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THE EFFECT OF COMPOSITION $MnFe_2O_4$/PANi NANOCOMPOSITE ON THE MICROSTRUCTURE THAT SYNTHESIZED BY SPIN COATING METHOD

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ABSTRACT

Manganese ferrite has the structure of MnFe2O4 which in the process of making it uses manganese mineral powder. Based on the MnFe2O4 testing is one material that is suitable for use as a composite by mixing other binders in it. Polyaniline (PANi) is a polymer that can be used as a binding material which is an attractive conductive polymer because it has unique properties and good thermal stability. This research was conducted by making three variations of the composition of MnFe2O4 / PANi namely 40%: 60%, 50%: 50%, and 60%: 40% made using the spin coating method. This research was conducted to investigate the effect of composition on the microstructure of the MnFe2O4 / PANi nanocomposite layer using the spin coating method. The microstructure to be investigated is the functional group of grain size and thickness of the nanocomposite layer. The tools used in this study are Fourier Transform Infrared (FTIR) used to obtain the functional groups of nanocomposite layers, X-ray Diffraction (XRD) is used to get the size of crystals and microstructure of crystals, and the Scanning Electron Microscope (SEM). Characterization and Scanning Electron Microscope (SEM) are used to obtain grain size and thickness of the nanocomposite layer. The results of the FTIR characterization are the functional groups of MnFe2O4 / PANi which produce absorption bands at wavenumbers 3214.85 cm-1, 3353.86 cm-1, and 3214.03 cm-1, which are O-H groups. The absorption band at wavenumbers 717.00 cm-1, 763.94 cm-1, and 747.31 cm-1 is the C-H group showing PANi. Absorption peaks that are below 1000 cm-1 that is at wavenumbers 874.78 cm-1, 924.18 cm-1, and 895.96 cm-1 show indications of Manganese Ferrit. The results of the XRD characterization were crystal size and microstrain, each of which had a crystal size composition of 49.90478417 nm, 45.29656118 nm, and 44.52213202, and then for the value of the microstrain, each variation was 0.116667149, 0.15983276, and 0.183718732. Then from SEM characterization results obtained grain size values of 0.445 μm, 0.426 μm, 0.318 μm, while the thickness obtained for each variation is 1.29 μm, 2.02 μm, and 2.20 μm. Based on the results of the study, the greater the addition of PANi composition given, the value of crystal size, grain size also increases while the value of microstructure and thickness decreases.

Keywords : Spin coating, nanocomposite, MnFe2O4 /PANi, thin film, micro structure.

I. INTRODUCTION

Nanocomposites are materials derived from a mixture of powdered nanoparticles, which are formed from two combinations, namely a matrix as a reinforcement for filler [1]. Nanocomposites themselves have new properties that are superior to the constituent materials [2]. The nanocomposite itself can be a thin layer that can be made using the spin coating method, where the formation of a thin layer is done by dropping a gel or solution on a substrate placed on a disk that can be rotated at a certain speed. After the substrate is rotated at a constant speed, the solution will spread to the edge of the substrate [3]. The method of making this thin layer can be applied to material processing in the form of iron sand made with soft ferrite, namely manganese ferrite.

MnFe2O4 is the structure of manganese ferrite which in the manufacturing process uses manganese mineral powder. Manganese has a high melting point of 1244oC so that it can withstand high heat treatment [4]. MnFe2O4 has several advantages such as; the material is a lightweight, low cost, design flexibility, and has wave-absorbing properties [5]. Another advantage of manganese ferrite is that it has strong mechanical properties, is not easily corroded, and has a fairly good temperature [6].

Manganese ferrite has high magnetic permeability which makes its application in many electronic devices, such as; microwave devices, computer memory chips, data storage media, radiofrequency coil fabrication, and transformer cores [7]. Ferrite material has a crystal structure which is a combination of ions that have magnetic abilities, which is the ability to come from magnetic ions called cations. The properties of manganese ferrite depend on the composition, morphology, and particle size, which are closely related to the preparation conditions [8]. Based on its crystal structure, manganese ferrite belongs to the cubic spinel crystal structure [9]. The cubic structure, spinel itself can be seen in Figure 1 below.

