Variation of Polietilena Glikol (PEG) Volume Ratio Addition to the Extraction and Synthesis of Hematite (Fe_2O_3) from Iron Ore of Pemalogan Village using the Precipitation Methods

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Abstract: - The synthesis of hematite iron oxide from iron ore in Pemalongan village was carried out using an easy and simple method, namely precipitation. The purpose of this study was to determine the extraction results, crystal structure and crystal size of the compounds synthesized using the precipitation method. Iron ore processing is carried out by reacting 37% (w/w) HCl by dripping 25% (v/v) NH_4OH as a precipitating agent and adding PEG-200 to control particle size by varying the volume ratio of PEG-200: Iron 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g). Then calcined at 500°C for 2 hours. The synthesis results were characterized using X-ray diffraction (XRD) and scanning electron microscopy (SEM). The results of XRD analysis with varying volume ratios of PEG-200:iron 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g) have formed a hematite iron oxide phase. The synthesis results with a volume ratio of 1:5 (mL/g) produced the highest purity and had a trigonal geometric structure, cell parameters a = 5.036340 Å; b = 5.036340 Å; and c = 13.345420 Å; $\alpha = \beta = 90^{\circ}$; $\gamma = 120^{\circ}$ and the space group is R3 c. Calculation of crystal size using the Debye Scherrer equation results in variations in the volume ratio of PEG-200: Iron 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g) is 50.9912 nm; 43.08837 nm; and 45.30663 nm. The SEM characterization results showed that the iron oxide hematite produced from the synthesis clumped and formed small non-uniform grains.

Key-Words: Iron ore, PEG-200, hematite, extraction, precipitation, synthesis.

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1 Introduction

Indonesia is a country with abundant mineral resources in nature, such as iron ore, [1]. Abundant iron ore is spread across the islands of Sumatra, Java, Kalimantan, and some eastern regions of Indonesia, [2]. Iron ore reserves in South Kalimantan amount to 7,472,600 tons. Especially in Bajuin Sub-district, Pemalongan Village, Tanah Laut Regency has abundant iron ore resources. However, the iron ore contained still has impurities, [3]

Natural iron ore is generally a compound of iron with oxygen containing about 73% iron oxides such as hematite, magnetite (Fe_3O_4), limonite ($FeO(OH).nH_2O$), or siderite (Fe_2CO_3), [2]. Iron ore is a rock containing iron minerals and a number of impure minerals such as silica, alumina, magnesia, and nickel, [4]. One of the most abundant metal elements on earth is formed around 5% in the earth's crust, [2]. The benefits of iron ore are as raw material for making iron and steel, [1], [5]. Hematite can be used as a dye, [6]. The hematite obtained can be applied as a ferroelectric material, namely by using hematite as a mixture for making $BiFeO_3$ ferroelectric materials, [7]. The compound Fe_2O_3 is the most stable iron oxide under ambient conditions so it is commonly used in catalysts, nanocatalysts [8], photocatalysts to degrade dyes remazol red, [9], gas sensors, and electrode materials. Fe_2O_3 is also commonly used as a basic material for making permanent magnets, [10].

This research refers to research, [2], namely by using the precipitation method because the method has the advantages of being the simplest, easiest, and cheapest. In his research, iron ore was initially separated manually (magnetic separation) using a bar magnet. Furthermore, the separation results were weighed. Then the iron ore is dissolved in HCl, while stirring and heating on a hot plate magnetic stirrer at a certain speed. Precipitation is done by dripping ammonium hydroxide (NH_4OH) into the solution. After the precipitation occurs, calcination is then carried out using a furnace. However, the research to be carried out refers to the research, [11] before calcination of the precipitate is added first with polyethylene glycol (PEG). The addition of

PEG aims to control particle size, so that it can change the particle size to be small. PEG on the particles will coat the particles so as to inhibit growth of the particles. The study succeeded in obtaining a smaller particle size and causing the peak formed to be wider. In this study, the volume ratio of PEG added to the sample mass will be varied to determine the composition that will form the optimal result. To get the shape and size of the particles more uniform, usually, polyethylene glycol (PEG) is added to the mineral to be synthesized, [12].

The characterization carried out is by X-ray fluorescence (XRF) to determine the composition of the compounds contained in iron ore and characterization using X-ray Diffraction (XRD) to determine its crystal structure, [13], [14], then continued with the Debye Scherrer equation to determine the particle size, [15]. Characterization of the crystals obtained was also carried out using the Rietveld method, [16]. Rietveld method, useful for matching between theoretical diffraction patterns and experimental diffraction patterns obtained from the results of XRD analysis until a match between the two curves is obtained as a whole. SEM analysis was carried out to determine the shape of the surface morphology, [17].

