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# Study of Magnetite Crystal Structure Extracted from Local Sands of Tegal Lenga Beach

**P. Suardana[1], M. Sumadiyasa[1], I Gede Hendrawan[2]**

[1]Physics Study Program, Faculty of Mathematics and Natural Sciences, Udayana University, Denpasar, Indonesia
[2]Marine Science Undergraduate Study Program, Faculty of Marine Affairs and Fisheries, Udayana University, Denpasar, Indonesia
Email: suardanaputu@unud.ac.id, sumadiyasa@unud.ac.id, gede.hendrawan@unud.ac.id

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## Abstract

Magnetite ($Fe_3O_4$) crystals have been synthesized from the natural iron sands of Tegal Lenga Beach with HCl (37%) as a solvent. Tegal Lenga Beach is a stretch of beach located in Kalisada Village, Seririt District, Buleleng Regency, Bali Province, Indonesia. Iron sand samples were obtained by extracting local natural sand from Tegal Lenga Beach by magnetic separation method. The iron sand sample was ground with agate mortar for 15 hours, then washed and rinsed with deionized water (DI water). Furthermore, the fine powder sample of iron sand was dried on a hotplate at a temperature of 80˚C until all the water had evaporated. Eighty grams of fine iron sand powder dissolved in 200 mL HCl (37%) while stirring with a hot plate magnetic stirrer at a temperature of 100˚C with a rotating speed of 600 rpm for 30 minutes. This solution was filtered through Wattman filter paper No. 42; then the filtrate was dried using a hot plate at a temperature of 100˚C to form a crust. This crust is then ground in an agate mortar until it becomes a fine powder of iron sand. Furthermore, the iron sand fine powder filtrate sample was divided into four equal parts which were then calcined at temperatures of 300˚C, 400˚C, 450˚C, and 500˚C for 30 minutes, respectively. It was found that the fine powder of iron sand from the natural sand of the Tegal Lenga Beach consisted mostly of 84.26% metal oxide magnetite. The synthesized magnetite crystal has a tetrahedral structure with lattice parameters of $a = b = (5.927 \pm 0.0180)$ Å and $c = (16.774 \pm 0.0145)$ Å. Magnetite crystals have granular and lumpy grains with an average grain size of (94,560 ± 10,1397) μm. In relation to the values obtained from the hysteresis curve, namely the remanent magnetization of 9.6672 emu/gr, saturation magnetization of 45.3491 emu/gr, coercive magnetic field strength of 0.0242 T, and saturation magnetic field strength of 0, 2938 T, then this magnetite crystal is classified as a weak ferromagnetic material.

**Keywords**

Ferromagnetic, Magnetite, Iron Sand, Tegal Lenga Beach

## 1. Introduction

Indonesia is an archipelagic country consisting of 17,504 islands with a coastline of 99,083 km which has the potential to find magnetite ($Fe_3O_4$), hematite ($\alpha$-$Fe_2O_3$), and maghemite ($\gamma$-$Fe_2O_3$), which are the main oxide minerals that make up iron sand. Research on the presence of iron sand with its content and characteristics has been carried out on iron sand on various beaches in the Indonesian archipelago, such as Losari Beach in Makassar, South Sulawesi [1], Ambal Beach in Central Java [2], Logending Beach in Central Java [3], Kulonprogo Beach in Central Java [4], Puger Beach in East Java [5], and Bugel Beach in Central Java [6], but no researchers have investigated the presence of iron sand along the coast of Bali Island whose coastline is about 633.35 km. Tegal Lenga Beach is a stretch of beach located in Kalisada Village, Seririt District, Buleleng Regency, Bali Province, Indonesia which is located close to a hilly area which is thought to have contributed to the presence of iron sand. The astronomical location of this beach is 8˚11'42.6"S 114˚52'35.6"E [7]. Magnetite particles are an interesting material to develop because of their potential in applications in various fields such as color pigments for industrial [8], biomedical material [9] [10] [11], heavy metal absorbing materials [6], anode materials for lithium batteries [12], etc. Based on the description above, a preliminary study has been carried out on the metal oxide content of the iron sand of Tegal Lenga Beach [13], and continued with the study of magnetite crystals extracted from the sand of Tegal Lenga Beach, which is reported in this paper.

