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# **Analysis of Optical Properties of Graphene Oxide from Bamboo Petung (Dendrocalamus Asper) Synthesized by Modified Hummer Method**

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\*Author Name: Indah Safira Email: Indahsafira360@gmail.com **Abstract:** Carbon, hydrogen and oxygen are abundant in the structure of graphene oxide (GO), often known as graphene. In this study, petung bamboo was used in the manufacture of graphene oxide. The aim of this research is to find out how the sintering temperature influences the ability of graphene oxide to absorb waves. The modified hummer method is used to oxidize graphite and produce Graphene Oxide. This research was divided into several parts, namely burning carbonized petung bamboo, production of graphene oxide, and sonication and neutralization of graphene oxide using sintering temperatures of 300°C, 350°C, 400°C and 450°C. The synthesis results were characterized using a UV-Vis spectrometer to obtain an absorption spectrum resulting in a band gap of 2.68 eV-4.38 eV in Petung bamboo at a sintering temperature of 300°C - 400°C, the bandgap increasing as the sintering temperature increased. At a sintering temperature of 400°C - 450oC the band gap value decreases which is influenced by the results of cell changes from refinement which can change the band structure thereby affecting the size of the band gap. The absorption value obtained in the optical properties decreases because the high sintering temperature causes the reaction rate to become faster so that the absorption value becomes lower.

**Keywords:** Bamboo petung, Energy gap, Graphene oxide, Hummer modification method, Optical properties.



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# **1. Introduction**

Indonesia is a nation with a wide range of creatures and flora. The bamboo is one of them. There are bamboo plants everywhere, both those that grow naturally and those that are produced

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specifically. There are 1200–1300 species of bamboo in existence worldwide. There are 143 different species of bamboo in Indonesia, 60 of which are found on the island of Java. The Indonesian people place great importance on the use of bamboo. Typically, bamboo plant stems are used in the manufacture of food, paper, building materials, handicrafts, and even medicines. However, it is still not ideal to use the roots, branches, and leaves of bamboo plants [1].

Rope bamboo, bamboo petung, andong bamboo, and black bamboo are often the ones that people in Indonesia utilize most frequently [2]. In comparison to other types of bamboo, such as bamboo tali 1,10%, bamboo hitam 2,93%, bamboo kuning 1,05%, bamboo andong 1,2%, and bamboo ampel 1,01%, Batang bamboo petung consist of 3,51% silicon. From the roots to the leaves, bamboo's silica concentration keeps rising [1]. Among carbon materials, graphene is the most promising material as an electrode for energy storage device applications because it has a high surface area, is relatively inexpensive, and has high electrical conductivity. Because of its huge surface area of 2630 m<sup>2</sup>/g, graphene is the perfect material for making supercapacitors.

Graphene was successfully synthesized in 2015 using the Zn reduction and Hummer process [3]. The most widely used method for the synthesis of graphene is chemical oxidation of graphite. This method involves the oxidation of graphite to GO (Graphene oxide) using strong oxidizing reagents, then GO can be converted into rGO through a reduction process using various reductants. Chemical oxidation of graphite is a method that uses concentrated acids (H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>, and H<sub>3</sub>PO<sub>4</sub>) and strong oxidizing agents (KMnO<sub>4</sub> and KClO<sub>4</sub>). There are several techniques for chemically generating GO, such as the Hummer, Staudeumaier, and Brodie procedures [4]. GO was first investigated by Brodie in 1859. Brodie investigated the structure of graphite by looking at the reactivity of the graphite sheets. Brodie uses  $KClO<sub>3</sub>$  and  $HNO<sub>3</sub>$  in the reaction process [5]. Subsequent research was carried out by Staudenmaier using H2SO4 and HNO3 materials. Staudenmaier mixed  $H_2SO_4$  and  $HNO_3$  and followed by the addition of KClO<sub>3</sub> little by little. The addition of  $H_2SO_4$  is done so that a strong oxidation process occurs. In 1958, Hummers discovered an alternative method for synthesizing GO using KMnO<sub>4</sub> and NaNO<sub>3</sub> and  $H<sub>2</sub>SO<sub>4</sub>$  [6].