2 Problem Formulation

The problem formulation in this study is as follows:

1. What is the iron content contained in iron ore from Pemalongan village?
2. What is the structure of hematite (Fe_2O_3) extracted and synthesized with the addition of various volume ratios of PEG-200 using XRD analysis?
3. What is the crystallinity of hematite (Fe_2O_3) extracted and synthesized using the precipitation method with the addition of variations in the volume ratio of PEG-200?
4. What is the particle size of hematite (Fe_2O_3) extracted and synthesized by adding variation of PEG-200 volume ratio using Debye Scherrer equation?
5. What is the morphology of hematite (Fe_2O_3) extracted and synthesized using precipitation method with the addition of PEG-200 volume ratio variation using SEM analysis?

3 Problem Solution

3.1 Characterization of Iron Ore using XRF (X-Ray Fluorescence)

Iron ore samples were previously crushed using a crusher and crushed into fine particles using a tor mill. Then, iron ore samples were manually separated (magnetic separation) using a bar magnet, [2]. Furthermore, the iron ore sample was sieved using a 200 mesh sieve to get a smaller particle size. The separation results were weighed as much as 5 grams for XRF (X-Ray Fluorescence) analysis.

Iron ore samples to be extracted were previously characterized to determine the metal composition and mineral content contained in iron ore. The results of XRF analysis are in the form of qualitative analysis that identifies the types of elements detected in iron ore, while quantitative analysis is to identify the number of elements contained in iron ore which is shown in the element content in percent (%). The result data was obtained in the form of graphs and tables detected by X-rays. The data provides information on the comparison of metal composition in iron ore before and after the extraction process.

Iron ore that has been analyzed is then extracted using the precipitation method. This method is a chemical separation process, [2]. Iron ore was previously separated first manually (magnetic separation) using an oval-shaped magnet. The purpose of separation is to separate magnetic minerals from non-magnetic ones. The results of iron ore separation were then dissolved in 37% HCl (equations 1), while stirring manually with a glass stirrer and heated at 90ºC for 60 minutes on a hot plate, [11]. The following reaction:

$$Fe_2O_3 + 6\ HCl \rightarrow 2\ FeCl_3 + 3\ H_2O \quad (1)$$

The addition of 37% HCl to the iron ore aims to dissolve the entire iron ore. Precipitation is done by dripping little by little 25% ammonium hydroxide (NH_4OH) into the solution until a precipitate form (equations 2), with the following reaction:

$$FeCl_3 + 3\ NH4OH \rightarrow Fe(OH)_3\downarrow + 3\ NH_4Cl \quad (2)$$

The $Fe(OH)_3$ precipitate formed was filtered using Whatman No. 42 filter paper. Filtering was carried out for 6 days to separate the filtrate from the precipitate. After the filtrate and precipitate were separated, the precipitate was then washed using aquademineral. Aqua demineral can not interfere

with the purity of the synthesis product, because it does not contain other minerals, which can add impurities to the synthesis product. The precipitate was then dried for 16 days. The dried precipitate was weighed and divided into 3 parts into alumina crucible, then PEG was added to each sample with variations in the volume ratio of PEG:Iron 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g). The variation aims to determine the optimal volume of PEG-200 to produce hematite metal oxide with a smaller particle size. The addition of PEG-200 to the particles serves to wrap the particles so that it can inhibit particle growth, [11]. Furthermore, it is stirred using a magnetic stirrer for 15 minutes, until perfect and homogeneous mixing occurs. The sample that has been obtained is then calcined at 500 ◦C for 2 hours using a furnace which aims to produce a synthesis that has a pure crystal structure (equations 3). The reaction:

$$2\ Fe(OH)_3 \rightarrow Fe_2O_3 + 3\ H_2O\uparrow \quad (3)$$

Tabel 1. XRF Characterization Results of Iron Ore

No	Metal type	Percentage (%) Before Extraction	Percentage (%) After Extraction
1.	Fe	97,56 %	97,69 %
2.	Si	0,88 %	1,7 %
3.	Ca	0,33 %	0,32 %
4.	Cr	0,094 %	0,086 %
5.	Cr	0,21 %	0,14 %
6.	Mn	0,1 %	0,1 %
7.	La	0,2%	0,00%
8.	P	0,13%	0,00%
9	Br	0,53%	0,00%

The $Fe(OH)_3$ powder obtained was then re-analyzed using XRF. XRF data on iron ore before and after the extraction process is shown in Table 1. Based on the characterization results of XRF (X-Ray Fluorescence) that have been carried out before extraction, that Pemalongan iron ore contains several types of metals such as Si, P, Ca, Cr, Mn, Cu, Br, La, and the main metal element Fe by 97.56%. Then after extraction, there was a decrease in other metal elements, even in metal types such as P, Cu, and Br could be removed and the extraction process succeeded in increasing the percentage of the main metal Fe to 97.69%. This is due to the loss of several other metal elements, causing the percentage of Fe metal to increase. The samples obtained from the extraction process were then synthesized to form hematite metal oxide compounds. The formed hematite metal oxide was analyzed for its crystal structure using XRD and surface morphology analysis of hematite metal oxide by SEM.

