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# Crystal Structure Analysis of Graphene Oxide Based on Bamboo "Betung" Synthesized By Modified Hummer Method

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## ABSTRACT

*Bamboo is a non-timber forest product with a very large population in Indonesia. One of them is bamboo betung which can be found from the lowlands to the highlands but has not been used properly. Therefore, research was conducted to utilize bamboo betung to benefit the society. In this study, the synthesis of graphene oxide (GO) from bamboo betung stems will be carried out. The purpose of GO synthesis to further observe the crystal structure and crystal size of GO itself because it can be applied in various fields such as wave absorbers, radio frequency electronics, data display panels, and photovoltaic cells. GO synthesis was carried out using the Modified Hummers Method. The sintering temperatures used in this study were 300oC, 350oC, 400oC and 4500C. GO characterization was carried out using XRD and FTIR to reveal the system of crystal, crystal structure, and size, in addition functional groups, and phases of graphene oxide. GO has an average crystal size of 23.30165 nm as the largest average crystal size. GO layer is formed due to the bond between Carbon (C), Hydrogen (H) and Oxygen (O). GO Layer can be typed at any temperature variations carried out..*

KEYWORDS : GO, Bamboo betung, crystal size, crystal structure



## INTRODUCTION

Bamboo is typically segmented and in the form of clumps. In the bamboo village, they live in yards, riverside, cliff edges or community land bunddaries. Bamboo is a very useful plant for the community because it has many avail. Indonesia has 157 species of bamboo out 10% in the world, Bamboo betung is one of its kind[1]. Bamboo is one of the natural resources that is widely used by the community because it has beneficial properties, robust, straight and easy to work stems[2].

Betung bamboo stems can be used as materials of economic value. The chemical nature of bamboo is composed of holocellulose in 50-70%, pentose in 30%, and lignin in 20-25% [3]. Bamboo is a source of raw resources that can be utilised in the manufacture of activated carbon. The carbon content in lignin and cellulose contained in bamboo betung, makes this material can be used as an alternative in the manufacture of activated carbon [4]. This carbon derived from processed bamboo

stems will replace graphite as a carbon source which is the primary component used to make oxide of graphene.

Graphene oxide comes from a single layer that has a hexagonal structure in its crystal. The graphene oxide layer can be typed from the synthesis of natural materials in the form of graphite. Simply, graphite is oxidized to graphite oxide (GO), then the graphite oxide sheets are exfoliated in water to form graphene oxide [5]. The main material in the form of natural graphite is a non-renewable mining material. In addition, the use of synthetic graphite which has good quality also has a high cost. Therefore, the basic material for making graphene oxide can be replaced by the use of betung bamboo stems in addition to increasing the utilization of the bamboo stem itself, this can also generate economic value for the society[6]. Bamboo betung stems can be processed into powdered activated carbon which can be synthesized using the hummers method, a modification of the process can produce graphene oxide. Hummers synthesized graphene from graphite oxide using oxidizing compounds such as sulfuric acid with a concentration of 98%, nitric acid, and potassium permanganate. The result discovered from this method is that the level of effectiveness of oxidation is influenced by the ratio between graphite oxide in the product or the ratio between carbon and oxygen. The optimal ratio for oxidation to occur is a ratio of carbon and carbon of 2.1 to 2.9 [13]. The hummers method was also modified to reduce the production of toxic gas evolution so that the modified hummers method eliminated the use of NaNO3 compounds for the production of the toxic gas. By omitting NaNO3, increasing the amount of KMnO4, and conducting the reaction in a 9:1 (by volume) mixture of H2SO4 and H3PO4, the hummers' method was modified. Increasing KmnO3 in the proper hummers method and giving rise to a new compound, namely H3PO4[10]. The resulting graphene oxide has a crystal size with a range of diffraction angles at an angle ($10^{o}$ – $90^{0}$) [5]. The distinguishable functional groups are O–H, C=O, C–OH, C–O, and C-H bonds. The development of graphene oxide in the industrial world has several applications incorporating translucent conductive electrodes for solar cells, radio frequency electronics, flat-panel displays, and other conductors with high current densities [7].

