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THE EFFECT OF COMPOSITION OF $NiFe_2O_4$ ON THE CRYSTAL STRUCTURE OF NANOCOMPOSITE $NiFe_2O_4$ / PVDF THAT SYNTHESIZED USING SOL GEL SPIN COATING METHOD

Yessy Arinda Putri[1], Ramli[1*]

[1] *Department of Physics, Faculty of Mathematics and Sciences, Universitas Negeri Padang, Jalan Prof. Dr. Hamka, Air Tawar Barat, Padang, West Sumatera, 25171, Indonesia*

Corresponding author. Email: ramli@fmipa.unp.ac.id

ABSTRACT

$NiFe_2O_4$ is a Ferrite Compound with the molecular formula MFe where M is a transition metal or alkaline earth is a magnetic semiconductor material with a spinel structure. The polymer used in this study is Polyvinilidene Flouride (PVDF). PVDF film is a material that is inexpensive, lightweight, flexible, has a wide frequency range, and is very sensitive. In this study 5 compositions were carried out namely ((50% : 50%), (33,3% : 66,6%), (25% : 75%), (75% : 25%), (66,6% : 33,3%)). Nanocomposites are synthesized using the sol-gel spin coating method. With making precursors ($Fe(NO_3)$ $3.9H_2O$ and $Ni(NO_3)$ $2.6H_2O$. For the growth of thin films, the solution is dripped on a 0.5 cm x 0.5 cm glass substrate. Characterized using XRD (X-Rey Diffraction), SEM (Scanning Electron Microscope), FTIR (Fourier Transform Infra Red).

Keywords : NiFe2O4,PVDF,Nanokompocite, Sol Gel, and Spin Coating

I. INTRODUCTION

The development of modern materials science has given birth to functional materials called smart materials, such as sensing materials (fiber optics and biosensors, etc.) [1]. Composite is a combination of two or more materials with different and different properties, where one material functions as a matrix component (binder) while the other material functions as a filler component (filler) [2].

In recent years, the material that has become one of the objects of research for composite materials is a conductive polymer mixed with ferrite spinels. Ferrite compounds with the molecular formula MFe where M is a transition metal or alkaline earth is a semiconductor material that is magnetic with a spinel structure.

Cubic ferromagnetic oxide which has high permeability at high frequencies, has high electrical resistance and has a magnetic moment of harvesting without a magnetic field externally exerted, which is located in transition metals including Fe, Co, Ni, and in rare earth metals such as Nd, and Gd are the meaning of NiFe2O4 nanocomposite.

Lots of binder materials are used to make composites, one of which is polymeric materials [3]. PVDF (Polyvinylidene Flouride) film is a material that is cheap, light, flexible, has a wide frequency range, and is very sensitive [4]. PVDF (Polyvinylidene Flouride) film is a piezoelectric material on the market, including a thin sheet.

The bonds between particles that occur in nanocomposite materials play an important role in enhancing and limiting material properties. The nano-sized particles have a high interaction surface area. What makes the bonds between particles stronger so that the mechanical properties of the material increase, namely the more particles that interact, the stronger the material will be.

The method used for synthesis in this study is the Sol-Gel method and spin coating. The solgel method is known as a fairly simple and easy method of synthesizing nanoparticles. This method is one of the "wet method" or wet method which in the process involves a solution as the medium. As the name implies in the solgel method, the solution undergoes a phase change to sol (colloids that have suspended solids in the solution) and then to gel (colloids but have a larger solid fraction than sol [5].

Easy composition control (good chemical composition homogeneity), Low process temperature, Low cost, are the advantages of the sol-gel method. Then to grow dielectric thin films with good quality and cheap spin coating method is used. With this method, the quality of the thin films grown is very sensitive to the fabrication parameters used, including solvent, substrate, and annealing temperature [6].

According to research conducted by [7] on the growth of a thin layer of magnetite using the spin coating method, it has been done many times before, such as growing a thin layer of magnetite on a Cu substrate with $FeCl_3.6H2O$ precursor. The quality of the film is determined by the crystal structure, morphology, and thickness of the film produced.

The intensity of the crystal structure of a thin film is linearly correlated with the thickness of the film produced [8,9] also conducted a study using the same polymer as this study, namely Fe_3O_4 / PVDF with the SEM test, namely the more PVDF composition, the smaller the resulting grain size, then using XRD it was found that the more Fe_3O_4 and PVDF used, the more Fe_3O_4 / PVDF peaks appear and the grain size will get smaller. The greater the amount of Fe_3O_4, the smaller the crystal size will be.

