



Effect of Fe₃O₄ Nanoparticle and Graphene Oxide Compositions on The Magnetic Properties of Fe₃O₄ : Graphene Oxide Nanocomposites

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Abstract: Magnetic material research aims to determine the effect of variations in the addition of Fe₃O₄ nanoparticles to graphene oxide. Graphene oxide made from coconut shell waste was synthesized using the modified hummers method and composited with Fe₃O₄ nanoparticles consisting of three composition variations, which are 20%:80%, 30%:70% and 40%:60%. The Fe₃O₄-Graphene Oxide nanocomposite was tested using three characterization tools, namely XRD, FTIR and VSM. Testing using XRD and FTIR was carried out to determine whether the Fe₃O₄-Graphene Oxide nanocomposite had been formed or not. In XRD testing, a crystal size of 39.57 nm was obtained, which means that this research has succeeded in forming nanocomposites where the size obtained is smaller than 100 nm. In FTIR testing, it can also be seen that this research has succeeded in forming Fe₃O₄-Graphene Oxide nanocomposites as seen from the functional groups obtained, which consist of oxygen, hydrogen, carbon and iron. In testing using VSM, it can be seen how the effect of adding Fe₃O₄ to graphene oxide, where the greater the addition of Fe₃O₄ composition, the greater the value of the coercivity field produced. In addition, the addition of Fe₃O₄ causes an increase in the magnetic properties of remanent magnetic, saturation magnetic and coercivity values. The coercivity values are 371.18 Oe, 387.59 Oe and 405.19 Oe, respectively, where the highest coercivity value is found in the 40% variation: 60%. This hail shows that the nanocomposite produced is ferromagnetic and classified as a hard magnet so that it can be applied as HDD.

Keywords: Magnetic Properties, Ferromagnetic, Graphene Oxide, Fe₃O₄, VSM



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1. Introduction

The development of technology in this increasingly sophisticated era cannot be separated from the development of material physics, especially in the magnetic field. One of the most influential

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technological products is the computer. The first computer had a storage media capacity of 5 MB until now increasing along with the development of science. One of the magnetic applications for media storage for computers is Hard Disk Drives (HDD) [1]. Hard Disk Drives (HDD) is a gadget launched by IBM America in 1956 in the form of electro-mechanical functions as a data storage area containing magnetic disks of ferromagnetic materials [2,3]. Ferromagnetic materials are materials that have very high magnetic susceptibility values, high Curie temperatures and are more stable at high temperatures. One of the ferromagnetic materials that can be used for HDD manufacturing is Fe_3O_4 . Fe_3O_4 nanoparticles are ferromagnetic materials at room temperature [4] which have regularity in the arrangement of atoms and their magnetic dipole moments, allowing for a good magnetic response when a material is composited with Fe_3O_4 nanoparticles. In this study, Fe_3O_4 nanoparticles were composited with graphene oxide because graphene oxide itself has good magnetic properties [5]. Magnetic properties in graphene oxide occur due to electron dispersion and sp^3 hybridized carbon molecules. These electrons are free to move and align with the direction of the magnetic field, increasing the magnetic moment of graphene oxide and giving it ferromagnetic characteristics.

Some of the magnetic properties of graphene oxide are that it has a negative curie temperature, has a magnetization range between 0.28-0.42 emu/g, the magnetic dipole of graphene oxide is parallel to the direction of the external magnetic field and shows paramagnetic behavior at high temperatures. In addition, graphene oxide can maximize the performance of Fe_3O_4 when composited. The addition of this graphene oxide into Fe_3O_4 can improve the durability and wear resistance of the material, as well as increase the electrical conductivity and porosity, without affecting the surface quality. Graphene oxide is usually produced from pure carbon. However, because the price of graphene oxide is expensive and difficult to obtain, one alternative is to produce graphene oxide from natural materials. There are many sources of carbon from natural materials, for example coconut shells, rice husks, corn cobs, candlenut shells, etc. Coconut shell charcoal is charcoal that has the highest carbon content among other natural materials so that in this study coconut shell is used as a material for producing graphene oxide.

Coconut shell has the main content of cellulose, lignin and hemicellulose consisting of C, O, H and N atoms with the highest elemental content of carbon as much as 51.09% [6]. Carbon material is one of the materials that has a variety of morphologies, including colloidal carbon, nanotubes, fullerenes, graphite, graphene, nanofiber, nanowire, and activated carbon. Carbon nanomaterials are one of the nanomaterials currently being researched by many scientists. This nanomaterial has physical properties that are very interesting to be studied by scientists including being able to flow electric current, having good thermal conductivity properties, and very strong mechanical properties [7]. Carbon is able to form many allotropes due to the valence number possessed by carbon atoms. Allotropes of carbon that have been known to date are diamond, carbon nanotubes, fullerene, fullerrite, graphene, and graphite. Graphite is an isomer of elemental carbon that has a hexagonal crystal structure [8].