## 2. Experiment

In this study, magnetite crystals were extracted from Tegal Lenga beach iron sand with HCl (37%) as a solvent. Iron sand is extracted from the sand of Tegal Lenga Beach by magnetic separation method. It is washed with deionized water (DI water) and further it dried on a hotplate (Hotplate SP 88857105) at 80˚C until all the water evaporates. Eighty grams of iron sand sample was ground with an agate mortar for 15 hours to make a fine iron sand powder. In an effort to produce magnetite crystal compounds, a sample of fine iron sand powder was dissolved in 200 mL of HCl (37%) while stirring with a hotplate magnetic stirrer (Hotplate SP 88857105) at a rotating speed of 600 rpm with temperature of 100˚C for 30 minutes. Furthermore, this suspension of fine iron sand powder and HCl (37%) was filtered using filter paper Watman No. 42. After that, the filtrate is dried using a hot plate at a temperature of 100˚C to form an iron sand crust. This iron sand crust is then ground in an agate mortar until it becomes a fine powder of iron sand sample. In an effort to obtain a metal oxide crystalline phase, this iron sand fine powder sample was divided into four equal parts which were then calcined on a hotplate at temperatures of 300˚C, 400˚C, 450˚C, and 500˚C, respectively, for 30 minutes. The four samples that had been calcined at different temperatures were further characterized by X-ray Fluorescence (XRF) brand: PANalytical, type: Minipal4), X-ray diffraction (XRD) brand: PANalytical, type: XPert PRO, scanning electron microscopy (SEM) brand: JSM-6510LA Analytical Scanning Electron Microscope, and the vibrating sample magnetometer (VSM) brand: VSM 250, each of which aims to determine the type and composition of the content, structure and phase, the physical state of surface granularity, and the magnetic properties of the metal oxides that make up the iron sand fine powder samples.

## 3. Results and Discussions

XRF characterization has been carried out on four samples of iron sand fine powder. The histogram of the metal oxide compounds that make up the iron sand sample of Tegal Lenga Beach is shown in **Figure 1**, it can be seen that the metal oxide compounds consist of 84.26% magnetite, 8.91% Titanium Dioxide ($TiO_2$), 2.75% Alumina ($Al_2O_3$), and other compounds with very small compositions. It also shows that the change in calcination temperature from 300˚C to 500˚C affect the composition of the content of each of its constituent components, even though it is very small. This shows that the chemical composition of the oxide compounds that compose iron sand is very stable at a temperature range of 300˚C to 500˚C. The change in the composition of the magnetite content in the iron sand fine powder sample due to the increase in the calcination temperature from 300˚C to 500˚C is shown in **Figure 2**, it can be seen that the composition of the magnetite content in the iron sand fine powder sample decreases with an increase in the calcination temperature from 300˚C to 500˚C with a decreasing gradient of $3.6 \times 10^{-3}$%/˚C.

XRD characterization has been carried out on the four samples of fine iron sand powder that have been calcined at temperatures of 300˚C, 400˚C, 450˚C, and 500˚C. XRD is set with anode material: Cu, $\lambda$ = 1.54060 Å, generator settings: 35 mA, 40 kV, and measurement temperature: 25˚C. The XRD pattern of the four samples measured at $2\theta$ angles with a range of 10˚ to 80˚ is shown in **Figure 3**. The peaks of the XRD pattern have been identified at an angle of $2\theta$ with values of 18.29˚, 30.11˚, 35.43˚, 37.01˚, 43.03˚, 53.27˚, 56.97˚, 62.53˚, 71.11˚, 74.05˚, and 78.93˚. All the peaks of the XRD pattern shown in **Figure 3** belong to the magnetite crystal phase, which correspond to PDF database No. 75-44. This indicates that the parameters that have been used in this extraction process have formed single-phase magnetite crystals, but the most perfect single-phase magnetite crystals is a single-phase magnetite crystal sample calcined at a temperature of 300˚C, this is indicated by the occurrence of sharp and narrow peaks in the XRD pattern of magnetite crystals for all possible $2\theta$ angle values, which does not occur in other XRD patterns. The lattice parameters and