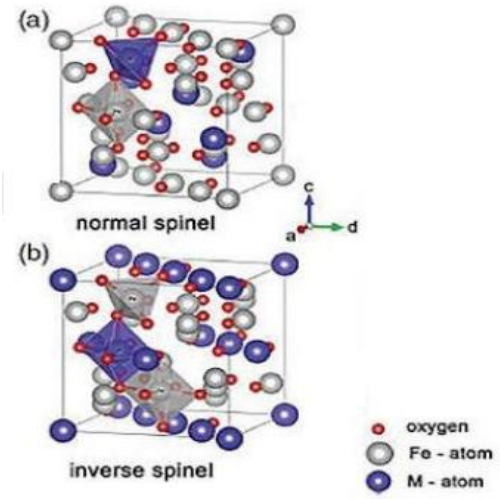

Fig. 1. Cubic spinel structure [10]

The spinel crystal structure has two sub-structures, namely the tetrahedral structure shown in Figure 1 (a) and the octahedral structure in Figure 1 (b). It can be seen in the tetrahedral section (a) In the center of the tetrahedron there are metal ions and the corners are occupied by oxygen ions, then in the octahedral (b) the monometallic is in the center of the octahedron and the corners are occupied by oxygen ions [10]. Figure 2 is a picture of the crystal structure of MnFe2O4.

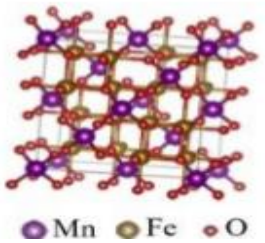

Fig. 2. Crystal structure of MnFe2O4 [11]

Figure 2 is a form of the MnFe2O4 crystal structure obtained by analyzing the resulting diffractogram using the working principle of x-ray diffraction. Mathematically it can be seen in the equation:

$$2\, d_{hkl} \sin\theta = \lambda \qquad (1)$$

Where:
n = integers 1,2,3,4, …
λ = wave length
dhkl = distance between grid planes
θ = diffraction angle

The microstructure obtained from characterization using X-rays is the crystal size (D) and micro-strain (ε) which can be found using the *Scherrer* equation in which equation (2) is for crystal size and equation (3) is for micro-strain [12].

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (2)$$

$$\varepsilon = \frac{\beta}{4 \tan \theta} \quad (3)$$

Based on several tests carried out, the nanocomposite is a material that is formed from two combinations, namely the matrix as a reinforcement (binder) and filler as the main material [1]. The polymer material is one of the binders that can be used in making composites [13]. Polyaniline (PANi) is a polymer that can be used as a binder because of its unique characteristics and good thermal stability and is easily synthesized electronically and chemically [14]. Polyaniline is obtained from the aniline polymerization process, aniline is an organic compound with the composition of C6H5NH4 which is a group of aromatic compounds that have a molecular weight of 93 g / mol, a boiling point of 183oC-186oC, a refractive index of 1.58, and a mass density of 1.002 kg/liter [15]. In this research, MnFe2O4 / PANi nanocomposite will be made, in which PANi will act as a matrix, namely the reinforcement of the filler (MnFe2O4).

The preparation of MnFe2O4 / PANi nanocomposites has the advantage of efficient photothallitic activity and excellent stability in the photocatalytic decomposition of RhB under visible light radiation [16]. In another study, this nanocomposite can also be applied to supercapacitor electoral because it shows a specific increase in capacitance and also has good stability [17]. A supercapacitor is an energy storage device that can be applied in the field of electromagnetism and transportation, which type of capacitor consists of double layers to store charge transfer energy that occurs at the electrodes and electrolytes [18]. The quality of the supercapacitor depends on the constituent material of the supercapacitor, the storage capacity will increase if it has a large surface area and the crystal size is in the nanometer order. The electrode material used will affect the filling mechanism of the supercapacitor material. The electrode materials that can be used in supercapacitors consist of 3 categories, namely carbon materials (activated carbon), conductive polymers, and transition metal oxides [19]. Ferrite metal is one of the transition metal oxides that has been widely used by researchers because it has good magnetic and electrical properties. Spinel ferrite with the formula MFe2O4, where M is a divalent metal ion that consists of several more materials such as Ni, Co, Cu, Mn, Mg, Zn, and Fe which all have the same cubic spinel structure [20].