Therefore, this is what motivates the researcher to investigate the effect of the $NiFe_2O_4$ composition on the structural properties of the $NiFe_2O_4$ nanocomposite in a study entitled "the effect of the $NiFe_2O_4$ composition on the crystal structure of $NiFe_2O_4$ / PVDF nanocomposite using the sol-gel spin coating method".

Iron sand is a type of sand with a significant concentration of iron. It is usually dark gray or blackish. This sand consists of magnetite, Fe_3O_4, and also contains trace amounts of titanium, silica, manganese, calcium, and vanadium. Ferrite compounds with the molecular formula MFe where M is a transition metal or alkaline earth is a semiconductor material that is magnetic with a spinel structure.

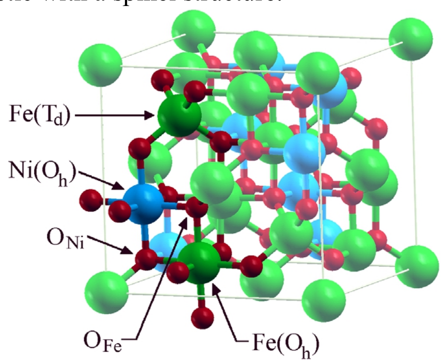

Fig. 1. Nickel ferrite crystal structure [10]

In Figure 1. Ni atoms are shown in blue, Fe in green and O atoms in red [10]. Due to its small melting point, large specific heating, large expansion coefficient, small magnetic moment saturation, and small magnetic transition temperature, nickel ferrite nanoparticles (NiFe2O4) are one of the most recently developed ferrite spinel magnetic nanocomposites.

According to Santoso, 2005 a material that is cheap, lightweight, flexible, has a wide frequency range, and is very sensitive is PVDF film. The PVDF film can be directly attached to the experimental material without mechanical interference with the material to be affixed or entered.

Composite according to Chung, 2010 is a combination of two or more materials with different and different properties, where one material functions as a matrix component (binder) while the other material functions as a filler component. In this research, NiFe2O4 as a filler and PVDF as a binder. Multi-phase solid materials, where each phase has one, two, or three dimensions less than 100 nanometers (nm), or solid structures with nanometer-scale dimensions repeating at distances between these different structural constituents is the definition of nanocomposites. The structure of the Nanocomposite itself can be seen in Figure 2

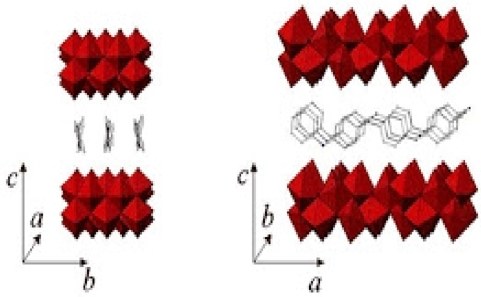

Fig. 2. Structure of nanocomposites [11].

The Sol Gel method is one of the most successful methods of preparing nanoscale metal oxide materials. Spin coating is a method of growing thin films on the substrate by dropping the liquid and then rotating it at a constant speed [12].

NiFe2O4 / PVDF nanocomposites were characterized using an XRD device, namely to see the structure of a crystal, an example of X-ray diffraction on XRD can be seen in Figure 3 below.

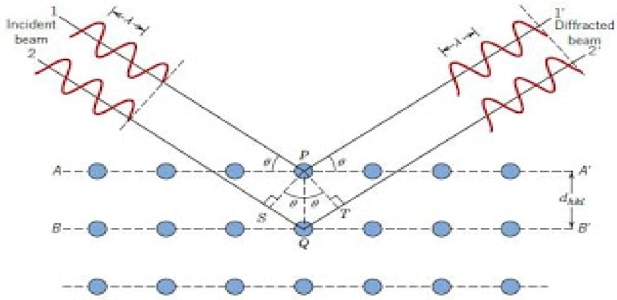

Fig. 3: An example of X-ray diffraction on XRD [13].

Then SEM is used to see the grain size, thickness of the sample, the working mechanism of SEM can be seen in Figure 4. Furthermore FTIR is used to see the functional groups of the sample.

