

Preparation and Characterization of Fe₂O₃ from Iron Sand of the Coastal Sea of Cidaun Beach-South Cianjur (Indonesia) using the Co-precipitation Method

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Abstract

The synthesis and characterization of Fe₂O₃ from Iron Sand of the coastal sea of Cidaun Beach - South Cianjur using the coprecipitation method has been carried out. The purpose of this research was to identify the crystal structure, magnetic mineral content, and magnetic properties of iron sands. The characterizations used in this research are X-Ray Diffraction (XRD), Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) and Vibrating Sample Magnetometer (VSM). Based on the XRD results, it shows that the iron sand sample produces multi-phases, namely hematite (α -Fe₂O₃), maghemite (γ -Fe₂O₃) and ilmenite (FeTiO₃). SEM-EDX results indicate that the iron sand samples have an average particle size of 12.396 μ m. VSM results indicate that the iron sand sample is soft magnetic (ferrimagnetic) with saturation, remanence and coercivity values of 47 emu/g, 1.53 emu/g and 102.97 Oe, respectively.

Keywords

Iron Sand, Maghemite, Hematite, Ilmenite, Milling

Received: 27 April 2023, Accepted: 1 August 2023

<https://doi.org/10.26554/sti.2023.8.4.594-598>

1. INTRODUCTION

In Indonesia, particularly along the southern coast of Java, iron sand resources of up to 160 million tons have been published in 2008 (Rianna et al., 2018; Togibasa et al., 2018; Togibasa et al., 2019). Iron sand is a magnetic material that has the properties of a ferromagnetic material (Gunanto et al., 2018; Karbeka et al., 2021; Yulia and Erwin, 2019). Iron sand has several mineral contents such as hematite (α -Fe₂O₃), maghemite (γ -Fe₂O₃), ilmenite (FeTiO₃), silica (SiO₂), magnetite (Fe₃O₄), alumina (Al₂O₃) and rutile (TiO₂) (Nengsi et al., 2016; Sihombing and Amiruddin, 2020; Susilawati et al., 2019). In addition, many unnecessary minerals (gangue) such as K, C, Mg, Na, Si, Al and Ca elements attached to iron ore can reduce the magnetic properties of iron sand. Gangue minerals (non-magnetic) can be reduced by washing the iron sand with acid or base solutions (Karbeka et al., 2020).

The iron ore (Fe) content of iron sands in each region varies, such as in West Sumatra, which has a high iron ore content of 62%, and in Trenggalek Regency (East Java), which has a total iron ore content of 22.28-51.26%. The utilization of iron sands is determined by the high and low Fe content in the sand, with low Fe contents (57-70%) being used for the production of cement raw materials and higher Fe contents (>70%) being

used for steel production (Fatmaliana et al., 2020).

In general, natural iron sands have a ferrite content of 58.39 - 60.23% in the form of hematite (α -Fe₂O₃) and maghemite (γ -Fe₂O₃). Fe₂O₃ is a dark red mineral that is easily contaminated by acids. The crystal structure of Fe₂O₃ is polymorphic, where some of the phases refer to the octahedral coordination geometry form, namely there are six oxygen ligand bonds in the Fe center (Muhajir et al., 2020). The phases in α -Fe₂O₃ and γ -Fe₂O₃ are different, namely the crystal structures in hematite and maghemite are cubic and tetragonal, respectively (Nurayni and Nofitasari, 2018; Saragi et al., 2012; Shokrollahi, 2017). The formation of the crystal structure of iron sand during the preparation process is influenced by the calcination or sintering temperature used, as at temperatures below 100°C, the phase formed is Fe₃O₄ with a cubic spinel crystal structure. At temperatures above 100°C to 600°C, a phase from magnetite to γ -Fe₂O₃ can be formed, and at temperatures of 1000-1100°C the phase changes to α -Fe₂O₃ (Kazeminezhad and Mosivand, 2014).