Graphene is one of the most researched materials and is called the "material of the future". Graphene is a single carbon allotrope that has a honeycomb-like structure. Graphene was discovered in 2004 by Andre K. Geim and Kostya Novoselov by using scotch tape attached to graphite to sample its carbon powders [9]. Graphene consists of carbon atoms that each carbon is bonded to three other carbon atoms with sp^2 hybridization [10]. Some of the unique properties of graphene are that it has great strength mechanical properties with Young's Modulus of 2.4 ± 0.4

TPa for single layer and 2 ± 0.5 TPa for double layer. In addition, graphene has a tensile strength of 130 GPa and is a good conductor of heat at 5000W/mK. Due to its good electronic transportation, graphene can inhibit fires by having a band gap value of zero [11]. Graphene has a very striking difference with graphene oxide where graphene consists of sp² hybridized atoms, while graphene oxide has a carbon structure equipped with various oxygen functional groups in it. The functional groups in graphene oxide that contain oxygen include =O, -OH, -O-, -COOH. Graphene oxide can have a single layer or multilayer structure, similar to other 2D carbon materials [12]. Graphene applications are very broad in various fields such as nanoelectrics, sensors, nanocomposites, batteries, supercapacitors, semiconductors, and transparent electrodes.

This research was conducted to utilize natural materials that were originally only used as waste to become more valuable materials and become an alternative to produce technology products, namely by synthesizing them into graphene oxide which was then composited with Fe₃O₄ with various composition variations. Furthermore, magnetic properties were tested using the Vibrating Sample Magnetometer (VSM) characterization tool. Previous research has been conducted by Lestari in 2015 by varying the speed and grinding time during the composite process [13]. This is what makes this research different from previous research. Due to the large content of carbon element in coconut shell, coconut shell can be processed into activated carbon through activation process. Coconut shell can be used as a viable biomaterial option to be produced into charcoal [14]. The activation process is carried out by chemical methods using NaOH solution [15]. After activation, this activated carbon can be utilized by converting it into graphene oxide through a synthesis process [16].

To produce graphene, graphite can be exfoliated using several methods. Some of these methods are the Modified Hummers Method, the chemical vapour deposition (CVD) method and the sonication method. In this study, graphene oxide was synthesized using the modified hummer method. The Modified Hummers method is a method developed from the Hummers method that can be used to synthesize graphene from graphite. This Hummers method is the most widely used method for graphene oxide synthesis because the oxidation process does not emit ClO₂ gas. In addition, the oxide process can take place quickly with lower temperatures and the materials used in the Hummers method are more readily available and harmless. The Hummers method uses chemical compounds including H₂SO₄, KMnO₄, HCl, NaNO₃ and H₂O₂ to exfoliate the graphite layer [17]. Fe₃O₄ and graphene oxide are composited to maximize the magnetic properties of both, namely ferromagnetic properties. Before being tested for magnetic properties, the Fe₃O₄-Graphene Oxide nanocomposite was tested using XRD and FTIR characterization tools with the aim of knowing whether the Fe₃O₄-Graphene Oxide nanocomposite had been formed. X-Ray Diffraction is a testing technique that can be used to examine a wide variety of materials by characterizing the position of atoms, their arrangement in each unit cell, and the distance between atomic planes [18]. Fourier Transform Infra-Red (FTIR) is a characterization tool that can be used to determine the functional groups of a material so that this tool can analyze the presence of a mixture of materials in a sample without damaging the sample [19].

A Vibrating Sample Magnetometer (VSM) characterization tool is used to determine the magnetic properties of a material. The hysteresis curve, which is produced by changes in the external magnetic field, shows the magnitude of magnetization. Change in the current cause change in the magnetic field from +H to -H. In addition to the direction of the magnetic field, changes in

current also cause changes in the magnetic flux density value which has the opposite polarity to +B or -B. when there is no external magnetic field, the current will be zero [20]. From the hysteresis curve, quantities can be known, namely saturation magnetization (Ms), remanent magnetization (Mr) and coercivity (Hc) [21]. The magnitude obtained can be a reference to determine the magnetic properties of the material, whether the material is superparamagnetic [22] or ferromagnetic [23].

2. Materials and Method

This type of research is experimental research. The research was conducted by giving treatment to the object of research, namely by varying the composition of Fe₃O₄-Graphene Oxide and characterized using a VSM characterization tool so that the effect of the addition of Fe₃O₄ on the magnetic properties of the research sample used (Graphene Oxide) can be known. This research begins with the manufacture of carbon from coconut shell waste from Koto Baru, Padang Pariaman Regency. Furthermore, this carbon was activated using NaOH solution to form activated carbon. This activated carbon was synthesized using the modified hummer method to form graphene oxide. The resulting graphene oxide was composited with nanoparticle-sized Fe₃O₄. After the Fe₃O₄-graphene oxide nanocomposite was formed, it was tested using characterization tools, namely X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR) and Vibrating Sample Magnetometer (VSM).

In this study, various tools were used, namely oven, furnace, magnetic stirrer, magnetic bar, beaker, buchner funnel, Ohaus balance, filter paper, fume hood, mortar and pestle, 120 mesh sieve, measuring flask, erlenmeyer, ultrasonic, micro centrifuge, and ball mill. While the materials used are old coconut shell waste activated carbon, NaOH, H₂SO₄, NaNO₃, distilled water, KMnO₄, H₂O₂, and Fe₃O₄. And the tool used for characterization is Vibrating Sample Magnetometer (VSM). Several stages carried out for this research are sample preparation, carbon activation, graphene oxide synthesis, sonication and neutralization stages of graphene oxide, Fe₃O₄-Graphene Oxide composite preparation stage, Fe₃O₄-Graphene Oxide nanocomposite characterization stage and data analysis.

