The electrical properties of NiFe$_2$O$_4$-PVDF nanocomposite prepared by sol-gel method

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Abstract. NiFe$_2$O$_4$ nanocomposite is a soft magnetic material with low saturation coercivity and magnetization but has high electrical resistivity making this material suitable for electric field applications. The purpose of this study was to determine the effect of NiFe$_2$O$_4$ composition on the electrical properties of NiFe$_2$O$_4$-PVDF nanocomposites prepared by the sol-gel method. Nickel Ferrite nanocomposites - polyvinylidene fluoride (NiFe$_2$O$_4$-PVDF) are made using the sol-gel method and then grown using a spin coating device on a glass substrate. NiFe$_2$O$_4$-PVDF nanocomposites are characterized using X-ray diffraction (XRD) which aims to determine the crystal system, whereas to determine the electrical properties of nanocomposite resistivity Four Point Probe are used. Based on the research conducted on the electrical properties of the NiFe$_2$O$_4$-PVDF nanocomposite, the resistivity value of the NiFe$_2$O$_4$-PVDF nanocomposite with variations in the composition of the PVDF used 10:10, 10:20, and 10:30 was 36.66913333 Ohm.m; 55.63242667 Ohm.m; and 20.85797333 (Ohm.m). While the results of the resistivity values of the nanocomposite NiFe$_2$O$_4$-PVDF with variations in the composition of NiFe$_2$O$_4$ used 10:10, 20:10, and 30:10 are 36.69613333 Ohm.m; 12.07853333 Ohm.m; and 9.616773333 (Ohm.m). It can be concluded that the greater the variation in the composition of NiFe$_2$O$_4$ and PVDF, the resistivity value decreases.

1. Introduction
Nanocomposite material is a material whose development is very promising in nanostructured research [1, 2]. Nanostructured material attracts many scientists because of its very small size and volume surface area ratio. This is due to the size that affects the chemical properties and physics which are very different from the large material in the same chemical composition [3]. NiFe$_2$O$_4$ nanocomposite is a soft magnetic material with low saturation coercivity and magnetization but has a high electrical resistivity making this material suitable for electrical field applications [4].

The nanocomposite is a material that is formed from two combinations, namely the matrix as a reinforcement filler and filler protector as an amplifier of the matrix [5]. The conductive polymers that are currently being developed are Polyvinylidene Fluoride or can be abbreviated as PVDF [6]. Polymers that have great thermal stability are Polyvinylidene Fluoride (PVDF). This happens because the chemical resistance of these polymers to aggressive reagents is widely used in the preparation process of nanofiltration (NF) and ultrafiltration (UF) [7].
The development of science and technology is growing rapidly in all applied fields, including in the field of nanoparticle technology. One of the advantages of using it is that the material produced can have better characteristics than the material that was previously available. Lately, a lot of research has been developed about ferrite spinel nanoparticles. This was developed because of its excellent electrical and magnetic properties and very broad application fields in storage systems, ferrofluid technology, magnetocaloric refrigerators, and medical diagnostics [8].

In this research, NiFe$_2$O$_4$-PVDF nanocomposite was made and tested the electrical properties of the nanocomposite. The method used to synthesize spinel ferrite nanocomposite is the sol-gel method then spin coating. One of the most successful methods of preparing nano-sized metal oxide material is the sol gel method. The sol is a colloidal suspension in which the dispersed phase is solid and the dispersing phase is liquid. Spin Coating is one method of making thin layers using around. The spin coating method is quite simple, can be done at room temperature, and is effective for making thin layers. The spin coating method is a process where the coating material as an individual particle is driven by gas flow pressurized to the surface temperature. These particles hit the substrate, stick and form a suitable thin surface [9].

2. Experimental

This type of research is an experiment, where in this study using a characterization tool that is XRD, FTIR, and Four Point Probe (FPP). XRD is used to see the peak of the sample used in this study, whether there is a peak of NiFe$_2$O$_4$ and the peak of PVDF, if it has been able to peak from both of these then proceed with FTIR characterization. Where in this characterization is used to determine the chemical bonds formed in the material that is NiFe$_2$O$_4$ and PVDF, if it has been shown the type of NiFe$_2$O$_4$-PVDF compound that has been bound to a number area then characterization can be continued using FPP, from this test obtained values of resistivity.

