Synthesis of Magnetite Based Iron Sand by Using Coprecipitation method

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Abstract: Iron sand is a natural material that is easy to obtain. The low utilization of iron sand in Indonesia causes the low price of iron sand material. So, needed a technology in processing iron sand in order to become a producer that has usability. In this study we studied the synthesis of iron oxide materials in the form of magnetite (Fe₃O₄) and hematite (α-Fe₂O₃). The process of synthesis is done coprecipitation method. For dilution of hydrochloric acid HCl sands (12M), while for the deposition of ammonia filtrate (NH₄OH). The coprecipitated precipitated powder of magnetite powder (Fe₃O₄) by XRD method (X-ray Diffraction) and XRF (X-ray Flourences). The characterization results indicate that the precipitate is identified as magnetite.

Keywords: Iron sand, Magnetite, Coprecipitation Method

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I. Introduction

Iron sand is one of the abundant natural magnetic materials in Indonesia, especially in Lombok Island, which is along the Ampenan coast of Ampenan District. Iron sand which is rich in Fe content is generally oxidized and forms iron oxides, such as Fe₂O₃ magnetite, hematite (α-Fe₂O₃), and maghemite (γ-Fe₂O₃) [9]. But so far the utilization of natural iron sand has not been optimal, that is only limited to be used as a mining material in the absence of processed. In a previous study by Fithriyani [4] explained that the iron sand samples obtained from Ampenan Beach-Lombok contain Fe 69.8%; Si 11.6%; Ti 6.71%; Al 4.49%; Ca 2.08% and other minor components.

Iron sand is one of the most widely used magnetic material in many fields such as electronics, energy, chemistry, ferofluid, catalyst, and medical diagnosis. The application of iron sand was inseparable from the development of studies of nanomaterials demanding that they be in the order of nanometers [8].

Iron contained in iron sand can be diektraski with existing technology at this time and have economic value. Extraction is the process of separating a substance from a mixture so as to separate the Fe metal from its source [2].

Various methods have been carried out in extracting Fe₂O₃ and Fe₃O₄ powders from natural iron sand into nanometer sizes such as Chloride Acid Dissolved Metals Method [6], Solid Reaction [5], Oxidation Process [7], Mill scale [1].

In this research, iron sand is synthesized through coprecipitation method. Coprecipitation method is one method of nanoparticle synthesis based on precipitation of the substance that is chosen. The choice of coprecipitation method is due to its perosenya using low temperature and easy to control particle size so that the required time is relatively short [3].

In this study characterization of purified natural iron sand, using XRF to reveal the iron sand content and XRD to reveal the phase, structure, and grain size.

II. Experimental

Material
The material used in this research is sand taken from Ampenan Beach –Lombok

Sample Preparation
The sample preparation begins with drying iron sand at a temperature of 100 °C to remove water content. Furthermore, the process of separating (extraction) the magnetic material content of the sand using a hard magnet.

Synthesis of Magnetite and Hematite is done by using Coprecipitation Method. The basic ingredients for forming Fe₂O₃ and hematite (α-Fe₂O₃) magnetic materials are natural iron sand, hydrochloric acid (HCL) and ammonia (NH₄OH). These basic ingredients are called percursors. Based on chemical reaction for the manufacture of Fe₂O₃ magnetite and hematite (α-Fe₂O₃) using coprecipitation method can be done as follows:

\[
\text{Fe}^3+ + 3\text{OH}^- \rightarrow \text{Fe} \cdot \text{OH}_3 \rightarrow \text{Fe}_3\text{O}_4
\]

\[
\text{Fe}^2+ + 2\text{OH}^- \rightarrow \text{Fe} \cdot \text{OH}_2 \rightarrow \alpha \text{Fe}_2\text{O}_3
\]

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50 Fe of Fe$_2$O$_3$ powder was dissolved in HCL (12 M) as much as 100 ml at 70 °C and stirred about 30 min with magnetic stirrer.

Reaction equation:

\[
3\text{Fe}_2\text{O}_3(s) + 8\text{HCl}(l) \rightarrow 2\text{FeCl}_3(l) + \text{FeCl}_3(l) + 3\text{Fe}_2\text{O}_3(s) + 3\text{H}_2\text{O}(l) + \text{H}_2(g)
\]

**Deposition Filtrate**

After the solution is shaped filtered by using filter paper. The result of the filtered solution was filtrate. Then to precipitate the filtrate NH$_4$OH added as much as 120 ml and allowed to stand for 60 minutes so that the sediment is obtained. The Fe$_3$O$_4$ precipitate formed in dark black color is separated from the solution which is then washed by using aquades 3 times so that the results obtained are completely clean.

