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CHARACTERIZATION STRUCTURE MICRO OF GRAPHENE OXIDE WHICH SYNTHESIS FROM BAGASSE WITH MODIFIED HUMMERS METHOD

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ABSTRACT

This study aims to form a graphene oxide layer from bagasse, which by knowing the microstructure of the layer graphene oxide can be used as microwave absorption. In the process, bagasse is made into activated carbon with various carbonation temperatures, namely 300ºC, 350ºC, 400ºC and 450ºC. Then it was synthesized using the modified hummers method by mixing carbon with H_2SO_4, $KMnO_4$, H_2O_2 and aquades. Then they were characterized using FTIR, XRD and SEM. Based on the FTIR data, it states that there is a relationship between carbon (C), hydrogen (H) and oxygen (O) which proves the formation of a graphene oxide layer. Also based on the results of XRD which states that the diffraction angle is in the range of 10º-90º and has the highest crystal size of 40.5267076 nm. Likewise, the results from SEM which stated that the best graphene oxide was at an average particle size of 81,4043598 nm. The resulting graphene oxide is also shaped like monolayer sheets.

Keywords : Graphene Oxide, FTIR, XRD, SEM, Modified Hummers Method.

I. INTRODUCTION

Nowadays, organic waste is very abundant in all regions in Indonesia. One of them is bagasse waste. Sugarcane is a plant that grows in tropical areas such as Indonesia. Sugarcane is often used as a natural sweetener, in the form of sugar and sugarcane juice which is sold in various areas, one of which is the city of Padang. However, from the amount of sugarcane that is processed, it also produces large amounts of bagasse. The more piled up and the more it becomes waste that is difficult to recycle. Sugarcane contains cellulose, hemicellulose, and lignin [1]. The carbon produced by bagasse has a high carbon content so that it can be used as activated carbon. Therefore, sugarcane bagasse that has become waste can be processed here into the basic material for making graphene oxide.

Graphene oxide is a single layer with high oxygen content, which has a mixture of chemical compounds carbon (C), oxygen (O) and hydrogen (H) with a strong oxidation process [2]. The process of forming graphene oxide can be carried out by synthesizing it with a mixing compound in the form of H_2SO_4, $KMnO_4$, H_2O_2 and aquades [3]. This synthesis process can be carried out using the modified hummers hummers method, namely by oxidizing activated carbon with H_2SO_4 and $KMnO_4$ then catalyzed using H_2O_2 with the final result in the form of graphene oxide powder. The purpose of graphene oxide formation is to determine the microstructure, functional groups and surface morphology, so that it can be used as microwave absorption.

II. METHOD

This research is an experimental research. This research was conducted at the Laboratory of the Department of Material Physics & Biophysics, FMIPA, UNP. In this study, the sample used as the basic material for the manufacture of graphene oxide is bagasse. The mixing materials used in the process of making graphene oxide are Sulfuric Acid (H_2SO_4), Potassium Permanganate ($KMnO_4$), Hydrogen Peroxide (H_2O_2) and Aquades. Successful samples will be characterized using three tools, namely FTIR to determine the functional group, XRD

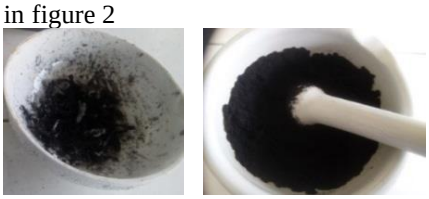
to determine the crystal structure as well as lattice parameters and SEM is used to determine the particle size and surface morphology of the sample.

