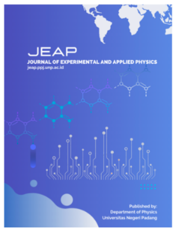
Journal of Experimental and Applied Physics
Journal Homepage: jeap.ppj.unp.ac.id
Vol. 2, No. 2, June 2024.
ISSN (Print): 2988-0378 ISSN (Online): 2987-9256



Effect of The Camposition Nanocomposite Fe_3O_4-Graphene Oxide on Optical Properties Synthesized from Coconut Shell Charcoal

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Article History
Received : June, 12th 2024
Revised : June, 30th 2024
Accepted : June, 30th 2024
Published : June, 30th 2024

DOI:
https://doi.org/10.24036/jeap.v2i2.60

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Abstract: This study aims to determine the effect of Fe_3O_4 nanocomposite composition: Graphene Oxide on the optical properties of Fe_3O_4 Graphene Oxide nanocomposite by obtaining the results of absorbance value, transmittance, reflectance, and band gap value based on UV-Vis test. Graphene oxide used in this study is made from biomass waste coconut shell charcoal which contains carbon elements, most of whose pores are still covered by hydrocarbons and other organic compounds and the charcoal is used as activated carbon through an activation process, then synthesized using the modified hummers method. In this study, three variations were used, namely 20%: 80%; 30%; 70%; 40%: The results show that the effect of Fe_3O_4-Graphene Oxide Nanocomposite Composition on coconut shell waste using a UV-Vis spectrometer, the absorbance value of each sample was obtained in the range of 200-290 nm.. In the results of gap energy research with 3 variations respectively 3.269 eV, 3.79 eV, 3.91 eV obtained that if the variation of graphene oxide is higher than the value of the energy gap produced is smaller, so the addition of graphene oxide from coconut shell waste has the effect of reducing the energy gap of graphene oxide.

Keywords: Energy Gap; Fe_3O_4; Graphene Oxide; Modified Method; Optical Properties.

1. Introduction

Magnetic iron oxide has been widely applied in human life, for example the application of iron oxide nanoparticles as a contrast agent for in vitro diagnostics. In the last decade, the synthesis of magnetic iron oxide nanoparticles has been intensively developed not only for fundamental scientific interests, but also for many technological applications, such as targeted drug delivery, magnetic resonance imaging (MRI) and magnetic resonance imaging (MRI)[1]. Nanocomposites are materials made by incorporating nanoparticles into macroscopic material samples. Nanocomposites result from mixing in a number of different phases. This mixing can result in new

How to cite:
S. Angraini, Gusnedi, R. Jonuarti, F.U. Jhora, 2024, Effect of The Camposition Nanocomposite Fe_3O_4-Graphene Oxide on Optical Properties Synthesized from Coconut Shell Carcoal, *Journal of Experimental and Applied Physics,* Vol.2, No.2*,* page 89-98. https://doi.org/10.24036/jeap.v2i2.60

properties that are superior to the original material [2].The measurement of optical properties uses electromagnetic waves from ultraviolet to infrared. The wavelength depends on the optical absorbance and transmittance spectrum. As for the absorbance spectrum, it will increase when the wavelength decreases. The wavelength used to measure the optical properties of graphene oxide is 200 nm to 400 nm [3].

Graphene is considered the thinnest material in the world, a sheet of sp bonded carbon atoms2 in a hexagonal two-dimensional lattice. Although it is very thin, the strength of graphene exceeds that of steel, this is due to the strong covalent bonds between its carbons, making it difficult to stretch.[4]. Graphene is the thinnest, strongest, and most advanced new material in the world today formed from a single layer of carbon atoms that have a hexagonal structure resembling a honeycomb [5]. Graphene is becoming a much sought-after raw material, however, the availability of graphene is still limited, so how to produce this material in large quantities is an interesting concern. The advantage of forming graphene in large quantities in powder form using the hummers method. In addition, the formation of graphene from this waste material is very effective for application to lithium battery electrodes.

The biomass waste used in this study is coconut shell, which is used is an old coconut shell that has a hard structure because it contains a high silicate content SiO_2[6].Coconut shell waste can be utilized as a basic material in the manufacture of graphene oxide because the waste contains a very high carbon element which can later be used as activated charcoal. One of the materials that can be used as activated charcoal is coconut shell. The utilization of coconut shell waste is what will increase the usability of coconut shells which were previously only as industrial waste which was generally just thrown away. This coconut shell charcoal is used as activated carbon through an activation process [7].