In this study, several characterization tools were used, namely Fourier Transform Infrared (FTIR), X-Ray Diffraction (XRD), and Scanning Electron Microscope (SEM). FTIR is used to see the functional groups contained in the nanocomposite layer by paying attention to the amount of wave number absorption that occurs due to infrared light emission. Based on these wavelengths the infrared region is divided into 3 parts, namely, near IR sensitive to overtone vibrations (14000-4000 cm-1), IR is in charge of providing information about functional groups in a molecule (4000-400 cm-1) and far IR for analyzed molecules containing heavy atoms (400-10 cm-1) [21]. The working principle of an FTIR spectrophotometer is the interaction between energy and matter where infrared light will shine on the sample through a gap, where the gap functions to control the amount of energy supplied to the sample. Part of the infrared light that reaches the sample will be absorbed and partially directed to be transmitted through the sample surface to the detector and the signal received by the detector will be forwarded to be displayed to the computer [22].

XRD is the use of X-ray diffraction to identify the crystal structure of a material. The components of XRD consist of 3 parts, namely, the source of X-Ray rays, specimens, and X-Ray detectors. The schematic of XRD work can be seen in Figure 3.

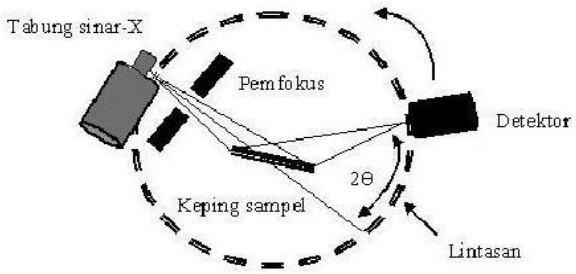

Fig. 3. Schematic of XRD [23]

Figure 3 is a schematic design of XRD based on Bragg's analysis, where the X-ray hits the specimen with an angle θ then the detector records the scattering angle and when is changed the detector will record its peak intensity visualized in the form of a diffractogram [23]. Scanning Electron Microscope (SEM) is used to determine the microstructure of solid objects including porosity and crack shape using electron rays. It should be noted that the material that is aerated using SEM must be a conductor because it will interact with electrons and if the material is not conductor then it must be coated first [24]. The working principle of SEM can be seen in Figure 4, can be seen that SEM has an electron gun to produce an electron beam, then a magnetic lens focuses the electrons towards the sample then the electron beam will scan the entire sample which is directed by the scanning coil, if the electrons hit the sample, new electrons will come out and received by the detector which is then sent to the monitor [25].

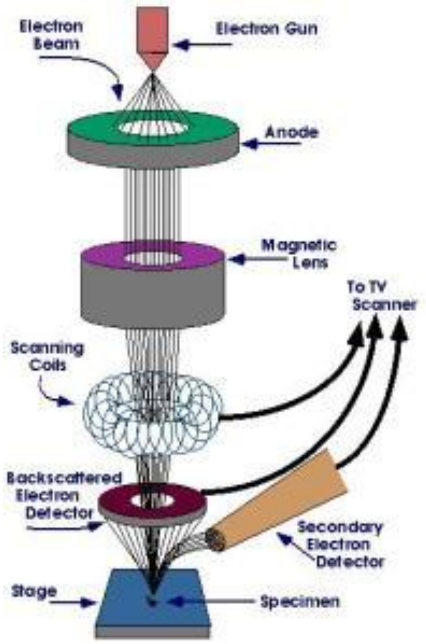

Fig. 4. SEM work scheme [25]