Radio frequency electronics, flat-panel displays, and solar cells as transparent conductive electrodes and conductors with high current densities are only a few of the industrial uses for graphene oxide [7]. Graphene is a plane of a single atom of graphite. Atomic fields are of course familiar to everyone as a bulk constituent of crystals but one-atom thick materials such as graphene remain unknown [8]. Graphene has a hexagonal crystal lattice like a honeycomb and is semi-metallic with a zero band gap [9]. Graphene can be used in photonic, electronic, biomedical and environmental pollution control as a biosensor, energy storage, polymer nanocomposite and adsorbent [10].

The advantage of the Hummer method which has been modified namely simplicity, lower production costs and environmentally friendly materials used, products produced more regularly and require a slightly faster time when conducting experiments [11]. The process of producing graphene oxide involves heating bamboo petung stems to various temperatures, including 300°C, 350°C, 400°C, and 450°C. This temperature varies because each increase in temperature affects the optical properties of graphene oxide and generates favorable conditions.

Among the several advantages of the Hummers method, namely the reaction process does not take a long time or only takes a few hours, the reaction process is very safe because it uses  $KMnO<sub>4</sub>$  which does not produce explosive materials (explosives), such as  $ClO<sub>2</sub>$  which is produced from KClO3 in previous studies. Using NaNO<sub>3</sub> instead of HNO<sub>3</sub> which can produce acid mist. The difference between the Hummers method and the modified Hummers method lies in the amount of  $NaNO<sub>3</sub>$  and  $KMnO<sub>4</sub>$ . In the modified Hummers method, the amount of NaNO<sub>3</sub> used is reduced, while the amount of  $KMnO<sub>4</sub>$  is increased by a ratio of 9:1. KMnO<sub>4</sub> is one of the strongest oxidizing agents, especially under acidic conditions [12].

Based on the modified hummer method used to produce graphene oxide from bamboo petung, bamboo petung is processed into activated carbon to produce a layer of graphene oxide. Bamboo petung can be dried, then burned in a furnace to manufacture graphene oxide. Activated carbon is created by converting burned bamboo petung stems into carbon and activating it with NaOH chemicals. The graphene oxide used to produce these results was made using reducing agents like  $KMnO_4$ ,  $H_2SO_4$ , and  $H_2O_2$ , as well as aquades.

In this research, variations of sintering temperature will be given to petung bamboo stems used for GO synthesis. This temperature variation is because each increase in temperature will affect the optical properties of the graphene oxide and the optimum conditions can also be obtained. The thermal process treatment of 200°C in the furnace will increase the number of pores formed on graphene so that it will increase the value of the supercapacitor capacitance on graphene, while temperatures above 500°C can increase the number of pores because the higher temperature will break down the bonds in the carbon structure so that the carbon becomes damaged and the pore walls rupture. Sintering is a change in the microstructure of a collection of particles due to heating at high temperatures [13]. Graphene oxide was made by varying the furnace temperature of petung bamboo i.e. 300°C, 350°C, 400°C and 450°C. This temperature variation is because every increase in temperature will affect the optical properties of graphene oxide and also obtain optimum conditions.

## **2. Materials and Method**

This research is experimental in nature. This paper reviews the optical characteristics of graphene oxide produced by the Hummer Modification Method from bamboo petung (Dendrocalamus asper). This work involved the following stages: preparation; synthesis of Bamboo petung (Dendrocalamus asper); carbon activation using NaOH; production of graphene oxide; and characterization using FTIR, XRD, SEM, and UV-VIS instruments. After that, data analysis was concluded. The characterization process used: The XRD used is type X'Pert PRO with the brand PANalitycal MPD PW/3040/60 at the Materials Physics &Biophysics Laboratory, The FTIR used is the Shimadzu brand FTIR with the IRPESTIGE 21 type at the Chemistry Laboratory, Faculty of Mathematics and Natural Sciences Universitas Negeri Padang and The SEM used is the HITACHI SU-3400 M SEM at LIPI Bandung. The UV-VIS used is the UV-2100 PC SPECTROPHOOMETER in the chemistry laboratory at Padang State University. In the implementation step, there are several steps, namely: Preparation of charcoal of bamboo betung step, Carbon Activation Step, GO Synthesis Step, GO Sonication and Neutralization Step, and the last Characterization of GO.