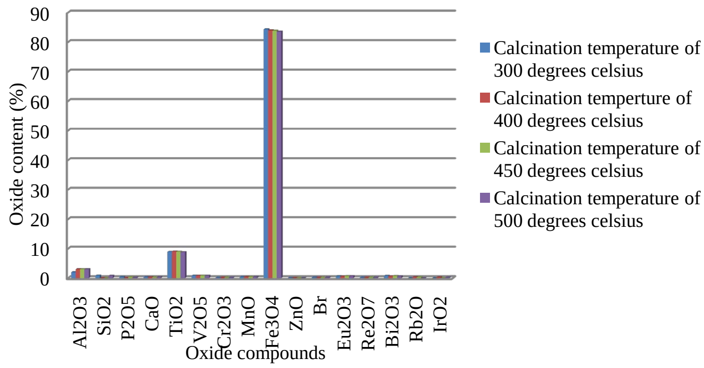


**Figure 1.** Histogram of the oxide content of fine powder sample of iron sand from Tegal Lenga Beach.

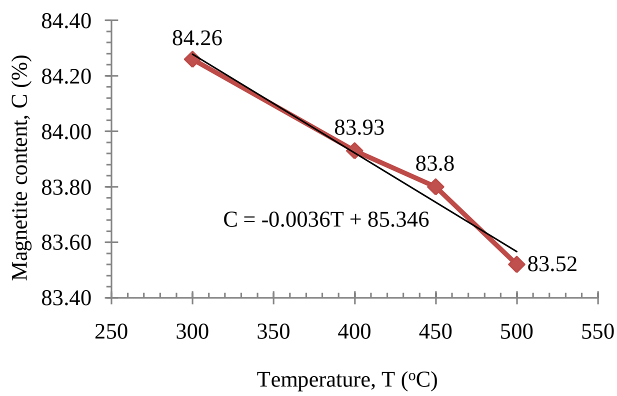


**Figure 2.** Graph of the relationship between Magnetite content (C) and calcination temperature (T).

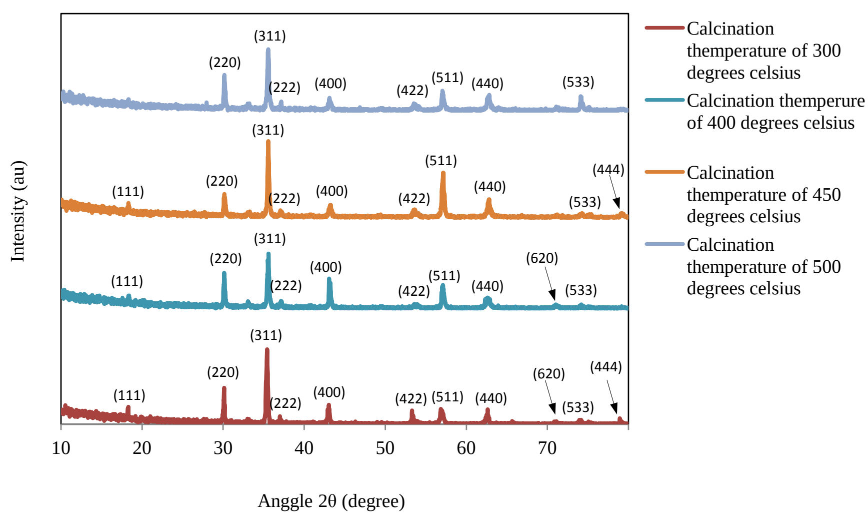


**Figure 3.** Magnetite crystal XRD pattern of iron sand fine powder sample from Tegal Lenga Beach.

figure of merit (FoM) of each magnetite crystal which is the metal oxide constituent of the iron sand fine powder sample are shown in **Table 1**. It can be seen that the goodness of fit (GoF) of each magnetite crystal is less than 4% [14], this indicates that the XRD pattern of the four magnetite crystal samples corresponds to the reference XRD pattern in the PDF database No. 75-44. Table 1 also shows that the crystal structure of magnetite contained in the iron sands of Tegal Lenga Beach is tetrahedral with an average lattice parameter of $a = b = (5.927 \pm 0.0180)$ Å and $c = (16.774 \pm 0.0145)$ Å.

The effect of changes in the calcination temperature of magnetite crystals on the lattice parameters is shown by the graph of the lattice parameters versus the calcination temperature as shown in Figure 4, as the calcining temperature increases from 300˚C to 400˚C the lattice parameter *a* increases by 0.026 Å, on the other hand the lattice parameter *b* decreases by 0.052 Å. As the calcining temperature increases from 400˚C to 500˚C the lattice parameter *a* decrease by 0.025 Å, on the other hand, the lattice parameter *b* increases by 0.008 Å. The increase in the lattice parameter *a* and the decrease in the lattice parameter *b* at the calcination temperature of 400˚C corresponded to a decrease in the number of peaks of the XRD pattern at the calcination temperature of 400˚C compared to the number of peaks of the XRD pattern at the other calcination temperatures. For changes in calcination temperature from 300˚C to 500˚C, the lattice parameter *a* increases by $3 \times 10^{-6}$ Å/˚C, conversely the lattice parameter *b* decreases by $2 \times 10^{-4}$ Å/˚C, while the lattice parameter *c* increases by $6 \times 10^{-5}$ Å/˚C. The offset changes in lattice parameters *a* and *b* and the increase in lattice parameters *c* are thought to be related to the stability of the magnetite crystal structure in the calcination temperature interval of 300˚C to 500˚C.

