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Synthesis and characterization of hematite (α-Fe_2O_3) from lathe waste using co-precipitation -calcination method

L M Khoiroh, M N Al-Chabib and A Prasetyo
Department of Chemistry, Faculty Science and Technology, Universitas Islam Negeri Maulana Malik Ibrahim Malang, Jl. Gajayana 50 Malang 65144, Indonesia

E-mail: lilikmfx@gmail.com

Abstract. Lathe waste contains high iron, therefore it has the potential to be a precursor in hematite (α-Fe_2O_3) synthesis. In this work, hematite was synthesized from lathe waste using the co-precipitation-calcination method with variation time of calcination: 1, 2, 3, 4 hours. The X-Ray diffraction data showed that the goethite (α-FeOOH) phase was obtained at precipitation stage then transformed to hematite (α-Fe_2O_3) phase after calcination and there is no impurities. The image of scanning electron microscopy showed that the distribution of particle size was not uniform. The color parameter showed that the highest chroma was achieved for 3 hours calcination.

1. Introduction

Iron craft industries produced lathe waste that contains 96 % of iron, which contributes to the environmental pollution significantly. The high iron content in lathe waste give opportunity to apply as precursor for iron compound synthesis. One of iron compound is hematite and this compound have advantages including non-toxic, having thermal stability [1], biodegradable [2], and durable [3]. In recent years, hematite becomes interesting in several applications as nanocatalyst, biomedical application, and especially in pigment application [3-6].

Many techniques have been developed to synthesized hematite, such as precipitation method [7], microwave-assisted calcination method [8], sol-gel method [9], hydrothermal technique [10], and thermal transformation technique [11]. Chemical precipitation method is a simple method, low cost, has high purity, short in preparation time, and high inhomogeneity [7, 11]. Synthesis of hematite by precipitation-calcination method results in high-crystallinity and nanoparticle sized product [7]. In synthesizing hematite, the compound change of structure, crystallite size, color, and distribution of morphology depend on the synthesis parameters, such as time of calcination [8]. Hematite have rhombohedral structure with crystallite size 40-41 nm, and lightness of 20.47 was calcined for 2 h [12], while for 3 h, hematite have crystallite size of 17 nm and lightness of 34.1 [11].

In this work, we investigated the effect of time calcination to structure, crystallite size, the color parameter, and morphology. We have used the co-precipitation-calcination method to synthesize hematite pigments. The sample products characterized using X-Ray Diffraction (XRD), color reader, and Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS).

2. Experimental

2.1 Materials

All reagents used to synthesis were analytical grades and without further purification. Nitric acid (HNO_3), sodium bicarbonate ($NaHCO_3$), and sodium hydroxide (NaOH), were purchased from Merck, Germany. Lathe waste used for synthesizing was from iron craft industry of Malang, Indonesia.

2.2 Methods

Samples of iron lathe were dissolved into 200 mL of HNO_3 (Merck) and heated until formed a slurry. The slurry product was then used as a precursor for synthesizing hematite. The precursor was dissolved in 500 mL of aqua de-mineral then was added with 50 mL of sodium bicarbonate solution and sodium hydroxide until reach pH 6. The homogenous mixture was heated at 70°C for 1 hour under magnetic stirring until formed the yellowish precipitated. The obtained precipitated was aged, filtered, and washed with distilled water until a neutral pH was obtained, then was dried at room temperature. The red dry powder obtained with calcining at 750°C for 1, 2, 3, and 4 hours. The structures of red powder synthesized were observed by X-Ray Diffraction (XRD), the value of lightness, redness degree, and yellowness degree were studied by a color reader and the morphology, and the compositions of the element were observed by Scanning Electron Microscopy-Energy Dispersive Spectroscopy (SEM-EDS).