II. METHOD

This research is experimental research to make $MnFe_2O_4$ / PANi nanocomposite layer which is done in several stages. In this study there are 3 types of variables, namely the independent variable which states the ratio of the composition of $MnFe_2O_4$ with PANi, namely (40:60)%, (50:50)%, (60:40)% with a total of 100% of each variation is 20 ml [26]. Then the control variables are spin coating speed and milling time for 30 hours [27], the last is the dependent variable, namely functional group, crystal size, micro-strain, grain size and thickness of the $MnFe_2O_4$ / PANi nanocomposite layer.

In the implementation of the research, there are several stages, namely the preparation stage, sample preparation, characterization stage, data collection stage, and data analysis stage. The preparation stage is the stage of theoretical assessment of the research topic being carried out. The sample preparation stage is the stage of making the sample, consisting of several steps, namely sample purification, Fe_3O_4 precursor manufacture, $MnFe_2O_4$ precursor manufacture, PANi manufacturing process, $MnFe_2O_4$/ PANi nanocomposite manufacturing, $MnFe_2O_4$/PANi nanocomposite thin-film manufacturing process, each of which will be explained. The purification of the iron sand sample uses a permanent magnet which is subjected to 30 times the pull, this is done so that the iron sand obtained is good. After that, the iron sand is sifted using a 100 mesh sieve and then washed using distilled water. After washing is done again pull the magnet 20 times. When the milling process has been completed on the sample using a High Energy Milling (HEM) tool for 30 hours, this is done to obtain the magnetic homogeneous phase of the sample. The milling process is carried out for 3 days in which the milling process is carried out for 10 hours per day [27]. If the milling process is complete the sample is again purified using aquabidest.

The process of making Fe_3O_4 is carried out by reacting 3.48 grams of Fe_3O_4 and 0.9 grams of oxalic acid with 8.4 grams of nitric acid, then these three ingredients are stirred for 20 minutes at a speed of 250 rpm at 110^oC

after that add 28.5 ml of ethylene glycol into the solution and return the stirrer for 2 hours at 80^{o}C using a speed of 250 rpm [28]. The process of making the $MnFe_2O_4$ precursor is carried out by mixing manganese nitrate with the iron solution that has been made, the mixing ratio of the two ingredients is Mn2 +: Fe3 + = 1: 2, this material is mixed while heated at 70^{o}C for 1 hour at 375 rpm. After that, add citric acid with a mole ratio of M: citric acid = 1: 1.5 in a stirrer for 1 hour at a constant speed at 70^{o}C until the material forms a gel. Then the gel was washed using ultrasonic and dried in an oven at 120^{o}C for 24 hours to obtain $MnFe_2O_4$ [29].

The manufacturing stage of PANi was carried out by mixing 0.46565 g of PANi with 0.57845 gr of ammonium persulfate and 50 ml of nitric acid, these three ingredients were stirred for 2 hours at a speed of 250 rpm and a temperature of 20^{o}C [30]. The next process is to make $MnFe_2O_4$/PANi nanocomposite with various composition variations. The variation used was (40:60)%, (50:50)%, (60:40)% with a total of 100% of the material was 20 ml. The method used is to hold each $MnFe_2O_4$ and PANi in each variation. PANi according to the desired variation in the stirrer with 0.05066 g of ammonium persulfate for 15 minutes at 20^{o}C, after that mix the manganese according to the variation and stirrer again for 48 minutes at 80^{o}C [30].