The Dendrocalamus asper, also referred to as bamboo petung, was produced in the Kerinci region. Bamboo petung (Dendrocalamus asper) is harvested, split, and cut into several pieces before being sun-dried for two days to reduce the water content. The bamboo petung (Dendrocalamus asper) is cut into small pieces for the oven and placed in a container or tray at 110°C for an hour to evaporate any leftover water. After the bamboo petung (Dendrocalamus asper) has dried in the sun, this procedure has been carried out. After spending time in the oven, the bamboo petung (Dendrocalamus asper) is then heated for 1 hour at 300°C, 350°C, 400°C, and 450°C to convert it to charcoal. Using a mortar and pestle, bamboo petung is first turned into charcoal. The charcoal powder is then sieved through a sieve with a mesh size of 200. Displays the bamboo's outcomes following its treatment in the furnace and sieving can be seen in Figure 1.



Figure 1. Charred bamboo from a furnace

Based on Figure 1, the following stage entails activating refined carbon, which weighs up to 8 grams, as well as NaOH, which weighs up to 8 grams. The NaOH is then combined with 100 cc of Aquades to dissolve the NaOH compound. Hold off until the NaOH solution has dissolved entirely. The NaOH solution was already in a 250 ml beaker when the carbon sample was added. After properly combining the mixture with the carbon, activated carbon was allowed to precipitate for 24 hours.

After each sample had been thoroughly filtered, the activated samples were dried in an oven at 110°C for three hours. The modified Hummer process was then used to manufacture 2 grams sample of activated carbon utilizing reducing chemicals. Next, the first mixing stirrer process was carried out using a sulfuric acid solution (H2SO4) where the solution was taken as much as 46 ml, put into a beaker glass, and placed into a water bath which acts as a thermostat. The solution is placed on a hot plate and mixed with 2 grams of activated carbon powder, do it stirring for two hours or 120 minutes. After that, mix 6 grams of potassium permaganate (KMnO4) then stir for 30 minutes with a temperature of  $200^{\circ}$ C – 350°C.

After 30 minutes the solution was diluted and added with 92 ml of distilled water. Then strain again for 20 minutes, then the solution is mixed with 2 ml of peroxide acid (H2O2) and 134 ml of second distilled water. The use of mixing peroxide acid solution is to reduce the bubbles resulting from mixing KMnO4. This mixing is done until the bubbles disappear or no longer appear, in which case bubbles can disappear after stringing. The mixture was ultrasonified for two hours to exfoliate the graphene oxide. The samples were then left to stand for a day to create precipitates of the liquid and solid phases. The graphene oxide sample was then cooked in the oven at 110℃ for three hours to produce the final result of graphene oxide as shown.

Characterization of graphene oxide was then performed using XRD of the final result. The purity and crystal structure of the produced material are assessed using XRD. Each peak on the XRD pattern corresponds to a crystal plane that is oriented in a certain way along the threedimensional axis. The resultant peaks display the composition of the produced compounds and the crystal structure. All crystal-containing materials will be identified using XRD, which will be seen at the apex of the waves displayed. The relationship between x-ray diffraction and the sample it is treated to reveals the workings of the XRD system. The findings of the characterization are represented by a relationship curve between 2 and I, which is utilized with the Scherrer formula to determine the crystal size [14].

The next characterization was FTIR. FTIR is used to detect the functional groups of the material in the form of graphene oxide. Analyze the chemical compounds contained in the sample without destroying the sample. According to [15], FTIR spectroscopy is a measurement technique that uses infrared light to detect the vibrational transition of a molecule at a wavelength of 4000 cm<sup>-1</sup> to 500 cm<sup>-1</sup>. The vibrations generated from each group provide an image in the form of a line which helps identify the functional groups of the compound.

The sample's morphology is found via SEM. The morphology of the sample surface used is analyzed in the visualization. When the electron beam is diffracted on the sample's surface, this happens. The sample's surface has been captured by the SEM. The sample's graphene oxide layer can be recognized using the provided parameters.