2.1. Sample Preparation

At the sample preparation stage, the old coconut shell waste that has been cleaned will be dried in the sun for two days. Then the coconut shell waste is oven at 105°C for 60 minutes and in the furnace at 350°C for 2 hours. After becoming charcoal, the coconut shell waste is pulverized and sieved using a 120 mesh sieve to separate the fine particles. The method can be seen through the flowchart below.

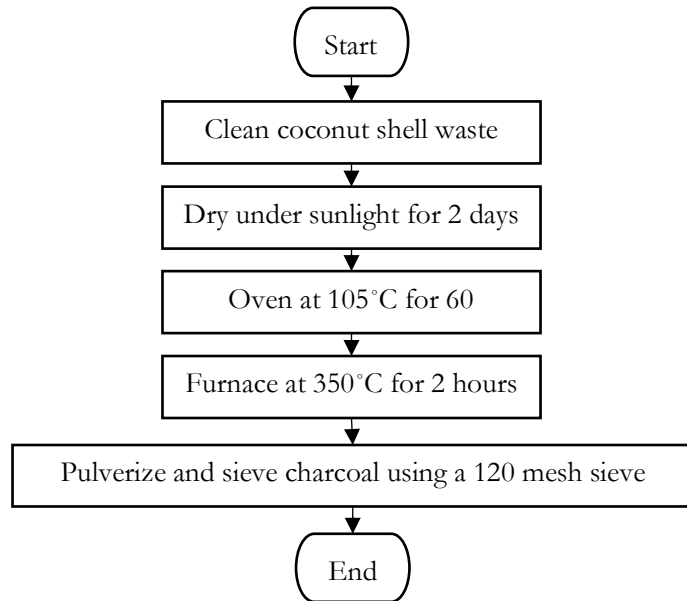


Figure 1. Flowchart of Sample Preparation

2.2. Carbon Activation

Carbon activation is done by putting 8 grams of carbon into a beaker and dissolving as much as 8 grams of NaOH using distilled water as much as 100ml into a measuring flask. NaOH that has dissolved is poured into a beaker that already contains carbon until it is submerged. Soaking is carried out for 24 hours. After the soaking process, the activated carbon precipitate is filtered to separate from the NaOH solution and then the activated carbon precipitate is baked for three hours at 105°C. The method can be seen through the flowchart below.

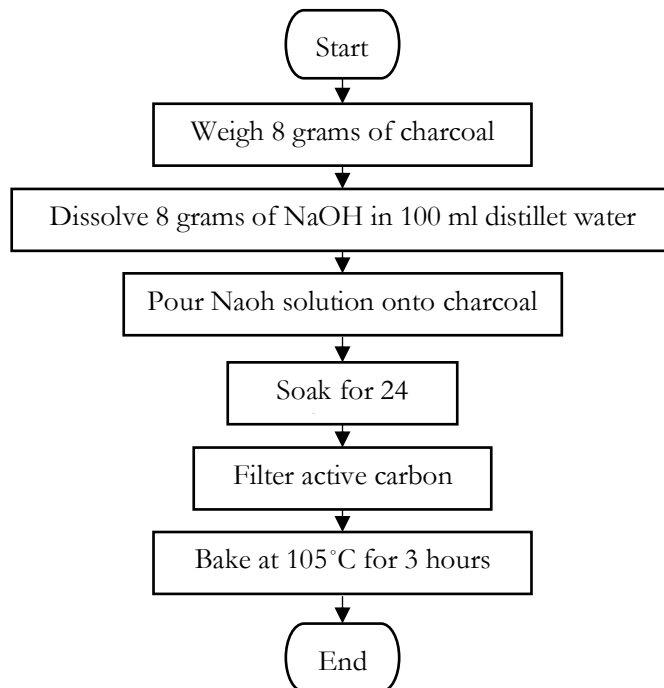


Figure 2. Flowchart of Carbon Activation

2.3. Graphene Oxide Synthesis

The third stage is the synthesis of graphene oxide using the modified Hummer method. In this Hummer method, KMnO_4 is used to avoid spontaneous explosion during the oxidation process while NaNO_3 is used to remove the resulting acid mist. This process only takes a few hours to produce high-quality graphene oxide [24]. The resulting graphene oxide will be composited with Fe_3O_4 nanoparticles using a ball mill [25]. The process is that 1.5 grams of activated carbon, 0.75 grams of NaNO_3 and 34.5 ml of H_2SO_4 are put into a 250 ml erlenmeyer which has previously been filled with a magnetic bar. The mixture was stirred for 20 minutes at 250 rpm at 0-5°C. Next, the erlenmeyer was placed in an ice bath and stirred for 2 hours on a hot plate. Then, KMnO_4 powder was added as much as 4.5 grams slowly and distirred again for 30 minutes. After 30 minutes, add distilled water as much as 69 ml using a drop pipette and stirring for 20 minutes. The final stage of this synthesis is to add 100 ml of distilled water, followed by the addition of 1.5 ml of H_2O_2 and adding another 50 ml of distilled water to the mixture until graphene oxide is formed.

