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The Synthesis of Fe_2O_3 from Domestic Waste as a Multipurpose Material

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ABSTRACT: Indonesia is categorized as an industrial country producing a lot of metal waste, so it is necessary to process this. One way of processing metal waste is by using it as a pigment. Pigments are natural substances that give rise to color, namely optical properties whose applications are endless; almost all industrial fields need pigments, from the ceramics, paint, and plastics industries to batik artisans who also need pigments or dyes. The method used in this test is hydrometallurgy, which consists of a leaching process and recovery. The pigment was produced oxide red from iron wool raw materials in this research. The weight of the oxide red pigment obtained for Experiment 1 was 0.20 grams, Experiment 2 was 4.96 grams, Experiment 3 was 2.63 grams, and Experiment 4 was 2.22 grams. Then the yield obtained in Experiment 1: 0.204 grams of pigment/gram of iron, Experiment 2: 1.984 grams of pigment/gram of iron, Experiment 3: 1.052 grams of pigment/gram of iron, and Experiment 4: 0.888 grams of pigment/gram of iron. The conversion obtained in Experiment 1 was 60%, Experiment 2 was 74%, Experiment 3 was 81%, and Experiment 4 was 78%. The pigment results obtained using this method can be further applied in industry because the conversion results are high.

Keywords: Hydrometallurgy, Iron Waste, Oxide Red, Pigments

1. Introduction

Indonesia can be categorized as an industrial country because the industrial sector contributes to the national economy, with a contribution reaching 20%. Still, apart from that, there is a high level of metal waste pollution in Indonesia, which comes from industrial processes related to machinery, metallurgy, metal coating, paint, skin, and mining and negatively impacts the human body and the environment. Iron metal is a chemical element that has many benefits for human life, starting from construction and various household equipment products. Multiple industries use iron metal as a primary material to make various products, for example, the synthesis of iron oxide, which can be used as a pigment. Pigments are inorganic dyes that give rise to color, namely optical properties that have countless applications; almost all industrial fields need pigments, from the ceramic industry, paint industry, and plastic industry to batik artisans who also need pigments or dyes[1], [2].

Metal waste will be more hazardous if it has been contaminated by the environment, such as when mining and metal welding processes contaminate water. As a result, the water cannot be consumed any longer, and if it is, it will have fatal effects on human health. To reduce imports of red pigment from foreign countries, efforts are needed to utilize natural resources in Indonesia, especially ferrous metal-based waste, which can be found in households, manufacturing industries, and lathe workshops. There are many ways to make pigments, including the hydrometallurgical process. Hydrometallurgy dissolves metals using acid or base solvents [3], [4]. Leaching is a process in hydrometallurgy. Several researchers have used leaching agents like hydrochloric acid (HCl) or sulfuric acid (H_2SO_4) at atmospheric pressure to dissolve metals during the leaching process [5], [6]. The valuable metal dissolved in the leaching agent will be precipitated using a solvent in the following stage, known as the precipitation process [7].

In this research, iron waste is processed into pigment using the hydrometallurgical method. This method was chosen. It has advantages over other processes because it can produce metal with high purity. Apart from that, another advantage is the low investment in equipment, which accelerates the production process and makes the processing process relatively shorter. This method is carried out in two stages: leaching and precipitation [8].

2. Experimental Method

2.1 Material

The materials used are spent iron wool, which is usually used for cleaning, sulfuric acid (H_2SO_4, Merck, Germany), sodium hydroxide (NaOH, Merck, Germany), hydrogen peroxide (H_2O_2, Merck, Germany), titanium dioxide (TiO_2, Merck, Germany), carboxymethyl cellulose (CMC, MTI, USA) and distilled water.

