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Composition Effect of Fe_3O_4 and Graphene Oxide on The Microstructure of Fe_3O_4-Graphene Oxide Nanocomposite

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Abstract: The manufacture of lithium ion batteries is a solution to future energy limitations, but lithium ion batteries have a high price. The best solution to overcome the high price of lithium ion batteriesis to make lithium ion battery electrodes from Fe_3O_4 nanoparticles.To optimize the performance of Fe_3O_4, it will be combined with graphene oxide made from coconut shell. This study aims to analyze the effect of Fe_3O_4:Graphene Oxide composition (40%:60%, 30%:70%, and 20%:80% ratio) on the microstructure of Fe_3O_4-Graphene Oxide nanocomposite which includes crystal size, functional groups, and surface morphology. The method used to make graphene oxide is by using the modified hummers method because this method is faster, safer, and has higher efficiency. The mixture of Fe_3O_4 variation with graphene oxide will be characterized using XRD, FTIR, and SEM. The result of XRD obtained crystal size for Fe_3O_4-Graphene Oxide nanocomposite composition 40%: 60%is 44.73 nm, for the composition of Fe_3O_4-Graphene Oxide nanocomposite 30%: 70%, the crystal size is 39.71 nm, and for Fe_3O_4-Graphene Oxide 20% nanocomposite composition: 80%, the crystal size is 45.64 nm. FTIR results showed the presence of C-H, O-H, C≡C, C=O, C-O bonds, and the presence of Fe-O absorption peaks indicating the success of the Fe_3O_4 with graphene oxide composite. SEM analysis results showed that the surface morphologyof Fe_3O_4-Graphene Oxide nanocomposite has a spherical structure, which can be seen that the more Fe_3O_4 composition in the composite, the larger the graphene oxide surface is covered.

Keywords: coconut shell waste; graphene oxide; microstructure; nanocomposites of Fe_3O_4.

1. Introduction

Energy demand will continue to increase in proportion to the increase in population and technological advances in various sectors. Energy is a necessity that will not be able to escape its use for humans in thepresent and the future. The limitation of energy which is non-renewable so that

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it becomes one of the problems for the future. The solution that can be done is in the manufacture of energy storage devices (batteries). Batteries are an environmentally friendly energy storage that has no harmful exhaust emissions that contribute to global warming. Batteries can guarantee large amounts of energy that can supply the power load required by various electronic devices [1]. The batteries that are currently widely used and developed are lithium ion batteries. This lithium ionbattery has the advantages of storage with better capacity, high absorption, light mass, high energy density, not having memory effect properties, can be recharged and can be used many times. However, these batteries have disadvantages, that is, a short life time and will degrade if completely discharged, and the priceis relatively expensive [2].

The solution to overcome the high price of lithium ion batteries is to manufacture lithium ion battery electrodes from Fe_3O_4 nanoparticles Fe_3O_4 can be separated from the media by a simple, efficient, economical and non-toxic magnetic process. Fe_3O_4 is also known as black iron oxide, magnetic iron ore, loadstone, ferrousferrite, or hercules stone which shows the strongest magnetism among transition metal oxides. So it has great potential to be used as an anode in lithium-ion batteries [3]. Fe_3O_4 is a magnetic material that has a magnetite phase and belongs to the iron oxide group [4]. Fe_3O_4 has unique nanoscale properties such as high surface area, high absorption, electron transfer, and superparamagnetic [5]. To optimize the performance of Fe_3O_4, it will be combined with graphene oxide. The addition of grapheneoxide to Fe_3O_4, will make the material more durable and have a high level of wear resistance withoutaffecting the surface quality (transparency) and will also improve its electrical conductivity and porosity [6].

Graphene oxide is a single-atom layered material that is composed of carbon, hydrogen, and oxygen molecules through the oxidation of graphite crystals [7]. Graphene Oxide has unique properties such as high mechanical resistance, good electrical properties, and high gas permeability, which makes Graphene Oxide an attractive material for various technological applications, such as air filtration, catalysts, and conductor materials [8]. Considering its advantages and potential, the requirement for graphene oxide will definitely continue to increase so that the availability of graphene oxide also needs to be increased.

