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Analysis of the Electrical Properties of Graphene Oxide from Bamboo Betung Synthesized by the Modified Hummers Method

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Abstract: The purpose of this study is to know the capacitance and conductivity of graphene oxide made from betung bamboo. Betung bamboo was synthesized using modified hummers method. The carbonization procedure, which will be the control variable in this study, will be carried out in the first stage at four different temperatures for an hour: 300°C, 350°C, 400°C, and 450°C. The results are identified by the initials GO300, GO350, GO400, and GO450. The samples were then characterized using the XRD (X-Ray Diffraction), SEM (Scanning Electron Microscope), FTIR (Fourier Transform Infrared), and LCR Meter instruments. At a temperature of 400 °C, the LCR Meter measured the greatest conductivity and conductance values, with values of 5.43×10^{-6} S/cm for conductivity and 5.66×10^{-7} F/m² for area capacitance. And according to the average conductivity and conductance values, this graphene oxide is situated in the region of semiconductor materials conductivity values.

Keywords: Betung bamboo, Electrical properties, Graphene oxide, LCR Meter

1. Introduction

In recent years, technological advancements have increased population growth throughout the world. The community's demand for electrical energy and power storage will increase as technology advances. A vital need in everyday life is electricity. Electrical energy storage technology is one of the technologies that needs to be created with the advancement of technology [1]. The majority of people so far only use batteries and capacitors as a means of storing electrical energy. Energy storage technologies for electrical energy are separated into a number of categories, including fuel cells, flywheels, and supercapacitors [2]. A supercapacitor is

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a double-layer capacitor that uses electrodes, an electrolyte, a separator (separator), and a current collector to store energy by charge transfer. Supercapacitors provide a number of benefits, including a longer lifespan, straightforward models and principles, quick recharging times, and secure use [3].

Supercapacitors, nanoelectrics, nanocomposites, sensors, batteries, semiconductors, and transparent electrodes are examples of graphene-based electrical applications [4]. The monoatomic two-dimensional substance known as graphene in 2004 from a single layer of graphite [5]. Researchers from a variety of scientific disciplines, including physics, chemistry, and biology, are interested in studying graphene. After the transparent band of graphite peels off and minerals adhere to the band, graphene is discovered. Graphene is a substance that conducts electricity or has the potential to do so. In place of copper in the construction of capacitors, graphene's plate structure is frequently employed as the primary component because it is stronger than steel [6].

Graphene possesses intrinsic strength of 130 Gpa, a Young's modulus of 1 Tpa, and electrical, mechanical, and thermal properties. It has a specific surface area of 2620 m^2/g. At ambient temperature, graphene has an electron mobility of 2.5 x 105 cm^2/Vs and a very high electrical conductivity. It has a thermal conductivity of roughly 3000 Wm/K [7] . Graphene is a substance comprised of carbon in the form of graphite, where each carbon atom possesses two-dimensional sp^2 bonds and is closely packed in a honeycomb-like crystal structure. The placement of the carbon atoms (C), which is exceedingly regular and nearly perfect, gives graphene its distinctive structure. Another benefit of graphene is its huge surface area (1 m^2) and light weight (0.77 mg). Two-dimensional material is exemplified by this tiny layer of graphene. Since two-dimensional (2D) materials like graphene are not found in nature, graphite must be used in their synthesis [8].

Graphene and graphene oxide are two distinct substances. Graphite oxidation produces graphene oxide, which contains more oxygen and has less sample thickness because some Van der Walls bonds have been freed. Graphene oxide has a large surface area and a functional group that contains oxygen. based on a 2011 study by Bin Xu et al. that contrasted the capacitance characteristics of graphene oxide and graphene as electrode materials in supercapacitors. Graphene oxide produces high capacitance values up to 189 F/g despite having half the surface area of graphene and having a large number of oxygen-containing functional groups on its surface [9].