After this is characterized by looking at its optical properties, how the material responds to electromagnetic, especially visible light. Characterization of optical properties is carried out using a UV-VIS Spectrophotometer, where if light is passed through a material, some of the light is reflected (Reflectance), some will be absorbed (Absorbance) and forwarded and spread (Transmittance). The wavelength used to measure the optical properties of graphene oxide is 200 nm to 400 nm [3]band gap characterization using a UV-VIS spectrophotometer in the wavelength range 200 nm -700 nm .UV-VIS spectrometer is an analytical method that uses near ultraviolet and visible light electromagnetic radiation sources in spectrometer instruments. The ultraviolet spectrum includes the ultraviolet region (190 nm-380 nm), and the visible spectrum of visible light (380 nm- 780nm). When the electron returns to the orbital of origin, the electron emits energy and that energy is detected as absorbance peaks [3].

Several studies have been conducted related to the characterization of optical properties. one of them by [8] entitled "analysis of the optical properties of Fe_3O_4 thin layer prepared from oyster beach iron sand Padang Pariaman Regency West Sumatra with Sol-gel spin method" where the results obtained optical properties in the form of transmittance, reflectance, adsorbansi and energy gap values obtained the average result of Fe_3O_4 thin layer of 3.75 eV.

According to research [9] which is entitled "fabrication and characterization of optical properties of magnetite nanoparticles Fe_3O_4 based on iron sand" which aims to determine the size and morphological structure and optical properties (absorbance) of nanoparticles. Where it is found that magnetite nanoparticles Fe_3O_4 have 2 absorption peaks. The appearance of 2 peaks is due to magnetite nanoparticles attached to each other causing conduction electrons on each particle surface not fixed position on 1 atom. Based on the results of the study, magnetite nanoparticles have a stable absorption peak at a wavelength of 230-400 nm. This result is in accordance with previous research, where the absorption peak of magnetite nanoparticles is in the wavelength range of 200-500 nm.

Successfully synthesized graphene with the rGO method modified by the Hummers metod using $KmnO_4$ mass ratio.The results obtained peaks that appear are at wavelengths of 230 nm and 310 nm.In the wavelenghth region is the characterization of graphene and graphene oxide.The purpose of analyzing using a Uv-Vis spectrophotemer is to determine the optical properties which form the response of the material to electromagnetic, especially visible light [9]. One method to characterize graphene is to use a Uv-Vis spectrophotometer. Graphene characterization can be analyzed by observing the wavelength results of the absorbance peaks indicated by the Uv-Vis results. Graphene characterization using Uv-Vis has been carried out by scientists and academics with different graphene synthesis methods [10].

2. Materials and Method

This type of research is experimental research. The coconut shell by cleaning the old coconut shell from the fiber until the remaining old coconut shell by scraping the remaining fibers after that sandpaper so that the surface of the coconut shell is smooth and smooth. Then the old coconut shell is dried for 3 days in the sun, after drying for 3 days the old coconut shell is cut into small pieces. Furthermore, to remove the water content in the old coconut shell using an oven with a temperature of 110˚Cfor 60 minutes. After the old coconut shell is finished in the oven, the furnace process is carried out for 2 hours with a temperature of 350˚C which is based on previous research [11].After the old coconut shell has become charcoal, it is pounded with a mortar or pestle overlap to produce charcoal powder and sieving is carried out using a 125 mesh sieve. After that, the carbon activation process by mixing the old coconut shell powder with NaOH and then dissolved using distilled water as much as 100 ml which has been measured using a measuring flask. Next, add 8 g of NaOH solids into the measuring flask using a spatula until the NaOH solids dissolve and the solution becomes homogeneous. Then provide a 250 ml beaker and then put 8 grams of coconut shell charcoal powder and add 100 ml of NaOH solution into a beaker that has been filled with old coconut shell charcoal powder so that the charcoal powder is submerged in the NaOH solution. After the mixture was submerged, the sediment at the bottom of the beaker occurred, the sediment was filtered using ordinary filter paper and a buchner funnel to facilitate the filtering process[12].