Graphene oxide can be made from various sources, one of which is coconut shell. Coconut shell is part of the coconut fruit in the form of endorap with a hard structure and covered with coconut husk [9]. Coconut shell can be used as activated charcoal because it has very high carbon content (76.32%) and it is supported by easily available materials, therefore making it the most suitable material to be used as activatedcharcoal. In addition, coconut shells contain more carbon than other natural materials such as corn stalks, rice husks, and cocoa shells, which are only around 12-20% [10].

Coconut shell has been widely used in previous researches with various treatment variables, such as in research [9], using variations in charring temperature and found that 350°C was the optimal temperature for charring. Therefore, in this research, the optimal charring temperature will be used, which has been carried out at a temperature of 350°C. The process of making graphene oxide from coconut shell involves dehydration to remove moisture from the sample and charring with a temperature of 350°C to produce granules with high absorption and regular structure [11]. The coconut shell charcoal will be ground into coconut shell charcoal powder, then the coconut shell charcoal powder is activated to enlarge the pores by breaking hydrocarbon bonds or oxidizing molecules on the surface so that the surface becomes larger and will affect its absorption power and synthesized using the modified hummers method which uses chemical reactions to form graphene

oxide because this method can be completed in just a few hours, is safe and has a higher efficiency [12].

Before mechanical testing, it is highly recommended to observe the microstructure because it can be used as supporting data that the microstructure of the sample is in accordance with the specifications stated in the manufacturing certificate (mill certificate) or in other words as a material identification process. Therefore, the completeness of the data from the microstructure test sample is very helpful in maintaining the quality of the manufactured products. The mixture of Fe3O4 variations with graphene oxide will be characterized using X-Ray Diffraction (XRD) to observe and determine the crystal structure formed. Characterization with Fourier Transform Infrared (FTIR) is used to determine the functional groups of the sample, where the data generated by FTIR will support SEM data in analyzing the nature of the microstructure of graphene oxide. Scanning Electron Microscope (SEM) will also be used to observe thesurface morphology of the sample and the particle size.

The microstructure characterization is used because it can affect the mechanical and physical properties of a material such as strength, hardness, durability and is needed for other characterizations. Microstructure characterization is carried out to determine the condition of the microstructure of a material. Observations are usually made by measuring the degree of criticality, crystal size and also surface morphology.

2. Materials and Method

This type of research is an experimental research. The study in this research was about the microstructure of Fe3O4-Graphene Oxide nanocomposite using coconut shell waste. The analysis was conducted using XRD, FTIR, and SEM characterization tools. The preparation of graphene oxide from coconut shell waste was carried out using the modified hummers method with the process that can be seen in Figure 1. Modified hummers method corresponding to the process in figure 1 uses many chemical compound solutions, such as NaOH, KMnO4, H2SO4, NaNO3, and H2O2 which were used to peel off the graphite layer [13]. This method has the advantages of other methods, such as, the reaction can be completed in just a few hours, replacing the explosive KClO3 with KMnO4 to improve safety, eliminating the formation of acid mist because it used NaNO3 instead of HNO_3 so it has higher efficiency, and the safety level of the reaction was also quite high [12].

The first thing to prepare the sample was to clean the coconut shell from the husk. After cleaning the coconut shell, it was dried for 3 days in the sun and cut into small pieces. To remove the remaining moisture content in the coconut shell, the coconut shell will be oven waste at 100℃ for 1 hour and furnace samples at 350℃ for 2 hours which was then pulverized and sieved with a sieve size of 125 mesh [9] which was carriedout at the LLDIKTI Region X Padang Laboratory.