There are different methods for making graphene. One of the methods discovered by Hummers is by reacting graphite with potassium permanganate ($KMnO_4$) and sodium ($NaNO_3$) in a solution of sulfuric acid (H_2SO_4). Since the modified hummers technique doesn't release ClO2 gas during the oxidation process, the oxidation process moves more quickly and at a lower temperature, and the materials used are straightforward to obtain, it is stated to be superior than the previous technique [8].

An organic material with a high carbon content serves as the primary basic component of activated carbon [10]. Due to Indonesia's abundant supply of bamboo, which hasn't been utilized to its full potential, bamboo was chosen as the research material. The potential for using Indonesian bamboo plants in research is quite intriguing. Bamboo is a plant that is simple to grow and has a short life cycle, only taking 3 – 4 years from planting to harvest [11]. Betung

bamboo grows in the lowlands to the mountains at an altitude of 2000 meters above sea level. And in Betung bamboo which is approximately 3 – 4 years old, has a silica content of 0.10% - 1.78%, cellulose content of about 52.9%, and lignin content of about 24.8% which contains a lot of high carbon [12].

In this study, bamboo betung materials were used to produce graphene oxide because of the carbon content in lignin and cellulose found in bamboo, this material is an alternative for producing activated carbon. And it will then be characterized using XRD (X-Ray Diffraction) to identify the phase that has developed on the material. Based on the 2θ and I angle values that were specified using XRD to analyze the phase generated on graphene oxide, the results from the examination of diffraction patterns with HighScore Plus Software were used. In the following circumstances, Scherrer's formula can be used to calculate the size of the crystal on :

$$D = \frac{k\ x\ \lambda}{\beta\ x \cos\theta} \quad (1)$$

Where :
D = the crystal size
K = the form factor of the crystal (0.9)λ = wavelength of X-rays (1.54056 Å)
β = the value of Full Width at Half Maximum (FWHM)
θ = angle of diffraction (degree)

The second characterization was SEM (Scanning Electron Microscopy) which is used to identify the graphene oxide's surface microstructure. Graphene oxide's functional group or atomic bond structure can be identified via FTIR (Fourier Transform Infra Red). The electrical characteristics of graphene oxide, particularly its area capacitance and conductivity values, will be assessed using an LCR meter. The data obtained from LCR meter measurements are resistance, frequency, capacitance and impedance. The first step is to use the following equation to determine the resistivity value. And then, to get conductivity we can use equation (3) when the resistivity value and the conductivity value are diametrically opposed.

$$R = \rho \frac{L}{A} \quad (2)$$

$$\sigma = \frac{1}{R} \quad (3)$$

Where :
R = resistance (Ω)
ϱ = resistivity (Ωm)
L = cross section length (m)
A = cross-sectional area (m^2)

And the formula below can be utilized to determine the area's capacitance value. Where the Cp iscapacitance (F)

$$C = \frac{Cp}{A} \quad (4)$$

The data obtained for the LCR meter are the capacitance and resistance values of graphene oxide varieties, then the data is processed to obtain the conductivity and capacitance area values using formulas (2), (3) and (4).

2. Materials and Method

This research is an experimental study using a modified Hummer method as a method for synthesizing graphene oxide. The samples were then characterized using the XRD (X-Ray Diffraction), SEM (Scanning Electron Microscope), FTIR (Fourier Transform Infrared), and LCR Meter instruments. The first step in preparing carbon for graphite preparation is to cut a rectangular-shaped piece of bamboo about 50 cm long lengthwise. After that, the bamboo betung was exposed to the sun for five days to evaporate any remaining moisture. The bamboo betung is then placed in the oven for 6 hours at a temperature of 110 °C to completely dry it out of any remaining moisture. The bamboo betung stem was then placed in a vaporizer cup, wrapped with aluminum foil, and heated for one hour at a range of four different temperatures: 300°C, 350°C, 400°C, and 450°C. The powdered bamboo charcoal was filtered through a sieve with a 200 mesh size [12]. The bamboo before the combustion process and after the combustion process can be seen in Figure 1.

