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Structure Analysis Of Fe_3O_4-Graphene Oxide Nanocomposite From Corn Cob Waste

Ismira, Rahmat Hidayat*, Ratnawulan, Riri Jonuarti

Department of Physics, Universitas Negeri Padang, Padang 25131, Indonesia

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Corresponding Author
*Author Name: Rahmat Hidayat
Email: r.hidayat@fmipa.unp.ac.id

Abstract: This research is a study of the structure of nanocomposites whose composition is varied between Fe_3O_4 and Graphene Oxide. The purpose of this study is to determine the structure of Fe_3O_4-Graphene Oxide nanocomposites from corn cob waste. This type of research is experimental research, which begins with sample preparation from corn cob waste, carbon activation, graphene oxide synthesis, sonication and neutralization of graphene oxide, and synthesis of Fe_3O_4-Graphene Oxide nanocomposites using the ball milling method by varying the composition. Crystal size and structure were characterized by XRD, functional groups were characterized by FTIR, surface morphology, particle size and porosity were characterized by SEM. The results of the research on the structure of Fe_3O_4-Graphene Oxide nanocomposites from corn cob waste, obtained XRD results show that the crystal structure is Hexagonal, Cubic, and Orthorhombic. FTIR results show that all compositional variation comparisons have C-H, O-H, C≡C, C=O, C=C, C-O, and Fe-O bond functional groups. SEM results show the morphology of Fe_3O_4 is spherical, while the morphology of graphene oxide is in the form of chunks. The particle size value gets smaller as the Fe_3O_4 composition increases, because the more Fe_3O_4 composition causes the surface of the graphene oxide particles covered to get bigger. In addition, particle size can also affect the porosity of nanocomposites, the smaller particle size can increase porosity because more particles can occupy space.

Keywords: Structure, Nanocomposite, Graphene Oxide, Corn Cob, Composition Variation.

1. Introduction

The population growth in Indonesia continues, causing the demand for energy to increase year by year. Energy is a key component in social development and plays an important role in protecting the environment and supporting technological advancement. Despite this, the use of petroleum-based fuels still dominates and has not been replaced by other alternative energy

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sources. One sector that relies heavily on petroleum is transportation, where energy options for this sector are limited and vulnerable to scarcity. With the declining supply of petroleum fuels, innovation is needed in providing energy for transportation, and one of the main alternatives is to use electric power, as proposed with the use of electric cars [1].

An electric car is a vehicle that uses electricity as an energy source to operate. The basic components of an electric car include a battery that stores energy, an electric motor that drives the wheels, and a controller that regulates the flow of energy to the motor. The batteries used in electric cars are usually rechargeable batteries such as lithium ion batteries. The advantage of lithium ion batteries as an option for electric cars is their ability to store large amounts of energy and be able to be recharged in a short time, less than ten minutes [2]. However, lithium ion batteries have disadvantages, such as a relatively short lifespan and relatively expensive price. To overcome this, one solution is to replace the negative electrode (anode) of lithium ion batteries with carbon based materials [3].

Graphene is a carbon-based material that has a honeycomb-like hexagonal arrangement [4]. Graphene has wide application capabilities, including in the fields of batteries, polymer fillers, sensors, energy conversion, and energy storage devices. Therefore, the demand for graphene is expected to continue to increase and needs to be improved [5]. In addition to graphene, there is also graphene oxide which is a mixed compound of carbon, hydrogen, and oxygen produced through a strong oxidation process from graphite [6]. The properties of graphene and graphene oxide are that both have a surface area of 1 m^2, but weigh only 0.77 mg. Both of these materials are examples of two-dimensional materials. However, this two-dimensional (2D) material cannot be found naturally and requires a synthesis process from graphite [7].

A fairly efficient method is the chemical method, which involves graphite oxidation and is followed by exfoliation using sonication. One of the popular chemical methods is the Hummer method, which is fast and considered safe [8]. Pure graphite is usually the raw material for graphene oxide production, although it tends to be expensive. On the other hand, abundant sources of graphene are naturally available in nature, such as activated carbon. Activated carbon is an organic material with high carbon content. Nowadays, many studies are using natural materials that are easy to find and environmentally friendly, which are often referred to as biomass.

