Analysis of magnetic minerals of iron sand deposit in Sampulungan Beach, Takalar Regency, South Sulawesi using the x-ray diffraction method

M Arsyad¹, V A Tiwow¹, and M J Rampe²

¹Department of Physics, Universitas Negeri Makassar, Kampus UNM Parangtambung Jl. Daeng Tata Raya, Makassar 90224, Indonesia
²Department of Chemistry, Universitas Negeri Manado, Kampus UNIMA di Tondano 95618, Indonesia

E-mail: m_arsyad288@unm.ac.id

Abstract. Takalar Regency has iron sand deposits along the coast. One of them is Sampulungan Beach, North Galesong District. In this study, an analysis of magnetic minerals of iron sand in Sampulungan Beach was carried out. The method used is X-Ray Diffraction (XRD). Measurements were made using the Rigaku MiniFlex II type XRD engine. An analysis of sample diffractogram using PDXL 2 software. The qualitative analysis using search and match techniques and adjusted to the 2011 version of the ICCD card. The quantitative analysis using the RIR method. The results showed that the dominant minerals contained in the iron sand of Sampulungan Beach is magnetite (Fe₃O₄).

1. Introduction

Today, iron sand is one of the sources of magnetic minerals that are useful in industry. Iron sand can be easily found in coastal areas. Iron sand was formed due to weathering of rocks in the mountains, then undergoes transportation by river flows and sea waves so that sedimentation occurs in more gentle areas. Of course, this can happen because Indonesia is an area that was passed by the Pacific and the Mediterranean Circums, so it has many active volcanoes.

Iron sand deposit, one of which was located in Sampulungan Beach, Takalar Regency, South Sulawesi Province. According to geological conditions, Sampulungan Beach was dominated by alluvium deposits and located between two Jeneberang tributaries. The source of sedimentation in Sampulungan Beach comes from Baturape-Cindako volcanic rock formations and Lompobattang volcanic rock formations which are volcanic rocks that are rich in magnetite [1]. This iron sand deposit was marked by black sand on the coast of Takalar Regency, especially in Sampulungan Beach. The black iron sand contains elements of iron (Fe) and magnetic minerals which are the determinants of the potential and quality of iron sand.

Iron sand generally contains magnetic minerals such as magnetite (Fe₃O₄), hematite (α-Fe₂O₃), maghemite (γ-Fe₂O₃), and ilmenite (FeTiO₃) [2-5]. But in nature, iron sand was obtained in an impure state, there are impurity elements such as titanium (Ti), silicon (Si), calcium (Ca), magnesium (Mg), aluminum (Al), vanadium (V) and so on. Magnetic minerals can be extracted or purified by magnetic separation method (rod magnet) and chemical separation [6-7].
The method used to investigate the mineral content of iron sand is X-Ray Diffraction (XRD). Various studies have been carried out using the XRD method. Iron sand from Ambal Beach in Kebumen Regency contains minerals magnetite ($\text{Fe}_3\text{O}_4$), hematite ($\text{Fe}_2\text{O}_3$), magnetite iron magnesium chromium oxide ($\text{Fe, Mg(Fe, Cr)}_2\text{O}_4$) [8]. Mineral sand iron content in Tanjung Bayang Beach, Makassar City obtained magnetite and iron-silicon oxide ($\text{Fe}_{5.36}\text{Si}_{0.64}\text{O}_8$), while iron sand in Bonto Kanang Village, Takalar Regency was dominated by magnetite minerals [9-10]. In this study, it was reported the potential of magnetic minerals and the types of magnetic minerals contained in Sampulungan Beach, Takalar District using the XRD method.

2. Experimental

2.1 Materials

Iron sand was taken at 5 different points randomly at Sampulungan Beach, Takalar District with a depth of 1 meter. At each point, iron sand was taken of 500 g. Taking iron sand using a drill tool.

2.2 Sample preparation

Iron sand was cleaned from dirt and washed using water. Then dried at room temperature for 1 week. Furthermore, iron sand was weighed as much as 100 g and extracted using a rod magnet 40 times to separate sand containing magnetic and non-magnetic minerals [11-12]. The bar magnet was coated with plastic. The extracted iron sand was sieved using a 200 mesh sieve to homogenize the size of the sand grains. Sieved iron sand was called a sample that is ready to be tested for mineral content.

2.3 Calculation of Magnetic Mineral Percentage

Iron sand that was attracted by the bar magnet or not then weighed. Next, the percentage of magnetic minerals for each sample was calculated using the following equation:

$$\% \text{ magnetic minerals} = \frac{\text{mass of magnetic minerals}}{\text{total mass}} \times 100\% \quad (1)$$

2.4 Magnetic Mineral Type Analysis

Samples of extracted iron sand, crushed for 15 minutes, then placed on the holder and polishing. Furthermore, the samples were characterized using the X-Ray Diffraction type Rigaku MiniFlex II. This tool works at a voltage of 30 kV, a current of 15 mA, a scanning width of 0.02°, a scanning rate per time of 4°/minute and a scanning range of 10°-70°. Qualitative analysis was carried out with search and match techniques by utilizing the default machine software, PDXL2, which was equipped with a 2011 version of the ICDD (International Center for Diffraction Data) card. Quantitative analysis (wt.%) was tested using the RIR (Reference Intensity Ratio) method [13-14].