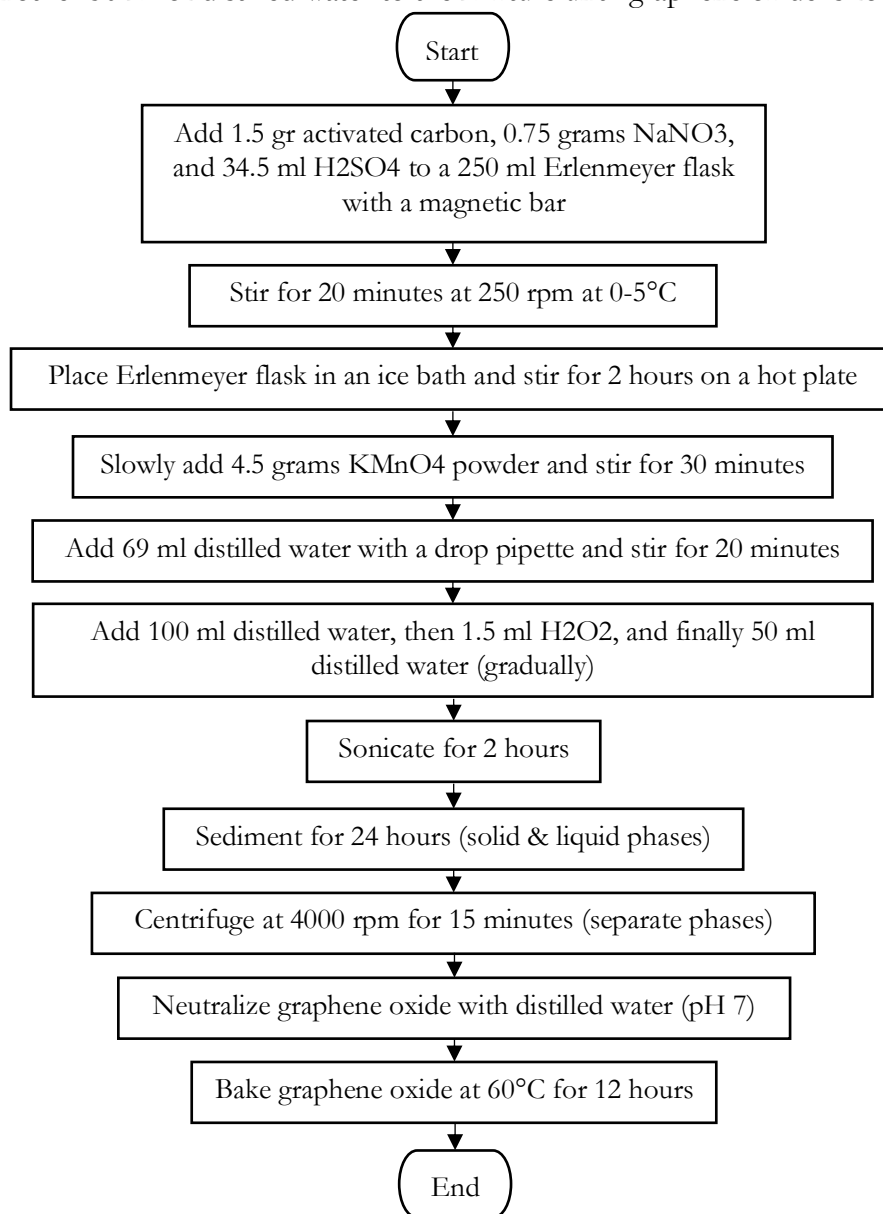


Figure 3. Flowchart of Graphene Oxide Synthesis

The next step is sonification and neutralization. In this stage, the mixture was sonicated for two hours and then sedimented for 24 hours to form the solid phase and liquid phase. Then, a centrifugation process was carried out to separate the two phases by setting the speed of the device at 4000 rpm for 15 minutes. Next, the graphene oxide was manually neutralized using distilled water until a pH of 7 was obtained before finally being baked for 12 hours at 60°C. The method can be seen through the flowchart below.

2.4. Compositing With Fe_3O_4

The fifth stage is compositing graphene oxide with magnetite (Fe_3O_4) using a Retsch Planetary Ball Mill PM 100. At this stage, three variations of the composition of the two materials were made where the composition of graphene oxide was more than the composition of magnetite (Fe_3O_4). The three variations are 40%: 60%, 30% : 70% and 20%: 80%. The speed used is 300 rpm within 30 minutes. The method can be seen through the flowchart below.

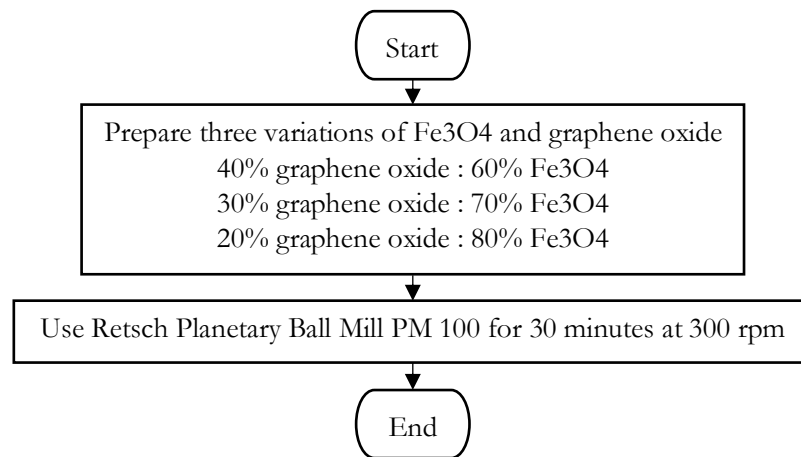


Figure 4. Flowchart of Compositing With Fe_3O_4

2.5. Characterization

The Fe_3O_4 -Graphene Oxide nanocomposite is then characterized using X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR) and Vibrating Sample Magnetometer (VSM) to determine whether the Fe_3O_4 -Graphene Oxide nanocomposite has been formed and determine the magnetic properties of the material which will be presented in the form of a hysteresis curve [20]. Then, the results will be analyzed using HighScore Plus software, Origin 2018 and MS. Excel. The method can be seen through the flowchart below.