UV-VIS is used for final characterization. In order to analyze the absorption characteristics of materials in the ultraviolet wavelength range (starting at around 200 nm and encompassing all visible light wavelengths), a UV-Vis spectrophotometer is utilized (up to about 700 nm). A spectrophotometer with a 500 nm wavelength was utilized for sample analysis [16].

## **3. Results and Discussion**

Characterization results have been obtained using XRD to determine the crystal structure and lattice parameters of graphene oxide, FTIR to determine the functional groups of graphene oxide of petung bamboo charcoal (Dendrocalamus asper), SEM to determine surface morphology and microstructure, and UV-VIS to measure the value of the relationship absorbance with wavelength and also energy gap. Graphene Oxide XRD characterization results for sintering temperature variations of 300°C, 350°C, 400°C, and 450°C can be seen in Figure 2.



Figure 2. XRD Characterization of Graphene Oxide sintering temperature variation 300°C, 350°C, 400°C, and 450°C

Based on Figure 2 it can be shown that at temperature of 400°C there are diffraction peak of 25.5832°. The Miller index values found at peaks associated with the emerging phase is (400). The crystal size obtained is 46.347612174 nm through the Scherrer equation. At a temperature of 450°C, there is a diffraction peak of 26.4669° and a peak associated with the emerging phase has a Miller index value of (002). The crystal size obtained is 35.45497252 nm through the Scherrer equation. According to the crystal size measurements, the largest crystal size is found at 400°C, while the smallest crystal size is found at 450°C.

Based on data from the XRD characterization, the findings at diffraction peaks between 10° and 44° are consistent with the claim that various properties of graphene oxide with diffraction peaks between  $2^{\circ} = 10^{\circ}$  to  $2^{\circ} = 44^{\circ}$ . Also, graphene oxide produced using graphite from carbonized agricultural waste has diffraction peaks of about 26.60°. And also, according to the other literature, the peak of graphene is around  $2\theta = 20$  to 26°, while for graphene oxide ranges between at  $2\theta = 24$  to  $26^{\circ}$ . FTIR characterization results with sintering temperature variations of 300°C, 350°C, 400°C, and 450°C can be seen in Figure 3.



Figure 3. FTIR Characterization of Graphene Oxide sintering temperature variation 300°C, 350°C, 400°C, and 450°C

Figure 3 shows that a number of absorption peaks display the outcomes of the FTIR characterization. The peak has peaks from the C-O, C=C, C-O and O-H functional groups. At various sintering temperatures, each has functional groups that correspond to the structure of the graphene oxide functional groups, namely C=O, O- H, C=C, C-OH and C-O [17]. The C-O functional group is in wave numbers  $1200-1275$  cm<sup>-1</sup> where in that wave number the types of compounds are alcohols, ethers, carboxylic acids, esters. The C=C functional group is at wave number 1566-1650 cm<sup>-1</sup> and has a cyclic alkane compound type. The C=O functional group is in wave numbers  $1685-1710$  cm<sup>-1</sup> with the type of aldehyde compound and O-H is in the wave number 3200-3550 cm<sup>-1</sup> with the type of alcohol compound [18]. Graphene oxide is a compound consisting of carbon (C), hydrogen (H) and oxygen (O), using FTIR characterization, this study can be said to have formed a layer of graphene oxide. SEM characterization results with sintering temperature variations of 300°C, 350°C, 400°C, and 450°C can be seen in Figure 4.



Figure 4. The results of characterization of graphene oxide using SEM at 1000x magnification (a)graphene oxide 300°C (b)graphene oxide 350°C (c)graphene oxide 400°C and (d)graphene oxide 450°C

Based on Figure 4, shows the outcomes of the characterization of graphene oxide using a SEM at a 1000x magnification. Each sample had similar morphology, with the only distinction being that the outcomes were uneven surface structures and dispersed lumps. In the SEM characterization analysis, there are differences from each sample, which can be seen in the distribution of the chunks. It can be seen that graphene oxide at 300°C and 350°C is scattered and not tightly packed with an almost non-uniform shape when compared to graphene oxide at 400°C and 450°C which looks dense and almost uniform. Graphene oxide at 400°C and 450°C

seems to have the characteristics of graphene oxide which are lots of large pores and thick exfoliated sheets. The UV-VIS characterization findings for sintering temperatures of 300°C, 350°C, 400°C, and 450°C, can be seen in Figure 5.