The used of methods for the graphene oxide synthesis include the method micromechanical exfoliation, the method of epitaxial growth method, Chemical Vapor Decomposition (CVD) [8], Improved GO [6], and the method of Hummers [9]. The micromechanical exfoliation method is inefficient to use, while the epitaxial and CVD growth methods are quite expensive. So, the most efficient method used today is the Hummers method. Among the advantages of the Hummers method are because it uses KMnO4, which does not produce explosive elements, the reaction process doesn't take very long and is quite safe (explosives), such as ClO2 which is produced from KCLO3, using NaNO3 as a substitute for HNO3 which can produce acid mist. [10]. The use of the modified hummers method is to create a safe working process in the treatment of the compound [6].

In the research on the synthesis and graphene oxide characterization from coconut shells by sonication and hydrothermal methods pursuant to the results of XRD, Graphite oxide still exists in the sonicated sample. The SEM data, which show a porous and not transparent shape, and the absence of 2D points in the Raman characterization, support this. There is a C=C bond present in the FTIR characterization. O-H bonds, C-H bonds, and C=C bonds were found in the FTIR characterisation. Compared to the sonicated graphite, the surface morphology appears more clear. The findings demonstrated that the hydrothermal procedure produced graphene oxide and a transparent, thin layer as a result of a prolonged sonication process and particle sizes of 200# and 230# [11].

This research is very beneficial for the community because it can become an economic value for the community itself and it can also make it easier to produce GO with a faster method and at a lower price. In this research, the synthesis of graphene oxide (GO) from betung bamboo stems will

be carried out. GO can be applied in various fields such as wave absorbers, radio frequency electronics, data display panels, and photovoltaic cells. Therefore it is very important to know the crystal structure and crystal size to produce good GO. In this research, the sintering temperature variation treatment will be given to bamboo stems used for GO synthesis. Sintering is a change in the microstructure of a collection of powders due to heating at high temperatures [12]. Making graphene oxide by varying the temperature of the bamboo stove, namely 300°C, 350°C, 400°C and 450°C. affect the microstructure of the graphene oxide and also obtain the optimum conditions.

## METHODS

This research is a experimental research type. This research about analysis of the microstructure of graphene oxide made from bamboo stems synthesized by the modified hummer method. This research was conducted from March 2022 to July 2022 at the Physics Laboratory of FMIPA UNP for XRD characterization and at the Chemistry Laboratory of FMIPA Padang State University, for the process of GO synthesis and FTIR characterization. This research was conducted in several stages, namely the sample stage for preparation, the stage for sample making, the stage for sample characterization, and analysis of data.

Hummers synthesized graphene from graphite oxide using oxidizing compounds such as sulfuric acid with a concentration of 98%, nitric acid, and potassium permanganate. The result discovered from this method is that the level of effectiveness of oxidation is influenced by the ratio between graphite oxide in the product or the ratio between carbon and oxygen. The optimal ratio for oxidation to occur is a ratio of carbon and carbon of 2.1 to 2.9 [13].

In carrying out the research, several stages must be carried out, the first is the stage of drying and smoothing the bamboo betung sample. Pursuant to the modified hummer method used in the manufacture of graphene oxide, the basic material in the form of bamboo stems is processed into activated carbon which can produce a oxide layer of graphene. The processing on oxide of graphene can be done by drying the bamboo stems first, the dried bamboo stems can be burned using a furnace. Bamboo stems that have been burned turn into carbon, where the carbon is activated with NaOH compounds to make activated carbon. These results were synthesized using reducing compounds in the form of KMnO4, H2SO4, and H2O2 as well as denim water to be used as graphene oxide. The synthesis processwith these compounds was carried out using the modified method of Hummers.