The most commonly found biomass is corn cobs. Corn cobs have a fairly high carbon compound content, reaching 43.42% carbon and 6.32% hydrogen [9]. Corn cob waste can also be utilized as an alternative raw material in the manufacture of nanocomposite materials [4]. One of the nanotechnologies used in the field of material science is nanocomposite material.

Nanocomposite is a type of material that combines two elements, namely matrix as reinforcement and filler as filler [10]. Fe_3O_4 is one type of nanoparticle known as magnetite. Meanwhile, graphene oxide has advantages in the synthesis process, solubility, customizable conductivity, large surface area, biocompatibility, and being an abundant and economically affordable material source [11]. Although these two materials have been the subject of intensive and separate research, research on Fe_3O_4-Graphene Oxide nanocomposites is still limited.

Research on graphene oxide from corn cob waste has been conducted previously by [12] with the title "Microstructure Analysis of Graphene Oxide Based on Corn Cob Waste Synthesized by the Modified Hummer Method." This research uses variations in combustion temperature of 300 ℃, 350 ℃, 400 ℃, and 450 ℃ with a combustion duration of 30 minutes.

The sample testing results show that graphene oxide material is formed with functional groups containing bonds between carbon (C), hydrogen (H), and oxygen (O). In addition, the results indicated that the smaller the grain size, the larger the surface area.

There has been no in-depth research on Fe_3O_4-graphene oxide nanocomposites from corn cob waste. By combining Fe_3O_4 and graphene oxide into nanocomposites, the advantages of these two materials can be integrated, creating new materials with improved properties and broader functions. Analysis of the structure of Fe_3O_4-graphene oxide nanocomposites is necessary because it is an important step in material development and characterization, as well as a guide for further research to study other properties. Therefore, the author proposes a study entitled "Structure Analysis of Fe_3O_4-Graphene Oxide Nanocomposites from Corn Cob Waste". By understanding the structure of nanocomposites, we can study the basic structure of the material using SEM for data interpretation in the form of images of surface morphology, particle size, and porosity. In addition, XRD is used to analyze X-ray diffraction patterns and crystal structure, and FTIR is used to determine functional groups.

2. Materials and Method

This research was basic research that aimed to increase scientific knowledge or discover new areas of research without specific practical objectives. This type of research was experimental research. The materials used in this research were Graphene Oxide Powder where the material was from Corn Cob waste, Sodium Hydroxide (NaOH), Sodium Nitrate ($NaNO_3$), 98% Sulfuric Acid (H_2SO_4), Potassium Permanganate ($KMnO_4$), 30% Hydrogen Peroxide (H_2O_2), and Distilled Water. The tools used included mortar and pestle, 100 mesh sieve, measuring cup, erlenmeyer, magnetic stirrer, oven, furnace, vaporizer cup, digital balance, funnel, volumetric flask, baking pan, volume pipette, centrifuge tube, centrifuge device, magnetic stirrer bar, fume hood, spatula, stirring rod, distilled water spray bottle, and ultrasonic.

The first step of this research was sample preparation, which involved cleaning the corn cob waste, cutting it into small pieces, and drying it in the sun for 3 days. After that, the water content in the corn cobs was removed using an oven at 100°C for 1 hour [6]. Next, the corn cobs were processed in a furnace at 400°C for 2 hours until they turned into biochar. After turning into biochar, the corn cobs were pulverized using a mortar and pestle until they reached the form of charcoal powder, and then were filtered using a 100 mesh sieve.

The second step was the activation of corn cob carbon. After the corn cob was ground into powder, the initial stage of activation began by preparing a NaOH solution using 100 ml of distilled water was measured by measuring flask and mixed with 8 grams of solid NaOH. The solid NaOH then dissolved completely in distilled water, and the solution became homogeneous. Next, a 250 mL beaker was filled with 8 grams of corn cob charcoal powder, and 100 ml of NaOH solution was poured into it so that the charcoal was submerged in the solution. This soaking process lasted for 24 hours, after which a precipitate formed at the bottom of the beaker. The next step was to filter the sediment using filter paper and funnel to separate the liquid from the corn cob powder. Filter paper that had been moistened with distilled water was placed in the funnel to filter the mixture of corn cob and NaOH solution. The filtered corn cob powder was then transferred into a vaporizer cup. Next, the activated corn cob powder was dried for 3 hours at 105°C in an oven [13].