3.2 XRD Characterization Results of Hematite Iron Oxide Variation of PEG:Iron Volume ratio 1:5

The results of hematite iron oxide synthesis with the addition of PEG:Iron volume ratio variation of 1:5 (mL/g) show the presence of hematite iron oxide phase which can be seen in Figure 1. The 2θ value shows the angle between the incident ray and the reflected ray, while the one that shows the number of X-rays diffracted by the crystal lattice is called the intensity value. The higher the intensity value indicates that more crystals are formed and have good crystal order in the sample. The diffractogram of the hematite iron oxide synthesis results in a variation of the PEG:Iron volume ratio of 1:5, which can be seen in Figure 1. The peaks that appear are then matched with standard X-ray diffraction data from JCPDS. The following is a comparison of the synthesis results with JCPDS data in Figure 2.

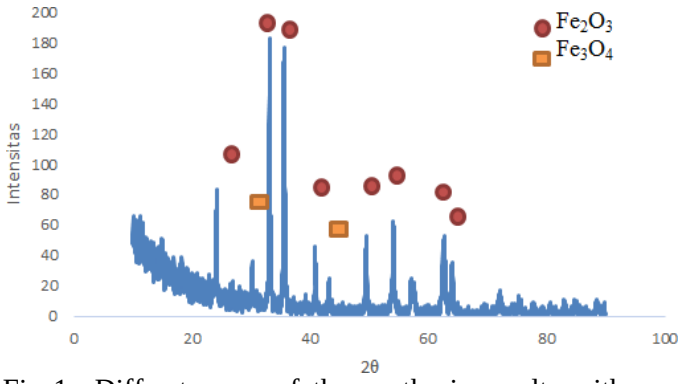

Fig. 1: Diffractogram of the synthesis results with variation of PEG:Iron volume ratio 1:5 (ml/g)

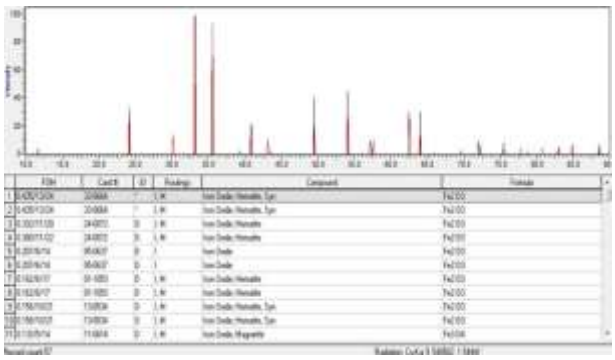

Fig. 2: Comparison of hematite iron oxide synthesis results of variation of PEG:Iron volume ratio of 1:5 (mL/g) with JCPDS

The results of hematite iron oxide synthesis variation of PEG:iron volume ratio of 1:5 (mL/g) are shown on the red vertical line, while the black vertical line shows the standard hematite iron oxide synthesis results from JCPDS X-ray diffraction. The presence of oxide phase peak of hematite iron oxide synthesis matches the diffraction peak of hematite iron oxide synthesis with JCPDS data 0.435/13/24 Card # 33-0664.

Based on the results of hematite iron oxide characterization, the highest intensity was obtained in the variation of the volume ratio of PEG: Iron, which was 117.5 with a 2θ angle value of 35.5842°. The high intensity indicates that the crystal has good crystal order. In addition, the hematite iron synthesis with variation of PEG:Iron volume ratio of 1:5 (mL/g) has a higher purity than the hematite iron oxide with variation of PEG:Iron volume ratio of 2:5 (mL/g). This is indicated by fewer impurity peaks appearing on the diffractogram pattern. The other identified impurity phase is Fe_3O_4. However, based on the diffractogram, the Fe_3O_4 impurity has a low intensity value, so it is not too significant.

3.3 XRD Characterization Results of Hematite Iron Oxide Variation of PEG:Iron Volume Ratio 2:5 (mL/g)

The results of hematite iron oxide synthesis with the addition of variations in the volume ratio of PEG:Iron 2:5 (mL/g) in Figure 3 show the presence of hematite iron oxide phase. The peaks that appear are then matched with X-ray diffraction standards from JCPDS. The diffractogram of hematite iron oxide synthesis results with variation of PEG:Iron volume ratio 2:5 (mL/g), shown in Figure 3.

Based on Figure 3, it can be seen that the intensity of the resulting peak of hematite iron oxide is quite low, namely 106.9 with a 2θ angle value of 35.5830, meaning that the hematite iron oxide product formed is small. This can be caused by many impurities characterized by the presence of other peaks that appear on the diffractogram, such as Fe_3O_4, SiO_2, and CaO. Some of the impurities that appear may come from extracted impurities that have not been removed, so the presence of these impurities can interfere with the process of forming the target compound. A comparison of the synthesis results with JCPDS data can be seen in Figure 4.