3. Results and Discussion

Magnetic mineral separation on iron sand samples from Sampulungan Beach by taking 5 samples. Separation was done by using a rod magnet that has been coated with plastic 40 times so that there are magnetic and non-magnetic minerals. Small fine sand and shiny black color attached to the rod magnet were called magnetic minerals. After the separation results were obtained, magnetic minerals were weighed to determine the mass of the magnetic minerals. Calculation of the percentage of magnetic mineral content using equation (1) is comparing the mass of magnetic minerals to total mass.

The results of the calculation of the percentage of magnetic mineral content per 100 g of samples from 5 samples were shown in table 1. The percentage of magnetic minerals of each sample were MA1 samples of 1.89 %, MA2 samples of 6.23 %, MA3 samples of 4.58 %, samples MA4 of 2.38 %, and the MA5 sample of 2.37 %. Based on these results it appears that the MA2 and MA3 samples have a high percentage of magnetic minerals compared to the other 3 samples. This result shows that MA2 and MA3 samples have more iron element content of Fe. In addition, it was also supported by MA2 and MA3 sampling points in the tidal zones, where there was a separation of heavy minerals and clay minerals. Heavy minerals were deposited while minerals such as clay are carried by seawater leaving the coast. This result is in accordance with what was revealed by [4] that there is a sand
washing process on the coastline so that clay minerals were suspended while sediments that have heavy mineral contents remain deposited.

Table 1. Percentage of magnetic minerals.

| Sample code | Mass of non-magnetic minerals (g) | Mass of magnetic minerals (g) | Percentage of magnetic minerals (%) |
|-------------|----------------------------------|------------------------------|-------------------------------------|
| MA1         | 98.12                            | 1.89                         | 1.89                                |
| MA2         | 93.78                            | 6.23                         | 6.23                                |
| MA3         | 95.42                            | 4.58                         | 4.58                                |
| MA4         | 97.62                            | 2.38                         | 2.38                                |
| MA5         | 97.63                            | 2.37                         | 2.37                                |

Furthermore, the types of magnetic minerals contained in Sampulungan Beach iron sand samples were measured using an XRD machine. Measurement with XRD was done by powder method. Measurement results were analyzed using PDXL2 software with search and match techniques. The results of the qualitative analysis were shown in figure 1, figure 2, figure 3, figure 4, and figure 5.

**Figure 1.** X-ray diffraction pattern of MA1 sample, M = magnetite (Fe₃O₄), R = rutile (TiO₂), and V = vanadium (IV) oxide (VO₂).
Figure 2. X-ray diffraction pattern of MA2 sample, M = magnetite (Fe$_3$O$_4$), R = rutile (TiO$_2$), V = vanadium (IV) oxide (VO$_2$), and MF = magnesioferrite [(MgFe$_2$)O$_4$].

Figure 3. X-ray diffraction pattern of MA3 sample, M = magnetite (Fe$_3$O$_4$), R = rutile (TiO$_2$), C = chromium oxide (CrO), and V = vanadium oxide (V$_2$O$_3$).
Figure 4. X-ray diffraction pattern of MA4 sample, M = magnetite (Fe$_3$O$_4$), C = chromium oxide (CrO), V = vanadium (IV) oxide (VO$_2$), and IT = iron titanium oxide [Fe(TiO$_3$)].

Figure 5. X-ray diffraction pattern of MA5 sample, M = magnetite (Fe$_3$O$_4$), R = rutile (TiO$_2$), and V = vanadium (IV) oxide (VO$_2$).
Table 2. Mineral composition of iron sand samples based on XRD analysis.

| Mineral                          | Weight percent (wt.%) |
|----------------------------------|-----------------------|
|                                 | MA1 sample | MA2 sample | MA3 sample | MA4 sample | MA5 sample |
| Magnetite (Fe$_3$O$_4$)          | 78         | 56         | 76         | 81         | 67         |
| Rutile (TiO$_2$)                 | 15         | 22         | 15         | 2          | -          |
| Chromium Oxide (CrO)             | -          | -          | 8          | 2          | -          |
| Vanadium (IV) Oxide (VO$_2$)     | 6          | 18         | -          | 8          | 5          |
| Vanadium Oxide (V$_2$O$_3$)      | -          | -          | 1          | -          | -          |
| Iron Tiranium Oxide [Fe(TiO$_3$)]| -          | -          | -          | 9          | -          |
| Magnesioferrite [(MgFe$_2$)O$_4$] | -          | 3          | -          | -          | -          |