Samples is consisted of bamboo stems were taken from Ambai, Kerinci Regency, Jambi Province. Then the bamboo stems are pured and dried by drying the bamboo stems in direct sunlight. Dry bamboo sticks are cut into small pieces and put in a baking dish, this aims to reduce the water content contained in the layer of betung bamboo sticks. Betung bamboo stems that have been cut into small pieces in the oven at 100oC for 1 hour. The completely dry sample was put into a ceramic crucible to be burned using a furnace with temperature variations (300, 350, 400, 450)$^{o}$C for 1 hour. Then the sample was ground using a pestle and mortar. Then the carbon sample of bamboo stems was sieved with a 200 mesh sieve to obtain a sample in the form of fine carbon. The results after being in the furnace and sifted can be seen in Figure 1.

(a) (b)

Figure 1. (a) Bamboo after being furnace and (b) Bamboo after being sifted

Based on Figure 1, it shown that the bamboo has changed and is sifted into powder. After the betung bamboo stems become powder, next is the activation stage. Carbon that has been refined weighed as much as 8 grams, as well as NaOH, weighed as much as 8 grams. Then the NaOH compound was dissolved by mixing NaOH into 100 ml of Aquades. Wait for the NaOH solution to completely dissolve. Then the NaOH solution is mixed into the carbon that has been in a 250 ml beaker. Stir the solution so that it is completely mixed, then let the carbon sample stand for 24 hours to form an activated carbon precipitate. The precipitate was dried by filtering the sample using filter paper and a Buchner funnel, this was done to facilitate the drying process. After all the samples were completely filtered, the activated samples were dried in a 105°C oven for three hours. The activated manufacture results carbon can be seen in Figure 2.

(a) (b)

Figure 2. (a) Filtering activated carbon and (b) Activated carbon after being oven-baked

Based on Figure 2, it shown that activated carbon has been typed and this activated carbon from bamboo betung will be used as the main ingredient in GO synthesis. GO synthesis was carried out using the Modified Hummers method. The first stage of this process is a solution of sulfuric acid (H2SO4) where 46 ml of the solution is taken into a beaker glass and placed into a water bath that acts as a thermostat. The solution was placed on a hot plate and mixed with 2 grams of activated carbon powder, stirring for 2 hours or 120 minutes. After that, mix 6 grams of potassium permanganate

(KmnO4) and stir for 30 minutes at a temperature of 20°C – 35°C. After 30 minutes 92 cc of distilled water were added to dilute the solution and then stirred again for 20 minutes, then the solution was mixed with 2 ml of peroxide acid (H2O2). This mixing is done until the bubbles disappear or no longer appear, which bubbles can disappear after stirring for 1 hour. Finally, 134 ml distilled water was poured into the mixture, which was then swirled for an hour to form graphene oxide. The synthesis of GO results can be seen in Figure 3.

(a) (b)

Figure 3. (a) GO stirring process (b) GO results

Based on Figure 3 it shown that the solution turns yellow which indicates the presence of graphene oxide. Then the mixture is sonicated using ultrasonic for 2 hours to peel off graphite oxide into graphene oxide [11]. Then the sample is allowed to stand for 1 day to form a solid phase precipitate and liquid. After that, the distilled water was replaced repeatedly to obtain a pH of 7 (neutral), then the graphene oxide sample was baked for three hours at 105°C.

# RESULTS AND DISCUSSION

## Results

Characterization results using XRD at a sintering temperature of of graphene oxide 300°C-450°C. This tes was conducted to see the crystal system, structure, and crystal size. These results can be seen in Figure 4.

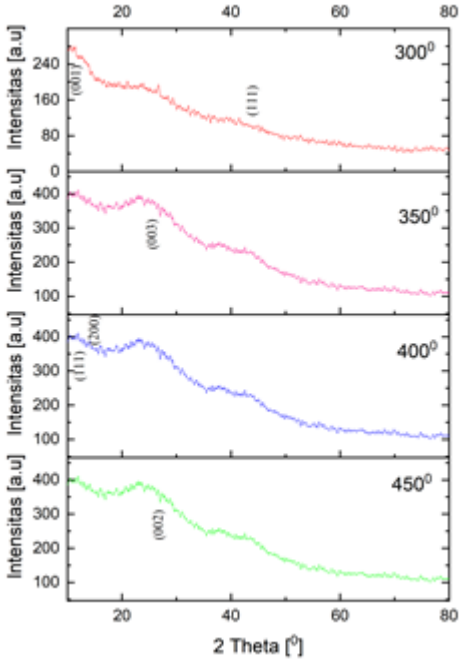


Figure 4. The Characterization of XRD Results of Temperature Variations 300°C, 350°C, 400 °C and 450 °C