The next process is to make a thin layer of $MnFe_2O_4$/PANi nanocomposite using the spin coating method. This is done by dropping the $MnFe_2O_4$/PANi sol solution on the glass substrate and rotating it with a spin coating at 1000 rpm for 10 seconds [28]. The magnetite thin layer that has been formed is dried in the oven for 15 minutes at 110^{o}C. after that, the thin films were annealed at 300^{o}C [31] for 3.5 hours [32]. Furthermore, the characterization stage was carried out for each variation of the $MnFe_2O_4$/PANi nanocomposite. Characterization was carried out using 3 tools, namely *Fourier Transform Infrared* (FTIR) which was used to see the functional groups in the nanocomposite. This test was carried out at the Chemical Laboratory of FMIPA UNP, *X-Ray Diffraction* (XRD) was used to see the crystal structure of the test from the Physics Laboratory of FMIPA UNP and *Scanning Electron Microscope* (SEM), which is used to see surface morphology and grain size, testing was carried out at the Bandung Geology Laboratory.

III. RESULTS AND DISCUSSION

From research conducted on $MnFe_2O_4$/PANi nanocomposites, the following results and discussions were obtained:

A. The results of characterization and discussion of $MnFe_2O_4$/PANi nanocomposites using FTIR

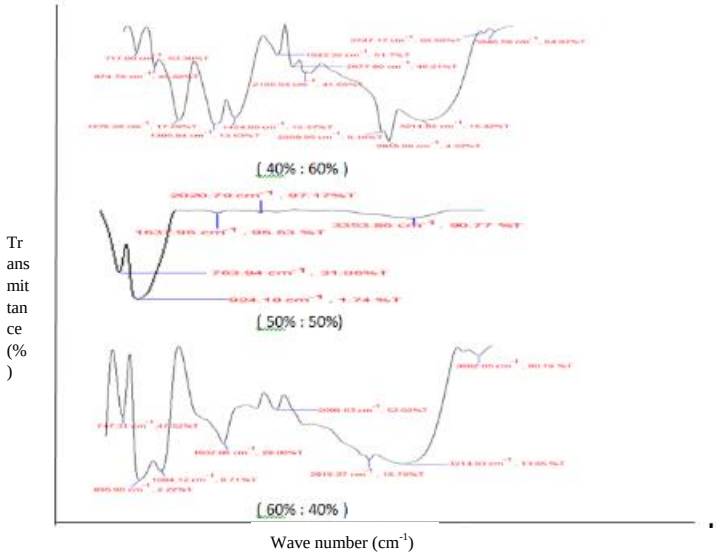

Fig. 5. Characterization results of 3 variations of the $MnFe_2O_4$ /PANi nanocomposite

Figure 5 is the results of the FTIR characteristics. The results of FTIR characteristics can be used to see the functional groups of the MnFe2O4 / PANi nanocomposite layer for each composition (40:60)%, (50:50)%, and (60:40)%. From the test results, it can be seen that the p absorption band at the numbers 3214.85 cm-11, 3353.86 cm-11, and 3214.03 cm-11 are the wavenumbers that show the state of the O-H group. The absorption bands found in the wavenumbers 717.00 ccm-1, 763.94 cm-11, and 747.31 cm-11 are C-H groups indicating PANi. This statement is following by Susmita, et al. [33] which states that the O-H functional group is in the

wavenumber 3000-3600 cm-1 and the wave number C-H is in the number 675-870 cm-1. Then the absorption bands in the wavenumbers 874.78 cm-1-, 924.18 cm-1- and 895.96 cm-1 indicated an indication of Manganese Ferrite. This statement is following that stated by Jaintha, et al. [34] who said that the absorption peak which is below 1000cm-1 indicates an indication of manganese ferrite.

B. The results of characterization and discussion of $MnFe_2O_4$ / PANi nanocomposites using XRD

1) X-ray diffraction pattern of $MnFe_2O_4$ /PANi nanocomposite at variation (40:60)%