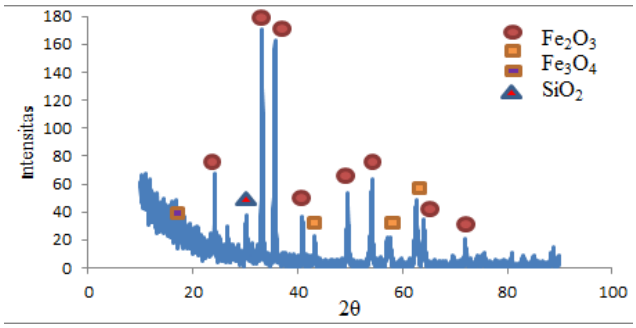

Fig. 3: Diffractogram of the synthesis results with the addition of variations in the volume ratio of PEG:Iron 2:5 (mL/g)

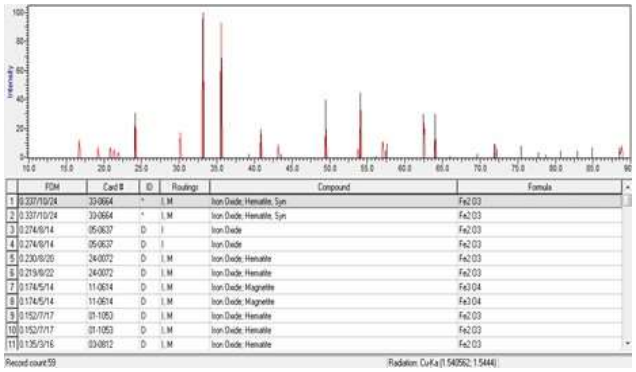

Fig. 4: Comparison of hematite iron oxide synthesis results of variation of PEG:Iron volume ratio 2:5 (mL/g) with JCPDS

The presence of the hematite iron oxide phase peak in Figure 4 is similar to the hematite iron oxide phase peak with JCPDS standard data 0.337/10/24 Card # 33-0664.

3.4 XRD Characterization Results of Hematite Iron Oxide Variation of PEG:Iron Volume Ratio 3:5 (mL/g)

The synthesis results on the hematite iron oxide variation of PEG:Iron volume ratio of 3:5 (mL/g) has shown the presence of hematite iron oxide phase as shown in Figure 5.

Based on Figure 5, it can be seen that the intensity of the hematite iron oxide peak produced is quite high, namely 114.3 with a 2θ angle value of 35.5932, meaning that the hematite iron oxide product formed is quite a lot. However, the synthesized oxide still contains other impurities such as Fe_3O_4. However, the impurity peaks that appear on the diffractogram pattern are fewer. This is because Fe_3O_4 from the extraction results still cannot be removed. Furthermore, the peaks obtained from the X-ray diffraction data were matched with

standard X-ray diffraction data from JCPDS. A comparison of synthesis results with JCPDS data can be seen in Figure 6.

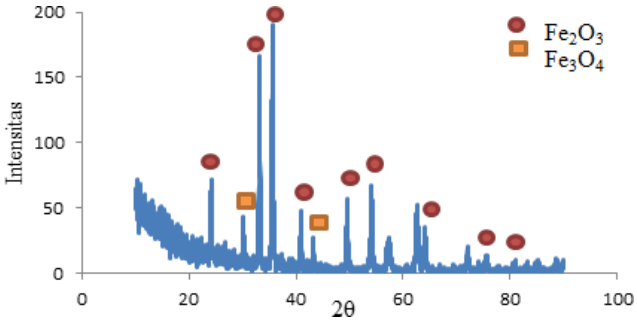

Fig. 5: Diffractogram of synthesis results with the addition of variations in the volume ratio of PEG: Iron 3: 5 (mL/g)

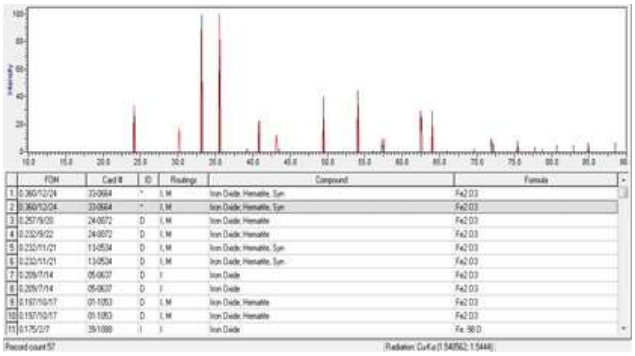

Fig. 6: Comparison of hematite iron oxide synthesis results of variation of PEG:Iron volume ratio 3:5 (mL/g) with JCPDS

The presence of the hematite iron oxide phase peak in Figure 11 matches the hematite iron oxide phase peak with JCPDS data 0.360/12/24 Card # 33-0664 standard data.

3.5 Comparison of XRD Characterization Results

The results of hematite iron oxide characterization by X-ray diffraction with the addition of variations in the volume ratio of PEG:Iron 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g) show the results of the formation of hematite iron oxide crystals shown in Figure 7. The synthesis results in Figure 7 are shown to have a crystal structure indicated by sharp diffractogram peaks. From the results of each synthesis, a comparison of the highest one peak diffractogram was carried out.