In this research, there are several step carried out including:

First, the bagasse samples obtained on Jl. Steba, Padang, West Sumatra. The bagasse obtained is dried for 2 days, then crushed by cutting the bagasse into small sizes. Furthermore, the bagasse samples were dried using an oven at 100ºC for 60 minutes. The following is a picture of the results of drying bagasse using an oven, it can be seen in Figure 1

Fig 1. Waste of bagasse that has been in the oven

The dry sample is then burned using a Furnace. The temperature variations used in the combustion of the samples used as independent variables are temperatures of 300ºC [4], 350ºC, 400ºC and 450ºC [5]. Samples were burned at these temperature variations for 20 minutes each. Then the results of this combustion will be crushed into carbon powder. As shown in figure 2



(a) (b)

Fig 2. (a) Sample of bagasse that has been in the furnace, (b) Sample of carbon that has been pounded with a pestle and mortar

At this step 8 grams of carbon that has been weighed is mixed with 8 grams of NaOH which has been dissolved with 100 ml of distilled water. Then the solution is stirred so that it is completely mixed, then for 24 hours or 1 day. Then filter the sample and then the filtered sample is dried using an oven at 105ºC for 3 hours [6]. At this step, 1.5 grams of bagasse activated carbon was weighed, mixed with 34.5 ml of H_2SO_4 and stirred for 2 hours. After that, $KMnO_4$ was added and continued stirring for 1 hour at a temperature of 20ºC–35ºC. Then add 69 ml of distilled water, continue stirring for 1 hour. Then add 2 ml of H_2O_2, then stir for 1 hour, finally add another 100 ml of distilled water and stir for 1 hour. At this step, the synthesized sample was sonicated using ultrasonic for 2 hours. After that, the graphene oxide sample was dried using an oven for 12 hours at a temperature of 60ºC [7]. The following is the final result of graphene oxide, it can be seen in figure 3

Fig 3. Final product of graphene oxide

The FTIR characterization data were analyzed by determining the wavelength with the functional groups formed. Furthermore, the data from the XRD characterization where to calculate the crystal size can be used the Scherrer formula, namely [8]

$$D = \frac{K \times \lambda}{B \times \cos\theta} \quad (1)$$

Next for the results of the data using SEM, the data is processed using Image-J software, calculated using Microsoft Excel and seen the average particle using the origin software.

III. RESULTS AND DISCUSSION

The results of the FTIR characterization analysis of graphene oxide at a temperature of 300° C, as shown in Figure 4

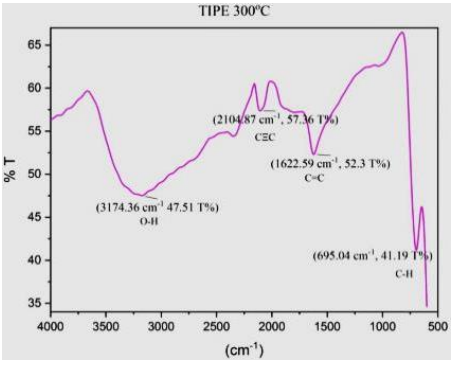


Fig 4. Results of graphene oxide FTIR characterization at 300°C

It can be seen in Figure 4, The results of the characterization using FTIR of graphene oxide with a sintering temperature of 300°C. This test was carried out with a wavelength range of (4000 - 500) cm-1. Graphene oxide at a temperature of 300°C, absorption at a wavelength of 3174.36 cm^{-1} indicates the presence of 0-H bonds. The absorption at a wavelength of 2104.87 cm^{-1} indicates the presence of a CΞC bond at its peak. At the peak of 1622.59 cm^{-1}, it shows the presence of a C=C bond. Likewise, at the wavelength of 695.04 cm^{-1} there is a C-H bond at the top of the graph.

The results of the FTIR characterization analysis of graphene oxide at a temperature of 350°C, as shown in Figure 5

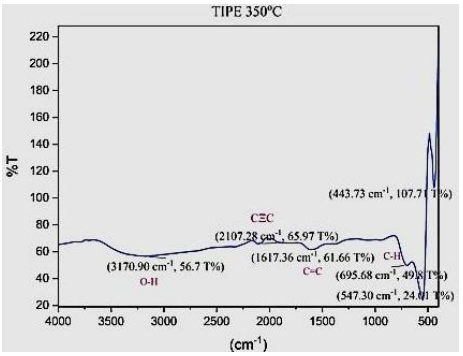


Fig 5. Results of FTIR characterization of graphene oxide at 350°C

Based on Figure 4, it can be seen that at a temperature of 350°C it has a functional group with chemical bonds which states that at a wavelength of 3170.90 cm^{-1} there is an O-H bond at its peak. Furthermore, at a wavelength of 2107.28 cm^{-1} there is also a triple bond in the C functional group such as CΞC at the peak of the chemical bond. At the wave number 1617.36 cm^{-1} there is a C=C functional group at its peak. Likewise, at the peak of 695.68 cm^{-1} there is a C-H bond.