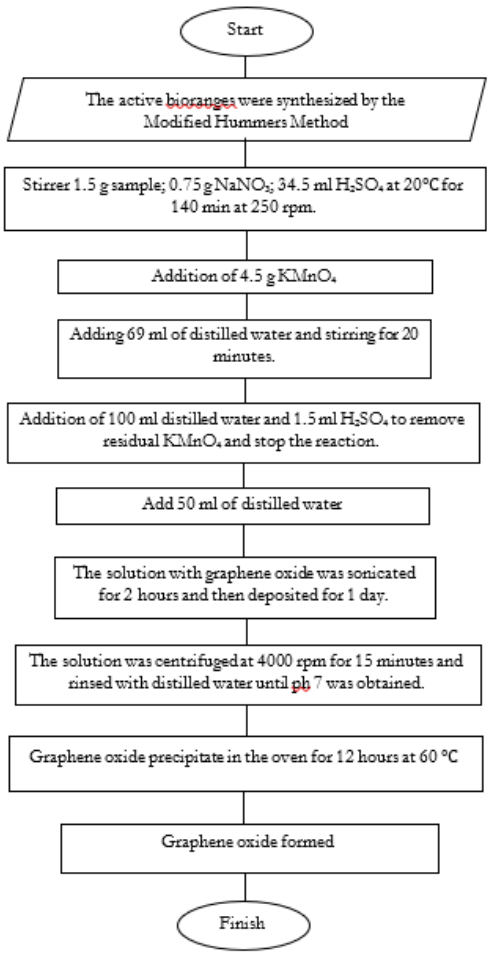


Figure 1. The process of forming graphene oxide by the modified hummers method

After the coconut shell becomes powder, carbon activation will be carried out by mixing coconut shell powder with NaOH. The method was to add 8 grams of NaOH solids into a measuring flask using a spatula until the NaOH solids dissolved and the solution becomes homogeneous. In a beaker, put 8 grams of coconut shell charcoal powder and add 100 ml of NaOH solution so that the coconut shell charcoal powder was submerged in the NaOH solution. This immersion was carried out for 24 hours [14]. After the mixture has been submerged, there will be a precipitate at the bottom of the beaker. To separate the coconut shell powder from the liquid, this precipitate will be filtered using filter paper that has been moistened with distilled water. The activated coconut shell powder will be dried using an oven at 105℃ for 3 hours [15]. The results of activation will then be checked for silica content contained therein using X-Ray Fluorescence (XRF). A good activated carbon the amount of silica contained should be less than 5% and if the silica content exceeds 5% then silica removal must be carried out first. Silica removal was carried out using NaOH.

Synthesis of graphene oxide was carried out using the modified hummer method. The first step was that 1.5 grams of coconut shell charcoal powder and 0.75 grams of NaNO3 are put into a 250 ml enlemeyer tube. Then added 98% H2SO4 as much as 34.5 ml and stirred for 20 minutes at a constant speed of 250 rpm [16]. The next step was to move the enlemeyer into an ice bath and stirrered again for 2 hours. After that, add KMnO4 as much as 4.5 grams which was done slowly to keep the temperature below 20℃ and stirred again for 30 minutes until the solution turned pale

brown [17]. After that, add distilled water as much as 69 ml, this process was also carried out slowly because there was an endothermic reaction so that the temperature was kept below 50℃ to see the oxidation process which was marked by the mixture turning dark brown. After the addition of distilled water, stirring was again carried out for 20 minutes. Next, 100 ml of distilled water, 1.5 ml of 30% H_2O_2, and 50 ml of distilled water were added to the mixture [18].

The solution will change color to yellow which indicated the presence of graphene oxide [19]. Then the solution will be sonicated for 2 hours [13] and precipitated for 1 day [20]. After that, the solution will be centrifuged at 4000 rpm for 15 minutes to separate the solid and liquid phases. Furthermore, the precipitated graphene oxide solution will be neutralized by repeated replacement of distilled water until a neutral pH is obtained [21] The preparation of Fe_3O_4-Graphene Oxide composites was started by mixing the two in a ratio of 20%: 80%; 30% : 70%; and 40%: 60%. The mixing of the two was carried out using a ball milling tool at a speed of 300 rpm for 30 minutes [6]. The mixing using this ball milling tool in addition to mixing the two also aims to homogenize the particle size.