The use of Fe₂O₃ material has been widely used in various applications, such as cancer cell targeting drugs, photocatalysts, waste water treatment and heavy metal removal. This is because Fe₂O₃ has the advantages of low cost, good magnetic properties,

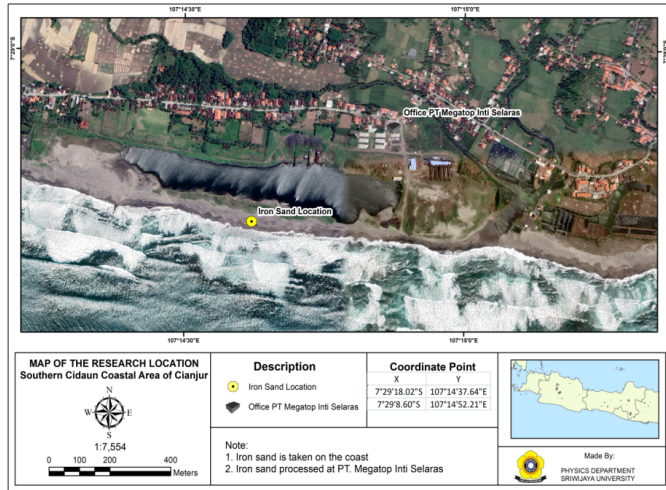


Figure 1. Map of the Iron Sand Sources of the Coastal Sea of Cidaun Beach

easy surface modification, high density, high stability, high porosity and high surface area (Fouda et al., 2021; Mishra and Chun, 2015; Widodo et al., 2020). The preparation of Fe_2O_3 can be done by several methods, namely hydrothermal, sol-gel, co-precipitation, green synthesis and precipitation (Ahmad et al., 2023; Babay et al., 2015; Lassoued et al., 2017; Liu et al., 1997; Tadic et al., 2014).

Currently, the preparation of Fe_2O_3 from natural iron sand has been widely carried out as shown in Table 1. Table 1 shows that many researchers have carried out the preparation of Fe_2O_3 from natural iron sands of various areas. However, the preparation of Fe_2O_3 from the iron sand of the coastal sea of Cidaun Beach-South Cianjur (Indonesia) has never been done and the information related to the Fe content is still very limited. Based on this, this research will be conducted on the preparation of Fe_2O_3 from marine iron sand on the coast of Cidaun-South Cianjur (Indonesia) using the co-precipitation method.

2. EXPERIMENTAL SECTION

2.1 Material

The materials used in this research are commercial iron sand from the coastal sea of Cidaun beach - South Cianjur (Indonesia) produced by PT Megatop Inti Selaras and map location of this iron sand is shown in Figure 1, 37% HCl (Merck), and 25% NH_4OH (Technical).

2.2 Preparation of Iron Sand

Commercial iron sand was first characterized by SEM-EDX to determine the composition of the iron sand. Iron sand is sieved 200 mesh to obtain a particle size close to nano, then iron sand is milled using High Energy Milling (HEM) with a composition ratio between iron sand and ball mill (1:10) wt% for 10 hours. Results of iron sand milling and then preparation using the co-