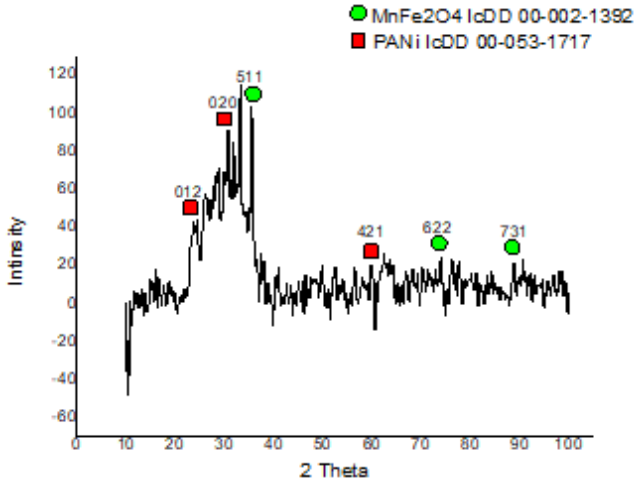

Fig. 6. Diffraction pattern of MnFe2O4 / PANi nanocomposite at various compositions (40:60)%

Figure 6 shows the diffraction pattern of the thin $MnFe_2O_4$/PANi nanocomposite layer. In this process, it can be seen that the results of the deposition formed are six peaks, namely with an angle of 23.2731°, 31.0991°, 36.9491°, 58.6071°, 74.9351°, 89.3391° with FWHM 0.3070°, 0.3070°, 0.3582°, 0.6140°, 0.3070°, 0.4368°. With the *HighScore Plus software*, it can be identified the peak miller index associated with the phases that appear are (012), (020), (511), (421), (622), (731). The average lattice parameters on the ICDD code 00-002-1392 and ICDD code 00-053-1717.

2) X-ray diffraction pattern of $MnFe_2O_4$/PANi nanocomposite with variation in composition (50:50)%

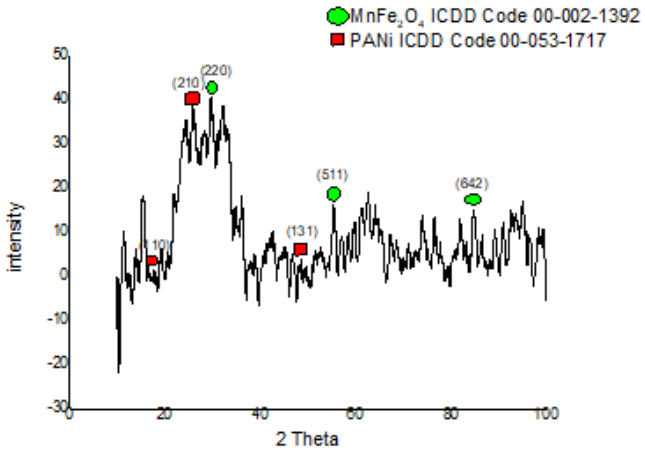

Fig. 7. X-ray diffraction pattern of $MnFe_2O_4$ / PANi nanocomposite variation in composition (50:50)%

In Figure 7, the diffraction pattern of the thin layer of $MnFe_2O_4$ nanocomposites analyzed with the *HighScore Plus software*, which in this process shows that there are six peaks formed with an angle of 19.0871°, 23.3251°, 29.6431°, 48.4411°, 55.6171°, 85.1011° with FWHM 0.3070°, 0.5117°, 0.3070°, 0.3070°, 0.5117°, 0.5117°, with the peak miller index associated with the phase that appears are (110), (210), (220), (131), (511), (642). The average lattice parameters on the ICDD code 00-002-1392 and ICDD code 00-053-1717.

3) X-ray diffraction pattern of $MnFe_2O_4$/PANi nanocomposite at variation (60:40)%

