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PENGARUH VARIASI KOMPOSISI TERHADAP UKURAN KRISTAL LAPISAN NANOKOMPOSIT MnO-Fe_2O_3/PS SEBAGAI *SELF CLEANING*

Dafri Zaldi[1], Ratnawulan[2]*

[1] *Department of Physics, Padang State University, Jalan Prof. Dr. Hamka, Air Tawar Barat, Padang, West Sumatra, 25171, Indonesia*
[2] *Department of Physics, Padang State University, Jalan Prof. Dr. Hamka, Air Tawar Barat, Padang, West Sumatra, 25171, Indonesia*

Corresponding author. Email: [1]dafrizaldi312@gmail.com [2]ratnawulan320@fmipa.unp.ac.id

ABSTRACT

This research is based on the abundance of natural materials such as manganese and iron ore. Manganese is usually used for metal coating and has durability so that it is rich in properties to be used for various purposes in the chemical, pharmaceutical, and technological industries. Meanwhile, iron ore is a stable type of oxide so that it has strong properties and is not easily corroded. This research is a type of experimental research conducted at the Laboratory of Material Physics and Biophysics Faculty of Mathematics and Natural Sciences (FMIPA), Padang State University (UNP), and the Chemistry Laboratory of FMIPA UNP. The tools used are HEM-3D, XRD, and UV-VIS. The precursors were prepared by varying the composition of the ratio of MnO-Fe_2O_3 and PS respectively 0,2:0,2:1 ; 0,4:0,4:1 ; 0,6:0,6:1 ; 0,8:0,8:1 ; 1:1:1. This coating is made by means of the spin coating method and uses a temperature of 60 ° using a oven. The results of this study are the maximum crystal size obtained in MnO is 61.26 nm, Fe_2O_3 is 50.45 nm, and MnO-Fe_2O_3 is 53.08 nm. Also, the maximum% degradation produced is 65.97% in a methyl orange solution, this shows that the MnO-Fe_2O_3/PS layer is well degraded. This degradation ability is known as self cleaning because it is able to break down dirt.

Keywords : *composition; nanocomposites MnO- Fe_2O_3/PS; self cleaning.*

I. INTRODUCTION

Recently, the use of nanoparticle technology is increasing in the world of research. One technology that is currently trending is technology that has photocatalyst properties. Photocatalysts can be applied to beautify properties and aid in energy saving processes. This photocatalyst application is applied to be able to clean yourself from contaminants or known as self cleaning [1]. Self Cleaning is a method to utilize the photocatalyst or lotus effect of chemical compounds so that it is able to clean itself from adhering particles. The two properties possessed by the action of water are hydrophobic and hydrophilic. Hydrophobic is the nature of being afraid of water or water repellent [2]. Meanwhile, hydrophilic is a water-like nature. However, hydrophilic has additional properties, namely that it can chemically break down impurities that are adopted under sunlight, known as the photocatalyst process [3].

Hematite (Fe_2O_3) is the most stable type of metal oxide and has a hexagonal or rhombohedral crystal structure. Hematite has the characteristics of anti-rust properties and has a mass density so that it is not easily disturbed by external disturbances.

Fig.1. Crystal Structure of the Hematite [4]

Hematite nanoparticles have a wide surface shape and good resistance so that small hematite can increase stability in the metal [5]. On the other hand, hematite which has self-cleaning properties already exists but is not resistant to UV radiation. Therefore, researchers used an additional material, namely manganese oxide (MnO).

Manganese is an element that is found in the earth's crust after iron, aluminum, and copper. Manganese has a characteristic color like gray. Manganese can be used for various purposes such as dry batteries, ceramics, dyes, and so on. Manganese oxide can be prepared in various ways such as heating, filtering, and precipitating [6]. A number of properties can be changed through controlling the size of the material, adjusting the chemical composition, and controlling the interactions between the particles.

One of the conductive polymers used in the manufacture of coatings is Polystyrene (PS). Polystyrene is a polymer that has a light molecular weight and has a very fragrant styrene monomer. Polystyrene is a multifunctional polymer because it has the characteristics of being transparent, easy to dye, easy processing, strong and cheap. Due to these characteristics polystyrene can be used in various applications [7]. The shape of the polystyrene structure can be seen in Figure 2.