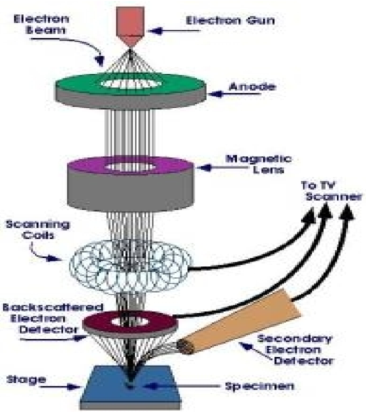

Fig. 4. SEM mechanism [14]

II. METHOD

This type of research is experimental research. This study examines the effect of composition variations on the structural properties of $NiFe_2O_4$/PVDF nanocomposites using the sol-gel method. In this study, some stages are carried out, namely: the preparation stage, the sample preparation stage, the characterization stage, the data analysis stage. For characterization using X-Ray Diffraction at the Physics Laboratory of Padang State

University, Scanning Electron Microscope (SEM) at P3GL Bandung, and for FTIR tests in the Chemistry Laboratory of State University of Padang.

a. Sample purification

The iron ore that has just been taken from nature is refined first, then filtered using a 100 mesh sieve and drawn with a permanent magnet for 30 times the pull to separate the sand containing iron from other mixed materials. After that the iron ore that has been withdrawn is washed using aquabidest, then after that, it is dried and pulled again with a magnet 20 times. Finally, milling for 30 hours.

b. Precursor preparation

1) $NiFe_2O_4$ - Precursor

Furnace Ni (powder) at 650°C for 3 hours. Weigh as much as 1.25 grams of Ni that has been in the Furnace. Weigh the Fe solution as much as 2.5 grams. Add 5.55 grams of citric acid. Enter 11.1 grams of PEG. Stir using a magnetic stirrer for 2 hours at a temperature of 90°C with a rotating speed of 250 rpm (until a gel is formed). Let stand for a while, then wash with aquabidest. Dry in oven at 110°C for 24 hours. The furnace at 400°C for 2 hours.

2) PVDF Precursor

Weigh the PVDF as much as 3 grams. Dissolve in THF (the material used to make PVDF) using a volumetric flask (closed) at a temperature of 75°C.

3) Nanocomposite Fabrication

Mix $NiFe_2O_4$ precursor with PVDF precursor with a composition ratio of 1: 2 or 2: 1. To make a thin layer of the nanocomposite use a glass substrate measuring 1 cm x 1 cm and 0.5 cm x 0.5 cm then drop the solution evenly on the glass using a dropper with a spin coating speed of 3000 rpm for 60 seconds.

c. Flow chart

This research was carried out according to the flow chart shown in Figure 5.