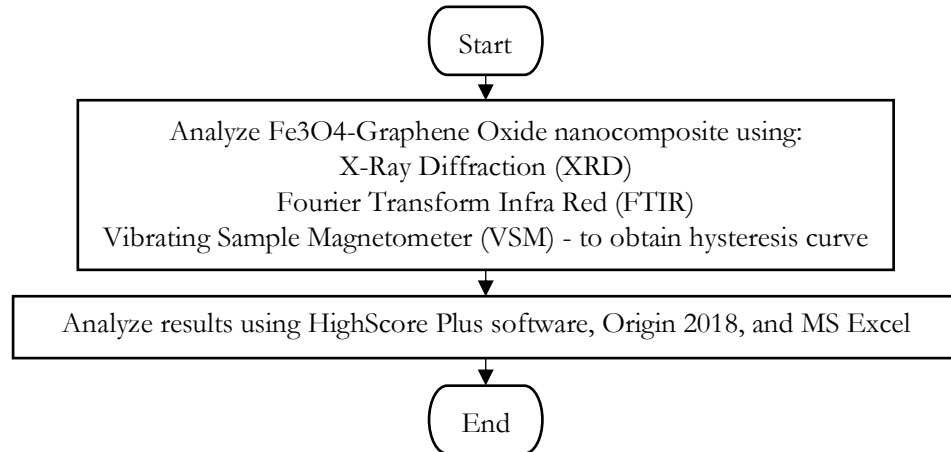
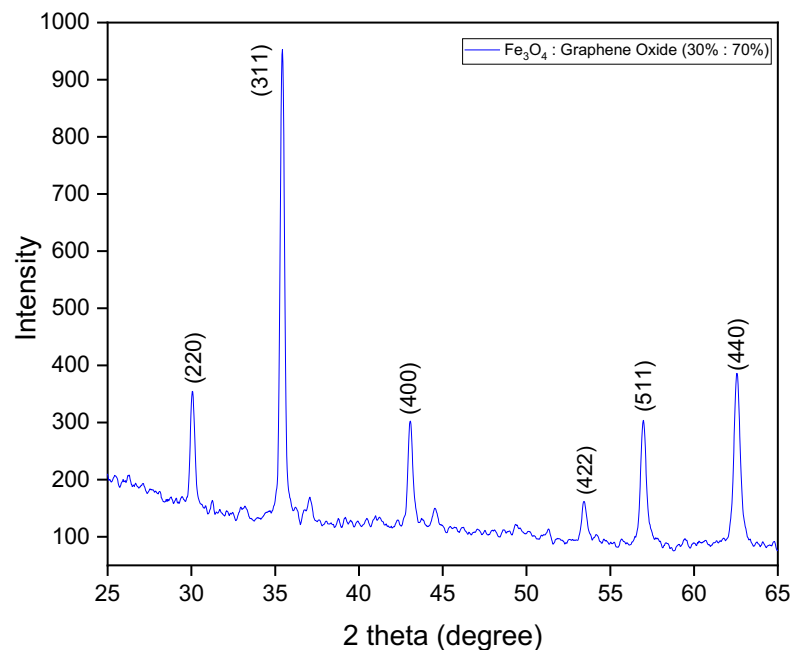


Figure 5. Flowchart of Characterization

3. Results and Discussion

Testing of Fe_3O_4 -Graphene Oxide nanocomposites was carried out with several characterization tools, namely XRD and FTIR to determine whether Fe_3O_4 -Graphene Oxide nanocomposites have been formed and VSM to determine the magnetic properties of these nanocomposites. Characterization using the XRD tool displays the peaks formed, the sparsity between crystal planes, FWHM and relative intensity. The results of data and data analysis of Fe_3O_4 -Graphene Oxide nanocomposites for variations of 30%: 70% variation can be seen in Figure 6 below.

Figure 6. XRD Data Results of Fe_3O_4 -Graphene Oxide Nanocomposite at 30%:70% Composition Variation

In Figure 6, the miller index value of each peak formed is (220), (311), (400), (422), (511) and (440). In addition, the values of 2theta, relative intensity, d-spacing and FWHM are obtained which can be seen in table 1 below.

Table 1. Diffraction Pattern Measurement Data of Fe₃O₄-Graphene Oxide Nanocomposite at 30% : 70% Composition Variation

| Peak To- | 2theta (°) | Intensity (%) | d-spacing (Å) | FWHM (°) |
|----------|------------|---------------|---------------|----------|
| 1 | 30.0357 | 25.28 | 2.97521 | 0.1791 |
| 2 | 35.4083 | 100 | 2.53513 | 0.2047 |
| 3 | 43.0147 | 20.35 | 2.10282 | 0.2047 |
| 4 | 53.4516 | 8.24 | 1.71425 | 0.2047 |
| 5 | 56.9506 | 25.54 | 1.61697 | 0.2558 |
| 6 | 62.5274 | 33.73 | 1.48549 | 0.307 |

The resulting X-ray diffraction pattern of the Fe₃O₄-Graphene Oxide nanocomposite of 30% : 70% variation can be seen in Figure 7.