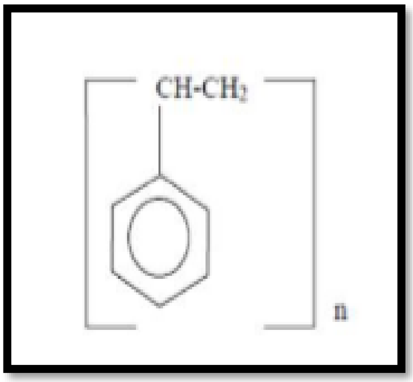

Fig. 2. Polystyrene structure [8]

The use of polystyrene is very much in the application because it has many characteristics so that it is easy to determine the crystal size, and the value can be expanded. The mechanical behavior of polystyrene is largely determined by its average molecular weight so strength increases with increasing chain length.

Many methods can be used in the growth of the MnO-Fe_2O_3/Ps layer. There are many ways to manufacture thin films, such as dip coating, sol gel, and spin coating because they are easy to do. Spin coating is a spinning method to spread the gel liquid evenly over the glass preparation with a certain time and speed [9]. This method is used because it is easy to do in the manufacture of thin films.

Based on the concept of this research, it can be seen how the effect of variations in the composition of the MnO-Fe_2O_3/Ps thin layer composition on crystal size as self cleaning. The manufacture of a thin nanocomposite layer is by combining two materials into one, which acts as a filler, namely MnO-Fe_2O_3 and acting as a matrix is Ps. the manufacture of this layer is made by the spin coating method. The parameter used in the study was the variation in composition. Composition is the amount of material used which will influence the properties in the manufacture of thin films. The properties that will affect in this study are the structure, crystal size, and so on. Determining this layer is self-cleaning, which is seen from the size of the crystal.

II. METHOD

This research is an experimental research. This study was studied to determine the effect of variations in the composition of $MnO-Fe_2O_3/Ps$ on the structure, phase, and crystal size tested using the X-Ray Difraction (XRD) characterization tool.

A. Tool

This research uses tools, namely; HEM-3D, digital scale, measuring cup, beaker, spin coating, ultrasonic cleaner, magnetic stirrer, glass substrate, litmus paper, syringe scale, XRD, and oven.

B. Ingredient

This study uses materials, namely; manganese oxide, iron ore, polystyrene, aquadest, tetrahydrofuran (THF), polyethylene glycol (PEG), and ethylene glycol.

C. Sample Preparation Stage

1. The Process of Making Manganese Nanoparticles

Preparing manganese to be used to obtain manganese oxide (MnO). Manganese is obtained from the warehouse of substances and materials in the Chemical Laboratory of Padang State University in the form of granules. Manganese (Mn) which is still in granular size is then refined again to get a tenorite scale using a HEM (High Energy Milling) tool for 20 hours.

2. The Process of making Iron Nanoparticles

Preparing iron ore to be used to obtain nanohematite (Fe_2O_3). Iron ore is obtained from the warehouse of substances and materials at the Chemical Laboratory of Padang State University in the form of granules. The iron ore (Fe_2O_3), which is still in granular size, is then refined to obtain the nanometer scale of hematite using a HEM (High Energy Milling) tool for 5 hours.

3. The Process of Making $MnO-Fe_2O_3/Ps$ Nanocomposite layer

Prepare 50 ml of Tetrahydrofuran (THF), 0.1 g PEG 400 and 0.5 g Polystyrene and vary the composition of MnO and Fe_2O_3 as much: 0.1 gram; 0.2 grams; 0.3 grams; 0.4 grams and 0.5 grams of each ingredient. Then put 50 ml of Tetrahydrofuran (THF), 0.1g PEG 400 and 0.5 g Polystyren into a 250 ml enlemeyer then heated using a magnetic stirrer at a speed of 250 rpm at 60 ° C. In the 15th minute, manganese oxide (MnO) was added with various compositions by doing five experiments. In the 25th minute then add iron ore (Fe_2O_3) with different composition variations with the same five experiments, the mixture is stirred until homogeneous for 60 minutes and produces a gel form.

4. Substrate Preparation

Nanocomposite coating using a glass preparation as a substrate. The preparatory glass is cut to a size of 1 cm × 1 cm. The glass was washed with alcohol using an ultrasonic cleaner for 2 hours. The purpose of this washing is to prevent dirt, dust, grease, and other particles from sticking to the substrate.

5. Coating Using the Spin Coating Method

The nanocomposite coating using the spin coating technique is carried out by dropping the nanocomposite gel on the substrate so that the entire surface is covered. The substrate is then rotated at a spin coating rate of 1000 rpm in 60 seconds. The sample was heated at 60 ° C using an oven.