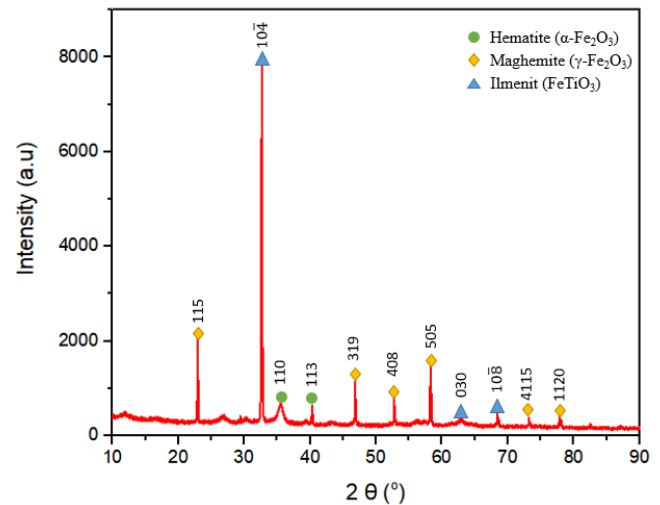


Figure 2. Diffraction Peak Pattern of Iron Sand Sample After Preparation

precipitation method. A total of 25 g of iron sand was slowly added to 150 mL of 37% HCl with stirring (350 rpm) at a temperature of 100°C within 1 hour. Adjust pH of solution to 6 by slow addition of 30% NH_4OH (per drop). Precipitate the solution overnight and wash with distilled water until the filtrate becomes clear, then dry the precipitate at 100°C for 19 hours. The precipitate is then calcined at 400°C for 1 hour and the sample is characterized (Rianna et al., 2018).

2.3 Characterization

The instruments used for the characterization of materials are X-Ray Diffraction (XRD; PANalytical AERIS), Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX; PhenomProX Desktop) and Vibrating Sample Magnetometer (VSM; VSM250). XRD is used to determine the phase and crystal structure formed in iron sand samples after preparation. SEM-EDX aims to determine the morphological state and particle size distribution as well as the content or composition of elements contained in the sample in iron sand, in this analysis there are two samples namely "iron sand before preparation" and "iron sand after preparation".

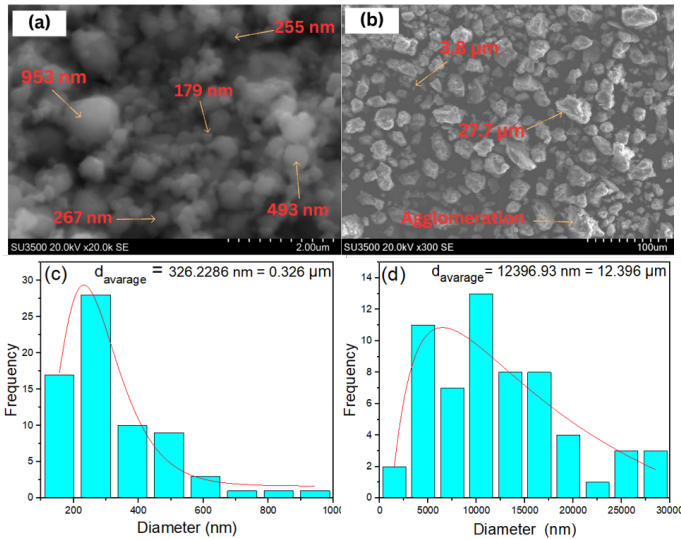
3. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction (XRD)

The phase and crystal structure formation in iron sand samples can be shown in Figure 2. Based on Figure 2, it shows that the phases formed in the sample are Hematite ($\alpha\text{-Fe}_2\text{O}_3$), Maghemite ($\gamma\text{-Fe}_2\text{O}_3$) and Ilmenite (FeTiO_3). The 2θ angle of 32.647°, which is the FeTiO_3 phase, has the highest intensity peak. The FeTiO_3 phase forms a hexagonal crystal structure. The appearance of the FeTiO_3 phase is caused by the presence of TiO_2 elements in the sample so that TiO_2 reacts with FeO to form the FeTiO_3 phase. The $\alpha\text{-Fe}_2\text{O}_3$ phase has fewer diffraction peaks than $\gamma\text{-Fe}_2\text{O}_3$, which has a rhombohedral