3. Result and Discussion

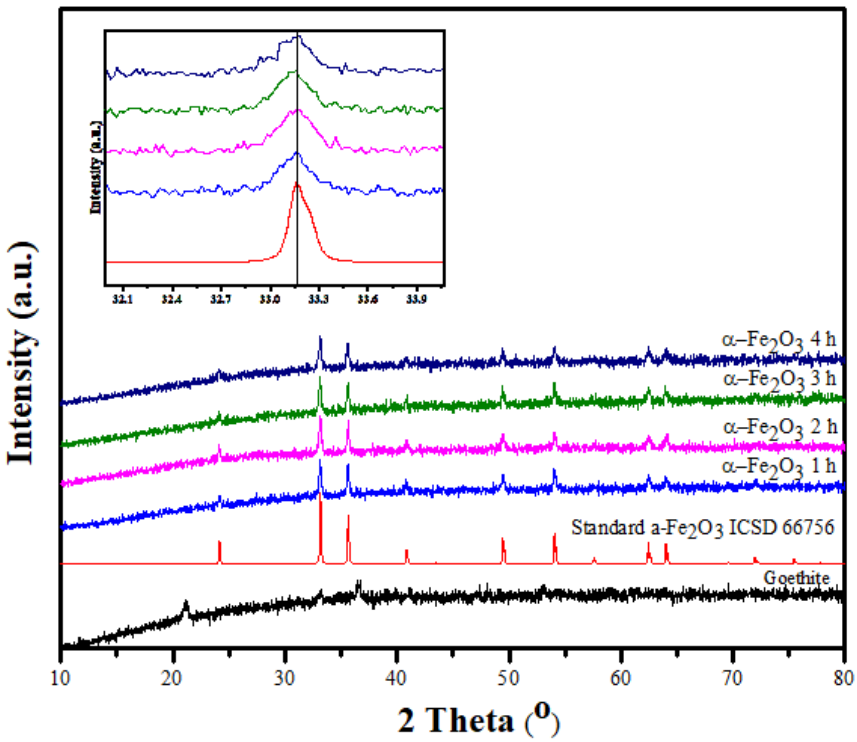


Figure 1. The X-Ray powder diffraction pattern of hematite obtained at calcination. Inset picture showed the peak position at around 33.1°

Figure 1 showed the XRD data of sample and showed that at precipitation stage at 70°C for 1 hour goethite phase was obtained and then transformed to hematite phase after calcination. XRD pattern in figure 1 shows that all sample after calcination has an agreement with pure of α-Fe_2O_3 (ICSD no

66756). Hematite successfully formed for 1-hour calcination and there was no changed phase when the calcination time was longer. All the reflection can be indexed in agreement with the expected rhombohedral structure of α-Fe_2O_3. The narrow peaks indicate that all of the hematite products are crystalline even though low crystallinity. The line broadening of the diffraction peak is affected crystallite size. Table 1 shows the average crystallites sizes are evaluated by Scherrer's equation at the highest intensity peak (104 planes). The average crystallite size associated with XRD data ranged between 14.84 and 24.34 nm, it means the size of hematite synthesized with longer calcination is increased due to a higher chance for crystal growth to take place in the calcination process. The highest crystallite size of α-Fe_2O_3 at 3 hours and decrease when calcined for 4 hours (20.83 nm).

Table 1. The crystallite size of hematite samples

The Compound	Crystallite Size (nm)
Hematite 1 h	14.83
Hematite 2 h	21.49
Hematite 3 h	24.34
Hematite 4 h	20.83

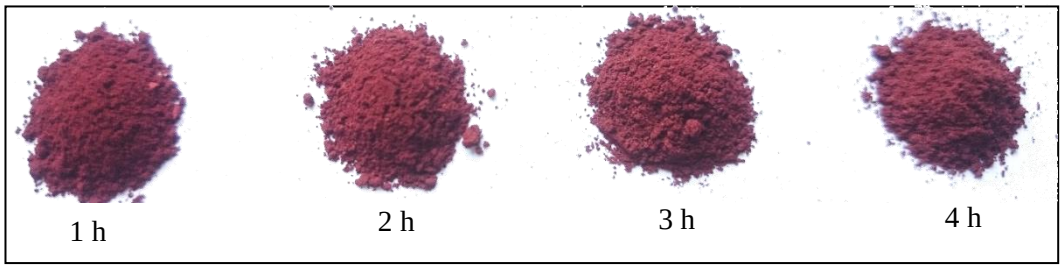
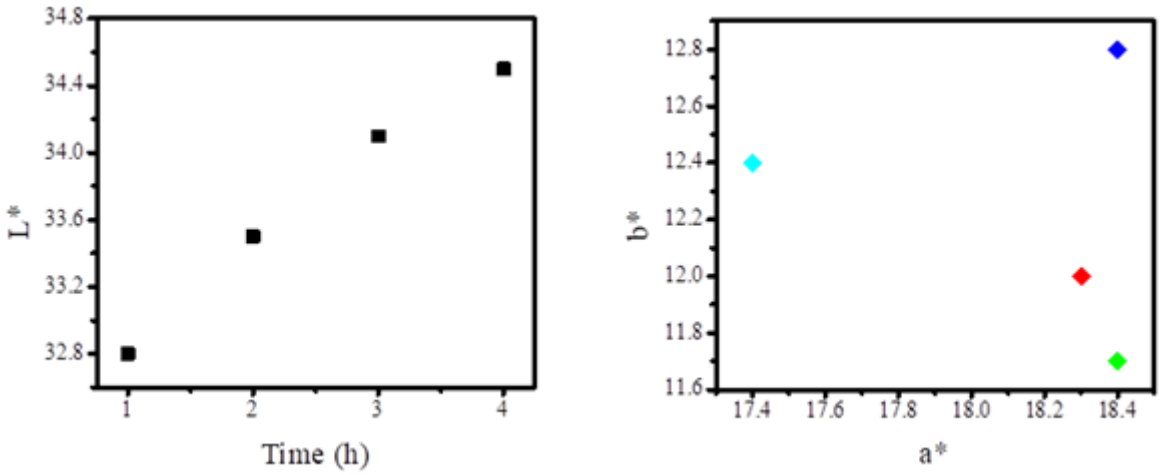