D. Analysis Phase

The $MnO-Fe_2O_3/Ps$ layer was varied with 5 variations in the composition of MnO and Fe_2O_3 materials using the spin coating method to grow the layers with several characterization tests including the XRD test. Based on the value of angles 2 and I obtained in characterization using XRD, the crystal structure of the $MnO-Fe_2O_3$ / PS layer was analyzed and the crystal size and phase information were obtained from the results of the diffraction pattern analysis using HighScore Plus software. Based on the FWHM angle to calculate the crystal size using the Scherrer equation.

$$t = \frac{0{,}9 \times \lambda}{\mathrm{B} \times \cos\theta} \tag{1}$$

Keterangan:
t = crystal size (nm)
λ = the X-ray wavelength is worth 1,54 Å
θ = diffraction angle
B = maximum half-peak width FWHM (rad)

III. RESULTS AND DISCUSSION

This research is about the effect of MnO-Fe_2O_3 composition on the structure, phase, and crystal size of MnO-Fe_2O_3 / PS nanocomposite for each composition variation.

The Results of Characterization Using XRD

The results of characterization using XRD of the MnO-Fe_2O_3 /Ps nanocomposite layer at a composition of 0,2: 0,2: 1 are shown in Figure 3.

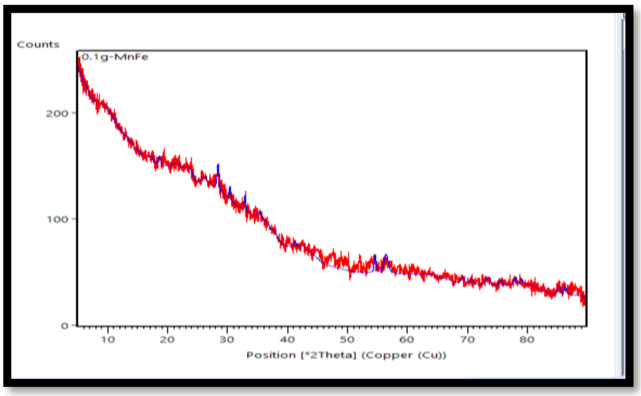

Fig. 3. The diffraction pattern of the MnO-Fe_2O_3 / Ps nanocomposite layer with a composition of 0,2: 0,2: 1

Figure 3 shows a diffraction pattern which shows the relationship between the intensity and the diffraction angle (2θ). The measurement data for the diffraction pattern of the MnO-Fe_2O_3 /Ps nanocomposite layer with a composition of 0,2: 0,2: 1 were obtained with the help of High Score Plus software.

Table 1. In the MnO-Fe_2O_3/Ps nanocomposite with a composition of 0,2: 0,2: 1

Angle (2θ)	Phase	Crystal Structure	Crystal Size (nm)
28,3823	*Pyrolusite*	*Tetragonal*	40,07
32,8543	*Hematite*	*Rhombohedral*	54,00
69,1311	*Hematite*	*Rhombohedral*	37,74
70,6235	*Jacobsite*	*Cubic*	47,61
73,3239	*Jacobsite*	*Cubic*	24,21
75,4837	*Hematite*	*Rhombohedral*	39,23
77,8809	*Hematite*	*Rhombohedral*	66,59
85,2173	*Hematite*	*Rhombohedral*	52,79

The results of characterization using XRD of the $MnO-Fe_2O_3$ /Ps nanocomposite layer at a composition of 0,4: 0,4: 1 are shown in Figure 4.

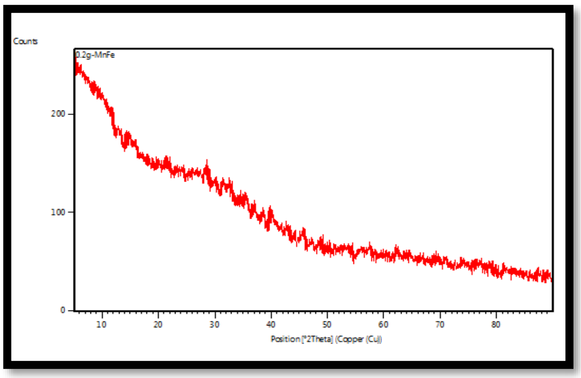

Fig. 4. The diffraction pattern of the $MnO-Fe_2O_3$ /Ps nanocomposite layer with a composition of 0,4: 0,4: 1

Figure 4 shows a diffraction pattern which shows the relationship between the intensity and the diffraction angle (2θ). The measurement data for the diffraction pattern of the $MnO-Fe_2O_3$ /Ps nanocomposite layer with a composition of 0,4: 0,4: 1 were obtained with the help of High Score Plus software.