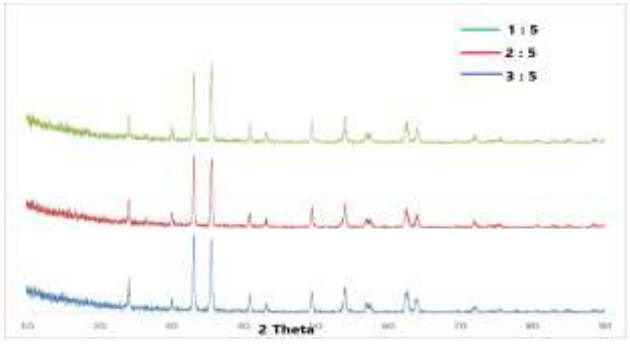

Fig. 7: Comparison of diffractograms of hematite iron oxide variation of PEG:Iron volume ratio of 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g).

The sharper the peak and the smaller the area, the higher the crystal quality of the synthesis. Figure 7 shows the highest peak intensity value of the synthesis at the variation of PEG:iron volume ratio of 1:5 (mL/g) with an intensity value of 117.5; 2θ = 35.5842°; FWHM of 0.2706, and area of 0.17. The highest peak of the synthesized 2:5 (mL/g) volume ratio has an intensity value of 106.9; 2θ = 35.5830o; FWHM of 0.2713; and an area of 0.17. The highest peak in the synthesis of 3:5 (mL/g) ratio has an intensity value of 114.3; 2θ = 35.5932°; FWHM of 0.2833; and an area of 0.18. The highest intensity indicates that more hematite iron oxide crystals are formed. states that the highest crystallinity in a material has a greater number of X-ray reflecting fields than the same material with a lower level of crystallinity. To determine the best results, it can be seen from the FWHM (Full Width at Half Maximum) value. This FWHM value is related to the crystallinity value.

Based on Table 2, it can be seen that hematite iron oxide is synthesized by adding variations in the volume ratio of PEG: Iron produces hematite iron oxide with different crystallinity. This can be seen from the FWHM value and the different areas in each XRD analysis result.

Table 2. Comparison of hematite iron oxide synthesis results with variations in PEG:Iron volume ratios of 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g)

No.	Comparative factor	Variation of PEG : Iron Ratio		
		1 : 5	2 : 5	3 : 5
1.	Peak Position (2θ)	35,5842	35,5830	35,5932
2.	Intensity	117,5	106,9	114,3

3.	FWHM	0,2706	0,2713	0,2883
4.	Area	0,17	0,17	0,18

From the results of XRD analysis if the smaller the FWHM value and area, the higher the crystallinity value, [18]. In the variation of PEG:Iron volume ratio 1:5 (mL/g), the hematite iron oxide formed has the smallest FWHM and area values of 0.2706 and 0.17 compared to the variation of PEG:Iron volume ratio 2:5 (mL/g), and 3:5 (mL/g). This indicates that the hematite iron oxide formed has a good level of crystallinity. Then the highest intensity value is also in the variation of PEG:Iron volume ratio of 1:5 (mL/g) which indicates that the synthesis results in more products produced. Based on the FWHM value, area, and the highest intensity value obtained, it can be seen that the hematite iron oxide synthesis with the best crystallinity is in the variation of PEG:Iron volume ratio of 1:5 (mL/g).

3.6 Hematite Iron Oxide Crystal Size

The crystallite size with a particular phase can be done using X-ray diffraction. The determination refers to the main peaks of the hematite structure of the diffractogram pattern through the Debye Scherrer equation approach. To calculate the crystal size can use the Debye Scherrer equation with the wavelength, intensity, 2θ, and FWHM values from the XRD analysis that has been produced. Based on the calculations obtained, a graph of the relationship between ln (1/cos θ) as the x-axis and ln β as the y-axis can be made. The relationship graph of ln (1/cos θ) with ln β for hematite iron oxide with PEG:Iron volume ratio variation of 1:5 (mL/g), 2:5 (mL/g), and 3:5 (mL/g) is as follows:

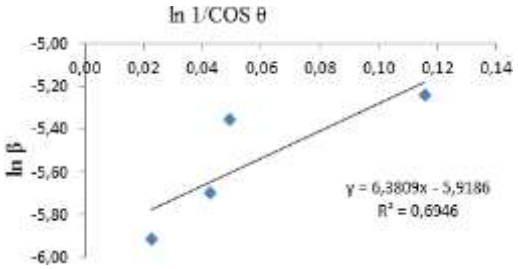

Fig. 8: Particle size graph of hematite iron oxide variation of PEG:Iron volume ratio of 1:5 (mL/g).

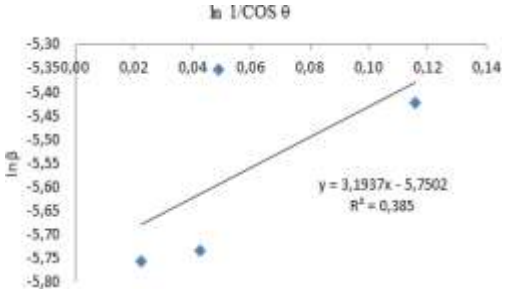

Fig. 9: Particle size graph of hematite iron oxide variation of PEG:Iron volume ratio of 2:5 (mL/g).