The third step was the synthesis of graphene oxide from corn cob waste. Graphene oxide was synthesized using the modified Hummers method. The synthesis process began by mixing 1.5 grams of activated carbon powder from corn cobs and 0.75 grams of $NaNO_3$ in a 250 ml Erlenmeyer. Then, 34.5 ml of 98% H_2SO_4 was added to the mixture [14]. The mixture was then stirred for 2 hours and 20 minutes in an ice bath at temperatures between 0 and 5°C at a constant speed of 250 rpm. Afterwards, the ice bath was removed and 4.5 grams of $KMnO_4$ was added slowly without using the ice bath for 30 minutes at a temperature of about 35ºC. After 30 minutes, 69 ml of distilled water was slowly added using a drop pipette while stirring for 20 minutes. The temperature would rise when adding distilled water due to an endothermic reaction, the temperature had to remain below 50℃ to see the oxidation process, which was marked by a dark brown color change and the appearance of bubbles. Then, another 100 ml of distilled water and 1.5 ml of 30% H_2O_2 were added. The addition of H_2O_2 aimed to remove the remaining $KMnO_4$ or stop the reaction, so that the solution changed color to yellow, indicating the presence of graphene oxide. Finally, 50 ml of distilled water was added, and graphene oxide was formed [15].

The fourth step was sonication and neutralization of graphene oxide. After the color of the solution changed to yellow, indicating the presence of graphene oxide, the mixture was sonicated for 2 hours to exfoliate the graphite into graphene [16]. Next, the solution was sedimented for 1 day until liquid and solid phases were formed [17]. After that, the centrifugation process was carried out using a micro centrifuge at 4000 rpm for 15 minutes to separate the solid and liquid phases. The centrifugation process was followed by manual neutralization of graphene oxide, where graphene oxide powder was precipitated and distilled water was replaced repeatedly until the pH reached neutral 7. Once a neutral pH was reached, the graphene oxide was oven dried at 105°C for 1 hour [18].

The fifth step was the synthesis of Fe_3O_4-Graphene Oxide Nanocomposite. The synthesis of Fe_3O_4-Graphene Oxide nanocomposites began by mixing the two materials in various composition ratios (20%:80%, 30%:70%, and 40%:60%). The mixing process was carried out through the ball milling method at a speed of 3000 rpm for 180 seconds [19]. The purpose of this step was not only to mix the ingredients, but also to achieve particle size homogenization.

The final step was sample characterization. The Fe_3O_4-Graphene Oxide nanocomposites derived from corn cobs were characterized using several analytical methods, such as characterization of crystal size and structure using X-Ray Diffraction (XRD), characterization of functional groups using Fourier Transform Infra Red (FTIR), and characterization of surface morphology and particle size using Scanning Electron Microscope (SEM).

3. Results and Discussion

The results of data analysis of Fe_3O_4-Graphene Oxide nanocomposites using XRD with composition variations (40%:60%, 30%:70%, and 20%:80%) can be seen in Figure 1 below.

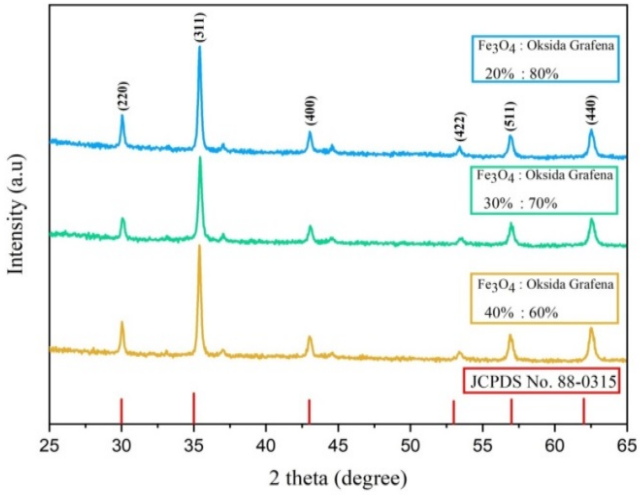


Figure 1. XRD Results of JCPDS No. 88-0315, and Variation of Composition of Fe_3O_4-Graphene Oxide Nanocomposite

Based on Figure 1, it can be seen that the diffraction patterns of Fe_3O_4-Graphene Oxide nanocomposites with three composition variations are compared with JCPDS standard No. 88-0315 [20]. Diffraction peaks appeared at angles 2θ = 30, 35, 43, 53, 57, and 62. These peaks correspond to the standard XRD data for Fe_3O_4-Graphene Oxide (JCPDS No. 88-0315). The Miller indices identified for each peak are (220), (311), (400), (422), (511), and (440).