Table 2. In the $MnO-Fe_2O_3$/Ps nanocomposite with a composition of 0,4: 0,4: 1

Angle (2θ)	Phase	Crystal Structure	Crystal Size (nm)
28,5934	*Pyrolusite*	*Tetragonal*	32,07
35,5613	*Jacobsite*	*Cubic*	27,20
45,8260	*Hematite*	*Rhombohedral*	24,10
48,8244	*Hematite*	*Rhombohedral*	28,44
53,6100	*Pyrolusite*	*Tetragonal*	21,76
62,5019	*Hematite*	*Rhombohedral*	36,35
81,5028	*Jacobsite*	*Cubic*	25,64
87,3922	*Jacobsite*	*Cubic*	42,98
88,4039	*Jacobsite*	*Cubic*	72,25

The results of characterization using XRD of the $MnO-Fe_2O_3$ /Ps nanocomposite layer at a composition of 0,6: 0,6: 1 are shown in Figure 5.

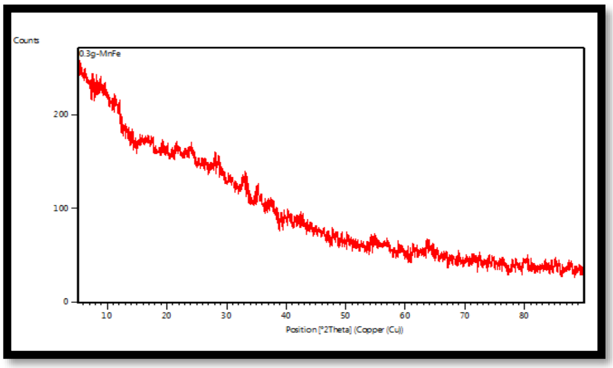

Fig. 5. The diffraction pattern of the $MnO-Fe_2O_3$ /Ps nanocomposite layer with a composition of 0,6: 0,6: 1

Figure 5 shows a diffraction pattern which shows the relationship between the intensity and the diffraction angle (2θ). The measurement data for the diffraction pattern of the $MnO-Fe_2O_3$ /Ps nanocomposite layer with a composition of 0,6: 0,6: 1 were obtained with the help of High Score Plus software.

Table 3. In the $MnO\text{-}Fe_2O_3$/Ps nanocomposite with a composition of 0,6: 0,6: 1

Angle (2θ)	Phase	Crystal Structure	Crystal Size (nm)
32,9814	*Hematite*	*Rhombohedral*	27,01
35,3185	*Jacobsite*	*Cubic*	20,38
46,3653	*Jacobsite*	*Cubic*	48,29
59,1757	*Pyrolusite*	*Tetragonal*	29,78
64,0792	*Jacobsite*	*Cubic*	30,55
65,3137	*Jacobsite*	*Cubic*	61,52
76,5234	*Pyrolusite*	*Tetragonal*	24,74
80,1461	*Jacobsite*	*Cubic*	33,84
85,1723	*Jacobsite*	*Cubic*	70,34
87,8388	*Jacobsite*	*Cubic*	53,94

The results of characterization using XRD of the $MnO\text{-}Fe_2O_3$ /Ps nanocomposite layer at a composition of 0,8: 0,8: 1 are shown in Figure 6.

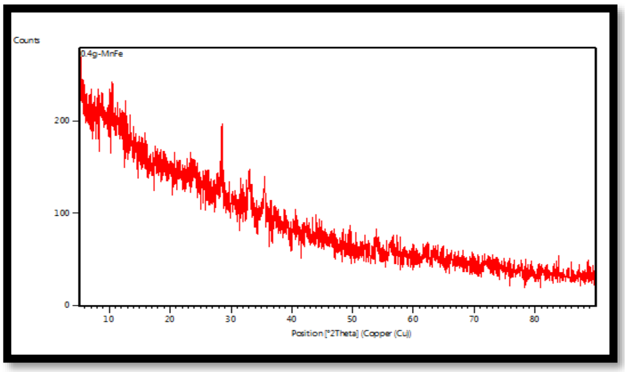

Fig. 6. The diffraction pattern of the $MnO\text{-}Fe_2O_3$ /Ps nanocomposite layer with a composition of 0,8: 0,8: 1

Figure 6 shows a diffraction pattern which shows the relationship between the intensity and the diffraction angle (2θ). The measurement data for the diffraction pattern of the $MnO\text{-}Fe_2O_3$ /Ps nanocomposite layer with a composition of 0,8: 0,8: 1 were obtained with the help of High Score Plus software.