The filtering process uses filter paper and then placed in a buchner funnel. After that, wet the filter paper using distilled water. Then filter the mixture of coconut shell and NaOH solution until the old coconut shell powder is completely separated from the liquid. After the old coconut shell powder is filtered, transfer the results of the old coconut shell powder into a vaporizer cup. The activated old coconut shell powder was dried using an oven at 105°C for 3 hours [11]. Furthermore, the graphene oxide synthesis process goes through a process of charring and pulverizing into coconut shell charcoal powder. Where the coconut shell charcoal powder is activated and synthesized using the modified hummers method which uses chemical reactions to form graphene oxide. The synthesis process is carried out by weighing 1.5 g of coconut shell charcoal powder that has been activated carbon and 0.75 g of graphene oxide.$NaNO_3$ as much as 0.75 g. Next, place a 250 ml erlenmeyer that has been inserted with a magnetic stirrer in it and insert the coconut shell and the weighed coconut shell powder.$NaNO_3$ that has been weighed. Next, add H_2SO_4 98% as much as 34.5 ml into the erlenmeyer. The mixture was then stirred for 20 minutes at a temperature of 0-5 ° C with a constant speed of 250 rpm and the solution will turn black because there is carbon content derived from old coconut shell charcoal [13].

Next, put the erlenmeyer on an ice bath and stirring for 2 hours on a hot plate. After that, add KMnO4 powder as much as 4.5 g. This is done slowly to keep the temperature below 20 ℃. This process must be done carefully to prevent the mixture from exploding and the synthesized coconut shell powder from decreasing. After adding $KmnO_4$ to the mixture, removing the ice bath from the hot plate and stirring the previous mixture at 35 ℃ for 35 min[14].This is so that the oxidation process can take place completely. This process is carried out until the solution changes color to pale brown. Then use a thermometer to ensure the temperature of the mixture reaches 35 ℃. Next, add distilled water as much as 69 ml slowly using a dropper and stirring for 20 minutes.

In this process, the temperature of the solution will increase because when adding distilled water an endothermic reaction occurs. In the addition of distilled water, the temperature is kept below 50 ℃ to see the oxidation process, and the mixture will change color to dark brown accompanied by bubbles. Next, add 100 ml of water and 1.5 ml of 30% H_2O_2 . The addition of H_2O_2 is done to remove the remaining $KmnO_4$ and to stop the reaction of the solution and then the solution finally turns into a yellow color [15]. The final process is to add 50 ml of distilled water to the mixture and graphene oxide will be formed. After carrying out the synthesis process which finally resulted in the solution turning yellow which indicates the presence of graphene oxide, the mixture was sonicated for 2 hours to exfoliate graphite into graphene [16].Then, the solution was precipitated for 1 day until liquid and solid phases were formed. After that, a centrifugation process was carried out using a micro centrifuge at 4000 rpm for 15 minutes to separate the solid and liquid phases [17]. The centrifugation process was followed by a manual graphene oxide neutralization process, namely by precipitating the powder. Graphene oxide and distilled water were replaced continuously and repeatedly until a neutral pH of 7 was obtained. If a neutral pH was obtained, graphene oxide was baked at 60℃ for 12 hours [18].Finally, the synthesis of Fe_3O_4-Graphene Oxide composites The preparation of Fe_3O_4 Graphene Oxide composites was carried out by mixing the two in the ratio 20%: 80%; 30%: 70% and 40%: 60%; .The mixing of these two was carried out using a ball mill tool at a speed of 3000 rpm for 30 minutes. In addition to mixing the two liquids, the ball mill tool can also homogenize the particle size [1]. After all these processes, sample characterization is then carried out. Graphene oxidation samples that have been made are characterized using XRD, FTIR. XRD characteristics use a sample of about 0.10 grams. Then the graphene oxide sample is put into a container to place a circular sample, then XRD testing will be carried out by firing X-rays.

UV-VIS spectrophotometer is based on the relationship between wavelength and transmittance, the relationship between wavelength and absorbance and the relationship between wavelength and reflectance [19]. According to [20]The characterization of optical properties using a UV-VIS spectrophotometer is transmittance, absorbance, reflectance and energy gap. From the data, a graph is then made using Origin and using the toch plot technique, so that the relationship between the wavelength and the transmittance, absorbance and reflectance values can be seen. Then from the transmittance value, the energy gap value of the Fe_3O_4-Graphene Oxide thin layer from coconut shell waste can be determined.