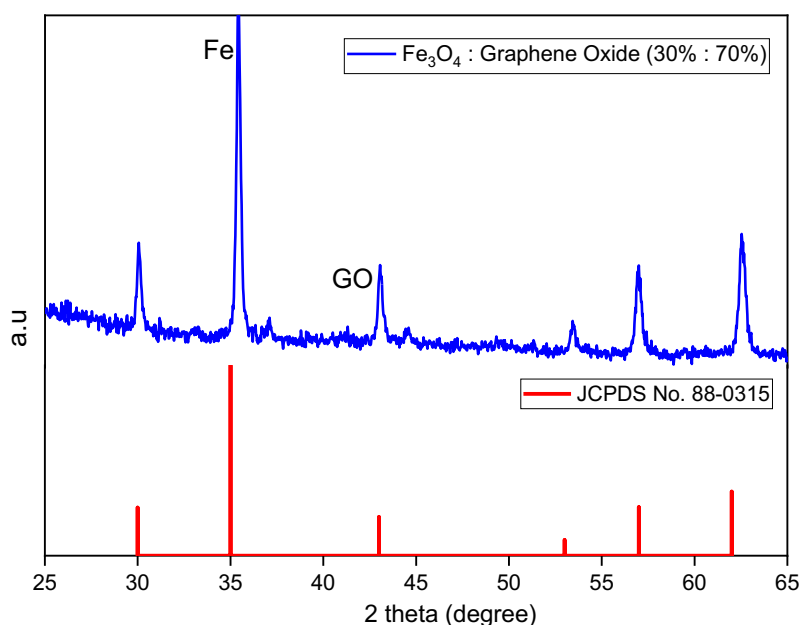


Figure 7. X-ray Diffraction Pattern of Fe₃O₄-Graphene Oxide Nanocomposite Compared to JCPDS Database No.88-0315

Figure provides information on the crystal size when the nanocomposite is formed. The XRD characterization results that have been obtained are compared with the JCPDS database No.88-0315. The crystallite sizes calculated using the Scherrer equation are 45.92 nm, 40.73 nm, 41.71 nm, 43.45 nm, 35.33 nm and 30.27 nm, with an average crystallite size of 39.57 nm. The peaks obtained from the diffractogram are 30.0357°, 35.4083°, 43.0147°, 53.4516°, 56.9506° and 62.5274° which when compared to the JCPDS database No.88-0315 by Rukman is suitable so that it can be ascertained that the research conducted has succeeded in forming Fe₃O₄-Graphene Oxide nanocomposites [26].

Furthermore, the characterization carried out using FTIR aims to determine the functional groups of the Fe₃O₄-Graphene Oxide nanocomposite. Data results from measurements using FTIR can be seen in Figure 3 below.

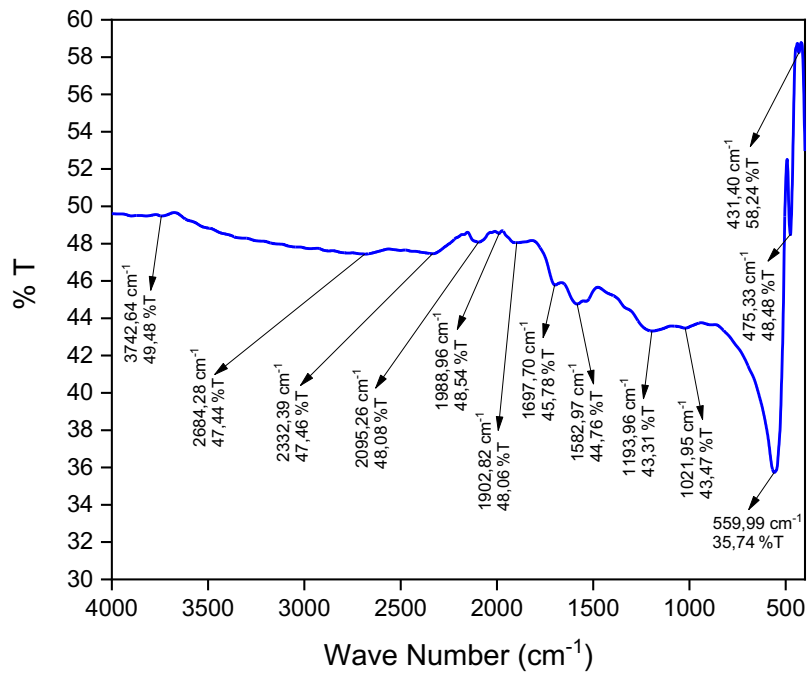


Figure 8. FTIR spectra of Fe₃O₄-Graphene Oxide Nanocomposite at 30% : 70% composition variation

Figure 8 shows the relationship between wave number and percentage transmittance where the transmittance spectrum shows the direction of the peak of the vibration band pointing downwards. From this figure, it can be seen that several wave numbers are formed where these wave numbers can determine the functional groups of the Fe₃O₄-Graphene Oxide nanocomposite. The functional groups of this nanocomposite can be seen in Figure 9.