Table 4. In the $MnO\text{-}Fe_2O_3$/Ps nanocomposite with a composition of 0,8: 0,8: 1

Angle (2θ)	Phase	Crystal Structure	Crystal Size (nm)
33,0649	*Hematite*	*Rhombohedral*	40,53
35,5164	*Jacobsite*	*Cubic*	54,39
37,4317	*Jacobsite*	*Cubic*	54,67
39,1192	*Hematite*	*Rhombohedral*	27,48
42,2834	*Pyrolusite*	*Tetragonal*	27,77
51,7701	*Hematite*	*Rhombohedral*	21,59
53,9378	*Jacobsite*	*Cubic*	43,59
56,3797	*Pyrolusite*	*Tetragonal*	29,38
59,4555	*Jacobsite*	*Cubic*	59,65
64,7880	*Pyrolusite*	*Tetragonal*	46,01
77,5541	*Hematite*	*Rhombohedral*	33,22

The results of characterization using XRD of the MnO-Fe_2O_3 /Ps nanocomposite layer at a composition of 0,8: 0,8: 1 are shown in Figure 7.

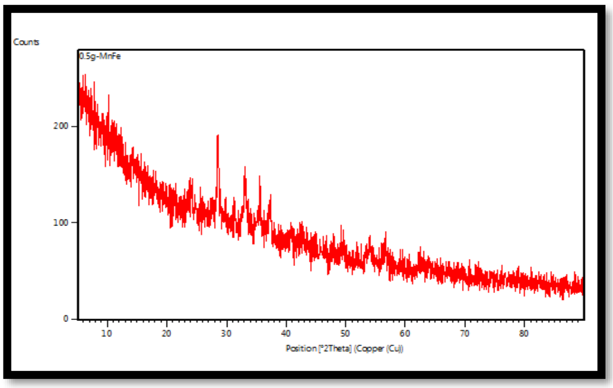

Fig. 7. The diffraction pattern of the MnO-Fe_2O_3 /Ps nanocomposite layer with a composition of 1: 1: 1

Figure 7 shows a diffraction pattern which shows the relationship between the intensity and the diffraction angle (2θ). The measurement data for the diffraction pattern of the MnO-Fe_2O_3 /Ps nanocomposite layer with a composition of 1: 1: 1 were obtained with the help of High Score Plus software.

Table 5. In the MnO-Fe_2O_3/Ps nanocomposite with a composition of 1: 1: 1

Angle (2θ)	Phase	Crystal Structure	Crystal Size (nm)
18,2260	*Jacobsite*	*Cubic*	31,48
33,1849	*Hematite*	*Rhombohedral*	54,05
49,5915	*Hematite*	*Rhombohedral*	28,53
54,0345	*Hematite*	*Rhombohedral*	58,14
63,9449	*Hematite*	*Rhombohedral*	61,06
64,5667	*Pyrolusite*	*Tetragonal*	61,26
72,0705	*Hematite*	*Rhombohedral*	32,03
74,4400	*Jacobsite*	*Cubic*	48,79
86,9845	*Jacobsite*	*Cubic*	53,55

XRD can determine the diffraction pattern of light on a sample. X-ray diffraction on the MnO-Fe_2O_3/Ps nanocomposite layer with various compositions. In analyzing the researcher used the highscore plus application to determine the 2 theta peak and intensity, to combine the plots from XRD data the researcher used the Origin 8.5 application, the following is a plot of the data results from XRD using the Origin 8.5 application in Figure 8.