2.2 Methodology

2.2.1 Synthesis of $Ni_{0.5}Mn_{0.5}C_2O_4$.

Oxygen red pigment is prepared by weighing 2.5 grams of iron waste (iron wool) and drying it in the oven for 30 minutes. Then, 10% (v/v) of H_2SO_4 200 mL was prepared by dissolving ~20 mL of concentrated sulfuric acid. Then, 200 mL of 4 M NaOH solution was prepared by dissolving 32 grams of NaOH in distilled water until the solution volume was 200 mL. The dried iron waste was dispersed into the H2SO4 solution at a temperature of 80 °C. After a few minutes, all of the iron was dissolved, which was indicated by forming a green ferrous sulfate solution. The solution was filtered using filter paper, and the residue was washed with 100 mL of water. NaOH solution was added to the stirred filtrate to obtain a greenish precipitate. H_2O_2 solution was added dropwise until the precipitate changed color from green to red. The precipitate was filtered and washed using 100 mL of water. The precipitate cake was dried in the oven until a powder was obtained.

2.2.2 Preparation of Iron Oxide Paste and its application as a pigment

Oxide red paste was prepared by weighing 2 grams of the as-obtained iron oxide powder on a watch glass. Three grams of CMC solution (3% v/v) was added to the iron oxide powder and mixed using a spatula until a slurry with homogenous characteristics was obtained. The paste was spread on filter paper and dried in the oven. The changes that occurred can be observed. Similar steps prepared another pigment by adding TiO_2 powder (Iron Oxide: TiO_2 was varied 1:1 and 1:3). The result of the pigment can be compared side-by-side.

3. Results and Discussion

3.1. The Fe_2O_3 synthesis process

The leaching process of iron wool using sulfuric acid results in a green ferrous sulfate solution and H_2 gas release, as described in eq.1.

$$Fe_{(s)} + H_2SO_{4\,(aq)} \rightarrow H_{2(g)} + FeSO_{4(aq)} \quad (1)$$

After the iron sulfate was separated using gravity filtration, precipitation occurred when NaOH solution was added to the filtrate, resulting in ferrous hydroxide precipitate formation (eq.2)

$$FeSO_{4(aq)} + 2NaOH_{(aq)} \rightarrow Fe(OH)_{2(s)} + Na_2SO_{4(aq)} \quad (2)$$

Red ferric hydroxide can be obtained by the addition of H_2O_2 solution in an alkaline condition which is indicated by the color change of the precipitate from green to red precipitate.

$$Fe(OH)_{2(s)} + \tfrac{1}{2}\, H_2O_{2(aq)} \rightarrow Fe(OH)_{3(s)\ (red)} \quad (3)$$

The residue was obtained via filtration and dried in an oven to obtain a stable oxide red material (Eq. 4)

$$2Fe(OH)_{3(s)} \rightarrow Fe_2O_3.\,3H_2O_{(s)} \quad (4)$$

The four experiments were conducted, and the production throughput and yield can be seen in Table 1.

No. Experiment	Oxide Red Weight (g)	Yield ($g_{oxide\ red}/g_{iron\ wool}$)	Conversion (%)
Experiment 1	0.20	0.204	60
Experiment 2	4.96	1.984	74
Experiment 3	2.63	1.052	81
Experiment 4	2.22	0.888	78
Average	2.50	1.032	73.25

Table 1. Experiment trials of Iron Oxide Preparation

Differences in experimental data are caused by several factors. The first factor is the difference in the iron wool waste used; namely, the iron content in each iron wool waste is different. Hence, the iron or Fe content extracted can also vary in each experiment. The second factor is the addition of NaOH, which does not have a precise dosage as it is a pH-dependent field [9]. Hence, the formation of precipitates in each experiment produces different precipitate weights. The third factor is the poor quality of the filter paper so that reiterates and sediment can escape during the filtering process. Apart from that, poor filter paper can cause the pigment powder from the oven to stick to the filter paper and cannot be removed, thus affecting the pigment application process.