3. Results and Discussion

In the data description, all data obtained from UV-VIS measurements are described. Then the data is analyzed in accordance with data analysis techniques. This UV-VIS test aims to determine the optical properties of graphene oxide, which is to find out how the absorbance value, transmittance, reflectance and the resulting GAP value. By doing 3 variations 20%: 80%, 30%: 70% and 40%: 60%. The results of characterization in this test are in the form of a wavelength spectrum and then the calculation of the band gap energy value using the touc plot method. Calculation of band gap energy values using the touc plot method utilizes data from the resulting UV-VIS spectrum. The determination of the band gap energy value utilizes a linear graph of the relationship between E (eV) on the x-axis and (ahv)2 on the y-axis.

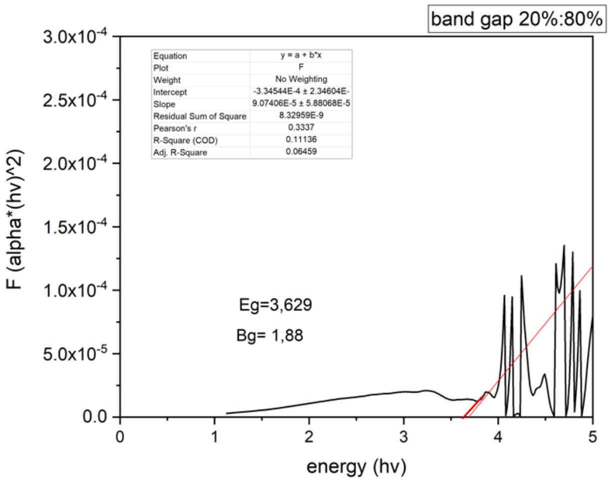

Figure 1. Tauc plot curve of Fe_3O_4 -Graphene Oxide nanocomposites with 20%:80% variation

Based on the Figure 1 above with a composition variation of 20%: 80%, the energy gap results are 3.629 eV .while for the band gap obtained using the COD value, the result is 3.71 eV.

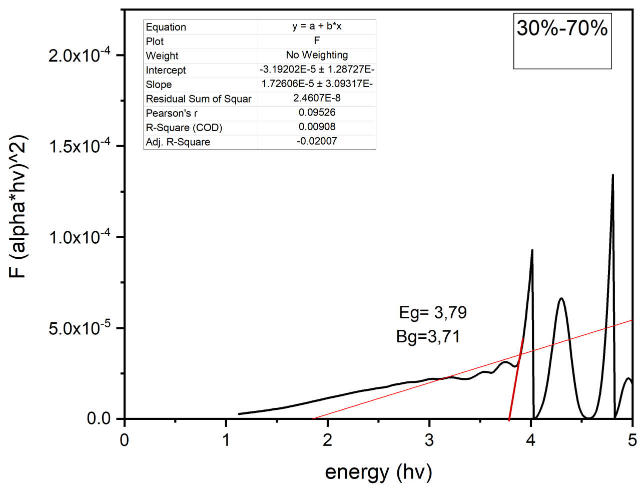

Figure 2. Tauc plot curve of Fe_3O_4 -Graphene oxide nanocomposites with 30%:70% variation

Based on the Figure 2 above with the 30%:70% composition variation, the energy gap result is 3.79eV. while for the band gap obtained using the COD value, the result is 1.88 eV.