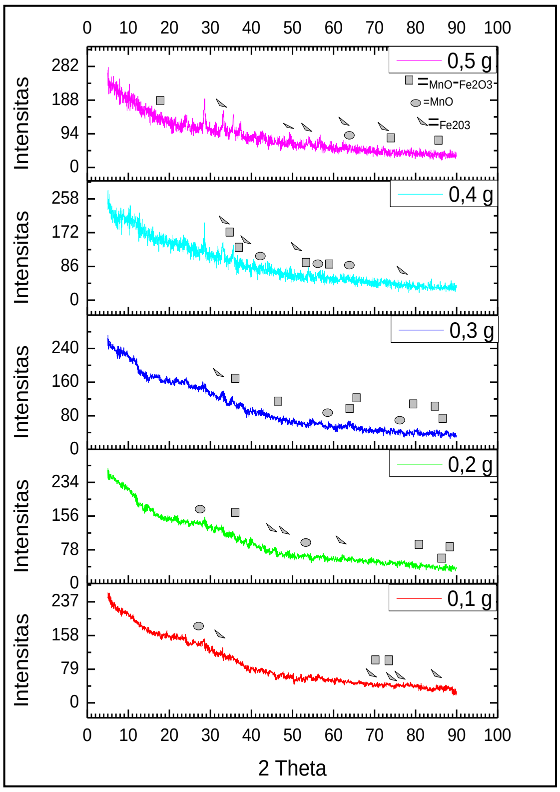

Fig. 8. XRD data analysis

Figure 8 shows the phases that arise from the MnO-Fe_2O_3 / PS nanocomposite sample for each variation in the composition. The phases found are Pyrolusite (MnO), Hematite (Fe_2O_3), Jacobsite (MnO-Fe_2O_3), each phase that appears has a different intensity. The crystal size of the MnO-Fe_2O_3 / PS nanocomposite layer can be determined using the Scherrer equation in Figure 9.

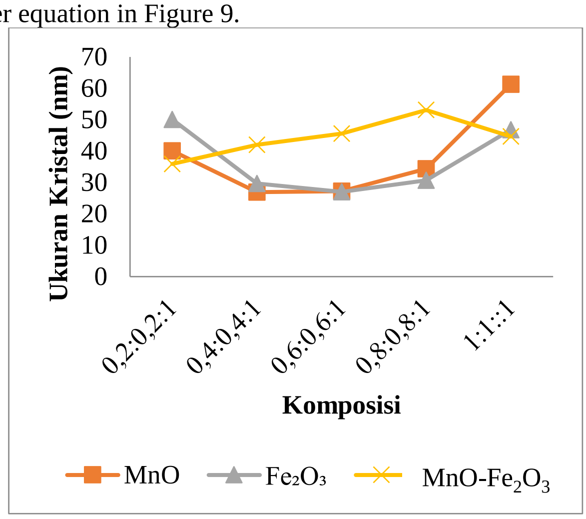

Fig. 9. Effect of composition variations on the mean crystal size

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Figure 9 shows the effect of the composition on the crystal size of the MnO-Fe_2O_3/Ps nanocomposite layer. The figure shows that when the composition is increased, the crystal size of MnO-Fe_2O_3 increases and decreases at a 1: 1: 1 composition. Meanwhile, MnO and Fe_2O_3 decreased in crystal size from a composition of 0,2: 0,2: 1 to 0,6: 0,6: 1 and increased from a composition of 0,6: 0,6: 1 to 1: 1: 1.
The crystal structure for each phase is MnO with a Tetragonal structure, Fe_2O_3 with a Rhombohedral structure, and MnO-Fe_2O_3 with a Cubic structure. The test results showed the crystal sizes of MnO, Fe_2O_3, and MnO-Fe_2O_3. In the variation of composition 0,2-1, the average crystal size is Fe_2O_3 crystal size: 50,07 nm, 29,63 nm, 27,01 nm, 30,71 nm, and 46,47 nm, crystal size. MnO: 40,07 nm, 26,91 nm, 27, 26 nm, 34,39 nm, and 61,26 nm,

and MnO-Fe_2O_3 sizes: 35,91 nm, 42,02 nm, 45,55 nm, 53,08 nm, and 44,41 nm. The size of the crystals can change due to different variations in composition. This can be seen from the FWHM value of XRD characterization. The FWHM value affects the crystal phase quality [10]. The smaller the crystal size because it has a high FWHM value [11]. The difference in the resulting peaks occurs because of the difference between the intensity and width of the peaks from the resulting diffraction angles. The difference in intensity and peak width from the diffraction angle is due to the change in crystal size [12] From these observations, the XRD test results have an effect in determining the crystal size, structure, and phase.

IV. CONCLUSION

Based on the research conducted, it can be seen that the variation in composition affects the crystal size, structure, and phase. The maximum crystal size obtained for MnO was 61,26 nm, Fe_2O_3 was 50,45 nm, and MnO-Fe_2O_3 was 53,08 nm. The crystal structures found are Cubic, Rhombohedral, and Tetragonal. This nanocomposite layer is self-cleaning because it uses polystyrene polymers as water repellent and is easy to escape from the dirt that sticks to it.

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