Figure 2. The visual aspect of hematite synthesized at variation of time calcination

Visual evaluation of calcined hematite at the variation of time showed dark red with no significantly different. The color parameter (L*) of hematite synthesized in figure 3 shows all samples high positive result for L* parameters. Hematite synthesized for 4 hours has the highest lightness. It was an increase in the intensity of lightness proportional to the time of calcination. Figure 4 shows that a* b* parameters of hematite synthesized accordance with standard [13]. From a*b* parameters, all hematite synthesized are more intense toward redness, except hematite which within 4 hours is showing more yellowness. Based on table 2 color parameter (C and H^o), the highest chroma parameter reached for 3 hours and decrease for 4 hours, meanwhile, the decreasing was not significant. It indicates that hematite can synthesize from 1 to 4 hours calcination. The parameter C tends to have a continuous increase as in increasing crystallite size. Decreased C parameter is caused differences of crystallite size in order to cause a broader distribution of electron transition band affected a less vibrant appearance.

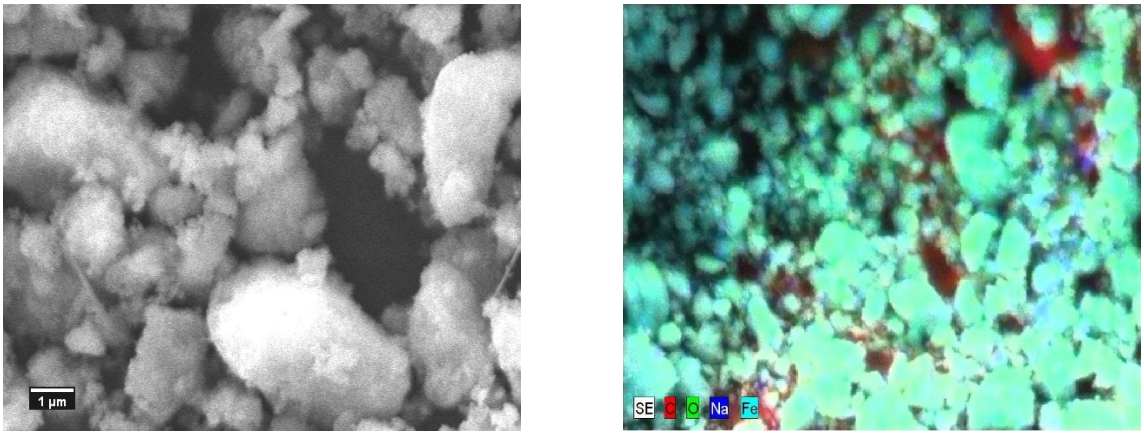
Table 2. Color parameter (C and H°) of hematite synthesized

Hematite (α- Fe_2O_3)	C	H°
Hematite 1 h	21.8	33.3
Hematite 2 h	21.8	32.5
Hematite 3 h	22.4	34.8
Hematite 4 h	21.4	35.5



(a) (b)

Figure 3. (a) Parameter of lightness at the variation of calcination time
(b) Value of a*b* color space 1 h 2 h 3 h 4h



(a) (b)

Figure 4. Synthesized hematite for 3 hours (a) SEM image of hematite (b) EDS image of hematite

The surface morphology of hematite obtained was studied using a Scanning Electron Microscope (SEM). Figure 5 showed the morphology of hematite calcined for 3 hours and can be seen that the particle shape of sample is not uniform and produces agglomerates. The results of energy dispersive

spectroscopy analysis as tabulated at Table 3 and showed there are found impurities in sample such as carbon and sodium. The presence of sodium element is derived from precipitating agents ($NaHCO_3$) and carbon is derived from raw material (lathe waste).

Table 3. The composition of an element in hematite

Element	Composition (%)
Fe	50.84
O	36.58
C	11.61
Na	0.97

4. Conclusion

Hematite was synthesized from lathe waste using precipitation-calcination method have a rhombohedral structure. The highest crystallite size of hematite was obtained for 3 hours (24.34 nm). The parameter of a*b* of hematite synthesized is directly to redness due to the highest chroma for 3 hours. The morphology of hematite particle was not uniform and there is agglomeration.

Acknowledgment

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