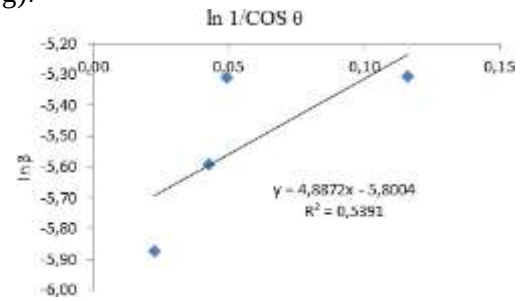

Fig. 10: Particle size graph of hematite iron oxide variation of PEG:Iron volume ratio of 3:5 (mL/g).

Based on Figure 8, 9 and 10 each sample shows an R2 value, and a slope value that is not close to 1 and does not have a 45° slope. This indicates that the 45° slope for a linear line is not achieved. Based on the data generated, the mismatch of the slope value and 45° slope is due to the values of β and 1/cos θ not being constant. The β value derived from FWHM has a different value at each peak, the FWHM value is influenced by the intensity of each crystal field. Then the value of 1/cos θ at each main peak has a different angle. The main thing that causes this difference is that the electromagnetic wave hitting the crystal causes diffraction of each atomic arrangement in the crystal which results in different X-ray diffraction patterns. R2 and slope can be close to one with a slope of 45° if the increase in β value is followed by an increase in 1/cos θ which means that the β value is directly proportional to 1/cos θ.

This also occurs because the hematite iron oxide crystals obtained are polycrystalline, which have more than one crystal orientation with different scattering fields, causing R2 not to approach one. Nevertheless, the equation can still be used to estimate one of the crystal sizes of the polycrystalline compound. Crystal size data that has been obtained from the calculation results are as follows :

Table 3. Crystal size comparison of iron oxide synthesis results..

Variation of PEG : Iron	2θ	FWHM	Crystal Size (nm)
1:5	35,5842	0,2706	50,99120
2:5	35,5830	0,2713	43,08837
3:5	35,5932	0,2833	45,30663

From Table 3, it can be seen that the addition of PEG with different volumes in the synthesis process can produce hematite iron oxide with different crystal sizes. This shows that the provision of variations in the volume ratio of PEG affects the crystal size. The variation of PEG volume ratio shows that the addition of PEG reaches the optimum volume at the variation of PEG:Iron volume ratio of 2:5 (mL/g) for an increase in Fe_2O_3 particles of 43.08837 nm. The decrease in crystal size is related to the molecular weight of PEG-200. The greater the molecular weight of PEG, the greater the number of PEG chains that coat the particle surface. Another factor that also affects the size of the crystals produced is the cooling temperature on the rate of nucleation and the rate of crystal growth. If the cooling process is fast, the rate of formation of nuclei is high and the rate of crystal growth will be faster, resulting in small crystal sizes, whereas in the slow cooling process, the rate of formation of nuclei is low, the slow crystal growth process will produce large size crystals.

3.7 Rietveld Analysis of Hematite Iron Oxide Variation of PEG: Iron Volume Ratio 1:5 (mL/g)

X-ray diffraction data is then processed by the Rietveld method using the Rietica program. The method serves to smooth the calculated diffraction pattern (model) with the measured diffraction pattern. From these results, no PEG phase was found in the sample, meaning that PEG is only useful for controlling particle size and does not react and only functions as a template that wraps particles, [19].

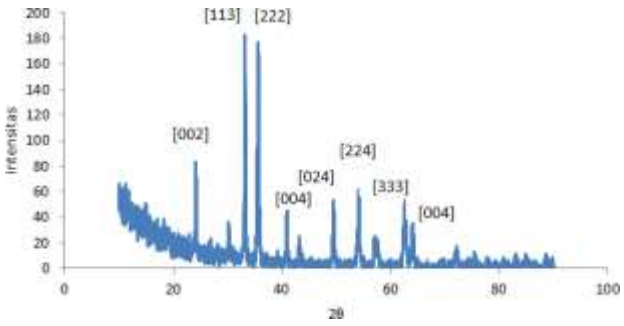

Fig. 11: The diffractogram of hematite iron oxide synthesized by the precipitation method adding a variation of PEG:Iron volume ratio of 1:5 (mL/g).

From data processing with the Rietveld method, it produces cell parameters, Miller index, space group and crystal system (geometry shape) of hematite iron oxide. Figure 11 shows the Miller index value showing the hkl value of each dominant peak at 2θ angles from 10° to 90°. The Miller index value provides information that shows the position of atoms in the unit cell and affects the properties and behavior of the synthesized material.

The diffractogram of the hematite iron oxide compound synthesized using the precipitation method that has been processed using the Rietveld method can be seen in Figure 12. The diffractogram with black dots is the diffractogram of the synthesized hematite iron oxide, while the red diffractogram is the diffractogram of the hematite iron oxide standard data in the Rietica program, [14]. The green diffractogram is the diffractogram of the difference between the data of the synthesized compound and the standard data of hematite iron oxide.