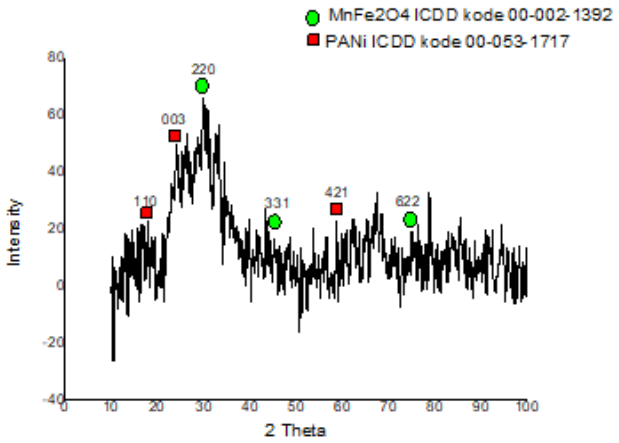

Fig. 8. X-ray diffraction pattern of $MnFe_2O_4$/PANi nanocomposite variation in composition (60:40)%

Figure 8 shows the diffraction pattern of the thin $MnFe_2O_4$/PANi nanocomposite layer. In this process, it can be seen that the results of the deposition formed are four peaks, namely with an angle of 19.2951°, 26.1331°, 29.6691°, 46.4651°, 58.6331°, 73.7911° with FWHM 0.307°, 0.8187°, 0.4093°, 0.307°, 0.307°, 0.5117°. With the *HighScore Plus software*, the peak miller index associated with the emerging phase can be identified, (110), (003), (220), (331), (421), (622). The average lattice parameters on the ICDD code 00-002-1392 and ICDD code 00-053-1717.

Based on the data obtained, it was found that the FWHM value was used to calculate the crystal size and micro-strain values. After calculating both using the *Scherrer* equation, the values for each variation are obtained as shown in Table 1.

Table 1. Values for crystal size and micro strains

Variation in composition	Crystal size	Micro Strains
(40: 60)%	49,90478417	0,116667149
(50:50)%	45,29656118	0,15983276
(60:40)%	44,52213202	0,183718732

From table 1 it can be seen that the greater the addition of the concentration of PANi addition in the MnFe2O4 material, the higher the crystal size value obtained, and whereas for the micro strains the more the addition of the PANi concentration in MnFe2O4, the smaller the value of the micro-strains. This statement is consistent with what Nurzam et al. [35] stated that the greater the variation in the filler concentration in the matrix, the smaller the crystal size will be. The two values are influenced by the FWHM value.

C. The results of characterization and discussion of $MnFe_2O_4$/PANi nanocomposites using SEM

The surface morphology of the $MnFe_2O_4$/PANi nanocomposite at each variation of the composition (40%: 60%), (50%: 50%), and (60%: 40%) showed the presence of grain size and the resulting homogeneity of the nanocomposite surface, from the comparison of SEM images to The surface morphology of the nanocomposites at each composition variation can be seen in Figure 9.

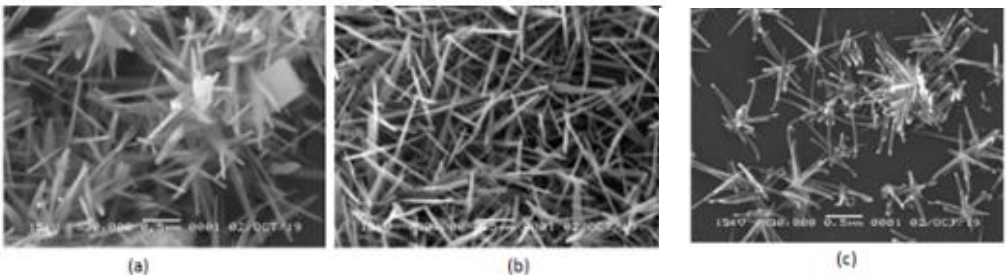

Fig. 9. Morphological form of $MnFe_2O_4$ / PANi

The shape of the morphology is influenced by the thickness of the material, the homogeneity of the material, the amount of substance that is dropped, and the thickness of the layer [3]. The results of morphological imaging were used to obtain nanocomposite grain size. The grain sizes obtained for each variation in the composition, namely, at 40%: 60%, 50%: 50%, and 60%: 40% were 0.445m, 0.426m, and 0.318m. Based on the resulting data for grain size values, it can be seen that the more MnFe2O4 is given to the material, the smaller the grain size value and vice versa. The shape of the morphology changes at each variation in composition, because the process of making the solution is too watery. The effect of the inequality of the amount of substance that is dropped, the homogeneity factor, and the flatness of the layer will also affect the morphological shape of a material [3]. The surface section of the $MnFe_2O_4$/PANi nanocomposite at each composition variation of 40%: 60%, 50%: 50%, and 60%: 40% shows the size of the surface thickness of the nanocomposites produced from each material. Based on the comparison of the results of the SEM images, it can be seen that the surface section of each variation can be seen in Figure 10.