(a) (b)

Figure 1. (a) rectangular-shaped piece of bamboo (b) charred bamboo from a furnace

The next step was carbon activation once the bamboo has been ground into charcoal powder. Bamboo charcoal powder is combined with a sodium hydroxide (NaOH) solution to complete the phases of carbon activation. A fume hood was used for the NaOH solution's production. First, using a spatula and 100 mL of distilled water in a volumetric flask that has already been pre-filled with distilled water, dissolve 8 grams of solid NaOH. until the NaOH solid disintegrates and the solution returns to being homogenous or transparent. Then, combine 100 mL of NaOH solution with 8 grams of bamboo charcoal powder in a beaker until the bamboo charcoal powder is completely covered. The mixture was submerged for 24 hours [13]. The submerged mixture will form a deposit on the beaker's bottom after 24 hours. After that, a Buchner funnel and filter paper were used to filter the precipitate that had developed. Bamboo charcoal powder was extracted from the NaOH solution and bamboo charcoal combination using filtration. After that, dry the carbon-activated bamboo charcoal powder in a 100°C oven for three hours using the filtered bamboo charcoal betung in a steamer cup.

The modified Hummers method was then used to create graphene oxide. Activated graphite or charcoal powder, $KMnO_4$, $NaNO_3$, H_2SO_4, and H_2O_2 are the ingredients utilized in this method. Weighing 2 grams of graphite powder, 1 gram of $NaNO_3$, and 46 mL of H_2SO_4 solution before adding them to a flask with a magnetic bar inside is the first step. Then stirred at a speed of 250 rpm for 20 minutes. The Erlenmeyer is then placed in anice bath, where it will remain for

the next two hours while being stirred at a temperature of 20°C. After that, gradually add 6 grams of $KMnO_4$ until the solution turns greenish black. After that, the Erlenmeyer was taken out of the ice bath and put back on the magnetic stirrer, where it was stirred for a further 30 minutes at ambient temperature until the solution turned dark brown. A 92 mL dropper pipette was used to drip the Aquades slowly after they had been stirred. Next, 134 mL of distilled water and 2 mL of H_2O_2 were slowly added so as not to start a fire. Until the solution turns a pale yellow, this H_2O_2 solution is added to cease the process.

The graphene oxide dispersion technique with sonication is then used to try to peel the graphene oxide into a thin layer once the solution appears to turn yellow. An ultrasonic cleaner was used to sonicate the material for two hours at room temperature, then the solution was left in place for 24 hours. After that, the solution is centrifuged using a microcentrifuge at 4000 rpm for 15 minutes to separate the solution's solid and liquid phases. In addition, the graphene oxide solution is once more neutralized by depositing it to create a solid phase and a liquid phase. The supernatant solution was made clear by washing the solid phase with distilled water. Measure the pH again until it is neutral. The graphene oxide precipitate is put in a container and heated to 105 °C in the oven for three hours [14]. After the synthesis step is complete, the next step is the material characterization test. The data obtained from the characterization with XRD were processed using formula (1) to determine the crystal size of graphene oxide. Then, data from the FTIR characterization results were collected to determine the elements contained in graphene oxide. The data obtained for the LCR meter are the capacitance and resistance values of graphene oxide varieties, then the data is processed to obtain the conductivity and capacitance area values using formulas (2), (3) and (4).

3. Results and Discussion

The first XRD characterization can be seen in Figure 2 that shows the graphene oxide diffraction pattern at temperatures of 300°C, 350°C, 400°C, and 450°C using Origin software.