The results of the FTIR characterization analysis of graphene oxide at a temperature of 400° C, as shown in Figure 6

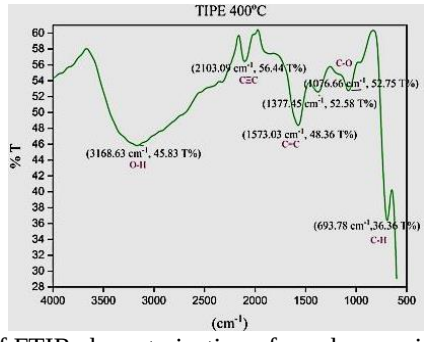


Fig 6. Results of FTIR characterization of graphene oxide at 400°C

In Figure 6 the FTIR data above at a type temperature of 400° C there are several chemical bonds, namely at the absorption wavelength of 3168.63 cm^{-1} which has O-H chemical bonds at its peak. Furthermore, at the absorption wavelength of 2103.09 cm^{-1} there is a CΞC chemical bond. At a wavelength of 1573.03 cm^{-1} there is a C=C chemical bond at the peak of the wave. Next, at a wavelength of 1076.66 cm^{-1} has a C-O chemical bond. Finally, at a wavelength of 693.78 cm^{-1} has a C-H chemical bond at the tip of the graphene oxide sample wave.

The results of the FTIR characterization analysis of graphene oxide at a temperature of 450° C, as shown in Figure 7

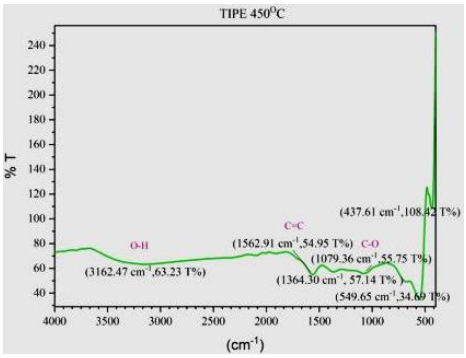


Fig 7. Results of FTIR characterization of graphene oxide at 450°C

Based on Figure 7, it can be seen that at a wavelength of 3162.47 cm^{-1} there is a 0-H bond at its peak. Furthermore, at the absorption wavelength of 1562.91 cm^{-1} there is a double c bond in the functional group such as C = C below. In the picture there is also a C-O bond at a wavelength of 1079.36 cm^{-1} in the graphene oxide wave spectrum at a temperature of type 450^{O} C. Based on the FTIR data above, it can be said that graphene oxide has been formed because it has a combination of functional groups C=C, C-O, C≡C, C-H and O-H [9].

The characterization data using FTIR that have been obtained can be said that graphene oxide has been formed, because based on the wave crests formed from the obtained bonds indicate the presence of C, H and O bonds which are in accordance with the statement, that graphene oxide is a a mixture of carbon (C), hydrogen (H), and oxygen (O) compounds obtained through a strong oxidation process from graphite or graphene.

Characterization testing using High Score Plus software and also being able to identify the peak Miller index in the emerging phase with the help of Origin software. The following is the result of graphene oxide XRD characterization data at 300°C. The data obtained can be processed using high score plus software to be able to calculate the Miller index. While the lattice parameters can be known from the ICDD data obtained from the characterization process.