Table 1. Preparation of Fe₂O₃ from Various Natural Iron Sand Sources

Material	Source of Iron Sand	References
Fe ₂ O ₃	Lhoong, Aceh	(Fatmaliana et al., 2020)
Fe ₂ O ₃	Paseban Beach, Jember	(Al Hadi et al., 2023)
Fe ₂ O ₃	Sijunjung, West Sumatra	(Soekansa et al., 2023)
Fe ₂ O ₃	Southern Coast of Yogyakarta	(Rusianto et al., 2015)
Fe ₂ O ₃	Sukabumi	(Wahyuningsih et al., 2016)
Fe ₂ O ₃	Banten	(Dewi and Adi, 2018)

**Figure 3.** Morphology and Particle Size Distribution of Commercial Iron Sand (a,c) Before Preparation and (b,d) After Preparation

and tetragonal crystal structure, respectively. The number of γ -Fe₂O₃ phases that appear can be caused by using a calcination temperature of 400°C, where the γ -Fe₂O₃ phase can form from temperatures of 325-400°C (Lepp, 1957).

3.2 Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX)

The morphological structure and particle size distribution of iron sand samples before and after preparation are shown in Figure 3. Figures 3(a) and 3(c) show the morphology at 20,000 times magnification and the particle size distribution of the iron sand sample before preparation. The results show that the sample has smooth spherical particles and there is no agglomeration, and the average diameter of the sample is 326.2266 nm. Its particle size is the result of the impact during the milling process, which has the effect of reducing the particle size. Figures 3(b) and 3(d) also show the morphology with a magnification of 300 times and the particle size distribution of the iron sand sample after preparation. The results show that the particles are of a bulk size and are agglomerated, with an average particle diameter of 12.396 μ m. There are factors that

influence agglomeration, such as instability between particles during the preparation process due to increased surface tension between the grains in the particles. The tension between the surfaces of the particle grains is caused by the small size of the small particles, so the surface area becomes large, resulting in nanoparticle instability. To achieve a stable state, the instability of the particles causes other particles to try to stick together and form bonds. These bonds create agglomerated particle grains (Kiswanto et al., 2021).

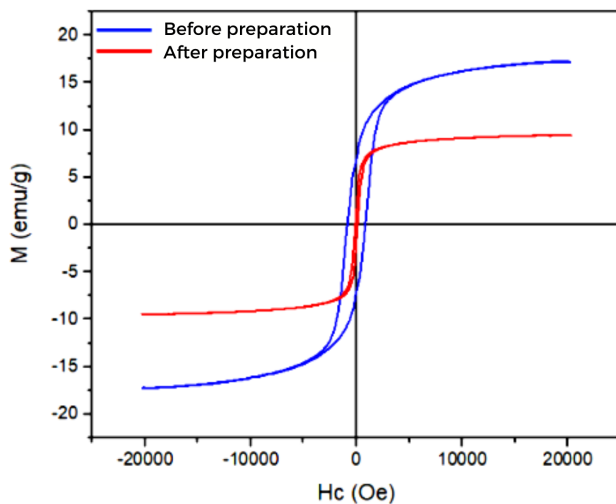
In addition, the EDX results of the iron sand samples before and after preparation are shown in Table 2. Table 2 shows the elemental contents of the iron sand samples before and after precipitation. In the iron sand sample before preparation, it shows the Fe and O content of 41.79% and 42.60%, the remainder is the content of elements in the form of gangue attached to iron ore. It can be said that iron sand has a low percentage of purity (<50%). The low Fe percentage value can be caused by the large size of the iron sand grains. The large size of the grains in iron sand can be caused by the large number of impurities, such as minerals, which envelop the magnetic part of the particles, resulting in a low percentage of dissolution of non-magnetic minerals. In the iron sand sample after preparation, the content of Fe and O decreased by 15.27% and 26.66%, respectively. This decrease may be caused by the increase in the percentage of impurities in the sample, such as the appearance of Cl element and the increase in the composition of C element. The Cl element in the sample is caused by the use of HCl solvents during the preparation process, so a longer wash is required to remove this element, while the C element is the material used during the EDX characterization process.