In the 40%:60% composition variation, the nanocomposite Fe_3O_4-Graphene Oxide has two phases, namely Hydrogen and Iron Oxide, with crystal structures namely Hexagonal, Cubic, and Orthorhombic, having significant diffraction angles of 30.03°, 35.38°, 43.03°, 53.34°, 56.91°, and 62.50° with FWHM of 0.15°, 0.20°, 0.25°, 0.30°, 0.12°, and 0.30° respectively. The results of the diffractogram pattern analysis used the Scherrer equation to determine the crystal size. The crystal sizes obtained were 53.58 nm, 40.75 nm, 33.38 nm, 28.95 nm, 70.64 nm, 30.26 nm, with an average crystal size of 42.93 nm.

In the 30%:70% composition variation, the nanocomposite Fe_3O_4-Graphene Oxide has two phases, namely Carbon and Iron Oxide, with a crystal structure that is Hexagonal, and Cubic, having significant diffraction angles of 30.05°, 35.44°, 43.05°, 53.48°, 56.99°, and 62.56° with FWHM of 0.15°, 0.17°, 0.17°, 0.40°, 0.25°, and 0.30° respectively. The results of the diffractogram pattern analysis used the Scherrer equation to determine the crystal size. The crystal sizes obtained are 53.58 nm, 46.57 nm, 47.68 nm, 21.73 nm, 35.33 nm, 30.27 nm, with an average crystal size of 39.19 nm.

In the composition variation of 20%:80%, the nanocomposite Fe_3O_4-Graphene Oxide has three phases namely Hydrogen, Carbon Oxide, and Iron Oxide, with crystal structures namely Hexagonal, and Cubic, having significant diffraction angles of 30.05°, 35.39°, 43.04°, 53.40°, 56.94°, and 62.48° with FWHM of 0.12°, 0.20°, 0.17°, 0.15°, 0.25°, and 0.35° respectively. The results of the diffractogram pattern analysis used the Scherrer equation to determine the crystal size. The crystal sizes obtained were 64.30 nm, 40.74 nm, 47.68 nm, 57.93 nm, 35.32 nm, 25.94 nm, with an average crystal size of 45.32 nm.

In the research conducted, the relationship between diffraction angle (2θ) and intensity (I) has been analyzed on Fe_3O_4-Graphene Oxide nanocomposite using XRD. The analysis results show that as the number of intensity readings received by the detector increases, the diffraction intensity becomes sharper and pointed. The diffraction patterns of the three variations of Fe_3O_4-

Graphene Oxide nanocomposite composition compared to the JCPDS standard No. 88-0315 showed peaks at 2θ = 30, 35, 43, 53, 57, and 62. The peak at 43° indicates the presence of graphene oxide in the Fe_3O_4-Graphene Oxide composite. These results are in accordance with the standard XRD data for Fe_3O_4-Graphene Oxide (JCPDS No. 88-0315) research conducted by [20]. The analysis showed that all peaks formed exhibited FCC (Face Center Cubic) phase, confirming the successful formation of Fe_3O_4 composite with graphene oxide, in line with the research findings by [21]. According to research [22] the Scherrer equation concluded that the higher the FWHM value of an XRD peak, the smaller the crystal size. From the results of XRD analysis of the average crystal size of the three variations of Fe_3O_4-Graphene Oxide nanocomposite composition, it was found that the composition of 30%:70% Fe_3O_4-Graphene Oxide was the best. This is because the crystal size produced is relatively small, namely 39.19 nm, compared to other composition variations. The results of data analysis of Fe_3O_4-Graphene Oxide nanocomposites using FTIR with composition variations (40%:60%, 30%:70%, and 20%:80%) can be seen in Figure 2 below.