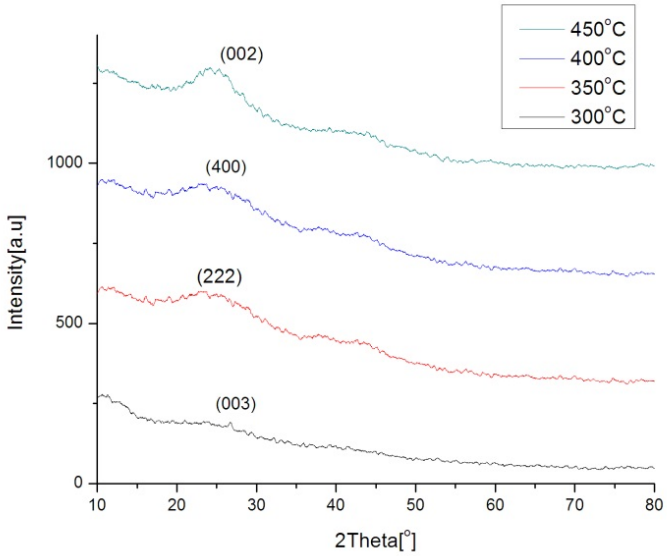

Figure 2. The graphene oxide diffraction pattern at temperatures of 300 °C, 350 °C, 400 °C, and 450 °C using XRD

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° = 10° to 2°= 44° [14]. Also, graphene oxide produced using graphite from carbonized agricultural waste has a diffraction peaks of about 26.60° [15]. And also, according to the other literature, the peak of graphene is around 2θ = 20 to 26°, while for graphene oxide ranges between at 2θ = 24 to 26°[16].

The second characterization, which includes the graphene oxide's surface dimensions as indicated in the picture, was discovered by SEM characterization. SEM (Scanning Electron Microscopy) is a characterization test used to analyze the surface microstructure of graphene oxide. The results from characterization of graphene oxide using SEM can be seen in Figure 3.

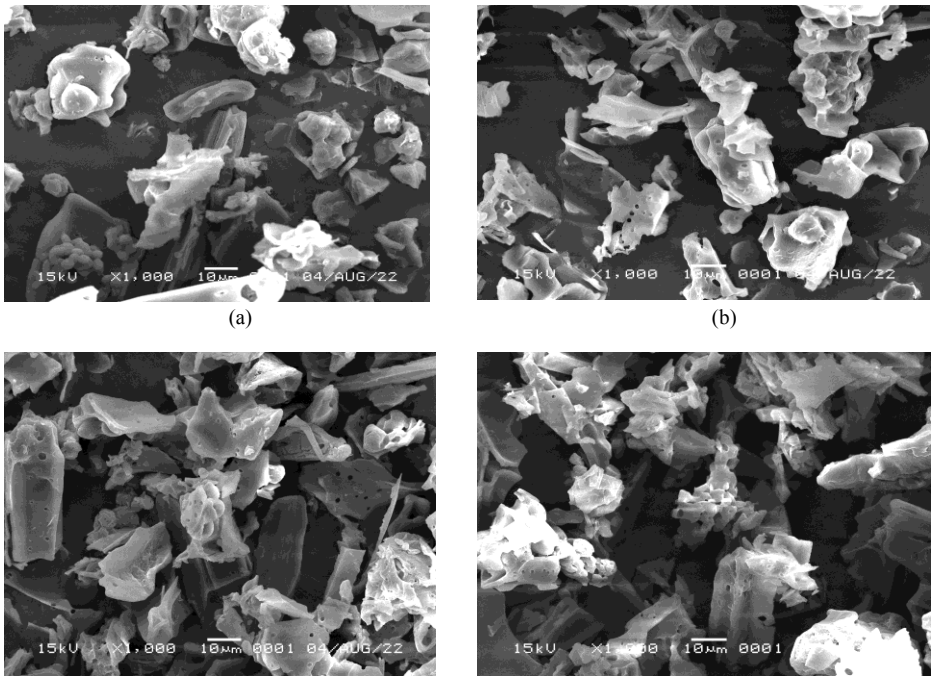

Figure 3. Results from characterization of graphene oxide using SEM at 1000x magnification (a) graphene oxide300℃ (b) graphene oxide 350℃ (c) graphene oxide 400℃ and (d) graphene oxide 450℃