3.3 Vibrating Sample Magnetometer (VSM)

The magnetic properties of the sample are shown in Figure 4. Figure 4 shows the difference in hysteresis curve area for iron sand samples before and after preparation. In the iron sand sample before preparation, it shows a not too narrow curve with the value of magnetic saturation (Ms), magnetic remanence (Mr) and high coercivity (Hc) of 6.90 emu/g, 17.26 emu/g and 852.13 Oe, respectively. As a result, the sample is classified as semi-hard magnetic as well as ferromagnetic. Materials with a coercivity between 125-5026 Oe or 10-400 kA/m are considered semi-hard magnetic. The iron sand sample after preparation shows a narrow curve with Ms, Mr and Hc values of 9.47 emu/g, 1.53 emu/g and 102.97 Oe, re-

Table 2. Elemental Content of Iron Sand Sample

Sample	Elemental Weight (wt%)										
	C	O	Mg	Al	Si	Cl	Ti	V	Mn	Fe	Ca
Iron Sand (Before Preparation)	5.85	42.60	1.21	1.31	0.48	-	6.06	0.28	0.30	41.79	0.11
Iron Sand (After Preparation)	41.46	26.66	0.10	0.30	0.14	13.97	2.00	-	0.10	15.27	-

**Figure 4.** Hysteresis Curves of Iron Sand Samples Before and After Preparation

spectively. As a result, the sample is classified as soft magnetic and ferrimagnetic. The decrease of the coercive field strength in the sample from 852.13 to 102.97 Oe can be caused by using the calcination temperature of 400°C for 1 hour. The use of temperature in the sample preparation process can have an effect on the magnetic properties of the sample. This is because temperature can disrupt the magnetic domain in a material, causing the spin-spin of the magnetic domain in the same direction to be reduced. The reduction in the spin-spin of the magnetic domain can cause the magnetic properties of the material to decrease so that the ferromagnetic iron sand sample becomes ferrimagnetic (Patimah and Saraswati, 2016).

4. CONCLUSION

The analysis of crystal structure, morphology and magnetic properties of iron sand from the coastal sea of Cidaun-Cianjur has been completed. The prepared iron sand produces multi-phases, namely α -Fe₂O₃, γ -Fe₂O₃ and FeTiO₃, and the average particle size obtained is 12.396 μ m. In addition, the change of coercivity value from 852.13 Oe (semi-hard magnet) to 102.97 Oe (soft magnet) in the sample can be caused by the use of calcination temperature of 400°C.

5. ACKNOWLEDGMENT

The author is grateful to the National Agency for Research and Innovation (BRIN) for the provision of the Talent Research Assistance (BARISTA) scholarship and the Postgraduate Program of the Faculty of Mathematics and Natural Sciences of Sriwijaya University for the opportunity to conduct research at the Research Center for Advanced Materials (PRMM)-BRIN.