Table 1. Lattice parameters and figure of merit (FoM) samples of iron sand fine powder.

| No. | Lattice parameter (Å) | | | Figure of Merit (FoM) (%) | | | | Calcination temperature (˚C) |
|---|---|---|---|---|---|---|---|---|
| | *a* | *b* | *c* | Rp | Rwp | Rexp | GoF | |
| 1 | 5.924257 | 5.957791 | 16.753927 | 25.45 | 34.68 | 30.43 | 1.3 | 300 |
| 2 | 5.950260 | 5.905344 | 16.771547 | 26.65 | 35.56 | 30.04 | 1.4 | 400 |
| 3 | 5.933054 | 5.907244 | 16.775064 | 25.58 | 34.99 | 29.56 | 1.4 | 450 |
| 4 | 5.924908 | 5.912825 | 16.794828 | 24.94 | 33.71 | 29.85 | 1.28 | 500 |

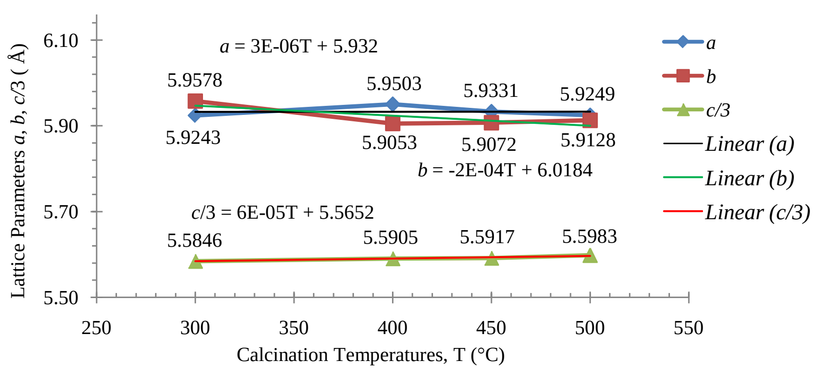


Figure 4. Graph of the relationship between lattice parameters and calcination temperatures.

SEM characterization has been carried out on magnetite crystals of iron sand fine powder samples that have been calcined at 300˚C. Figure 5 shows the SEM image of magnetite crystals of iron sand fine powder samples at a magnification of 100 times, it is seen that some of the magnetite crystal grains are more than 100 μm in size with granular and lumpy grains. This shows that the magnetite of the iron sand fine powder sample produced is already in the crystalline phase, this is in accordance with the XRD pattern shown in Figure 3 where the peaks of the XRD pattern are sharp and narrow. It also appears that all the magnetite crystal grains are almost the same color; this indicates that the magnetite crystals extracted from the iron sand fine powder sample are homogeneous.

As shown in Figure 5, the size of the magnetite crystal grain of the iron sand fine powder sample is not uniform, therefore the size of the crystal grains was measured manually using the Image J software application to the SEM image in Figure 5. The grain size of magnetite crystals is determined by measuring the length and width of each crystal grain twice in mutually perpendicular directions in Figure 5. Furthermore, the grain size data of these crystals is analyzed to obtain the average grain size of magnetite crystals grains. Figure 6 is a histogram of magnetite crystal grain size data for iron sand fine powder samples measured by the Image J software application based on SEM images in Figure 5. It was found that the average grain size of magnetite crystals of iron sand fine powder samples was (94.560 ± 10.1397) μm with R-Square or the coefficient of determination (COD) of 0.82838.

The magnetic properties test has been carried out with the VSM 250 tool on magnetite crystals that make up the iron sand fine powder sample which has been calcined at 300˚C. The magnetic properties test has been carried out at room temperature (300 K) with a magnetic hysteresis curve shown in Figure 7, it can be seen that the remanent magnetization ($M_R$) of magnetite crystals from this iron sand fine powder sample is 9.6672 emu/gr, this value is much smaller than the saturation magnetization ($M_S$) value of 45.3491 emu/gr, as well as the strength of the coercive magnetic field ($H_C$) is 0.0242 T, this value is much smaller than the saturation magnetic field strength ($H_S$) with a value of 0.2938 T. The values of remanent magnetization and the coercive magnetic field which are