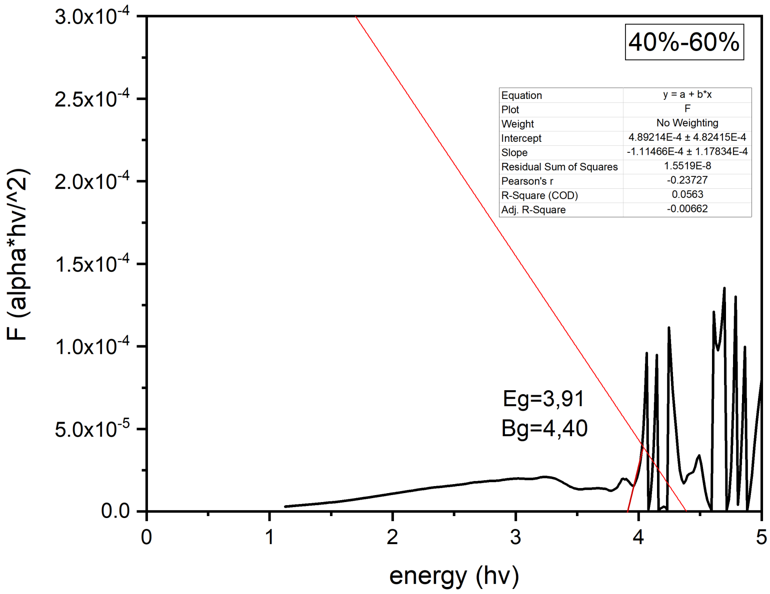

Figure 3. Tauc plot curve Fe_3O_4 -Graphene Oxide nanocomposites with 40%:60% variation

Table 1. Band Gap Energy

Variations	Band Gap Energy (eV)
20% Fe_3O_4 -80% graphene oxide	3.269 Ev
30% Fe_3O_4 -70% graphene oxide	3.79 eV
40% Fe_3O_4 -60% graphene oxide	3.91 eV

Based on the data Table 1 above, the band gap energy from highest to lowest in a row is in the composition of 40% Fe_3O_4: 60% graphene oxide with 3.91 eV, 30% Fe_3O_4: 70% graphene oxide with 3.79 eV and 20% Fe_3O_4: 80% graphene oxide with 3.269 eV. This indicates that in the variation of 20% Fe_3O_4: 80% graphene oxide low band gap energy value, then in this variation if the variation of graphene oxide is higher, the resulting energy gap value is smaller, so the addition of graphene oxide from coconut shell waste has the effect of reducing the energy gap of graphene oxide. Likewise, the opposite is if the Fe_3O_4 nanocomposite variation is added, a high energy gap value is obtained. The band gap energy values in the three samples are within the band gap energy value for graphene oxide with semiconductor properties.

Where the more the composition of Fe_3O_4 nanocomposites used, the higher the band gap energy value: 70% variation obtained a band gap of 1.18 eV according to the resulting COD value where the COD value is very important because it is a parameter whether the graph value is good or not. This value is close to the ideal band gap value where the ideal gap is 1.1 eV. So the effect of this energy gap on the optical properties of Fe_3O_4 -Graphene Oxide nanocomposites is that the smaller the energy gap, the better the optical properties produced.

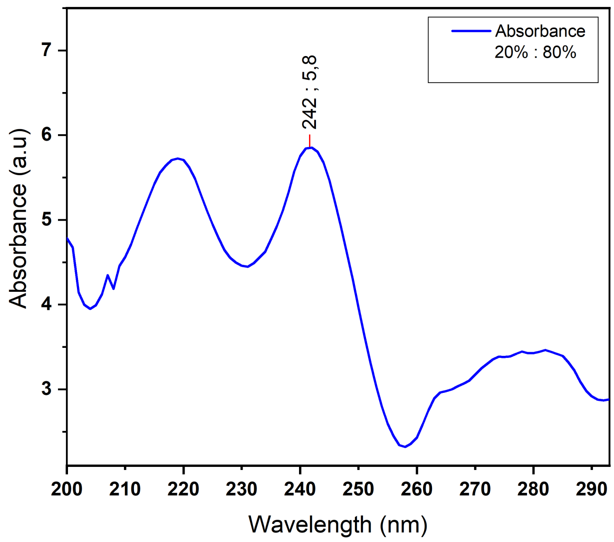

Figure 4. Uv-vis spectra of Fe_3O_4:Graphene oxide nanocomposites with 20%:80% variation

Based on the Figure 4 above in the variation of 20%: 80% absorbance with a wavelength of 200 nm - 290 nm with a large absorbance of 5.8%, which is with a maximum peak 242 nm.