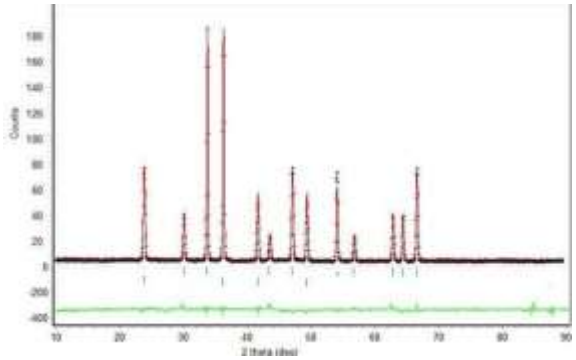

Fig. 12: Diffractogram of hematite iron oxide synthesized using precipitation method with variation of PEG:Iron volume ratio of 1:5 (mL/g) processed with Rietica program.

Based on table 4 of data processing using the Rietica program, it shows that hematite iron oxide synthesized by the precipitation method with the addition of variations in the volume ratio of PEG: Iron 1: 5 (mL / g) has a trigonal geometry with space group R3 c and cell parameters a = 5.036340 Å; b = 5.036340 Å; and c = 13.345420 Å. Hematite iron oxide has a trigonal structure. This means that this crystal structure has 4 axes of symmetry consisting of 1 main symmetry axis and 3 additional symmetry axes. The 3 axes (a, b, and d) are the same length and are located in the horizontal plane while the 1 c

axis can be shorter or longer as shown in Figure 13. The trigonal system has an axial ratio, namely a = b = d ≠ c, meaning that the length of the a axis is equal to the b axis and equal to the d axis, but not equal to the c axis and also has crystallographic angles α = β = 90˚; γ = 120˚. This means that in this system, the angles α and β are perpendicular to each other and form an angle of 120˚ to the γ axis.

Table 4. Crystal system, space group, and cell parameters of hematite iron oxide compounds result. indexing of Rietica program

Parameters	Iron Oxide
Crystal System	Trigonal
Space Group	R3 c
Parameter Cell Unit (Å)	a : 5,036340
	b : 5,036340
	c : 13,345420
Angle	α : 90°
	β : 90°
	γ : 120,0000°

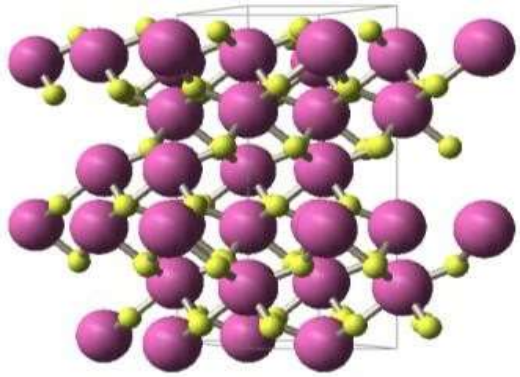

Fig. 13: Crystal structure of hematite iron oxide ((Fe_2O_3))

The results of figure 11 are obtained from the data in Table 5 which has the following fractional coordinates.

Table 5. Hematite Iron Oxide Fractional Coordinates

ATOM	NTYP	X	Y	Z	B	N	
		B11	B22	B33	B12	B13	B23
Fe	Fe	0.00000	0.00000	0.35530	0.78285	0.02083	
		0.00000	0.00000	0.00000	0.00000	0.00000	0.00000
O	O	0.30590	0.00000	0.25000	0.81035	0.04166	
		0.00000	0.00000	0.00000	0.00000	0.00000	0.00000

3.8 SEM Characterization of Hematite Iron Oxide

The results of the synthesis using SEM were carried out to determine the surface morphology of the solid sample. The image formed by a very small electron beam in this analysis is focused on the surface of the material. The results of SEM analysis with variations in the volume ratio of PEG obtained have an influence on crystal morphology. Figure 14 shows that the effect of PEG addition can cause agglomeration in hematite iron oxide, [20]. The following morphology of hematite iron oxide crystals was analyzed using Scanning Electron Microscope (SEM) with magnification of 10,000 and 20,000 times. Based on Figures 14(a) and 14(c), the morphology of the synthesized particles with variations in the volume ratio of PEG:Iron 1:5 (mL/g) and 3:5 (mL/g) shows that particle agglomeration occurs, causing the particle size to become large. This is because the pores formed are smaller. While Figure 14(b) shows that in the addition of variations in addition to variations in the volume ratio PEG:Iron 2:5 (mL/g) shows more particles forming small clumps.