The results of the characterization of graphene oxide using a SEM at a 1000x magnification are shown in the Figure 3. Each sample showed similar morphology, as evidenced by the results in the form of sporadic lumps anderratic surface patterns. Differences between each sample may be detected in the distribution of the chunks in the SEM characterization analysis. In contrast to graphene oxide at 400°C and 450°C, which seems thick and nearly uniform, graphene oxide at

300°C and 350°C is dispersed, not densely packed, and almost irregularly shaped. At 400°C and 450°C, graphene oxide appears to exhibit the properties of graphene oxide, including a lot of big pores and thick sheets that peel off [17].

The third data known as the FTIR characterization. The purpose of the FTIR test is to identify the chemical bonds or functional groups of graphene oxide. Data was obtained from the graph of the characteristic vibration method with transmittance and then examined by figuring out the wavelength with the functional groups formed. The result of graphene oxide at temperatures of 300 °C, 350 °C, 400 °C, and 450 °C can be seen in Figure 4.

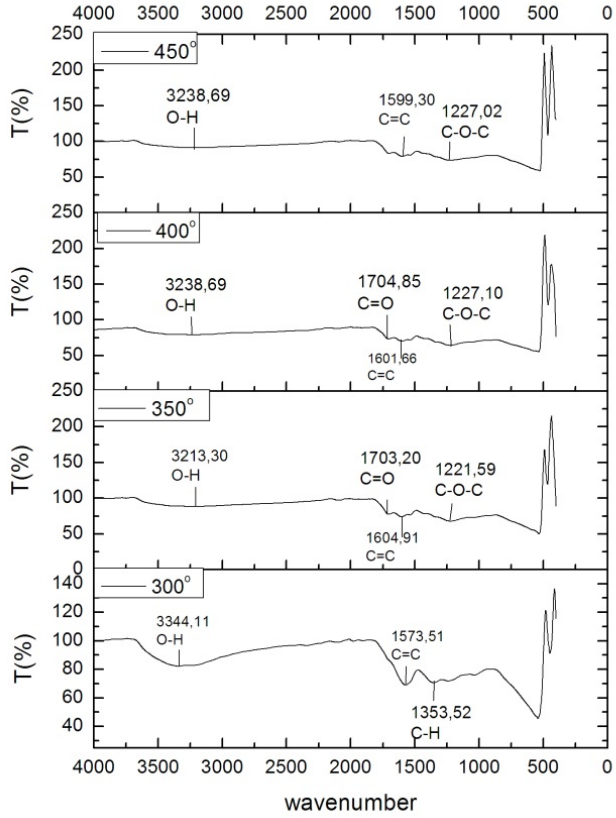

Figure 4. Result of graphene oxide at temperatures of 300 °C, 350 °C, 400 °C, and 450 °C using FTIR.

In the Figure 4, graphene oxide graphs for the four temperature variations can be seen at wavelengths from 3000 to 3750 cm^{-1}, indicating the presence of an O-H bond peak; 1600 to 1900 cm^{-1}, indicating the presence of a C=O peak bond; 1500 to 1675 cm^{-1}, indicating the presence of a C=C peak bond; and at 1230 to 1270 cm^{-1}, indicating the presence of a C-O-C peak bond [18]. The test results for graphene oxide at a temperature of 300℃ obtained O-H vibrations at 3344.11 cm^{-1}, C=C at 1573.51 cm^{-1} and C-H at 1353.52 cm^{-1}. Graphene oxide at 350℃ obtained O-H vibration at 3213.30 cm^{-1} , C=Oat 1703.20 cm^{-1}, C=C at 1604.91 cm^{-1} and C-O-C at 1221.59 cm^{-1}. the results of the graphene oxide test at 400℃ obtained O-H vibrations at 3238.69 cm^{-1}, C=O at 1704.85 cm^{-1}, C=C at 1601.66 cm^{-1} and C-O-C at 1227.10 cm^{-1}. And the test results of graphene oxide at a temperature of 450℃ obtained O-H vibrations at 3221.36 cm^{-1} , C=C at 1599.30 cm^{-1}, C-O-C at 1227.02 cm^{-1}.