Figure 1 shows that the minerals contained in MA1 samples are magnetite, rutile, and vanadium (IV) oxide. Figure 2 shows MA2 samples containing minerals magnetite, rutile, vanadium (IV) oxide, and magnesioferrite. The minerals contained in the MA3 sample are magnetite, chromium oxide, vanadium oxide, and rutile (figure 3). The MA4 sample contained minerals magnetite, chromium oxide, vanadium (IV) oxide, and iron titanium oxide (figure 4). Meanwhile, MA5 samples contain minerals magnetite, rutile, and vanadium (IV) oxide. The results of the analysis showed that the samples containing dominant magnetic minerals are magnetite. The presence of Ti elements in iron sand can reduce the concentration of Fe. These results are in accordance with [15] expression which states that iron sand was dominated by Fe and there is a Ti element as a disturbance which can reduce Fe concentration. Magnetic minerals were included in the titanomagnetic group that is ferrimagnetic, namely the material in which there is a magnetic moment that each atom is not the same so that it has spontaneous magnetization even in the absence of a magnetic field [16].

Quantitatively, the mineral composition was analyzed by the RIR method. The results of the analysis were shown in table 2. In table 2 it appears that the dominant minerals contained in all samples of iron sand are magnetite and rutile. This shows that iron sand samples have a high content of Fe. Thus, these results indicate that the potential of Sampulungan Beach iron sand to be explored further, extracted and used as metal industry materials. In addition, the iron sand sample also contained vanadium oxide. This mineral was not included in the classification of magnetic minerals [16]. However, vanadium oxide comes from algae, shellfish, and crabs [17].

4. Conclusion
Iron sand deposit in Sampulungan Beach contains magnetic minerals which were indicated to contain high iron (Fe) elements and were located in tidal zones. The dominant magnetic minerals are magnetite (Fe$_3$O$_4$) which belong to the titanomagnetic group and ferrimagnetic. In addition, there is also vanadium oxide which was not included in the classification of magnetic minerals and was indicated to come from algae, shellfish, and crabs.

Acknowledgements
We would like thank to the Rector of Universitas Negeri Makassar for funding this research through the Fund of DIPA UNM 2018. We would like to thank Winda Indira B. Tiro, S.Si., and Nurfadilah, S.Si. who have helped in sampling. And we also to thank Nur Akifah, S.Si. an operator of the Microstructure Laboratory Department of Physics FMIPA UNM who has helped in data analysis.

References
[1] Zulkiifli M D, Tholib A, Franklin, A. Sofyan, Sudiaman 2002 *Inventarisasi dan Evaluasi Mineral Logam di Kabupaten Takalar dan Kabupaten Gowa Provinsi Sulawesi Selatan* (Kolokium Direktorat Inventarisasi Sumber Daya Mineral).
[2] Yulianto A, Bijaksana S, Locksmanto W, Sekaran J R, and Pati G 2003 *Indones. J. Phys.* **14** 1–4.
[3] Findorak R, Froehlichova M and Legemza J 2014 *Metalurgija* **53** 9–12.
[4] Nugraha P A, Sari S P, Hidayati W N, Dewi C R, and Kusuma D Y 2016 *Proc. The 2016 Conf. on Fundamental and Applied Science for Advanced Technology (Yogyakarta)* **1746** (New York: AIP Conf. Proc.) p 20028.
[5] Bassez M-P 2017 *Procedia Earth Planet. Sci.* **17** 492–5.
[6] Sunaryono, Taufiq A, Mashuri, Pratapa S, Zainuri M, Triwikantoro and Darminto 2015 *Mater. Sci. Forum* **827** 229–34.
[7] Setiadi E A, Sebayang P, Ginting M, Sari A Y, Kurniawan C, Saragih C S, and Simamora P. 2016 *J. Phys. Conf. Ser.* **776** 12020.
[8] Bilalodin 2010 *Molekul* **5** 105-108.
[9] Tiwow V A, Arsyad M, Palloan P, and Rampe M J 2018 *J. Phys.: Conf. Ser.* 997 012010.
[10] Fahlepy M R, Tiwow V A, and Subaer 2018 *J. Phys.: Conf. Ser.* 997 012036.
[11] Yulianto A, Sulhadi, Azis A L I and Dayati E 2013 *Malaysian J. Fundam. Appl.* **9** 211–215
[12] Rahmawati R, Melati A, Taufiq A, Sunaryono, Diantoro M, Yuliarto B, Suyatman S, Nugraha N, and Kurniadi D 2017 *IOP Conf. Ser. Mater. Sci. Eng.* **202** 12013.
[13] Pratapa S 2004 *Bahan Kuliah Difraksi Sinar-X* (Surabaya: Jurusan Fisika FMIPA ITS).
[14] Subaer, Nurhayati, Nurhasmi, dan Nurfadillah 2014 *Indones. J. of Appl. Phys.* **4** 134-141.
[15] Jahidin 2012 *Jurnal Aplikasi Fisika* **8** 20-24.
[16] Dearing J 1999 *Environmental Magnetik Susceptibility: Using the Bartington MS2 System.* (British Library Cataloguing in Publication Data).
[17] Baranova V N and Fortunatov A V 2012 *Vanadium: Chemical Properties, Uses, and Environmental Effects* (Nova Science Publisher, Inc, Book).