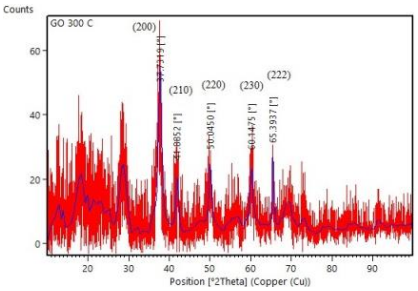


Fig 8. Results of graphene oxide FTIR characterization at 300°C

Based on the figure 8 above, it can be explained that the deposition results formed were five peaks with angles of 37.7644°, 41.9215°, 50.0893°, 60.2024°, and 65.4546°. Based on the identification of the Miller index using the High Score Plus software, the phases that appear are (200), (210), (220), (230) and (222). The crystal size calculated using the Scherrer equation is 18.98051563 nm.

The results of the XRD characterization analysis at a temperature variation of 350°C as shown in Figure 9

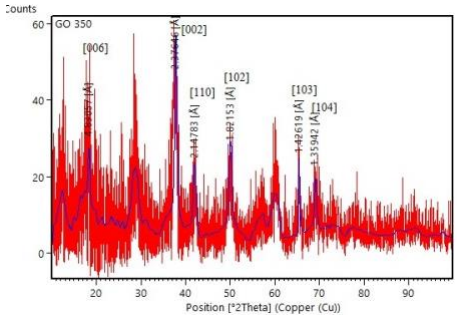


Fig 9. Results of graphene oxide FTIR characterization at 350ºC

It can be seen in the figure 9 that the 350ºC variation has peaks at angles of 18.3668º, 37.8591º, 42.0699º, 50.0782º, 65.443º, and 69.0974º. which also have FWHM, namely 0.5117º, 0.614º, 0.8187º, 0.8187º, 0.307º, and 0.614º. The miller index identification is (006), (002), (110), (102), (103), and (104). Test results from XRD at 350o C variation. Based on ICDD data obtained, the lattice parameters obtained are based on Bravais lattice with $a = b \neq c$, $\alpha = \beta = 90^0$, $\gamma = 120^0$ which has a hexagonal crystal system. The crystal size calculated using the Scherrer equation is 25.93692581 nm.

The results of the XRD characterization analysis at a temperature variation of 400ºC as shown in Figure 10

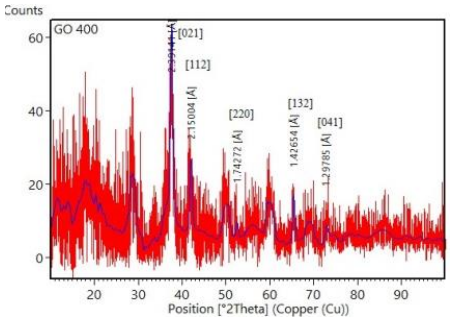


Fig 10. FTIR characterization results for graphene oxide at 400ºC

Judging from the figure 10 above, the graph of the peak deposition is at an angle of 37.6135º, 42.0246º, 52.5111º, 65.4252º, dan 72.8844º. The results of the Miller index identification are (021), (112), (220), (132), and (041). Based on the ICDD data obtained, the lattice parameters obtained are based on the Bravais lattice with a b c, and the value of (alpha) α = (bheta) β = (gamma) $\gamma = 90^0$ then the crystal system is orthorhombic.The crystal size calculated using the Scherrer equation is 21.84037863 nm.

The results of the XRD characterization analysis at a temperature variation of 450ºC as shown in Figure 11

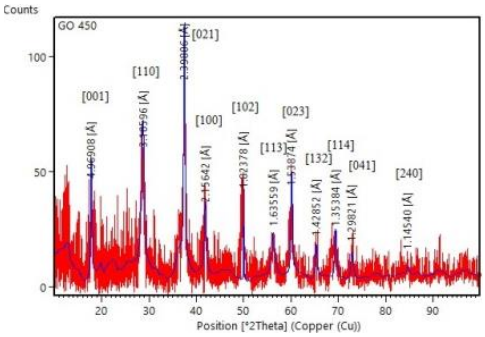
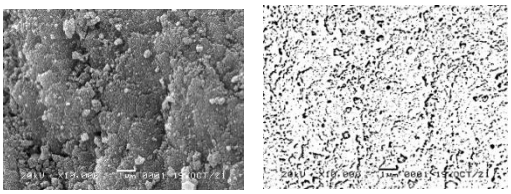