Based on Figure 4 shows the diffraction pattern of graphene oxide at temperatures of 300°C, 350°C, 400 °C and 450 °C using Origin software. At a temperature of 300oC, it shown that there is an angle of for the peak deposition graph 11,340 and 43,630 Miller index values contained in the peaks associated with the emerging phase, namely (001) and (111). The lattice parameters found are in accordance with the Bravais lattice's a = b = c, = 90°, which has a f system orhombohedral crystal, based on ICDD data. The Scherrer equation determined the crystal size to be 20.36156 nm. The crystal size of the produced graphene oxide has a diffraction angle range of (2 θ) at an angle (10° – 90°)[14].

At a temperature of 350oC, it shown that there is an angle of for the peak deposition graph 26.60 and the Miller index value found at the peak associated with the emerging phase is (003). Pursuant to the data of ICDD discoveredThe lattice parameters were found to be in accordance with the Bravais lattice, which has a crystal cubic system and the coordinates a = b = c, = 90 °. The Scherrer equation determined the crystal size to be 22,184 nm.

At a temperature of 400 ° C, it shown that there is an angle of for the peak deposition graph 10,810 and 12,490 Miller index values contained in the peaks associated with the emerging phase, namely (111) and (200). Pursuant to the data of ICDD discovered, the lattice parameters found are in accordance with the crystal cubic Bravais lattice with a = b = c, = 90°. The Scherrer equation determined the crystal size to be 23.30165 nm.

At a temperature of 450°C, it shown that there is an angle of for the peak deposition graph and 26,420 Miller index values contained in the peak associated with the emerging phase, namely (002). Pursuant to the data of ICDD discoveredThe lattice parameters were found to be in accordance with

the hexagonal crystal structure of the Bravais lattice with a = b c, = 90 °, and = 120 °. The Scherrer equation determined the crystal size to be 18.86597 nm.

In XRD data processing using Origin, the size of crystal in average and the average size of the graphene oxide micro stain were also discovered. These results can be seen in table 1.

Table 1. Results of XRD Characterization, Average Crystal Size, and Stain Micro Size at 300 °C, 350 °C, 400 °C, and 450 °C.

| GO Temperature Variation (°C) | Crystal Average (nm) | Average Micro Stain ($10^{-3}$) |
|---|---|---|
| 300 | 20.36156 | 0.7148 |
| 350 | 22.184 | 0.4118 |
| 400 | 23.30165 | 0.4279 |
| 450 | 18.86597 | 0.3404 |

Based on table 1, it shown that the average size of crystals and the average size of the largest micro stains are 23.30165 nm and 0.4279 which are discovered at a temperature variation of 400 °C. The crystal size of the produced graphene oxide has a diffraction angle range of (2 θ) at an angle (10 ° – 90 °). Then it is proven that Pursuant to the test results there is already a graphene oxide layer typed [14].

In the FTIR test for an understanding of the functional groups in graphene oxide, the temperature variations are 300°C, 350°C, 400°C, and 450°C. These results can be seen in Figure 5.