Data processing on XRD was used to determine the crystal structure and purity of the results formed. Each peak in the XRD pattern formed describes one crystal plane that has a certain orientation in the three- dimensional axis. In addition, the pattern formed was also confirmed in accordance with the JCPDS (Joint Committee on Powder Diffraction Standard) standard.

Data processing with FTIR was used to detect functional groups, analyze mixtures and identify changes in chemical bonds in samples. This quantitative analysis was carried out using standard compounds from the spectra produced at various concentrations. The data generated by FTIR will support SEM data in analyzing the microstructure properties of graphene oxide.

Data processing with SEM analysis was to observe the morphology on the surface of the sample and the particle size of the sample. SEM test results data in the form of curves, graphs, and images of electron morphology (structure). The results of the crystal size analysis will be adjusted to the COD value on the Origin software.

3. Results and Discussion

The XRD test was conducted to determine the crystallinity phase of the Fe_3O_4-oxide graphene nanocomposite. This method uses a 2θ angle range with a range of 10-70º. The results of XRD characterization of Fe_3O_4-oxide graphene nanocomposites will produce intensity peaks along the 2θ value with varying shapes. The results of data analysis of Fe3O4-Graphene Oxide nanocomposites using XRD with three composition variations, which are 40% : 60%, 30% : 70%, and 20%: 80% can be seen in Figure 2. The crystal size can be calculated using the Scherer equation obtained from the FWHM peak value in the resulting diffractogram pattern. At the composition of Fe_3O_4-Graphene Oxide nanocomposite 40% : 60%, the calculated crystal size is 44.73 nm. At the composition of Fe_3O_4-Graphene Oxide nanocomposite 30% : 70%, the calculated crystal size is 39.71 nm. And at the composition of Fe_3O_4-Graphene Oxide nanocomposite 20% : 80%, the calculated crystal size is 45.64 nm.

Based on the figure 2 shows the diffraction pattern of Fe3O4-Graphene Oxide nanocomposite of 40% composition: 60%, 30% : 70%, and 20%: 80% using Origin software and will be compared with JCPDS No.88-0315 as a standardization of XRD results from Fe3O4-Graphene Oxide, which are at peaks 2θ = 30, 35, 43, 53, 57, and 62. From the figure it can be seen that each composition comparison looks the same. It can be ascertained that the data generated is appropriate.

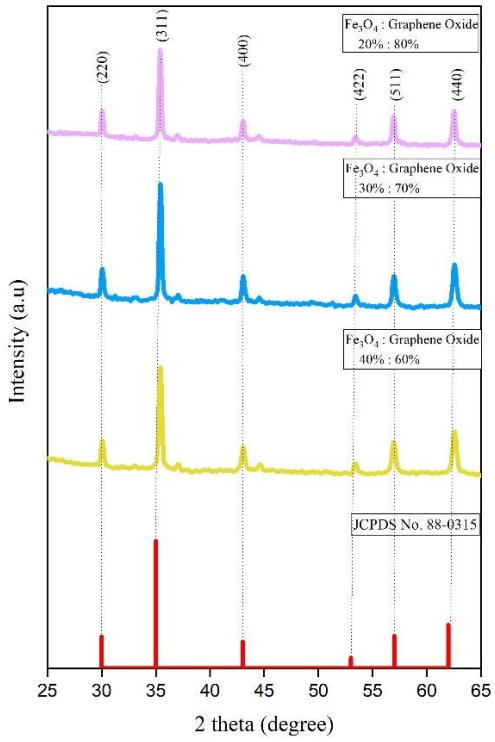


Figure 2. XRD result data JCPDS no. 88-0315, Fe_3O_4-graphene oxide nanocomposite composition 40%: 60%, Fe_3O_4-graphene oxide nanocomposite composition 30%: 70%, and, Fe_3O_4-graphene oxide nanocomposite composition 20%: 80%