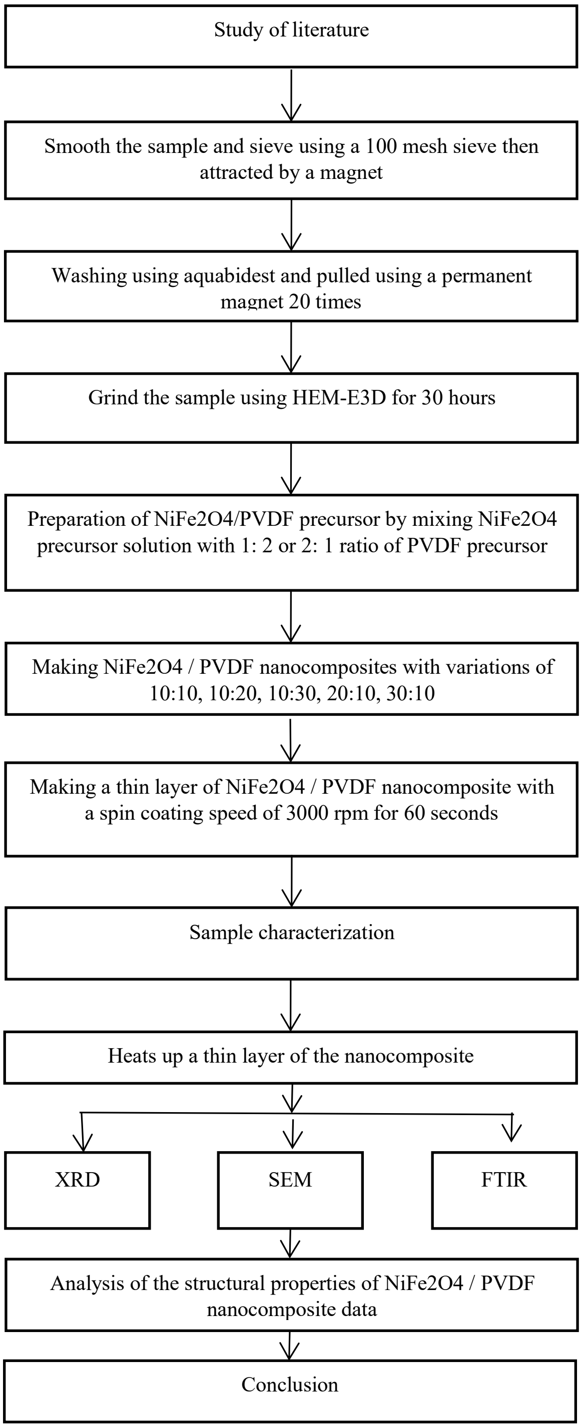

Fig. 5. Research flowchart

III. RESULTS AND DISCUSSION

Effect of $NiFe_2O_4$ Composition on the Crystal Structure of $NiFe_2O_4$/PVDF Nanocomposites. XRD testing was carried out to determine the crystallinity structure with variations in the composition of the $NiFe_2O_4$/PVDF nanocomposite, ((50%: 50%), (33.3%: 66.6%), (25%: 75%), (75%: 25%).), (66.6%: 33.3%)), can be seen in Figure 6.

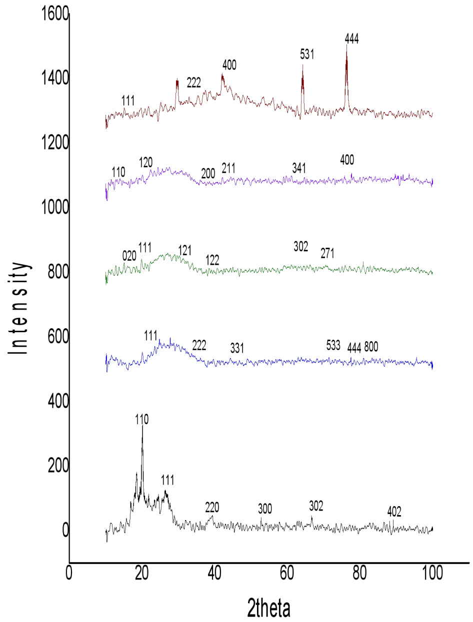

Fig. 6. Effect of composition on crystal structure

The size of the crystals formed in the composition variation of 10 ml: 10 ml the average crystal is 91,436, 10 ml: 20 ml the average crystal is 55,710, 30 ml: 10 ml the average crystal is 50,527, 10 ml: 30 ml with average size the crystals were 51.646 and 20 ml: 10 ml with an average crystal size of 53.672. At variations in the composition of 10: 10, 20: 10, 30: 10, the composition of NiFe2O4 varies, where 30 ml: 10 ml is smaller than the average size of the crystals compared to the composition variations of 10 ml: 10 ml, and 10 ml: 20 ml. This means that the more the composition of NiFe2O4 that is entered, the smaller the crystal size, while for the variation of 10 ml: 10 ml, 10 ml: 20 ml, 10 ml: 30 ml, because here what varies is the PVDF, the results are not in accordance with the existing theory. In accordance with previous research conducted by Rahmi, et al, 2014, namely the more the amount of Fe_3O_4, the smaller the crystal size.

Effect of composition on grain size of $NiFe_2O_4$ / PVDF nanocomposites. The variation in the composition of the $NiFe_2O_4$ / PVDF nanocomposites affected the average grain size formed in the layers synthesized using the sol-gel spin coating method. Average grain size value for each variation of composition 10 ml: 10 ml is 0.503 µm, 10 ml: 20 ml is 0.207 µm, 10 ml: 30 ml is 0.167 µm, 20 ml: 10 ml is 0.336 µm, 30 ml: 10 ml is 0.367 µm. In the NiFe2O4 / PVDF nanocomposite the smallest average grain size was found in the composition 10 ml: 30 ml, namely 0.167 µm. Based on the results of the analysis using SEM, it was found that the more PVDF was entered, the smaller the grain size. Rahmi, et al., 2014 have conducted previous research on Fe_3O_4 / PVDF and conducted an analysis using SEM, namely the more PVDF, the smaller the grain size.

