coast of North Sulawesi. Two sampling sites (Lolan are Lalow beaches) are located on the west coast and two others (Hais and Minanga beaches) are located on the east coast of North Sulawesi province. The coordinates of sampling location can be seen in Table 1. Five samples were taken at different points at each location. The samples are then prepared through the drying process. The 200 mg of sand then sieved using two different sizes American Standard Test Sieve Series (ASTM), screen number 35 and 60. It was done in order to classify the grain size of sand in three categories such as coarse, medium, as well as fine sand. Weighing was done to find out the mass percentage of each grain size. Morphological and elemental composition analysis of iron sand particles then was carried out using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX). SEM-EDX observation was conducted in the Central Laboratory of Malang State University using the Scanning Electron Microscope FEI Inspect S50.

|          | Coordinate |              |  |
|----------|------------|--------------|--|
| Location | Longitude  | Latitude     |  |
| Hais     | 0°57′42″ N | 124°49′49″ E |  |
| Minanga  | 0°58′03″ N | 124°51′06″ E |  |
| Lalow    | 0°54′04″ N | 124°02′26″ E |  |
| Lolan    | 0°57′06″ N | 124°10′03″ E |  |

Table 1. Position of the sampling location

#### 3. Results and Discussion

Grain size distribation is obtained by separating sand grains into three fraction sizes, namely coarse grain fore than 0.5 mm), medium grain (0.25 mm until 0.5 mm), as well as fine grain (less than 0.25 mm). Figure 1 shows the mass fraction of the three classifications aforementioned. Grains in samples from Lalow, Lolan, and Hais are dominated by fine-sized grain fractions, followed by medium grain size and then coarse grain fractions. The weight percent ratio (in the form of fine: medium: coarse) for the sample from Lalow is 58.52%: 36.75%: 4.73%, the sample from Lolan is 84.33%: 8.42%: 7.25%, while the sample from Hais is 66.08%: 27.92%: 6%. In contrast, samples from Minanga were dominated by medium-sized grain fractions followed by fine-grained fractions and then coarse grains with ratio of 21.25%: 74.59%: 4.15% (fine: medium: coarse). Grain size distribution is related to wave energy in carrying and settling sand on the beach [9] as well as transport process [10]. From this, it probably means that Minanga beach is influenced by waves with higher energy than other sampling locations or the Minanga area is relatively close to rock as a source of sand formation.

Figure 2a – 2d shows the morphology of iron sand grains in samples from Lolan, Lalow, Hais, and Minanga. SEM observations show that the morphology of sand grains is irregular shape, rounded, and bitetrahedral. Grains with irregular shapes were commonly formed due to weathering or destruction of rocks, while grain morphology which tended to be rounded formed as an effect of the transportation process. Bitetrahedral grains are lithogenic titanomagnetite or magnetite minerals which are formed due to weathering of rocks. These grains generally contain high Fe and have strong magnetic properties [11]. SEM images also show the roughness of grains surface. There are grains with a rough surface as well as the smooth one. The surface roughness of the grain is estimated to be related to the length of the grains experiencing the process of transport or erosion.

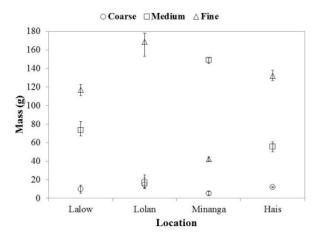


Figure 1. Grain size distribution expressed in mass

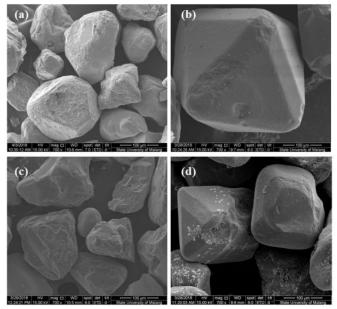


Figure 2. Morphological observations of iron sand by using SEM, (a) grains with relatively rough surface (samples from Lolan), (b) bitetrahedral grains with smooth surface (samples from Minanga), (c) irregularly shaped grains (samples from Hais), (d) bitetrahedral and poorly rounded grains with a smooth surface (sample from Hais)