Based on the peaks formed, it is in accordance with the standard XRD data for Fe3O4-Graphene Oxide (JCPDS No. 88-0315), that is for the composition variation of Fe3O4-Graphene Oxide nanocomposite 40%: 60%, 2θ = 30.07; 35.41; 42.97; 53.39; 56.98; and 62.50 were obtained. For the composition variation of Fe3O4-Graphene Oxide nanocomposite 30% : 70%, 2θ = 30.03; 35.40; 43.01; 53.45; 56.95; and 62.52 were obtained. For the composition variation of Fe3O4-Graphene Oxide nanocomposite 20%: 80%, 2θ = 30.07; 35.41; 42.05; 53.36; 56.98; and 62.54 were obtained.

Characterization using FTIR was carried out to determine the functional groups contained in the Fe3O4-Graphene Oxide Nanocomposite. The results of data analysis of Fe3O4-Oxide Graphene nanocomposites using FTIR with three composition variations, which are 40% : 60%, 30% : 70%, and 20%: 80% can be seen in the Figure 3.

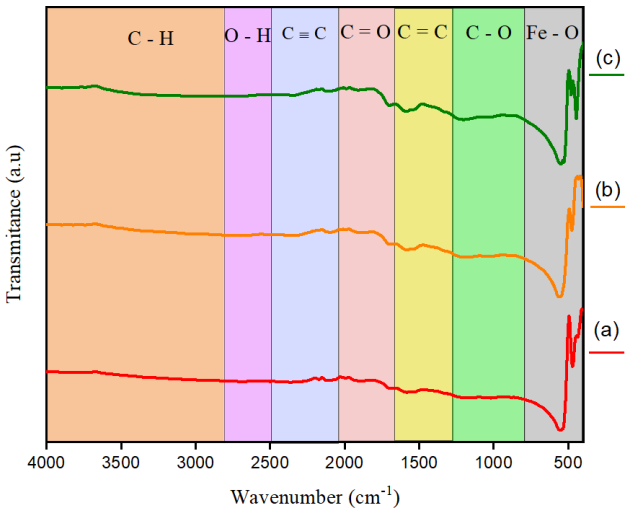


Figure 3. FTIR result data of Fe_3O_4-graphene oxide nanocomposite 40%: 60%, Fe_3O_4-grapheneoxide nanocomposite 30%: 70%, and Fe_3O_4-graphene oxide nanocomposite 20%: 80%

Based on the Figure 3 showing the FTIR spectrum of Fe3O4-Graphene Oxide Nanocomposites from three variations of composition, which are 40 %: 60%, 30% : 70%, and 20%: 80%, the presence of C-H, O- H, C≡C, C=O, C-O bonds, and the presence of Fe-O absorption peaks indicate that Fe3O4 has been successfully composited with graphene oxide. The surface morphology and particle size of Fe_3O_4-Graphene Oxide nanocomposite can be analyzed using SEM characterization tool. The morphology image produced will be analyzed using Image-J software. SEM characterization results for Fe3O4-graphene oxide nanocomposite composition 40%: 60% with 10,000 x magnification can be seen in Figure 4.

