Preparation of Iron Oxide Magnetic Nanoparticles Natural Sand of Rokan River Synthesis with Ball Milling

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Abstract. Natural sand samples taken from the Rokan river, Riau Province, Indonesia, have been ball milled for 100 (one hundred) hours using a ball with a diameter of 1.5 cm and a constant rotation angular velocity of the tube of 100 revolutions / minute. Neodymium Iron Boron (NdFeB) strong magnets are used to separate magnetic minerals from their non-magnetic minerals. Magnetic induction was measured using the Pasco PS-2162 probe and its value was used to calculate the magnitude of magnetic susceptibility, it turns out that the magnetic susceptibility value of the 100-hour Ball Milling results increased significantly when compared to the magnetic susceptibility before Ball Milling, \(968.245 \times 10^{-5}\) becomes \(19.471 \times 10^{-5}\). The results of the X-Ray Fluorescence (XRF) test showed that the percentage of Fe milled increased from 1.669% to 35.187%, the particle size obtained from the scanning electron microscope (SEM) results was in the nanometer order of 78 nm and the X-Ray Difractometer (XRD) results shows that the magnetic nanoparticles produced are dominated by the hematite phase \((\alpha-Fe_2O_3)\) and the others are in the magnetite \((Fe_3O_4)\) phase.

1. Introduction

Natural sand generally contains magnetic and non-magnetic materials. Magnetic materials are usually iron oxides such as Magnetite (Fe3O4), Hematite (\(\alpha\)-Fe2O3) and Maghemite (\(\gamma\)-Fe2O3)[1]. Separation of the magnetic material from the non-magnetic material of natural sand needs to be done so that the magnetic material can be further processed so that it can be applied for a wider range of purposes. Chemical methods such as electrochemistry, sonochemistry, coprecipitation; biological methods such as fungal mediation, bacterial mediation, protein mediation; and physical methods such as gas phase deposition, ball milling [2-6] can be used for the separation.

The synthesis of magnetic materials using physical methods, especially ball milling is preferred because of its simplicity, low cost, and smaller magnetic particle size. In the Ball milling method, the sample and grinding ball are inserted into a tube, the tube is then rotated which results in a continuous collision between the ball and the sample on the tube wall. This collision causes the magnetic material to separate from the non-magnetic material and become smaller in size, reaching micrometer to nanometer in size [7].

Magnetic nanoparticles have very wide applications, including drug delivery, tissue engineering [8], magnetic sensors [9], printer / photocopy ink [10], magnetic resonance imaging [11,12], separation of industrial waste and water pollutants [13], and others.

In order for magnetic nanoparticles to have high performance qualities, at least two important parameters Must be fulfilled, that is particle size and surface area [14]. In this paper, we will report the
size of the magnetic phase of magnetic nanoparticles synthesized by Ball milling which will later be applied for environmental discoveries.

2. Experimental Method
Natural sand samples taken from the Rokan river, Riau Province, Indonesia, are dried naturally under the sun. Magnetic minerals are separated from non-magnetic minerals using an Iron Sand Separator (ISS), magnetic minerals from the ISS are then synthesized physically using a ball milling tool for 100 hours using an iron ball with a diameter of 1.5 cm and a constant rotating speed of the milling tube 100 revolutions / minute To obtain magnetic minerals that are purer and smaller in size, the ball milling results are further separated between magnetic minerals and non-magnetic minerals using a strong magnet, Neodymium Iron Boron (NdFeB). The magnetic induction of river sand, magnetic minerals from ISS and magnetic minerals from ball milling for 100 hours were measured using the Probe Pasco PS-2162, magnetic induction values are needed to calculate their magnetic susceptibility. The magnetic mineral from 100 hours ball milling that has been separated with a strong NdFeB magnet is then carried out by the X-Ray Fluorescence (XRF) test to determine the percentage change in its elements, the scanning electron microscope (SEM) test to determine the size of the magnetic particles and the X-Ray Diffractometer test. to find out the structure and phase of the magnetic particles produced.

3. Results and Discussion

3.1. Magnetic Susceptibility Value
The magnetic susceptibility value of natural sand in the Rokan River, the results of Iron Sand Separator, and the 100-hour Ball milling results are shown in Table 1.

| NO | Name                                    | Susceptibility($x 10^{-5}$) |
|----|-----------------------------------------|------------------------------|
| 1  | Rokan River Natural Sand                | 1874.128                     |
| 2  | Iron Sand Separator Results             | 3246.082                     |
| 3  | Results of Ball milling for 100 hours   | 19471.568                    |

From Table 1, it can be seen that the Ball milling process is successful in separating magnetic particles from non-magnetic particles. This is confirmed by the significant increase in the value of magnetic susceptibility.

3.2. Element Composition
The composition of natural sand elements before and after ball milling is shown in Figure 1.

![Figure 1](attachment:image.png)
From Figure 1 it can be seen that there is an increase in concentration for magnetic particles such as Fe, Mn and V while non-magnetic particles such as Si, Al and K experience a decrease in concentration. Fe magnetic particles experienced a significant increase in concentration, while non-magnetic particles Si experienced a significant decrease in concentration. This indicates that the Ball milling process has succeeded in separating magnetic particles from non-magnetic particles, besides the increase in Ti concentration because it combines with Fe to form FeTiO$_3$.

3.3. Microstructure and Particle Size
The results of the Scanning Electron Microscope (SEM) test for Ball milling time of 100 hours with a magnification of 10000 times are shown in Figure 2.

![Figure 2](image1)

Figure 2. Particle size of 100 hour ball milling with magnification of 10000 times.

Figure 2 shows the surface shape and particle size of the 100 hour ball milling results. It appears that the surface is more regular and the particle size is nanometer order for about 78 nm. This particle size is smaller than the results of previous studies [15]. This proves that the ball milling time affects the size of the particles obtained, of course this applies to a certain time limit.

3.4. Magnetic Phase
The X-Ray diffraction pattern is shown in figure 3.

![Figure 3](image2)

Figure 3. X-ray diffraction pattern of ball milling for 100 hours.
The X-ray diffraction pattern (Figure 3) shows that the magnetic materials contained in the natural sand of the Rokan River after ball milling for 100 hours are magnetite (Fe3O4) and Hematite (Fe2O3). Hematite (Fe2O3) appears at the 2θ diffraction angle of 24.60°, 32.78°, 36.71°, 68.78°, corresponding to the reflection fields of hkl (012,104,110, and 208). Magnetite (Fe3O4) appears at 2θ diffraction angles of 28.62°, 41.72°, 58.83°, 61.98°, which correspond to the reflection fields of hkl (202, 400, 333, and 404). In addition, diffraction peaks still appear for non-magnetic materials, such as SiO2, this is in accordance with the XRF test results which show the presence of silica oxide and other non-magnetic materials.

4. Conclusion
Synthesis of natural sand by ball milling has succeeded in separating magnetic particles from non-magnetic particles, this is confirmed by the XRF test results.

Ball milling for 100 hours has also succeeded in obtaining a minimum particle size of 78 nanometers, this is confirmed by the SEM test results. While the XRD test results show that the magnetic particles, this is confirmed by the XRF test results. The XRD test results show that the magnetic particles produced are dominated by the hematite (α-Fe2O3) phase while the others are in the magnetite (Fe3O4) phase.

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