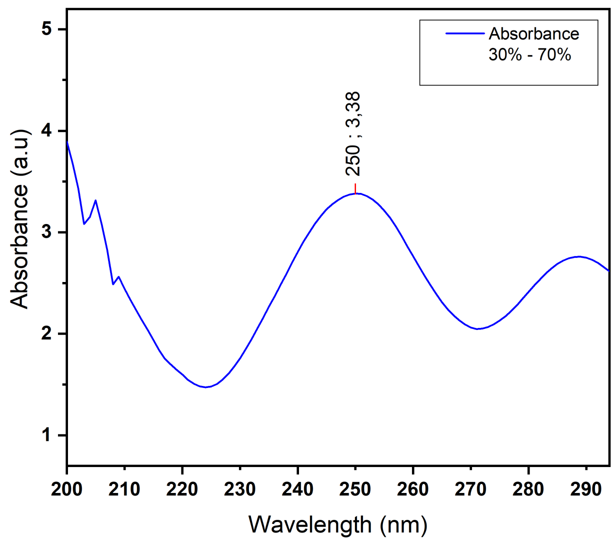

Figure 5. Uv-vis spectrum of Fe_3O_4:Graphene oxide nanocomposite with 30%:70% variation

Based on the Figure 5 above in the variation of 30%: 70% absorbance with a wavelength of 200 nm - 290 nm with a large absorbance of 3.38%, which is with a maximum peak 250 nm.

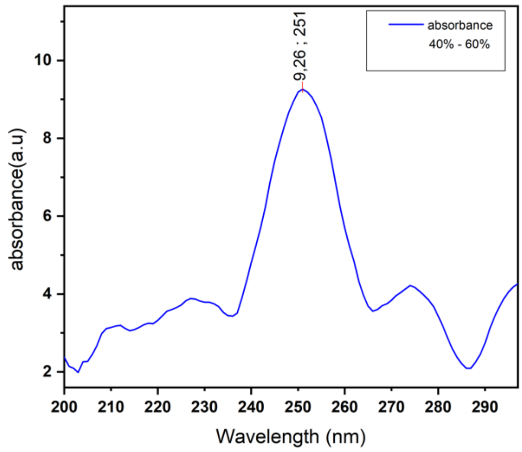

Figure 6. Uv-vis spectra of Fe_3O_4:Graphene oxide nanocomposite with 40%:60% variation.

Based on the Figure 6 above in the 40% variation: 60% absorbance with a wavelength of 200 nm - 290 nm with a large absorbance of 9.26%, which is with a maximum peak of 251 nm. Based on the results that have been obtained, the wavelength results are obtained with a wavelength range of 200-290 nm. Based on the picture above that the author took the absorbance value with the highest peak with a large absorbance of 5.8%. The highest absorbance value is in the composition of Fe_3O_4 -Graphene oxide 40%-60% which is 9.26% and the lowest is in the composition of Fe_3O_4 -Graphene oxide 20%-80% which is 5.8% while in the composition of Fe_3O_4 -Graphene oxide 30%-70% which is 3.38%. Where this absorbance value is obtained the highest peak at the sixth peak.in the results of research with energy gap with 3 variations, it is found that if the variation of graphene oxide is higher, the value of the resulting energy gap is smaller, so the addition of graphene oxide from coconut shell waste has the effect of reducing the energy gap of graphene oxide.by analyzing linear fitting by showing the COD value because it is very important to know the parameters whether the graph results are good or not, if it is close to 1 eV then the better. In the results of the study, the lowest band gap value was obtained in the 20%-80% composition, which is 1.88 eV, the value is close to the ideal band gap value of 1.1 eV. while in the 30%-70% composition, it is 3.71 eV; 40%-60% is 4.40 eV.

4. Conclusion

Magnetite nanoparticles have a stable absorption peak at a wavelength of 200-290 nm. This result is in accordance with previous research, where the absorption peak of magnetite nanoparticles is in the wavelength range of 200-500 nm. In the results of research with energy gap with 3 variations, it is found that if the variation of graphene oxide is higher, the value of the resulting energy gap is smaller, the addition of graphene oxide from coconut shell waste has the effect of reducing the energy gap of graphene oxide. Vice versa, if the Fe_3O_4 nanocomposite variation is added, a high energy gap value is obtained. As the purpose of this study where the composition of graphene oxide is very influential for reducing the energy gap. The band gap energy values of the three samples are within the band gap energy value for graphene oxide with semiconductor properties.

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