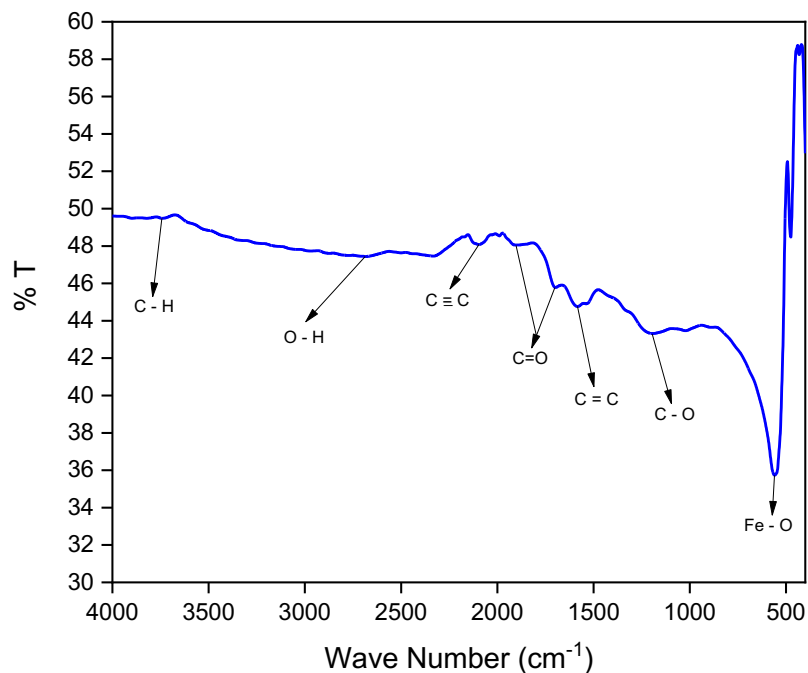


Figure 9. Functional Groups of Fe₃O₄-Graphene Oxide Nanocomposite at 30% : 70% Composition Variation

In Figure 9, several functional groups are obtained, namely C-H, O-H, $C\equiv C$, C=O, C=C, C-O and Fe-O bonds. With the existence of these bonds, it shows that the Fe_3O_4 -Graphene Oxide nanocomposite has been formed where the nanocomposite contains carbon (C), hydrogen (H), oxygen (O) and iron (Fe) bonds, which the results of this test match the research previously conducted by Perveen et al. which states that some of the peaks obtained are as out-of-plane bending vibrations for C-O-H and -C-O groups [27]. Graphene oxide (GO) or graphene acid is a mixed compound of carbon (C), hydrogen (H), and oxygen (O) obtained through a strong oxidation process from graphite [28]. Fe_3O_4 or magnetite contains a mixed compound of iron (Fe) and oxygen (O). In addition to the C, O and H groups, there is also an Fe-O group which if the peak appears around the 578 cm^{-1} number is considered to come from Fe-O, thus confirming the Fe_3O_4 exfoliation in the graphene oxide matrix in the Fe_3O_4 -Graphene Oxide spectrum.

Testing to determine the magnetic properties of the Fe_3O_4 -Graphene Oxide nanocomposite is by using a VSM tool. Testing using this tool consists of three variations of Fe_3O_4 addition into graphene oxide with a comparison of 20%: 80%, 30% : 70% and 40%: 60%. The test results for the three variations can be seen in Figure 10 below.

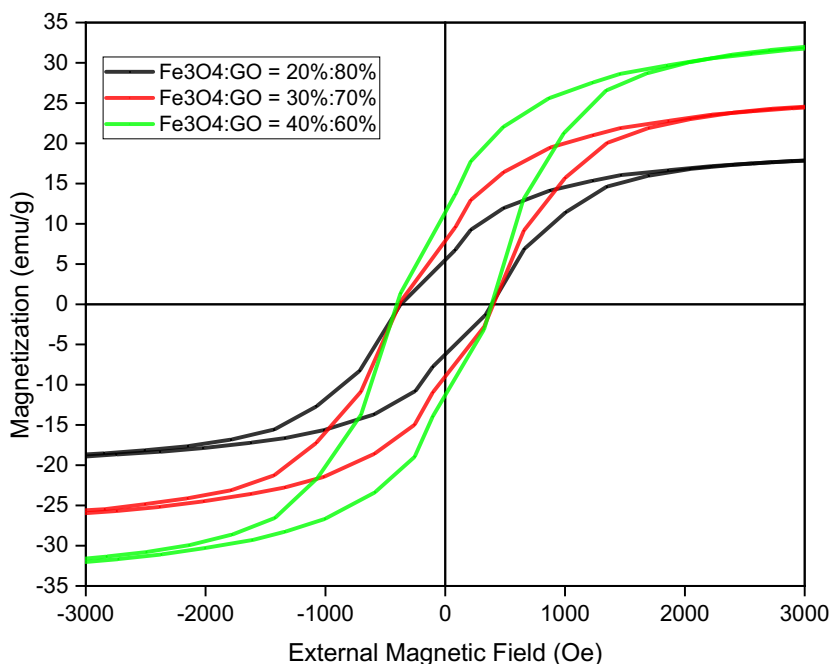


Figure 10. Hysteresis Curve of Fe_3O_4 -Graphene Oxide Nanocomposite for Three Composition Variations