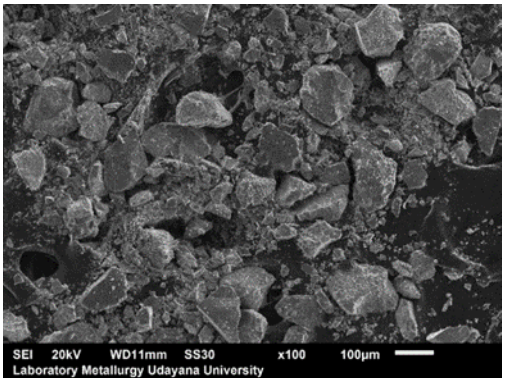


Figure 5. Magnetite crystal SEM image of iron sand fine powder sample.

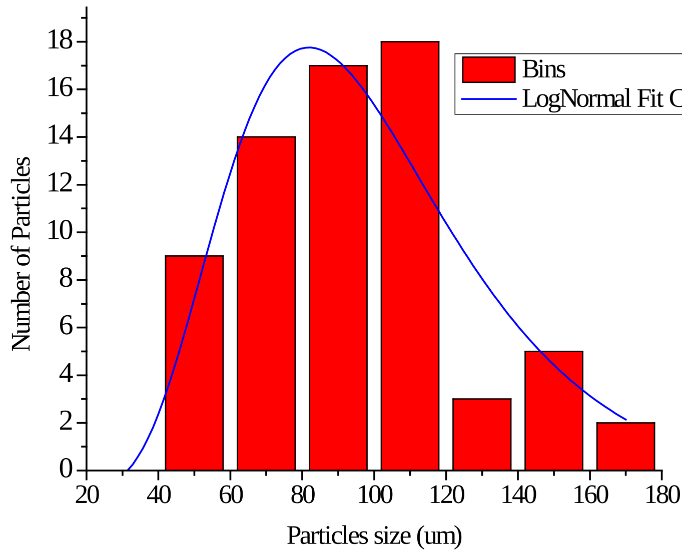


**Figure 6.** Histogram of Magnetite particles size data of iron sand fine powder sample.

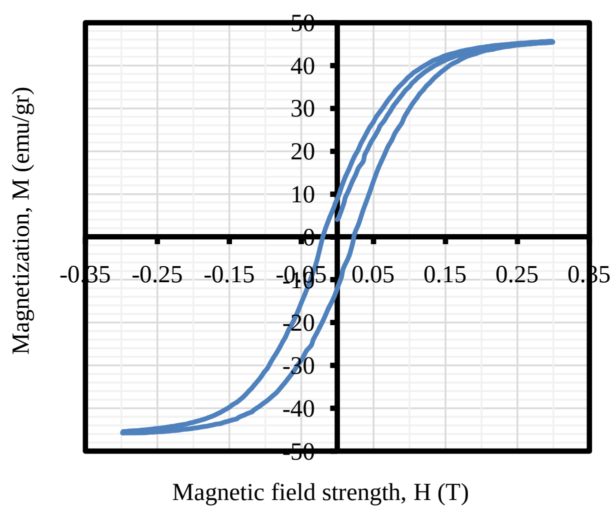


**Figure 7.** The magnetic hysteresis curve of the iron sand fine fowder sample from Tegal Lenga Beach

much smaller than the saturation value have caused the narrow area of the magnetic hysteresis curve. This magnetic hysteresis curve with a narrow area shows that the magnetite crystals that make up the fine powder of iron sand from natural sand of Tegal Lenga Beach are classified as weak ferromagnetic materials [15].

## 4. Conclusion

From the results of this study, it is known that magnetite crystals can be synthesized from the iron sands of Tegal Lenga Beach. The main constituent of the iron sand of Tegal Lenga Beach is magnetite metal oxide with a content of 84.26%. The magnetite crystal phase that has been obtained in this study has a tetrahedral structure with lattice parameters $a = b = (5.927 \pm 0.0180)$ Å and $c = (16.774 \pm 0.0145)$ Å. The magnetite crystal grains that have been formed are not homogeneous in size with the average grain size of (94,560 ± 10,1397) μm with granular and lumpy grains. The magnetite crystals from the iron sands of Tegal Lenga Beach are classified as weak ferromagnetic material with a remanent magnetization of 9.6672 emu/gr, a saturation magnetization of 45.3491 emu/gr, a coercive magnetic field strength of 0.0242 T, and a saturation magnetic field strength of 0, 2938 T.

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## Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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