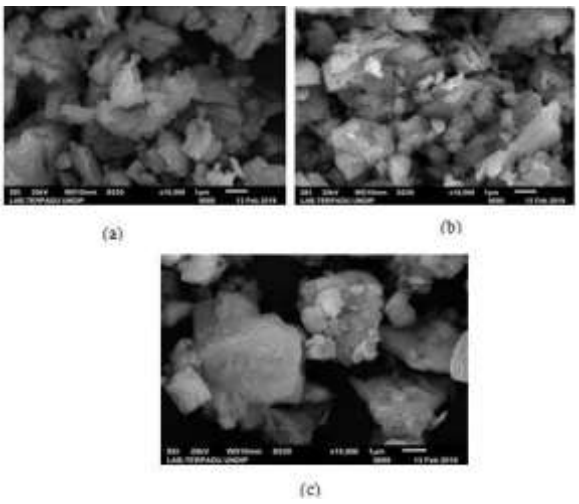

Fig. 14: SEM Characterization Results of hematite iron oxide (a) 1:5 (mL/g) (b) 2:5 (mL/g) (c) 3:5 (mL/g) with magnification of 10,000 times

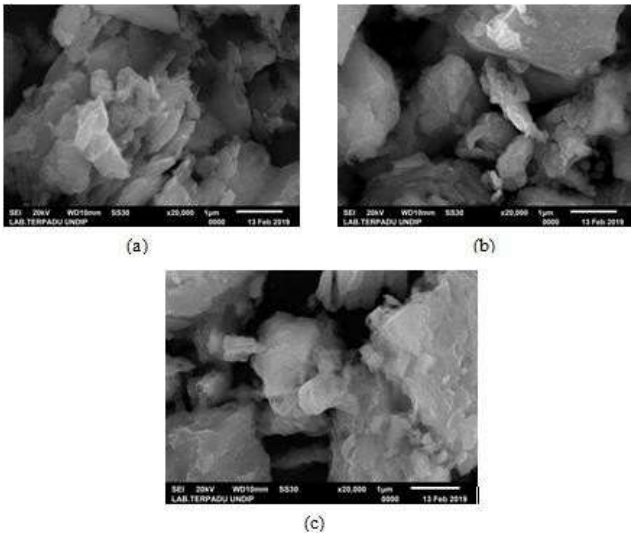

Fig. 15: SEM characterization of hematite iron oxide synthesis results (a) 1:5 (mL/g) (b) 2:5 (mL/g) (c) 3:5 (mL/g) with a magnification of 20,000 times.

Figures 15(a), 15(b), and 15(c) of hematite iron oxide synthesis results from SEM characterization with a magnification of 20,000 times show in more detail that the hematite iron oxide crystals have agglomerated. The agglomeration occurs due to the relatively slow diffusion speed of PEG which causes the possibility of crystals to agglomerate and rearrangement occurs which causes smaller pores. According to [21], the pores in the crystal will gradually shrink to a smaller pore size, so that the hematite iron oxide particles (Fe_2O_3) are agglomerated. Meanwhile, Figure 15(b) shows that the crystals do not agglomerate and rather form small clumps. This is because the more volume of PEG added, the more PEG is trapped on the surface of the particles, and during the calcination process PEG will be decomposed which causes the formation of pores in the crystal. However, the presence of pores in hematite iron oxide indicates that crystals with the addition of PEG at a variation of the PEG:Iron volume ratio of 2:5 (mL/g) is the optimum volume, where the resulting particle size is smaller than 1:5 (mL/g) and 3:5 (mL/g). This is in accordance with the calculation of Debye Scherrer. The smallest particle size can be applied as a catalyst material. According to [22], [23], stated that different particle sizes and non-uniform morphology indicate that the addition of PEG has a role in controlling crystal nucleation, but the results obtained, PEG has not been seen to have a role in controlling crystal nucleation.

4 Conclusion

Based on the results of the research and discussion, the following conclusions can be drawn:

4.1 Conclusion

1. Iron ore from Pemalongan contains iron metal (Fe) of 97.56%, while the iron metal content after the extraction process that has been dissolved in HCl with NH4OH as a precipitating agent is 97.69%.
2. Hematite iron oxide can be synthesized by the precipitation method, where the structure obtained by varying the volume ratio of PEG: Iron 1: 5 (mL / g) has cell parameters, namely, a = 5.036340 Å; b = 5.036340 Å; and c = 13.345420 Å, and has a space group R3 c with a trigonal crystal system.
3. Hematite iron oxide synthesized with variation of PEG:Iron volume ratio 1:5 (mL/g) has the best crystallinity with FWHM value of 0.2706, and an area of 0.17.
4. The crystal size of the synthesized hematite iron oxide calculated by the Debye Scherrer equation has a crystal size that is in the variation of the volume ratio of PEG:iron 1:5 (mL/g) of 50.9912 nm, 2:5 (mL/g) of 43.0883 nm, and 3:5 (mL/g) of 45.30663 nm. 5. The morphology of hematite iron oxide formed tends to agglomerate and has an inhomogeneous grain distribution.

4.2 Suggestion

Based on the results of the research that has been carried out, there are several things that can be of concern for conducting this research, namely further researchers to vary the calcination temperature to obtain optimum data resulting from synthesis products such as high crystallinity

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Contribution of Individual Authors to the Creation of a Scientific Article (Ghostwriting Policy)

-Edi Mikrianto extracted, synthesized, and analyzed the structure of hematite iron metal oxide.
-Rahmat Yunus conducted the analysis using XRF, XRD and SEM instrumentation.

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Conflict of Interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.