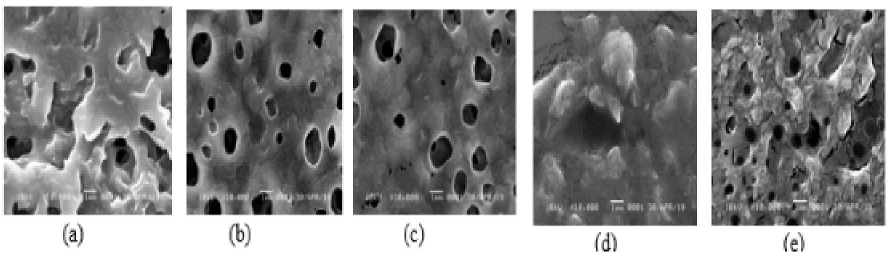

Fig. 7. Results of the SEM morphology of $NiFe_2O_4$ / PVDF nanocomposites with a magnification of 10,000X, for each composition variation (a) 50%: 50%, (b) 33.3%: 66.6%, (c) 25%: 75% , (d) 75%: 25%, (e) 66.6%: 33.3%

Effect of Composition on the thickness size of $NiFe_2O_4$ / PVDF Nanocomposites. The variation in the composition of the $NiFe_2O_4$ / PVDF nanocomposites affected the average grain size formed in the layers synthesized using the sol-gel spin coating method. The rotation when dripping the liquid on the substrate causes the result of the distribution of the thin film solution partly unevenly on all parts of the substrate so that the thickness is different. Therefore, spin coating speed (ω) must be sought in order to produce an even distribution of thin films. The value of the size of the average thickness for each variation of composition 10 ml: 10 ml is 7,961 μm, 10 ml: 20 ml is 9,147 μm, 10 ml: 30 ml is 9.745 μm, 20 ml: 10 ml is 7,957 μm, 30 ml: 10 ml is 9.787 μm.

Based on the analysis carried out using SEM for the composition of 10 ml: 10 ml, 10 ml: 20 ml, 10 ml: 30 ml where the more PVDF is entered, the greater the thickness, at the composition of 20 ml: 10 ml the thickness is 7,957 μm, it can be seen that the greater The composition of $NiFe_2O_4$ then the thickness is getting smaller [15] with his research the addition of PVDF concentration fraction which is added with Fe_3O_4 get, the more Fe_3O_4 composition is added, the thinner the thickness will be.

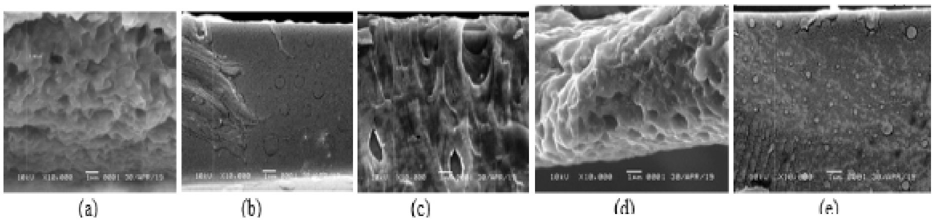

Fig. 8. Results of the SEM sections of the $NiFe_2O_4$ / PVDF nanocomposite surface section with a magnification of 10,000x, for each composition variation (a) 50%: 50%, (b) 33.3%: 66.6%, (c) 25%: 75%, (d) 75%: 25%, (e) 66.6%: 33.3%

Effect of $NiFe_2O_4$ Composition on the $NiFe_2O_4$ / PVDF Nanocomposite functional groups. Testing using FTIR to find out how the functional groups of $NiFe_2O_4$ / PVDF nanocomposites by looking at the relationship between wavelengths and the percent transmittance