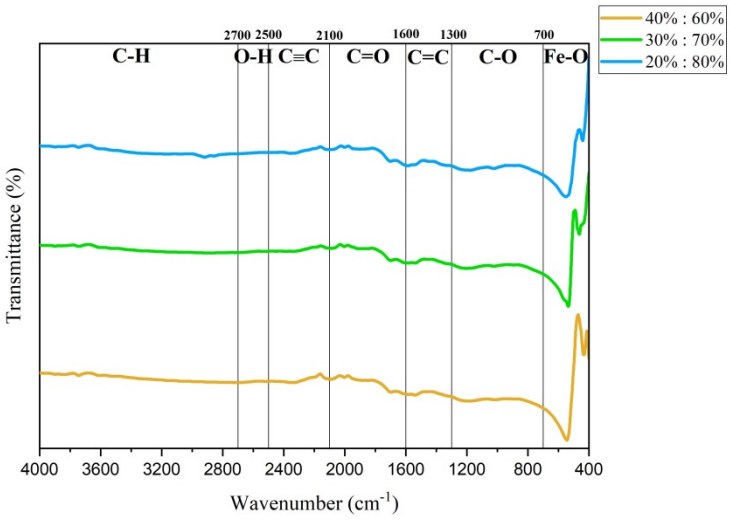


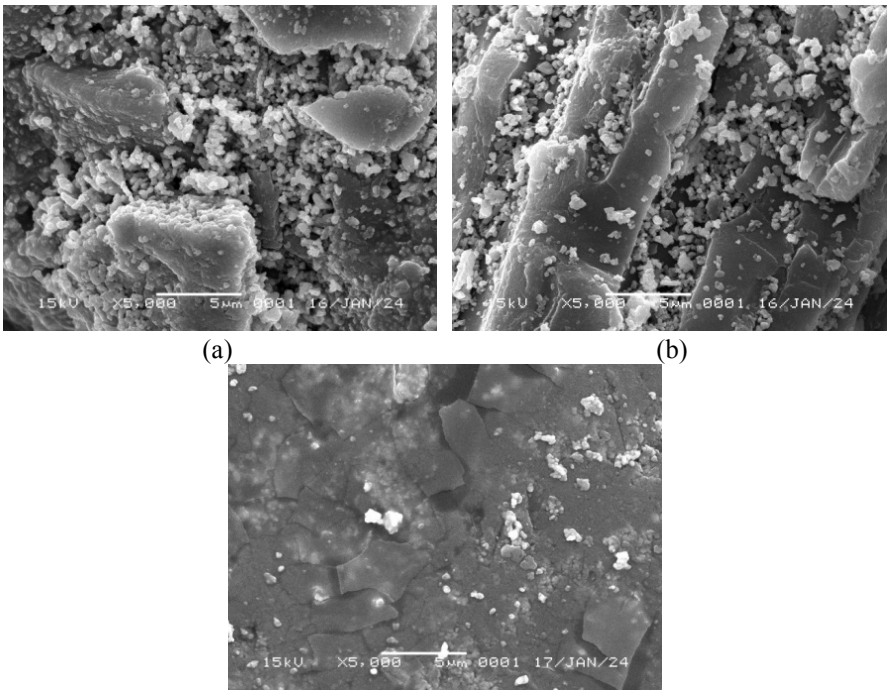
Figure 2. FTIR spectra of variation of Fe_3O_4-graphene oxide nanocomposite composition (40%:60%, 30%:70%, and 20%:80%)

Based on Figure 2, we can see the FTIR spectrum of Fe_3O_4-Graphene Oxide nanocomposite with three variations in composition. Characterization of the composition variation of 40%:60% Fe_3O_4-Graphene Oxide nanocomposite, C-H bonds are formed at wave numbers 3852.51 cm-1, and 3744.27 cm-1, O-H bonds are formed at 2714.98 cm-1, C≡C bonds are formed at 2328.23 cm-1, C=O bonds are formed at 2091, 24 cm-1, 2001.30 cm-1, and 1698.52 cm-1, formed C=C bonds at 1540.64 cm-1, formed C-O bonds at 1190.47 cm-1 and 1017.54 cm-1, and formed Fe-O bonds at 543.26 cm-1, 436.90 cm-1, 431.29 cm-1.