Elemental observation of sample from the four locations was done by EDX scan in the area. Until energy is around 7 keV, there were seven similar elements detected in the four samples such as O, Na, Mg, Al, Si, Ti, Fe, while elements that were not present in all samples such as Co, Cl, and Ca. Concentration ratio of these elements (expressed in percentage weight) is provided in Table 2. In general the concentrations of titanium (Ti) and iron (Fe) in all samples are more than 4.35 wt% and 45 wt% respectively. The highest Ti and Fe concentrations were found in samples from Lolan, namely 7.1 wt% and 61.48 wt%, while the lowest Ti and Fe concentrations were found in Minanga samples, which are 4.35 wt% and 45 wt%. Titanium and iron in natural samples such as sand are commonly compounded to form iron titanium oxide (Fe - Ti - O) in ferri- and canted antiferomagnetic states, namely titanomagnetite in the ulvospinel (Fe $_2$ TiO $_4$ ) – magnetite (Fe $_3$ O $_4$ ) series as well as titanohematite in the ilmenite (FeTiO $_3$ ) – hematite (Fe $_2$ O $_3$ ) series [5].

| Elements - |       | Weight % |       |         |  |
|------------|-------|----------|-------|---------|--|
|            | Lolan | Lalow    | Hais  | Minanga |  |
| О          | 21.52 | 26.58    | 20.81 | 25.38   |  |
| Na         | 1.23  | 3.61     | 5.83  | 3.25    |  |
| Mg         | 1.80  | 3.13     | 2.76  | 4.03    |  |
| Al         | 3.24  | 4.58     | 4.09  | 5.97    |  |
| Si         | 3.24  | 5.39     | 5.07  | 7.54    |  |
| Ti         | 7.10  | 5.20     | 5.72  | 4.35    |  |
| Fe         | 61.48 | 49.04    | 51.58 | 45.18   |  |
| Others     | 0.38  | 2.46     | 4.14  | 4.30    |  |

Table 2. Elemental Comparison of samples from four locations

Figure 3 provides the results of SEM and EDX in a single grain. Observation of EDX in the whole samples shows varying of Ti and Fe content. The highest value of Ti and Fe content are 9.99 wt% and 88.27 wt%, respectively. Sampling locations if arranged based on the average Fe content in some representative grains, starting from the highest value, are Lolan, Hais, Lalow, and Minanga. There is a correlation between the size of the grain and the Fe content. In samples with finer grain sizes tend to have a higher Fe content compared to the more coarse ones. Some EDX observations in a single grain also indicate that bitetrahedral grains tend to have higher Fe content compared to another shapes. In general, mineralogy of grains in the shape of bitetrahedral is titanomagnetite derived from the results of erosion or weathering of volcanic rocks.

#### 4. Conclusions

Iron sand deposits on the coast of North Sulawesi have grain size ranges from fine to coarse grain. The sediments on the Lolan, Lalow, and Hais beaches are dominated by sand grains on fine grades, while Minanga is dominated by sand with medium grade. SEM observations show that the morphology of sand grains is varied, but generally shaped irregularly, rounded and bitetrahedral. Based on the EDX scan area, samples from the Lolan beach have relatively higher Fe and Ti content than other samples. Fe content reaches 61.48 wt% while the Ti content is around 7.1 wt%. Sequentially, sches with Fe content ranging from the highest are Lolan, Hais, Lalow, and Minanga. Analysis of the relationship between grain size and Fe content in the sample showed a good correlation, in this case the smaller the grain the higher the Fe content.

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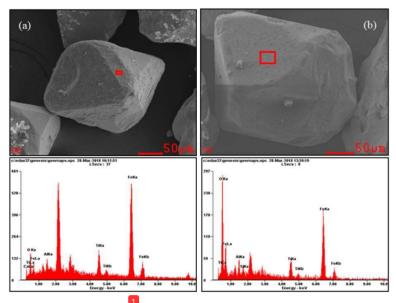


Figure 3. (a) The bitetrahedral grain was observed by SEM in fine-sized samples from Hais beach. EDX result shows that the grain has a high Fe content (Fe = 88.27 wt% and Ti = 7.03 wt%), (b) Rounded grain in sample from Lalow containing Fe = 68.3 wt% and Ti = 6.52 wt%. Note: red square is EDX spot.

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