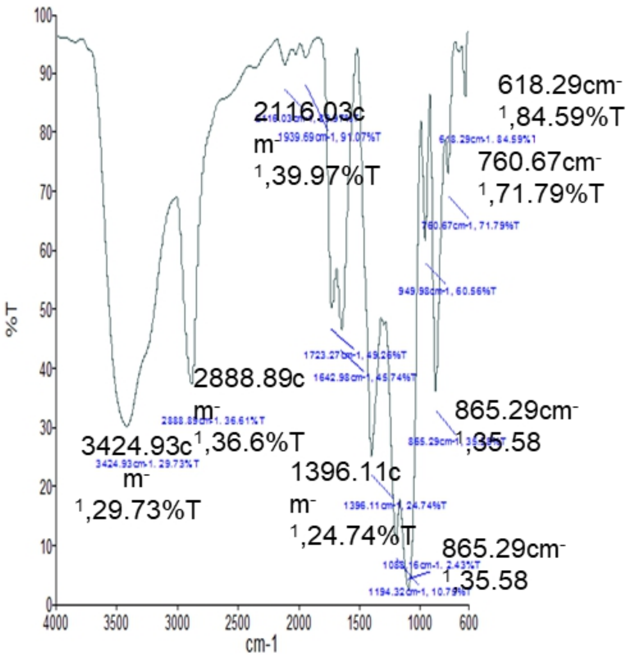

Fig. 9. FTIR spectra of the sample

Characteristics of the sample were carried out with 5 variations in the composition of the first 10 ml: 10 ml, namely 3449.57 cm^{-1}, 1925.36 cm^{-1}, 1728.31 cm^{-1}, 1398.44 cm^{-1}, 1192.25 cm^{-1}, second 10 ml: 20 ml which is 2919.22 cm^{-1}, 1722.19 cm^{-1}, 1429.05 cm^{-1}, 926.71 cm^{-1}, 721.90 cm^{-1}, the third 10 ml: 30 ml is 2946.27 cm^{-1}, 1733.72 cm^{-1}, 772.03 cm^{-1}, 926.41 cm^{-1}, fourth 20 ml: 10 ml that is 2616.60 cm^{-1}, 1726.20 cm^{-1}, 924.08 cm^{-1}, 771.79 cm^{-1}, 30 ml: 10 ml, namely 3424.93 cm^{-1}, 2888.89 cm^{-1}, 2116.02 cm^{-1}, 1939.69 cm^{-1}, 1939.69 cm^{-1}. Based on the results of $NiFe_2O_4$ testing using FTIR, it can be seen that the absorption peaks appear at wave number 3425.58; 1627.92; 601.79 and 347.19 cm-1. At the wave number 3425.58 cm-1 the absorption peak that appears is a symbol of the stretching of O-H (hydroxide) absorption. And the absorption peak that appears at the wave number 1627.92 cm^{-1} is the absorption symbol of O-H bending. At wave numbers 400 cm^{-1} and 600 cm^{-1}, two main peaks of the metal ion and oxygen absorption groups are observed which are attached to the ferrite spinel structure, each of which is related to the siteoctahedral and tetrahedral position of the metal ions on the ferrite spinel, the absorption peak. which appears at wave number 347.19 cm^{-1} belongs to the sub-lattice of the siteoctahedral (vibrations between Ni, Fe and oxygen atoms), while the absorption peak seen at wave number 601.79 cm^{-1} is the character of atomic absorption groups. -atoms that are in cytetetrahedral (vibrations between Fedanoxygen) [16].

IV. CONCLUSION

Synthesized using the sol-gel spin coating method. Synthesis was carried out by varying NiFe2O4 and PVDF by 5 variations in the composition, namely ((50%: 50%), (33.3%: 66.6%), (25%: 75%), (75%: 25%), (66.6%: 33.3%)). With a spin coating speed of 3000 rpm for 60 seconds. The results show that the variation in the composition of NiFe2O4 / PVDF greatly affects the structure of the crystal size. It can be seen in the composition of 30 ml: 10 ml the average size of the crystals is smaller than the composition of 10 ml: 10 ml and 10 ml: 20 ml. Based on SEM analysis, it was found that the more PVDF was entered, the smaller the grain size. In terms of thickness, the more the NiFe2O4 composition, the smaller or thinner the thickness will be. And for the functional group of NiFe2O4 the absorption peak at number 3425.58 cm-1, namely O-H (stretching) between H atoms and O atoms, at the peak of absorption at numbers 1627.92 cm-1 (bending). At the wave numbers 400 cm-1 and 600 cm-1, two main peaks and the metal ion and oxygen absorption groups are observed which are attached to the ferrite spinel structure.

Based on the research that has been done, the researchers suggest to be more careful in carrying out the sol gel process. So that the results obtained can be in the form of a good gel. So that when the spin coating process is carried out, the growth of the nanocomposites can produce a thin film that is sticky and perfect by looking for a more precise spin coating acceleration, so that the resulting crystal structure matches the varied composition.

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