In the composition variation of 30%: 70% Fe_3O_4-Graphene Oxide nanocomposite, C-H bonds were formed at wave numbers 3899.97 cm-1, 3743.56 cm-1, and 2906.49 cm-1, C=O bonds were formed at 2085.68 cm-1, 2002.62 cm-1, 1902.21 cm-1, and 1698, 85 cm-1, formed C=C bonds at 1584.59 cm-1 and 1540.96 cm-1, formed C-O bonds at 1203.75 cm-1 and 1023.32cm-1, and formed Fe-O bonds at 534.65 cm-1, and 462.37 cm-1. In the composition variation of 20%: 80% Fe_3O_4-Graphene Oxide nanocomposite, C-H bonds were formed at wave numbers 3899.60 cm-1, 3744.33 cm-1, and 2918.80 cm-1, O-H bonds were formed at 2352.99

cm-1, C=O bonds were formed at 2089.42 cm-1, 2000, 83 cm-1, 1902.09 cm-1, and 1699.98 cm-1, formed C=C bonds at 1586.09 cm-1, formed C-O bonds at 1178.61 cm-1, and 1022.28 cm-1, and formed Fe-O bonds at 551.36 cm-1, 466.28 cm-1 and 439.30 cm-1.

Analysis of Fe_3O_4-Graphene Oxide nanocomposite using FTIR showed the presence of C-H, O-H, C≡C, C=O, C=C, C-O, and Fe-O bonds. An Fe-O absorption peak in the range of 400-700 cm-1 was detected. According to research [23] the presence of Fe-O absorption peak indicates that Fe_3O_4 has been successfully composited with graphene oxide. Graphene Oxide vibrations on Fe_3O_4-Graphene Oxide can be observed at absorption peaks around 1100-1300 cm-1 and 1400-1600 cm-1 [24]. The results of data analysis of Fe_3O_4-Graphene Oxide nanocomposites using SEM with composition variations (40%:60%, 30%:70%, and 20%:80%) can be seen in Figure 3 below.

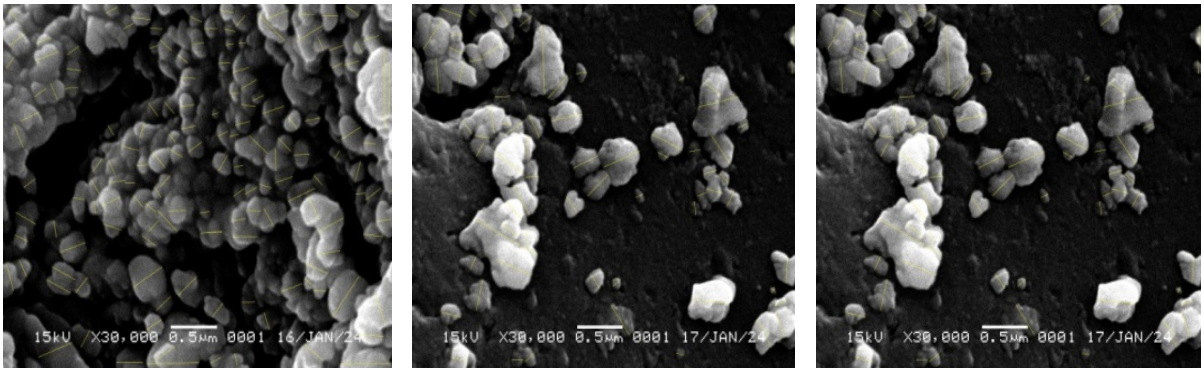


(a) (b)

(c)

Figure 3. SEM Result Data of Fe_3O_4-Graphene Oxide Nanocomposites Magnification 5.000× (a) 40%:60%, (b) 30%:70%, (c) 20%:80%

Based on Figure 3, the results of the analysis of Fe_3O_4-Graphene Oxide nanocomposites using SEM show that the surface morphology of Fe_3O_4 is round, while the morphology of Graphene Oxide is chunk-shaped. This is in accordance with the theory that states the morphological structure of graphene oxide is chunk-shaped. The chunks have thin particles and there are parts that clump inhomogeneously and overlap irregularly. The morphology of graphene oxide is formed in the form of chunks consisting of several layers that are stacked on top of each other and arranged irregularly. The data were analyzed using ImageJ software to measure the particle size of the samples, and then plotted using Origin software for particle size visualization. The coefficient of determination (R-Square or COD) was used to measure the extent of the relationship between the dependent and independent data. R-Square values range from 0 to 1, where closer to 1 indicates a better degree of association between the two types of data.