The differences in experimental data also influence the results of pigment application from each experiment, which can be seen in Table 2 as follows:

Table 2. Pigment Application Results Data

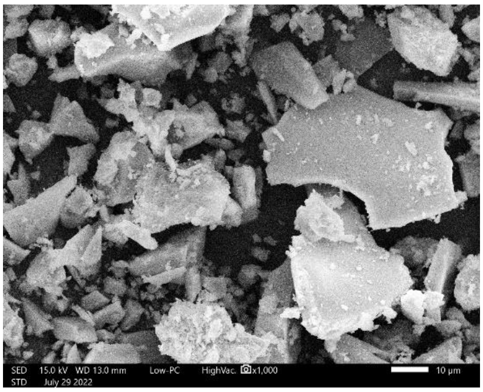
No. Experiment	Pigment (Full iron oxide)	Pigment : TiO_2 (3:1)	Pigment : TiO_2 (1:1)
1			
2			
3			
4			

Taken from the camera of Iphone 11, Samsung A52s, OPPO A5 2020, ASUS ROG Phone 5S

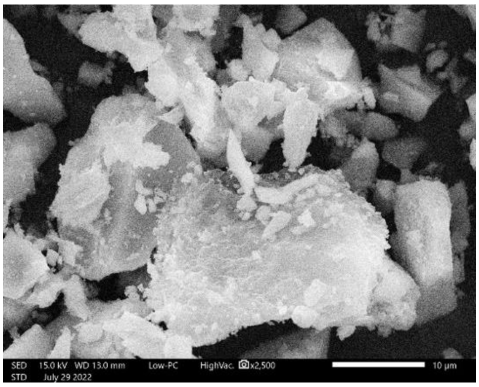
Other factors that influence the production of iron oxide from iron waste are as follows [3]:

1. Metal size
   The larger the metal size, the more complex the metal leaching process will take longer, but the smaller the metal size, the faster the leaching process.
2. Temperature
   The higher the temperature, the faster the metal leaching process.
3. Addition of solvent
   Adding an acidic solvent affects the speed of the reaction rate in the metal being reacted so that the metal absorption process will increase[10]–[12].

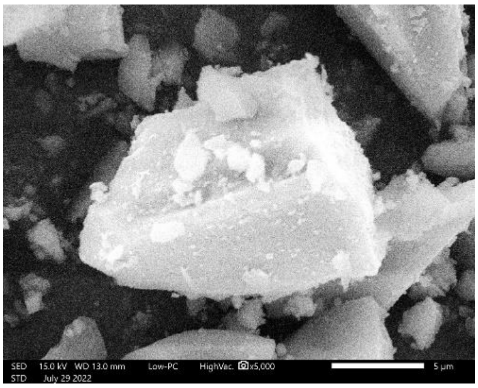
SEM Analysis



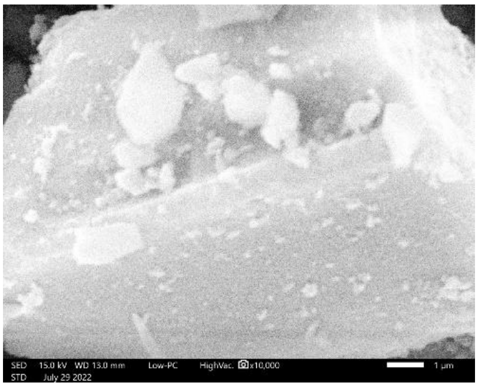
(a)



(b)



(c)



(d)

Fig 1. SEM Analysis (a) 1.000x (b) 2.500x (c) 5.000x (d) 10.000x

Figure 1 shows the results of analysis of iron oxide samples at various magnifications such as (a) 1,000x (b) 2.500x, (c) 5.000x and (d) 10,000x. Iron Oxide samples have a flake shape with a size of 10-60 microns. These particles are not homogeneous.

4. Conclusion

The hydrometallurgical method is effectively used in making iron waste pigments because it produces high conversions. In this study, the average pigment weight was 2.50 grams, the yield was 1.032 grams of pigment/gram of iron, and the conversion was 73.25%.

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Author Contributions

Aleida Dwi Rahmawati, Alma Putri Margaretta, Dyah Ayu Mutiara A, and Fajar Aszhari, out the experiment and wrote the manuscript with the support of Himmah Sekar Eka Ayu Gustiana. The final report was committed by all contributors.

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