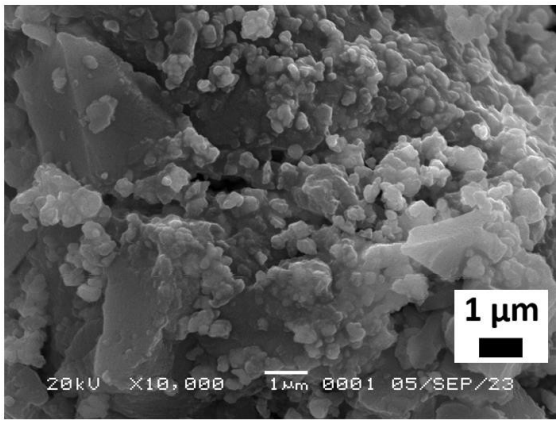
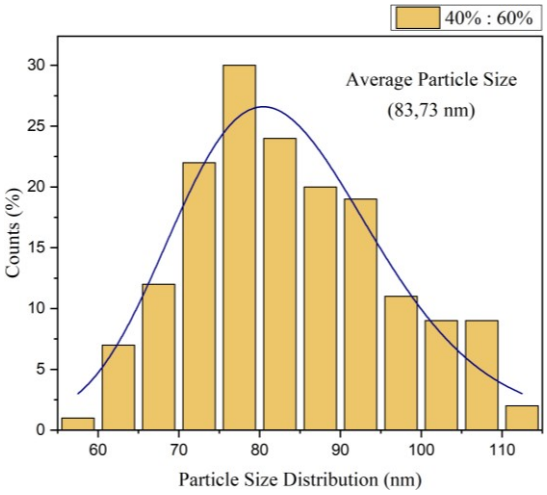


Figure 4. SEM Results data with 10,000x magnification on Fe_3O_4-oxide graphene nanocomposite composition 40%: 60%

Based on Figure 4 SEM characterization results on the composition of Fe3O4-Graphene Oxide nanocomposite, it can be seen that the surface morphology of Fe3O4 is in the form of small bright grains attached to graphene oxide which looks like a larger slab. for Fe3O4-Graphene Oxide 40%:60% composition, it can be seen that almost the entire surface of graphene oxide is covered by Fe3O4.

The particle size from the characterization using SEM for variations in the composition of Fe3O4- Graphene Oxide nanocomposites 40%: 60% can be further processed through Image-J digital processing software and plot the particle size distribution graph using the Origin application which can be seen in the following Figure 5.



40%:60%

Figure 5. Particle size distribution chart for Fe_3O_4-graphene oxide nanocomposite composition

Based on Figure 5 which has been plotted using image-J and Origin applications, the average particle size for the Fe3O4-Graphene Oxide 40%:60% composition is 83.73 nm with a COD value in the Origin application of 0.9317 which proves the accuracy of the data obtained.

The surface morphology and particle size of Fe_3O_4-Graphene Oxide nanocomposite can be analyzed using SEM characterization tool. The morphology image produced will be analyzed using Image-J software. SEM characterization results for Fe3O4-graphene oxide nanocomposite composition 30%: 70% with 10,000 x magnification can be seen in Figure 6.

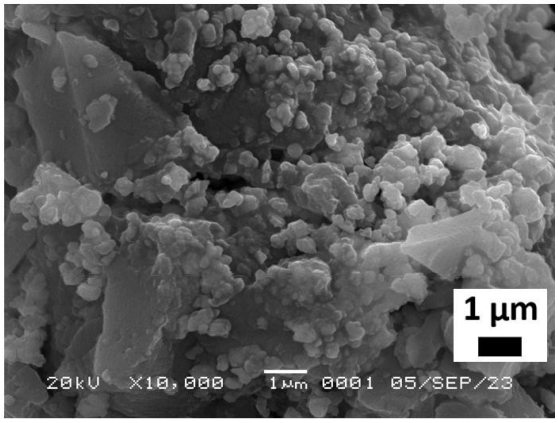


Figure 6. SEM results data with 10,000x magnification on Fe_3O_4-oxide graphene nanocomposite composition 30%: 70%

Based on Figure 6 SEM characterization results on the composition of Fe3O4-Graphene Oxide nanocomposite, it can be seen that the surface morphology of Fe3O4 is in the form of small bright grains attached to graphene oxide which looks like a larger slab. for Fe3O4-Graphene Oxide 40%:60% composition, it can be seen that less Fe3O4 covers the graphene oxide surface.