REFERENCES

- Ahmad, W., H. C. Joshi, S. Pandey, V. Kumar, and M. Verma (2023). An Overview of Green Methods for Fe₂O₃ Nanoparticle Synthesis and Their Applications. *International Nano Letters*, **13**(2); 117–130
- Al Hadi, F. H., T. Haryati, N. Andarini, and Y. A. Sulistiyo (2023). Hydrothermal Synthesis of Hematite (α -Fe₂O₃) from Indonesia Iron Sand. *Indonesian Chimica Letters*, **2**(1); 10–13
- Babay, S., T. Mhiri, and M. Toumi (2015). Synthesis, Structural and Spectroscopic Characterizations of Maghemite γ -Fe₂O₃ Prepared by One-step Coprecipitation Route. *Journal of Molecular Structure*, **1085**; 286–293
- Dewi, S. H. and W. A. Adi (2018). Synthesis and Characterization of High Purity Fe₃O₄ and α -Fe₂O₃ from Local Iron Sand. *Journal of Physics*, **1091**; 012021
- Fatmaliana, A., M. Maulinda, and N. Sari (2020). Synthesis and Characterization of Hematite (Fe₂O₃) of Iron Ore and Magnetite (Fe₃O₄) from Iron Sand Through Precipitation Method for Industrial Raw Materials. *Jurnal Neutrino: Jurnal Fisika dan Aplikasinya*, **12**(2); 37–42
- Fouda, A., S. E. D. Hassan, E. Saied, and M. S. Azab (2021). An Eco-friendly Approach to Textile and Tannery Wastewater Treatment Using Maghemite Nanoparticles (γ -Fe₂O₃-NPs) Fabricated by Penicillium Expansum Strain (Kw). *Journal of Environmental Chemical Engineering*, **9**(1); 104693
- Gunanto, Y., M. Izaak, E. Jobiliong, L. Cahyadi, and W. Adi (2018). High purity Fe₃O₄ from local iron sand extraction. **1011**(1); 012005
- Karbeka, M., F. V. Koly, and N. M. Tellu (2021). Characterization of Magnetic Content From Puntaru Beach Iron Sand. *Conference Proceedings*, **2349**(1)
- Karbeka, M., F. V. L. Koly, and N. M. Tellu (2020). Karakterisasi Sifat Kemagnetan Pasir Besi Pantai Puntaru Kabupaten Alor-NTT. *Lantanida Journal*, **8**(2); 108–116
- Kazeminezhad, I. and S. Mosivand (2014). Phase Transition