In this study, tools used were spatulas, permanent magnets, mortars and pestles, 100 mesh filters, plates, beakers and measuring cups, drop pipettes, glass substrates, digital scales, Hem-E3D, magnetic stirrers, furnaces, ovens, cleaners ultrasonic, spin coating, XRD, FTIR, and Four Point Probe (FPP). The materials used to make Nanocomposite Nickel Ferrite-Polyvinylidene Fluoride (NiFe$_2$O$_4$ - PVDF) are iron ore, aquabidest, flour, Alcohol Antiseptic, Nickel Oxide (NiO), Nitric Acid (HNO$_3$), citric acid, Polyethylene Glycol (PEG), Oxalic acid, ethylene glycol, Polyvinylidene Fluoride (PVDF) and Tetrahydrofuran (THF).

Implementation This research was carried out in stages, starting with iron ore purification that is by being crushed as smooth as possible using mortar and pestle, filtered with ordinary sieves and pulled with permanent magnet as much as 20 pulls then washed using aquabidest, dried and pulled again with permanent magnet as much as 30 times to separate it from residual impurities. At the stage of refining the sample using HEM-3D was carried out for 30 hours to be made into nanoparticles. Making Fe$_3$O$_4$ sol-gel by mixing iron sand after milling as much as 17.4 grams and 4.5 grams of oxalic acid using magnetic stirrer at 110°C for 15 minutes, then adding ethylene glycol as much as 55 ml then stirring for 2 hours at 80°C.

Making NiFe$_2$O$_4$ is by weighing NiO which has been in the furnace as much as 1.25 gr with a scale tool; Fe$_3$O$_4$ as much as 4.35 gr; Citric Acid as much as 5.55 gr; PEG as much as 11.1 gr. All weighed ingredients are mixed and put the magnetic bar into the beaker then in the stirrer with Magnetic Stirrer for 2 hours at 90°C with a rotating speed of 250 rpm until a gel is formed. Let stand for a while, then dry in the oven for 24 hours at a temperature of 110°C. Furnished at 400°C for 2 hours. After finishing and forming NiFe$_2$O$_4$, precursor NiFe$_2$O$_4$ is formed by weighing 3 grams of NiFe$_2$O$_4$ and dissolved with 70 ml THF in a beaker, then cleaning using Ultrasonic for 2 hours (until dissolved). Preparation of precursor PVDF is by weighing 3 grams of PVDF with a weighing device then dissolved with THF of 70 ml in a measuring flask that already has a temperature thermometer closed using a magnetic stirrer for 2 hours [10].

In the manufacture of NiFe$_2$O$_4$-PVDF nanocomposites using the Tetrahydrofuran (THF) solvent system. Makes five variations for precursor NiFe$_2$O$_4$ with precursor PVDF. Sol-gel NiFe$_2$O$_4$ solution
was dissolved into THF using ultrasonic Cleaner for 2 hours, after adding PVDF and THF with a ratio (3 gr PVDF: 70 ml THF) mixed using a magnetic stirrer at a temperature of 70°C until the PVDF was dissolved continuously into the solvent. Varying PVDF + THF with NiFe2O4. The variation is 30ml: 10ml, 20ml: 10ml, 10ml: 10ml: 20ml, 10ml: 30ml. The THF-NiFe2O4 solution was added to the PVDF solution for 1 day.

The nanocomposite is dripped on a glass substrate and rotated at 3000 rpm for 60 seconds using a spin coating. The thin film that has been formed is then brushed with an oven for 30 minutes at 60°C. The thin film of NiFe2O4-PVDF nanocomposite is characterized using X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) to determine nanocomposite chemical bonds and to determine the characteristics of resistivity of electrical properties using the Four Point Probe (FPP).

3. Results and discussion

The results of this study were in the form of identification of data, structure and grain size tested using XRD, the capacitance values tested using FPP and FTIR were used to look at the chemical compound structure of the NiFe2O4-PVDF nanocomposite. Characterization using XRD showed that the NiFe2O4-PVDF nanocomposite thin films were developed on glass substrates, characterized by peaks in X-Ray diffraction patterns with variations in composition of NiFe2O4 and PVDF can be seen in Figure 1.