Reaction equation:

\[
2\text{FeCl}_3(l) + \text{FeCl}_2(l) + \text{H}_2\text{O}(l) + 8\text{NH}_4\text{OH}(l) \rightarrow \text{Fe}_3\text{O}_4(s) + 8\text{NH}_4\text{Cl}(l) + 5\text{H}_2\text{O}(l)
\]

**Making Magnetite Nanoparticles (Fe$_3$O$_4$)**

The subsequent precipitate was heated in an oven with a temperature of 100 °C for 4 hours. Furthermore, it was crushed using mortar and formed Fe3O4 hematite powder.

### III. Results And Discussion

The analysis carried out in this study begins with a magnetic separation process in natural iron sand in the Ampenan-Lombok Beach area and then on iron sand samples which have been prepared to be separated chemically using coprecipitation method.

Reprinted iron sand is characterized using XRF to determine the iron oxide content and other metal elements therein. The XRF characterization results obtained by chemical element data in natural iron sand as in Table 1.

<table>
<thead>
<tr>
<th>No</th>
<th>Element</th>
<th>%mass</th>
<th>Compounds</th>
<th>%mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fe</td>
<td>69.8</td>
<td>Fe$_2$O$_3$</td>
<td>60.8</td>
</tr>
<tr>
<td>2</td>
<td>Si</td>
<td>11.6</td>
<td>SiO$_2$</td>
<td>17.8</td>
</tr>
<tr>
<td>3</td>
<td>Ti</td>
<td>6.71</td>
<td>TiO$_2$</td>
<td>7.46</td>
</tr>
<tr>
<td>4</td>
<td>Al</td>
<td>4.49</td>
<td>Al$_2$O$_3$</td>
<td>6.18</td>
</tr>
<tr>
<td>5</td>
<td>Ca</td>
<td>2.08</td>
<td>CaO</td>
<td>1.99</td>
</tr>
<tr>
<td>6</td>
<td>Other Elements</td>
<td>&lt;1</td>
<td>Other Compounds</td>
<td>&lt;1.5</td>
</tr>
</tbody>
</table>

Furthermore, iron sand that has been diseparasi through chemical separation process that is by using method of copresipitasi. The results of elemental compositions and compounds are shown in Table 2.

<table>
<thead>
<tr>
<th>No</th>
<th>Element</th>
<th>%mass</th>
<th>Compounds</th>
<th>%mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Fe</td>
<td>72.06</td>
<td>Fe$_2$O$_3$</td>
<td>75.01</td>
</tr>
<tr>
<td>2</td>
<td>Cl</td>
<td>22</td>
<td>Cl</td>
<td>17.5</td>
</tr>
<tr>
<td>3</td>
<td>Ti</td>
<td>3.16</td>
<td>TiO$_2$</td>
<td>4.05</td>
</tr>
<tr>
<td>4</td>
<td>Ca</td>
<td>1.1</td>
<td>CaO</td>
<td>1.2</td>
</tr>
<tr>
<td>5</td>
<td>Mn</td>
<td>0.731</td>
<td>MnO</td>
<td>0.691</td>
</tr>
<tr>
<td>6</td>
<td>Other Elements</td>
<td>&lt;0.5</td>
<td>Other Compounds</td>
<td>&lt;0.5</td>
</tr>
</tbody>
</table>

Characterization results using XRF showed that the metal content of natural iron sand in Ampenan-Lombok Beach was dominated by Fe metal with presentation of 69.8% element composition. Then followed by Si metal exposure of 11.6%. For Fe$_2$O$_3$ compounds prior to the chemical separation process (Coprecipitation Method) has a 60.8% content presentation. Furthermore, the natural iron sand samples that have been synthesized based on quantitative data results show that the Fe metal element increased to 72.06%. Followed by an increase of Fe$_2$O$_3$ compounds by 75.01%. While the Si and SiO$_2$ compounds are not visible. Then after going through the process of oxidation showed an increase in the presentation of Fe metal content elements and Fe$_2$O$_3$ compounds of 90.02% and 87.99%. This indicates that in this process has formed Hematite ($\alpha$-Fe$_2$O$_3$).

The result of identification of chemical compound of iron sand sample which has been through chemical process that is coprecipitation method shows that the highest peak at 20 angle can be indicated as magnetite (Fe3O4). With magnetite presentations of 28.7% and hematite of 71.3% as shown in Figure 1.
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IV. Conclusion

Based on the results of the description and discussion that have been described, it can be concluded that the Magnetite (Fe₃O₄) have been synthesized from the natural sand material of Ampenan-Lombok Beach using the method of Copresipitasi.

References


Figure 1. XRD result of iron sand Coprecipitation Method


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