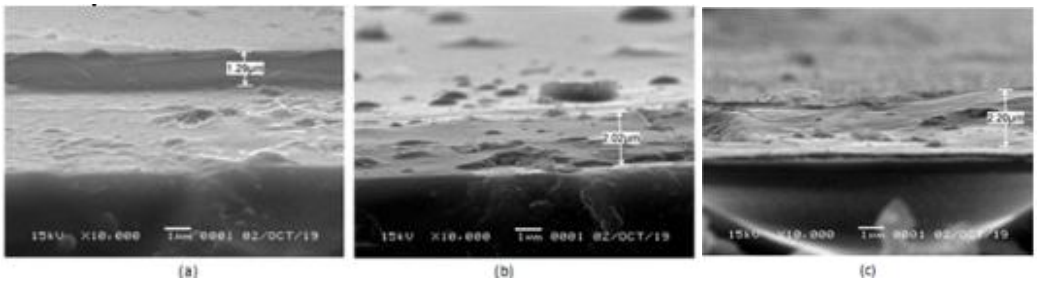

Fig. 10. SEM image results cross section of the $MnFe_2O_4$/PANi nanocomposite surface. (a) variation (40:60)%, (b) variation (50:50)% (c) variation (60:40)%

From Figure 9, it can be seen that the morphological form of the $MnFe_2O_4$ In Figure 10, it can be seen that the results of the SEM image of the surface section of the $MnFe_2O_4$/PANi nanocomposite obtained the size of the surface thickness for each variation of variation (40:60)% of 2.20 μm, variation (50:50)% of 2.02 μm, and variations (60:40)% 1.29 μm. Broadly speaking, it can be seen that the greater the variation in the composition of the addition of PANi to $MnFe_2O_4$, the greater the layer thickness obtained. This statement is consistent with what Sahanaya [36] did with Fe_3O_4/PANi which obtained greater thickness results as the variation in the composition of the material increased. This difference in thickness can be caused by differences in the mass and concentration of the gel that is dropped on the glass substrate when doing the spin coating process in each variation is not the same.

IV. CONCLUSION

The process of making the $MnFe_2O_4$/PANi nanocomposite layer using a spin coating tool has been successfully carried out. Based on the results of the functional group test, it can be said that the $MnFe_2O_4$/PANi layer includes a nanocomposite layer with almost the same wavenumbers for each variation. The absorption bands of the wavenumbers 3214.85 cm-^{1}, 3353.86 cm^{-1}, and 3214.03 cm^{-1} are the wavenumbers showing the state of the O-H group. The absorption bands at the wavenumbers of 717.00 cm^{-1}, 763.94 cm^{-1}, and 747.31 cm^{-1} are the CH groups indicating PANi while the absorption bands at the values of 874.78 cm^{-1}, 924.18 cm^{-1}, and 895.96 cm^{-1} indicate Manganese Ferrite.

In the next XRD characterization, the calculation was carried out using FWMH data on $MnFe_2O_4$/PANi which obtained the results of the crystal size and micro-strains which were inversely proportional. The greater the addition of the PANi concentration to $MnFe_2O_4$, the greater the crystal size value obtained, while for the micro strains the smaller the addition of PANi to $MnFe_2O_4$ is enlarged. In the grain size, the less PANi added to $MnFe_2O_4$, the smaller the grain size value. As for the thickness, the less variation in the addition of PANi to $MnFe_2O_4$, the greater the resulting thickness.

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