- of Electrooxidized Fe_3O_4 to γ and α - Fe_2O_3 Nanoparticles Using Sintering Treatment. *Acta Physica Polonica A*, **125**(5); 1210–1214
- Kiswanto, H., A. H. P. Yuniarto, N. I. Istiqomah, and E. Suharyadi (2021). Struktur Kristal dan Sifat Kemagnetan Nanopartikel Mn-Ferrite yang Disintesis dari Bahan Alam Pasir Besi. *Jurnal Fisika Unand*, **10**(4); 413–420 (In Indonesia)
- Lassoued, A., B. Dkhil, A. Gadri, and S. Ammar (2017). Control of the Shape and Size of Iron Oxide (α - Fe_2O_3) Nanoparticles Synthesized Through the Chemical Precipitation Method. *Results in Physics*, **7**; 3007–3015
- Lepp, H. (1957). *Stages in the oxidation of magnetite*. Mineralogical Society of America
- Liu, X., S. Tao, and Y. Shen (1997). Preparation and Characterization of Nanocrystalline α - Fe_2O_3 by a Sol-gel Process. *Sensors and Actuators B: Chemical*, **40**(2-3); 161–165
- Mishra, M. and D. M. Chun (2015). α - Fe_2O_3 as a Photocatalytic Material: A Review. *Applied Catalysis A: General*, **498**; 126–141
- Muhajir, M., P. Puspitasari, and J. A. Razak (2020). Synthesis and Applications of Hematite α - Fe_2O_3 : A Review. *Journal of Mechanical Engineering Science and Technology (JMEST)*, **3**(2); 51–58
- Nengsi, S. W., A. Budiman, and D. Puryanti (2016). Karakterisasi Struktur Kristal dan Sifat Magnetik Maghemite (γ - Fe_2O_3) yang Dioksidasi dari Magnetit (Fe_3O_4) dari Pasir Besi Batang Sukam Kabupaten Sijunjung Sumatera Barat dengan Variasi Waktu Oksidasi. *Jurnal Fisika Unand*, **5**(3); 248–251 (In Indonesia)
- Nurayni, S. and R. N. Nofitasari (2018). Characterization of Mineral Content of Iron Sand at Depok Beach, Sigandu Beach and Muara Beach in Batang Regency. *Journal of Natural Sciences and Mathematics Research*, **4**(1); 23–26
- Patimah, P. and T. E. Saraswati (2016). Pengaruh Suhu Kalsinasi Pada Sifat Kemagnetan Material Besi Oksida Hasil Elektrolisis. *Jurnal Kimia Dan Pendidikan Kimia*, **1**(3); 149–156 (In Indonesia)
- Rianna, M., T. Sembiring, M. Situmorang, C. Kurniawan, E. A. Setiadi, A. P. Tetuko, S. Simbolon, M. Ginting, and P. Sebayang (2018). Characterization of natural iron sand from Kata Beach, West Sumatra with high energy milling (Hem). *Jurnal Natural*, **18**(2); 97–100
- Rusianto, T., M. W. Wildan, and K. Abraha (2015). Characterizations of Ceramic Magnets from Iron Sand. *International Journal of Technology*, **6**(6); 1017–1024
- Saragi, T., E. Noelik, and D. Gustaman (2012). Pengembangan Bahan Magnetik Barium Heksaferite dari Mineral Yarosit Alam dan Karakterisasinya. *Bionatura*, **14**(2); 156–163
- Shokrollahi, H. (2017). A Review of the Magnetic Properties, Synthesis Methods and Applications of Maghemite. *Journal of Magnetism and Magnetic Materials*, **426**; 74–81
- Sihombing, M. and E. Amiruddin (2020). Sintesis Dan Karakterisasi Nanopartikel Fe_2O_3 Dari Pasir Alam Desa Logas Kabupaten Kuantan Singingi. *Komunikasi Fisika Indonesia*, **17**(2); 68–73 (In Indonesia)
- Soekansa, A. F., N. Sudirman, and S. Aini (2023). Sintesis dan Karakterisasi Pigmen Merah Hematit (α - Fe_2O_3) dari Pasir Besi Kabupaten Sijunjung, Sumatera Barat, Indonesia. *Jurnal Periodic Jurusan Kimia UNP*, **12**(1); 9–13 (In Indonesia)
- Susilawati, S., A. Doyan, M. Taufik, W. Wahyudi, E. R. Gunawan, A. Fitriani, and N. Nazarudin (2019). Characterization of Barium M-Hexaferrite with Doping Zn and Mn for Microwaves Absorbent. *Materials Science Forum*, **966**; 282–289
- Tadic, M., M. Panjan, V. Damjanovic, and I. Milosevic (2014). Magnetic Properties of Hematite (α - Fe_2O_3) Nanoparticles Prepared by Hydrothermal Synthesis Method. *Applied Surface Science*, **320**; 183–187
- Togibasa, O., M. Akbar, A. Pratama, and S. Bijaksana (2019). Distribution of Magnetic Susceptibility of Natural Iron Sand in the Sarmi Coast Area. *Journal of Physics: Conference Series*, **1204**(1); 012074
- Togibasa, O., S. Bijaksana, and G. C. Novala (2018). Magnetic properties of iron sand from the Tor River Estuary, Sarmi, Papua. *Geosciences*, **8**(4); 113
- Wahyuningsih, S., A. Ramelan, H. Pranata, Q. Hanif, Y. Ismoyo, and K. Ichsan (2016). Preparation of Fe_2O_3 - TiO_2 Composite from Sukabumi Iron Sand Through Magnetic Separation, Pyrometallurgy, and Hydrometallurgy. *Journal of Physics*, **776**(1); 012026
- Widodo, R., S. Anis, A. Ichwani, B. Setiawan, D. Fitriyana, and L. Rochman (2020). Synthesis and Characterization of Iron (III) Oxide from Natural Iron Sand of the South Coastal Area, Purworejo Central Java. *Journal of Physics*, **1444**(1); 012043
- Yulia, W. and E. Erwin (2019). Analisis Sifat Magnetik dan Struktur Partikel Pasir Besi Pantai Arta Pariaman Sumatera Barat Menggunakan Ball Milling. *Komunikasi Fisika Indonesia*, **16**(2); 107–112 (In Indonesia)