Figure 10 shows the hysteresis curves of Fe_3O_4 -Graphene Oxide nanocomposite for three composition variations. The X-axis shows the external magnetic field (H) and the Y-axis shows the amount of magnetization (M). And from the figure above, it can be observed that the value of the external magnetic field (H) in each variation is in the range of $-22,000 \leq H \leq 22,000$ Oe. The black colored hysteresis curve displays the hysteresis curve of the Fe_3O_4 -Graphene Oxide nanocomposite with 20% variation: 80% obtained M_s value of 19.24 emu/g, M_r of 5.82 emu/g and H_c value of 371.18 Oe. The red hysteresis curve displays the hysteresis curve of the Fe_3O_4 -Graphene Oxide nanocomposite with a variation of 30%: 70% obtained M_s value of 26.55 emu/g, M_r of 7.41 emu/g

and Hc value of 387.59 Oe. While the green hysteresis curve displays the hysteresis curve of the Fe₃O₄-Graphene Oxide nanocomposite with a variation of 40%: 60%, where the Ms value of 34.22 emu/g, Mr of 11.09 emu/g and Hc value of 405.19 Oe were obtained. The analysis results of the magnetic properties of Fe₃O₄-Graphene Oxide nanocomposites can be summarized in Table 3.

Table 2. Analysis Data of Magnetic Properties of Nanocomposites with Variations in Composition of Fe₃O₄-Graphene Oxide

| Composition Variation (Fe ₃ O ₄ : Graphene Oxide) | Saturation Magnetization (emu/g) | Remanent Magnetization (emu/g) | Coercivity (Oe) |
|--|----------------------------------|--------------------------------|-----------------|
| 20% : 80% | 19.24 emu/g | 5.82 emu/g | 371.18 Oe |
| 30% :70% | 26.55 emu/g | 7.41 emu/g | 387.59 Oe |
| 40% : 60% | 34.22 emu/g | 11.09 emu/g | 405.19 Oe |

From Table 3, it can be seen that the Saturation Magnetization (Ms), Remanent Magnetization (Mr) and Coercivity (Hc) values of each composition variation have different values. The ability of the magnetic field when magnetization reaches a saturation point when the value no longer changes is known as saturation magnetization (Ms). The magnetization that is still present when the external magnetic field is zero is known as remanent magnetization (Mr). The amount of external magnetic field required to achieve zero magnetization is known as coercivity (Hc).

From the data obtained, it can be seen that the highest Saturation Magnetization (Ms), Remanent Magnetization (Mr) and Coercivity (Hc) values are obtained in the 40% : 60% composition variation. The 20%:80% composition variation yielded the lowest saturation magnetization (Ms), remanent magnetization (Mr), and coercivity (Hc) values. The difference in coercivity values occurs due to differences in the composition of the addition of Fe₃O₄ in graphene oxide. Based on the coercivity value (Hc) obtained from the three hysteresis curves, it can be seen that the Fe₃O₄-Graphene Oxide nanocomposite includes a hard magnet which is in accordance with research by Przybyl et al. which states that the coercivity value for hard magnetic materials is greater than 125 Oe [29] and research by Maniur et al. that permanent magnets have a wide hysteresis curve so they must have high remanence and coercivity values to achieve high energy density. Therefore, to magnetize a permanent magnet that has a high coercivity value requires a large external magnetic field [30].

The Vibrating Sample Magnetometer (VSM) measurement results show that the smaller the grain size (diameter) of magnetite nanoparticles, the higher the magnetic response of the nanoparticles. The magnetic properties of nanocomposites are largely determined by the crystallite or grain size, crystallite shape and distribution, and the type of magnetic phase that makes up the composite structure. Intergranular exchange interactions as well as dipolar interactions play an important role in determining the magnetic properties of nanocomposite magnets. The effect of the exchange interaction of two magnetic phases is to combine the high coercivity (Hc) of the hard phase and the high magnetization (Ms) of the soft phase. In addition, the variation in saturation magnetization values is also thought to be due to the effect of particle size. The larger the particle size, the higher the coercivity value. This result also confirms the suitability of the theory which states that the smaller the particle size, the easier it is for particles to undergo magnetization [31].

In theory, the greater the addition of Fe₃O₄ composition, the greater the coercivity field value produced [32]. In addition, the addition of Fe₃O₄ causes an increase in the magnetic properties of remanent magnetic (Mr), saturation magnetic (Ms) and coercivity value (Hc) [33]. This can be found in the research that has been done, where the highest coercivity value is found in the Fe₃O₄-Graphene Oxide nanocomposite variation of 40%: 60%. In this variation, the composition of Fe₃O₄ in graphene oxide is more than the other two variations so that it can be seen that the research conducted has matched the existing theory.

4. Conclusion

Based on the research results that have been obtained, it can be concluded that the effect of variations from the addition of Fe₃O₄ into graphene oxide in this study affects the magnetic properties of the resulting Fe₃O₄-Graphene Oxide nanocomposite. The more Fe₃O₄ added to graphene oxide, the stronger the magnetic properties produced. This is evidenced by the high value of saturation magnetization (Ms), remanent magnetization (Mr) and coercivity value (Hc) in the 40% variation: 60% where the amount of Fe₃O₄ is more than the other composition variations. The Fe₃O₄-Graphene Oxide nanocomposite that has been produced is expected to be applied as one of the technologies, namely hard disk drives which are characterized by high coercivity values (greater than 125 Oe) where this nanocomposite is classified into a type of hard magnet or hard magnet.

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