Fig 11. Results of FTIR characterization of graphene oxide at 450ºC

Based on the figure 11, it can be seen that the peak deposition graph is at an angle of 17.8506º, 28.7435º, 37.5054º, 41.8945º, 49.968º, 56.244º, 60.1347º, 65.323º, 69.4231º, 72.8607º dan 84.6092º. The results of the Miller index identification are (001), (110), (021), (100), (102), (113), (023), (132), (114), (041), and (240). Based on the ICDD data obtained, the lattice parameters obtained are based on the Bravais lattice with a b c, and the value of (alpha)α = (bheta)β = (gamma)γ = 900 then the crystal system is orthorhombic. The crystal size

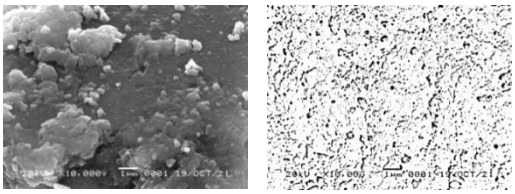
calculated using the Scherrer equation is 40.5267076 nm. The result of graphene oxide has a crystal size with a diffraction angle range (2θ) at an angle (10^o – 90^o) [10]. So based on the characterization using it can be said that there is a graphene oxide layer formed.

The following is the result of SEM characterization of graphene oxide at 300ºC, with a magnification of 10.000x. then the image can be processed using image-J software. This can be seen in Figure 12, figure 13 , figure 14 and figure 15



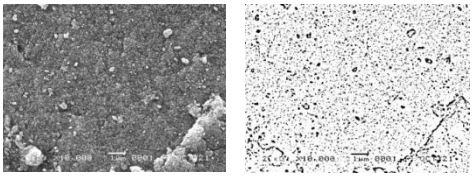
(a) (b)

Fig 12. SEM GO 300◦C test results at 10.000x magnification (a) the original sample (b) the sample after the threshold



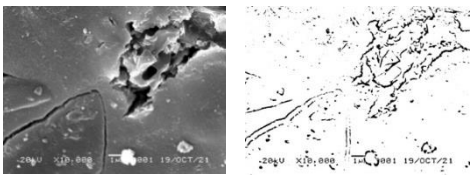
(a) (b)

Fig 13. SEM GO 350◦C test results at 10.000x magnification (a) the original sample, (b) the sample after the threshold



(a) (b)

Fig 14. SEM GO 400◦C test results at 10,000x magnification (a) the original sample, (b) the sample after the threshold



(a) (b)

Fig 15. SEM GO 450◦C test results with a magnification of 10,000x (a) the original sample, (b) the sample after the threshold

Based on Figure 12, figure 13 , figure 14 and figure 15, SEM sample analysis results using Image-J software, with a magnification of 10,000x. It can be seen that graphene oxide at a temperature of 300◦C can be seen that the average diameter of the particles is 63.79740769 nm. At a temperature of 350◦C, the average particle diameter is 60.69622036 nm. At a temperature of 400◦C, the average particle diameter is 47.50449759 nm. At a temperature of 450◦C, the average particle diameter is 81,4043598 nm. This can be known by processing data using image-J software.

The data results show that the average diameter of the particles is in the range of 33 – 84 nm. So it can be said that this research is successful and is in accordance with the statement that graphene oxide can be formed in the size range of 32 nm to 84 nm. At a temperature of 300◦C and 400◦C, it is in the form of lumps according to the graphene oxide formed in the form of lumps [11]. At 350◦C and 450◦C graphene oxide can form sheets [12]. At a temperature of 450ºC graphene oxide material with a surface morphology in the form of sheets with a particle diameter of 81.4043598 nm. better results because the sample is seen from the surface shape and particle size.

IV. CONCLUSION

Based on the research that has been done, it can be concluded that the graphene oxide with the best results is found at a temperature of 450ºC in the form of sheets and the highest particle size is 81,4043598 nm. Where the larger the particle size, the surface pores of the graphene oxide sample are also getting bigger so that the microwave absorption is getting better [13].

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