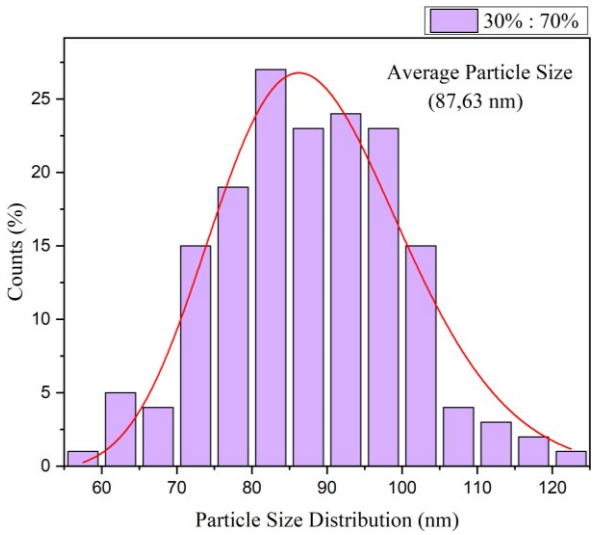


Figure 7. Particle size distribution chart for Fe_3O_4-graphene oxide nanocomposite composition 30%:70%

The particle size from the characterization using SEM for variations in the composition of Fe3O4- Graphene Oxide nanocomposites 30% : 70% can be further processed through Image-J digital processing software and plot the particle size distribution graph using the Origin application which can be seen in the following figure 7.

Based on Figure 7 which has been plotted using image-J and Origin applications, the average particle size for the Fe3O4-Graphene Oxide 30%:70% composition is 87.63 nm obtained using Origin software with a COD value of 0.9372 which proves the accuracy of the data obtained.

The surface morphology and particle size of Fe_3O_4-Graphene Oxide nanocomposite can be analyzed using SEM characterization tool. The morphology image produced will be analyzed using Image-J software. SEM characterization results for Fe3O4-graphene oxide nanocomposite composition 20%: 80% with10,000 x magnification can be seen in Figure 8.

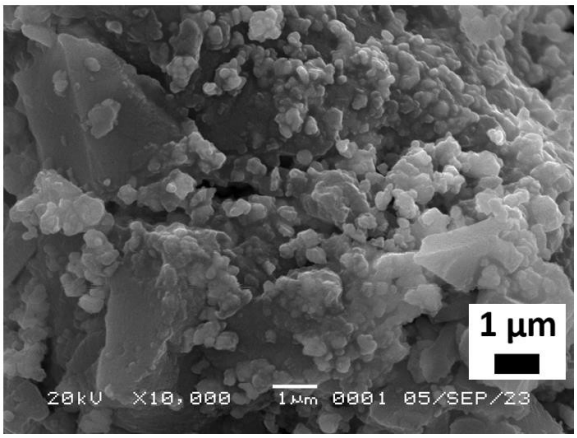


Figure 8. SEM results data with 10,000x magnification on Fe_3O_4-oxide graphene nanocomposite composition 20%: 80%

Based on Figure 8 SEM characterization results on the composition of Fe3O4-Graphene Oxide nanocomposite, it can be seen that the surface morphology of Fe3O4 is in the form of small bright grainsattached to graphene oxide which looks like a larger slab. for Fe3O4-Graphene Oxide 20% : 80% composition, It can be seen that very little Fe3O4 covers the surface of graphene oxide.

The particle size from the characterization using SEM for variations in the composition of Fe3O4- Graphene Oxide nanocomposites 40%: 60% can be further processed through Image-J digital processing software and plot the particle size distribution graph using the Origin application which can be seen in the following Figure 9.