From the results of the FTIR, graphene oxide is obtained based on bond peaks containing wave bonds which indicate the presence of a C=C peak bond which is an aromatic functional group, a C=O peak bond which is a carbonyl and carboxyl functional group [5], the presence of an O-H bond peak which is an indication of a hydroxyl functional group, and the presence of a C-O-C peak bond which is an epoxy functional group which is in accordance with the statement [19] Graphene oxide is an oxidation state of graphene with oxygen functional groups (epoxide, hydroxyl, carbonyl and carboxyl groups). All peaks indicate the presence of graphene oxide functional groups with a mixture of carbon (C), hydrogen (H), and oxygen (O) compounds [20]. The capacitance and resistance values are derived from the LCR meter's characterization findings. The data will also be analyzed using equations (3) and (4) in order to derive the conductivity, area capacitance, average conductivity, and average area capacitance of the four temperature variations.

The value of capacitance and resistance, which are derived from the results of characterization using an LCR meter, are then processed by the electrical conductivity data using the computation of the resistance value based on equation (2) to get resistivity. Then to get the value of electrical conductivity obtained using the following formula (3). And for the area capacitance calculated using the capacitance formula based on the cross-sectional area measurement findings as determined by equation (4). The material was compressed into a tube-shaped pellet with a diameter of 2 mm, a height of 3 mm, and a cross-sectional area data (A) of 33 mm before being examined. Table 1 shows the results of data processing for resistivity, conductivity, and area capacitance.

Table 1. Result of resistivity, conductivity and area capacitance of graphene oxide

Sample	**Resistivity (Ω)**	**Conductivity (S/cm)**	**Area capacitance (F/m²)**
GO300	2.17×10^{5}	4.86×10^{-6}	7.02×10^{-7}
GO350	1.56×10^{5}	6.54×10^{-6}	7.44×10^{-7}
GO400	1.29×10^{5}	3.43×10^{-5}	5.07×10^{-6}
GO450	1.03×10^{5}	2.02×10^{-5}	4.62×10^{-6}

The resistivity and conductivity values are impacted by changes in combustion temperature in the furnace process, as can be seen in the table above. The conductivity and capacitance area increase and the resistivity value decreases as the combustion temperature rises. Figure 5 shows a graph illustrating the correlation between temperature and conductivity values.

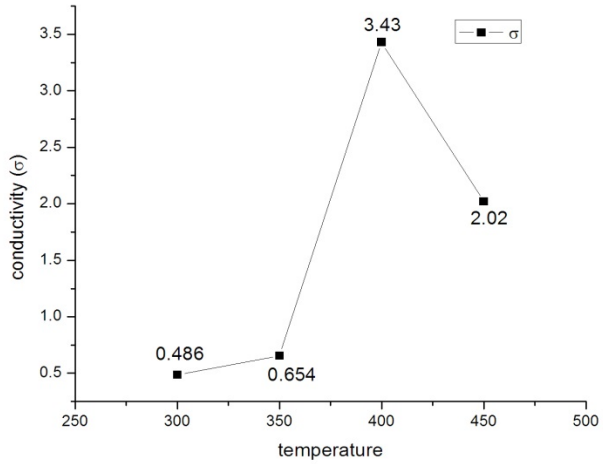

Figure 5. Graph that shows the relation between conductivity and combustion temperature

In Figure 5 can be seen that the conductivity value increases from 300 ℃ to 400 ℃ and decreases at 450 ℃. Figure 6 shows a graph illustrating the correlation between temperature rise and area capacitance values.