Figure 5. UV-VIS characterization with sintering temperature variations of 300°C, 350°C, 400°C, and 450°C

Based on Figure 5, is a UV-VIS characterization chart created from bamboo petung with different sintering temperatures that illustrates the correlation between wavelength and absorbance. The ultraviolet wavelengths seen in each peak at sintering temperatures of 300°C, 350°C, 400°C, and 450°C were, respectively, 270 nm, 267 nm, 265 nm, and 263 nm. Based on the absorbance measurement data above, it can be seen that with increasing wavelength, the absorbance spectrum will be smaller. At the maximum absorbance point, it indicates that the electrons cannot absorb energy at that wavelength so that when the given wavelength increases, the given energy is just passed.

The wavelength values at the four sintering temperature variations are in the range of 220 nm–270 nm, where these values are the characteristic wavelengths of graphene oxide (GO) with multilayer properties so that they can be categorized as GO. This could be due to the ability of the vibrations generated from the sonication tool to exfoliate the graphite solution. It is observed from the spectrum that the absorbance of the sample decreases slightly with increasing temperature. This absorbance is related to the size of the energy gap. Because absorbance indicates the amount of light that can be absorbed by a material used for electrons to move from the conduction band to the valence band, this is also known as the energy gap.

Figure 6 showed the size of the energy gap was related to this absorption. The energy gap, which is determined by the tauc plot method, is also known as absorbance because it reveals how much light can be absorbed by a substance when it is being utilized to transport electrons from the conduction band to the valence band.



Figure 6. Graph of Uv-Vis energy gap width (Eg) on graphene oxide from bamboo petung stems (a)300<sup>o</sup>C, (b)350<sup>o</sup>C, (c)400<sup>o</sup>C, (d)450<sup>o</sup>C

Based on Figure 6, the graph plot between  $(\alpha h\nu)^2$  and hu is shown at the intersection of the graphs and the flat axis shows the width of the energy band gap. The energy gap indicates the movement of electrons across the valence band towards the conduction band. It can be seen that the largest energy gap comes from the GO sample at  $400^{\circ}$ C, namely 4.38 eV, and its value increases with increasing sintering temperature, namely 2.68 eV, 3.92 eV, 4.38 eV, for sintering temperature  $300^{\circ}$ C,  $350^{\circ}$ C,  $400^{\circ}$ C respectively. The quality of the resultant coating may be the reason for the rise in the energy band gap value and the rise in sintering temperature. Free electrons travelling in the conduction band are to blame for this as they speed up conductivity. Some electrons are liberated when the sintering temperature rises, improving conductivity [19]. The increase in the energy band gap value as the sintering temperature increases can occur due to the quality of the layers produced. This occurs when electrons are free to move in the conduction band and accelerate conductivity. The smaller the energy gap value of a material, the smaller the energy band gap (between the conduction band and valence band), so that it will be easier for electrons in the valence band to jump (excite) to a higher band, namely the conduction band and vice versa, if The greater the energy gap value of a material, the electrons in the valence band require greater energy to move to the conduction band.

The results of this study are the same as the research conducted by [20] it can be concluded that UV-Vis shows an increase in the energy band gap as the heating temperature increases. At

the sintering temperature of  $400^{\circ}$ C –  $450^{\circ}$ C, the value of the energy gap decreases. The decrease in the value of the energy gap is influenced by the results of changes in cell parameters from refinement. Cell changes can change the band structure so that it affects the size of the energy band gap [3].

# **4. Conclusion**

Based on the findings of the research, it is possible to draw the following conclusions about the optical properties of graphene oxide produced by the modified Hummer method from bamboo petung: as the sintering temperature is raised, the resulting energy gap widens, leading to a rise in absorbance coefficient with each increase in sintering temperature. The absorbance value was decrease because the reaction rate was accelerated by the increaseing sintering temperature. It can be seen that the peak point of absorption is at the sintering variation of  $300^{\circ}$ C. It can be concluded that the most effective GO results are at the most optimal  $300^{\circ}$ C sintering variation based on the wavelength value produced.

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