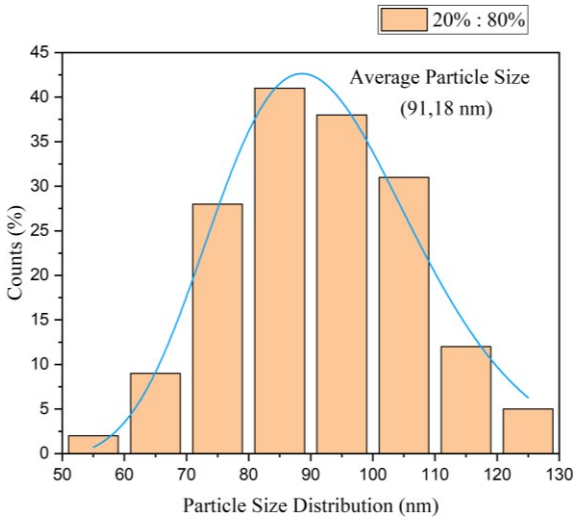


Figure 9. Particle size distribution chart for Fe_3O_4-graphene oxide nanocomposite composition 20% : 80%

Based on Figure 9 which has been plotted using image-J and Origin applications, the average particle size for the Fe3O4-Graphene Oxide 20% : 80% composition is 91.18 nm obtained using Origin software with a COD value of 0.9834 which proves the accuracy of the data obtained.

Based on the results of characterization using XRD, we obtained the crystal size of the three variations of Fe3O4-Graphene Oxide nanocomposite composition 40%: 60%, 30% : 70%, and 20%: 80%. Based on the peaks formed, it is in accordance with the standard XRD data for Fe3O4-Graphene Oxide (JCPDS No. 88-0315). The miller index of each peak formed is (220), (311), (400), (422), (511), and (440). From all the peaks formed, it shows the FCC (Face Center Cubic) phase, which shows that the formation of Fe3O4 composites with Graphene Oxide has been well implemented [22].

Based on the results of characterization using FTIR, the presence of C-H, O-H, C≡C, C=O, C-O bonds, and the presence of Fe-O absorption peaks indicate that Fe3O4 has been successfully composited with graphene oxide. The peak at 3000-3400 cm-1 is the carboxylic acid peak of Fe3O4-Graphene Oxide [23]. The absorption peaks of 1100-1300 cm-1, and around the absorption peaks of 1400-1600 cm-1 show the vibrations of graphene oxide in Fe3O4-graphene oxide [24]. The absorption peak at 400-700 cm-1 shows the Fe-O functional group which indicates the presence of Fe3O4 in graphene oxide [25].

Based on the results of characterization using SEM, the surface morphology of the Fe3O4-Graphene Oxide nanocomposite has a spherical structure [26], which can be seen that the more Fe3O4 composition in the composite, the greater the surface of graphene oxide that is covered. It can also be seen that the particles formed tend to form bonds with other particles so that they look clumpy which is called agglomeration [27]. The three composition variations can be seen that the more Fe3O4 composition in the composite, the particle size will be smaller [28] and the presence of Fe3O4 seems

to cover and stick to the surface of the graphene oxide layer [25]. This is also in accordance with the research [6] where the composite with the ratio of Fe3O4:graphene oxide = 0.7: 0.3, almost the entire surface of graphene oxide particles is covered by Fe3O4 whilethe ratio of Fe3O4: graphene-oxide = 0.3: 0.7, the distribution of Fe3O4 is very uneven. This proves that the more the composition of Fe3O4 particles, the greater the surface of the graphene oxide particles covered.

4. Conclusion

Based on the research that has been done about Composition effect of Fe3O4 and Graphene Oxide onthe microstructure of Fe3O4-Graphene Oxide nanocomposite, it can be concluded that for variations in the composition of Fe3O4-Graphene Oxide nanocomposites 40% : 60%, the crystal size is 44.73 nm. For the composition variation of Fe3O4-Graphene Oxide nanocomposite 30%: 70%, the crystal size is 39.71 nm. For the composition variation of Fe3O4-Graphene Oxide nanocomposite 20%: 80%, the crystal size is 45.64 nm. The presence of C-H, O-H, C≡C, C=O, C-O bonds, and the presence of Fe-O absorption peaks indicate the success of Fe3O4 composite with graphene oxide. The surface morphology of the Fe3O4- Graphene Oxide nanocomposite has a spherical structure, which can be seen that the more Fe3O4 composition in the composite, the larger the surface of graphene oxide is covered.

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