![Figure 1. X-Ray Diffraction Pattern of Nanocomposite Thin Films(NiFe2O4 : PVDF)](image)

Figure 1 is an XRD data analyst, X-Ray diffraction patterns obtained from nanocomposite NiFe2O4-PVDF with variations in composition 10:10, 10:20, 10:30, 20:10, and 30:10 there are crystal structures of nanocomposites that grow on the substrate glass when used as a thin layer. The XRD characterization results that have been obtained are then compared with the ICDD database. All variations of the nanocomposite phases NiFe2O4, PVDF, and NiFe2O4-PVDF appear. At variation
30:10 shows some of the highest peaks that stand out from several variations resulting from XRD diffraction patterns.

The results of FTIR characterization of NiFe$_2$O$_4$-PVDF nanocomposites with 30:10 composition variation can be seen in Figure 2.

![Figure 2. Results of FTIR Nanocomposite Characterization of NiFe$_2$O$_4$-PVDF with a variation of 30 ml NiFe$_2$O$_4$: 10 ml PVDF](image)

NiFe$_2$O$_4$-PVDF nanocomposite with a composition of 30:10 obtained observations from the IR spectrum of NiFe$_2$O$_4$-PVDF nanocomposites synthesized using the sol-gel method with FT-IR spectroscopy using KBr method. The NiFe$_2$O$_4$ - PVDF nanocomposite IR spectrum can be observed at wavenumbers 600-4000 cm$^{-1}$. In Figure 2 can be seen from the FTIR graph of NiFe$_2$O$_4$-PVDF nanocomposites in the area of 1194.32 cm$^{-1}$ which shows the types of NiFe$_2$O$_4$-PVDF compounds that have been bound.

Data analysis results of the characterization of the electrical properties of the NiFe$_2$O$_4$-PVDF nanocomposite using the FPP method obtained the resistivity value of a material. The cross-sectional area used in this study is 1 cm because the size of the glass substrate used for this study is 1 cm. The relationship between the composition of the NiFe$_2$O$_4$-PVDF nanocomposite using the FPP method with the resistivity for NiFe$_2$O$_4$ changes is shown in Figure 3.

![Figure 3. The relationship between the composition of the NiFe$_2$O$_4$-PVDF nanocomposite using the FPP method with the resistivity for NiFe$_2$O$_4$ changes](image)
Based on Figure 3, the resistivity value of nanocomposite NiFe₂O₄-PVDF with variations in the composition of NiFe₂O₄ used 10:10, 20:10, and 30:10 is 36.69613333 Ohm.m; 12.07853333 Ohm.m; and 9.616773333 (Ohm.m). Can be seen the relationship between the composition of NiFe₂O₄-PVDF with resistivity for NiFe₂O₄ changes where the greater the value of the composition given to NiFe₂O₄, the resistivity value will be smaller. In the variation of NiFe₂O₄-PVDF 30:10 the smallest resistivity value is 9.616773333 (Ohm.m). This is influenced by the large crystal size of the sample variation. The relationship between the composition of the NiFe₂O₄-PVDF nanocomposite using the FPP method with the resistivity for PVDF changes is shown in Figure 4.

![Figure 4](image-url)

**Figure 4.** The relationship between the composition of the NiFe₂O₄-PVDF nanocomposite using the FPP method with the resistivity for PVDF changes

The resistivity value of nanocomposite NiFe₂O₄-PVDF with variations in the composition of the PVDF used 10:10, 10:20, and 10:30 is 36.6913133 Ohm.m; 55.63242667 Ohm.m; 20.85797333 (Ohm.m). Based on Figure 4, it can be seen the relationship between NiFe₂O₄-PVDF composition and resistivity for PVDF changes where the greater the value of the given PVDF composition, the smaller the resistivity value. Comparison with journals [11] the greater the value of the matrix, the resistivity value will decrease. Journals [12] stated that the more PVDF in the solution, the smaller the resistivity value. This proves that the experiment is in accordance with the theory.

4. Conclusion
Based on the research conducted on the electrical properties of the NiFe₂O₄-PVDF nanocomposite, the resistivity value of the NiFe₂O₄-PVDF nanocomposite with variations in the composition of the PVDF used 10:20, 10:10, and 10:30 was 55.63242667 Ohm.m; 36.6913333 Ohm.m; 20.85797333 (Ohm.m). While the results of the resistivity values of nanocomposite NiFe₂O₄-PVDF with variations in the composition of NiFe₂O₄ used 10:10, 20:10, and 30:10 are 36.6913133 Ohm.m; 12.07853333 Ohm.m; and 9.616773333 (Ohm.m). It can be concluded that the greater the variation in the composition of NiFe₂O₄ and PVDF, the resistivity value decreases.

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