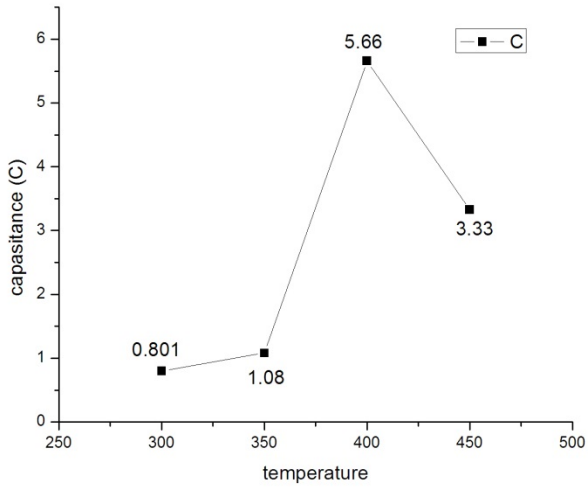

Figure 6. Graph that shows the relation between area capacitance and combustion temperature

Figure 5 and 6 shows that after analyzing the LCR Meter data, the highest conductance and capacitance values occur at 400°C and drop at 450°C. This is consistent with the claim [21] that conductivity and area capacitance values rise with increasing carbonization temperature. Additionally, in this study, the surface area will increase as the activation temperature rises, and if it has reached an optimum point, the surface area and conductivity value will decrease. The statement that the value of electrical conductivity is exactly proportionate to a rise in temperature is not supported by the research findings. The increase in crystal size is what causes the capacitance value to rise from 300°C to 400°C. The capacitance and conductivity values increase with crystal size. While the decrease in conductivity and capacitance values at 450°C is brought about by a reduction in particle size, this also results in smaller capacitance and conductivity values [22]. The size of the crystal increases as the carbonization temperature rises. The atomic arrangement's increased orderliness may also be correlated with the crystal size.

It is evident that the conductivity and capacitance values fall at 450°C. According to research by [23] who optimized the synthesis of activated carbon from Schizostachyum brachycladum reed bamboo with variations in carbonization temperature for the absorption of iron in peat well water, increasing carbonization temperatures and temperatures above 500°C can increase the number of pores as a result of increasing temperature. Due to the volatile nature of bamboo, high carbon content will cause the carbon structure's bonds to break down, causing the carbon to become damaged and produce ash. If the remaining component of the combustion results is the ash element, the predominant component of the ash is silica minerals, and it has a negative impact on the calorific value produced [24].

Bin Xu et al. in a 2011 study that compared the capacitance properties of graphene oxide and graphene as electrode materials for supercapacitors. Graphene oxide has half the surface area of graphene and has many oxygen-containing functional groups on its surface, so that graphene oxide produces high capacitance values. The conductivity value area of semiconductor materials, which is located at a value of 10^{-8} S/m to 10^{3} S/m, is where the Betung bamboo graphene oxide is placed based on the area conductivity and capacitance values[25], with average conductivity values ranging from $4.86\text{x}10^{-6}$ S/cm to $2.02\text{x}10^{-5}$ S/cm and average area capacitance values ranging from $8.01\text{x}10^{-8}$ F/m2 to $3.33\text{x}10^{-7}$ F/m^2. And the capacitance value of this area is not too small compared to research conducted by [25] in a research on the manufacture of reduced graphene oxide (rGO) materials made from chicken feathers with a capacitance value in the research conducted of $1.29\text{x}10^{-7}$ F/m^2.

4. Conclusion

The conductivity and capacitance area of graphene oxide are precisely proportional to the rise in combustion temperature from 300°C to 400°C, according to the results of the LCR Meter's characterization. The levels of conductivity and area capacitance increase with increasing combustion temperature. However, due to the excessively high combustion temperature at 450°C, the conductivity and capacitance values fell. Accordingto the conductivity and conductance values of the region, the graphene oxide of betung bamboo is situated in the range of semiconductor materials' conductivity values, which is between 10^{-8} S/m and 10^{3} S/m. The results of this study also showed that graphene oxide produced from betung bamboo has the potential to be employed as anelectrode material for supercapacitors.

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