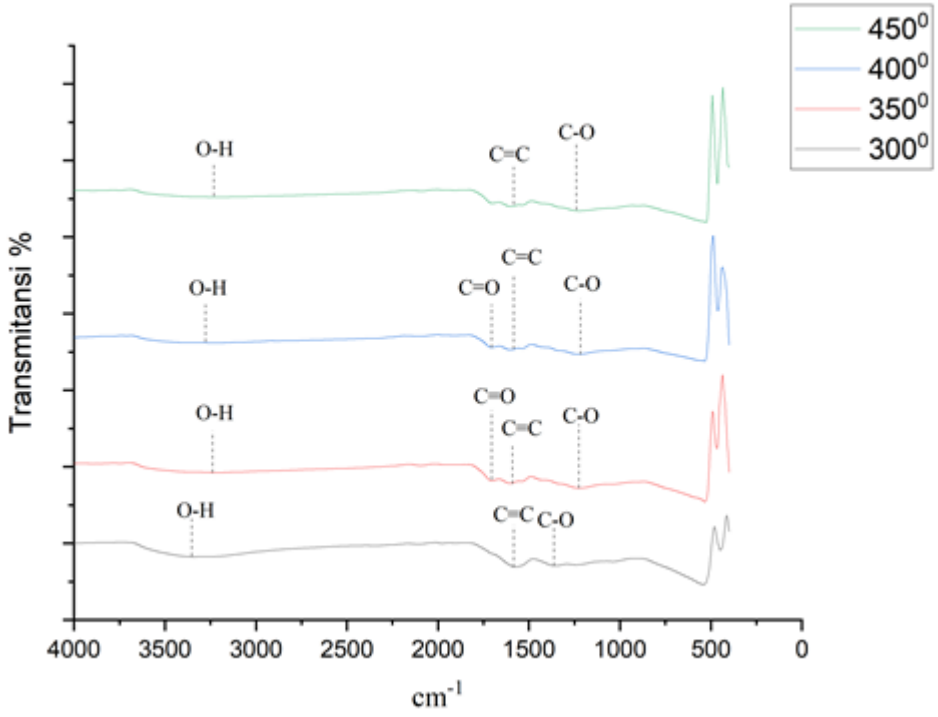


Figure 5. The Characterization of FTIR Results of Temperature Variations 300oC, 350oC, 400oC, 450oC

Based on Figure 5, there are several absorption peaks shown from results of GO used FTIR. GO consists of the functional groups C-O, C=O, C=C, and -OH in combination [15]. The C=C group of functional with the kind of alkene compound is in wave number 1600–1680 cm-1, and the aromatic ring with the C–O functional group is in wave number 890–1300 cm-1, which has the type of alcohol, ether, acid, carboxylic, and ester compounds. The O-H group of functional is in number of wave 3000-3640 cm-1 with the types of alcohol and hydroxy compounds and C=O is in number of wave 1640-1820 cm-1[16].

In Figure 5 (GO 300), there are 3 GO groups of functional, namely the C-O group of functional which is at number of waves is 1303.52 cm-1, the functional group C=C and is at number of wave 1673.51 cm-1, and the O-H group of functional. is at number of wave 3344.11 cm-1. In (GO 350) there are 4 functional groups GO, namely the C-O group of functional which is at the number of waves 1221.59 cm-1, the C=C group of functional which is at the number of waves 1604.91 cm-1, the C=O group of functional located at a number of waves on 1703.2 cm-1 and also the O-H group of functional which is at a number of waves are 3213.3 cm-1. In (GO 400) there are 4 group of functional, namely the C-O functional group which is at a wavelength of 1227.10 cm-1, the C=C group of functional which is at a number of waves are 1601.66 cm-1, the C=O group of functional which is at number of waves 1704.85 cm-1 and also the O-H group of functional which is at number of waves 3238.68 cm-1. In (GO 450) there are several groups of functional, namely the C-O group of functional which is at the number of waves 1227.02 cm-1, the C=C group of functional which is at the number of waves 1603.3 cm-1, and also the O-H group of functional at the number of waves 3221. ,36 cm-1. Graphene oxide or graphene oxide (GO) or graphitic acid is a blend of carbon (C), hydrogen (H), and

oxygen (O) molecules that was isolated from graphite by a vigorous oxidation process [17]. Bonds including Oxygen (O), Hydrogen (H), and Carbon (C).

## .CONCLUSION

Microstructure of Graphene Oxide synthesized from bamboo betung by modified hummers method. To sum up the Graphene Oxide which has been characterized contains bonds between Oxygen (O), Carbon (C), and Hydrogen (H) in the FTIR characterization test, as well as the XRD results which have an average crystal size of 23.30165 nm as largest average crystal size. Graphene Oxide Layer can be typed at any temperature variations carried out. However, the best results were discovered at a temperature variation of $400^0$C.

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