(a) (b) (c)

Figure 4. Particle Measurement of Fe_3O_4-Graphene Oxide Nanocomposite Magnification 30,000× (a) 40%: 60%, (b) 30%: 70%, (c) 20%: 80%.

Based on Figure 4, the data is processed using Imagej software to see the particle size of the sample and then plotted with Origin software to see the particle distribution. By using Imagej Software, the average diameter of the 40%:60% composition variation of Fe_3O_4-Graphene Oxide nanocomposite is 74.60 nm, and in Origin Software the COD result is 0.99. In the average diameter of the composition variation of 30%:70% -Graphene Oxide nanocomposite is 84.71 nm and in Origin Software the COD result is 0.99. In the average diameter of the composition variation of 20%:80% Fe_3O_4-Oxide Graphene nanocomposite is 90.49 nm and in Software Origin the COD result is 0.99. The results of porosity calculations using Origin software, porosity in the 40%:60% composition variation of Fe_3O_4-Graphene Oxide nanocomposite is 68.5%, in the 30%:70% composition variation of Fe_3O_4-Graphene Oxide nanocomposite is 64.5%, and in the 20%:80% composition variation of Fe_3O_4-Graphene Oxide nanocomposite is 64.3%.

In the research that has been conducted based on the results of the analysis of Fe_3O_4-Graphene Oxide nanocomposites using SEM shows that the morphology of Fe_3O_4 is spherical, while graphene oxide is in the form of chunks, this result is in line with the findings of the theory [25] which reveals that the morphological structure of Fe_3O_4 tends to be spherical, and the theory [26] which states that the morphological structure of graphene oxide is chunk-shaped. The chunks consist of thin particles with inhomogeneous parts and irregular overlaps, as described in previous research [27]. The morphology of graphene oxide formed consists of chunks composed of several layers that are stacked and irregular, in accordance with the findings in a previous study [28]. The particle size tends to decrease as the Fe_3O_4 composition increases because the more Fe_3O_4 particles in the composite, causes greater closure of the graphene oxide particle surface, in accordance with research [19]. In addition, particle size can also affect the porosity of nanocomposites, smaller particle sizes can increase porosity as more particles can occupy space. With more particles occupying space, more and smaller pores can be created in the composite, which in turn increases the overall porosity. High porosity has benefits in various fields, one of which is in energy storage such as batteries. Battery electrodes with high porosity allow the electrolyte to permeate more easily, increasing the contact between the electrode and electrolyte, and increasing the battery's energy storage capacity. The most optimal composition in terms of particle size and porosity is 40%:60%.

4. Conclusion

The results of research on the structure of Fe_3O_4-Graphene Oxide nanocomposites derived from corn cob waste have been analyzed using XRD, FTIR, and SEM tools. Based on XRD results, all composition variations showed the presence of Hydrogen, Carbon, Iron Oxide, and Carbon Oxide phases, with crystal structures consisting of Hexagonal, Cubic, and Orthorhombic. In terms of crystal size, the best composition variation was detected in the 30%:70% ratio because it produced an average particle size of 39.19 nm, which is smaller than the other composition variations. FTIR analysis showed that all composition variations have functional groups such as C-H, O-H, C≡C, C=O, C=C, C-O, and Fe-O bonds. Meanwhile, SEM results illustrate the spherical morphology of Fe_3O_4, while graphene oxide is in the form of chunks. The particle size value is getting smaller as the composition of Fe_3O_4 increases, because the increasing number of Fe_3O_4 particles in the composite, the surface of the graphene oxide particles covered is getting bigger. In addition, particle size can also affect the porosity of nanocomposites, the smaller particle size can increase porosity because more particles can occupy space. The best composition variation in terms of particle size and porosity is 40%:60%, as it manages to produce particles with a size of 74.6 nm, which is smaller than the other composition variations, and porosity with a size of 68.5%, which is larger than the other composition variations.

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