

Microwave Absorption Properties of Graphene Oxide Derived from Coconut Shell Waste by Modified Hummer's Method

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ABSTRACT

Coconut shells are organic waste, so they can be used as an alternative for carbon source. In this study, the synthesis of graphene oxide (GO) from coconut shell waste will be carried out. The results of the synthesis of GO will then be tested for microwave absorbing properties because it can be applied in various fields, such as information technology, medical equipment, industry, polymer synthesis, and organic synthesis. GO synthesis was carried out using the modified Hummers method. There are several stages in this study, namely the stage of preparing old coconut shells, the stage of carbon activation, the stage of GO synthesis, and the stage of sonication and neutralization of GO. The coconut shell was treated with variations in the sintering temperature to see its effect on the microwave absorbing properties. The sintering temperatures used in this study were 250°C, 300°C, 350°C, 400°C, and 450°C. GO characterization was carried out using X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR), and Vector Network Analyzer (VNA) to determine the phase, functional groups, and microwave absorption properties of GO. In the results of GO characterization using VNA, it was found that there was an effect of temperature variations. GO can be synthesized from old coconut shell waste using the modified Hummers method and has a GO phase result. The best microwave absorbing properties are at a sintering temperature of 400°C with a reflection loss value of -24.40 dB. Absorbing coefficient 93.97% at 10.40 GHz.

Keywords : Coconut Shell, Graphene Oxide, Modified Hummer's Method, Reflection Loss



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I. INTRODUCTION

Indonesian coconut is one of the export commodities from the Indonesian plantation sub-sector. Coconut is processed into several products, one of which is crude coconut oil and virgin coconut oil [1]. Coconut shell is organic waste, so it can be used as an alternative carbon source. The coconut shell contains organic compounds, such as cellulose, lignin, and hemicellulose. Organic compounds can be used as a carbon source if further processed. Carbon materials have had increasing attention due to their remarkable properties like conductivity and chemical stability. The morphology of the carbon materials at the macro-scale provides for a diversity of application, such as active carbon with a large number of pores as a catalyst, carbon fiber for structural natural material [2].

Graphene oxide has been demonstrated to be a good precursor material for the fabrication of graphene-based materials in large quantities due to its low-cost and easily scalable synthesis process [3]. Graphene applications are very wide in various fields such as nano electricity, sensors, nanocomposites, batteries, supercapacitors, semiconductors, and transparent electrodes [4].

Coconut shell is organic waste, but it is difficult to decompose by microorganisms because of its hard nature. The coconut shell used is an old coconut shell that has a hard structure due to the high silicate (SiO₂) content in the coconut shell. This waste has a large weight and size, so it can cause accumulation in garbage disposal [5]. In addition, coconut shell is a material with a carbon content of 49.86% (old coconut type) which consists of cellulose and hemicellulose and mostly consists of lignin [4].

Graphene has good conductivity, fast electron mobility, an excellent catalyst, a large surface area for contact, and great optical transparency. Graphene is a 2D carbon allotrope with a honeycomb lattice structure. Individual carbon atoms are bonded by sp^2 hybridization [5]. Graphene is one of the carbon families with low density, thinness, high electrical conductivity, corrosion resistance, and large surface area that is very suitable for being made as a radar wave absorbing material [6]. Graphene can be obtained in the form of reduced Graphite oxide, sometimes also referred to as Graphene oxide [7].

Carbon is able to form many allotropes because of the valence number possessed by the carbon atom. The carbon allotropes that have been known to date are diamond, carbon nanotubes, fullerenes, fullerites, graphene, and graphite [8]. One of the advantages of GO is its easy dispersibility in water and other organic solvents, as well as in different matrixes [9].

In this study, variations in combustion temperature of coconut shell were used for GO synthesis. Temperature were change the microstructure of charcoal powders from coconut shell due to heating at high temperatures [6] as well as an analysis of the properties of the microwave absorber.

II. METHODS

GO synthesis was carried out with a modified Hummer's method. The stages in this study were the old coconut shell casting stage, carbon activation stage, GO synthesis stage, and GO sonication and neutralization stages. Treatment of variations in the combustion temperature of coconut shells to see the effect on the absorption of microwaves. The combustion temperatures used were 250°C, 300°C, 350°C, and 400°C and 450°C. The materials used in this study are as follows; coconut shell waste, NaOH, Sulfuric Acid (H_2SO_4), Sodium Nitrate ($NaNO_3$), Potassium Permanganate ($KMnO_4$), Hydrogen Peroxide (H_2O_2), and distilled water.

The research was carried out at the Laboratory of Physics and Laboratory of Chemistry, Universitas Negeri Padang, Laboratory of LLDIKTI Region X Padang. The characterization VNA was carried out at the Telecommunications Laboratory, Electrical Engineering Department, Padang State Polytechnic, XRD at the Materials Physics & Biophysics Laboratory, and FTIR at the Chemistry Laboratory, FMIPA UNP.

The independent variable is the combustion temperature of coconut shell waste. The temperature variations used were 250 °C, 300 °C 350 °C, and 400 °C, 450 °C. The control variables in this study were the temperature of the coconut shell charcoal oven which was 100°C, the time of the coconut shell charcoal oven process was 60 minutes, the processing time of the shell charcoal furnace was 30 minutes, the concentration of H_2SO_4 (98% wt.), the concentration of H_2O_2 (30% wt.), the concentration of NaOH, the time of the centrifugation process, the duration of the stirring process for each stage, the GO pH, the GO oven temperature at 60°C for 12 hours, the composition of the materials used at each stage, and the size of the mold for testing using VNA, namely 2 cm x 1.5 cm with a thickness of 2 mm. The dependent variable in this study is the microwave absorption properties of GO.

2. 1. Preparation of charcoal from coconut shells

The coconut shells used are old coconut shells that have been collected and then dried for 2 days in the sun to reduce the water content in the old coconut shells. Then, the coconut shell is cut into small pieces and heated for 60 minutes at 110°C. The coconut shells that have been baked are then subjected to a furnace process for 30 minutes at temperatures of 250°C, 300°C, 350°C, 400°C and 450°C so that the coconut shells turn into charcoal. The coconut shell charcoal is then crushed with a mortar and pestle to produce charcoal powder and sifted with a sieve size of 125 mesh.

2.2. Carbon activation of charcoal from coconut shells

The carbon activation stage was carried out by mixing coconut shell charcoal powder with NaOH solution. As much as 8 grams of coconut shell charcoal powder and 100 mL of NaOH solution are put into a beaker until the charcoal powder is submerged in the NaOH solution. The soaking of this mixture was carried out for 24 hours. The activated coconut shell powder was dried in an oven at 105°C for 3 hours.

2.3. GO synthesis

GO synthesis was carried out using the modified Hummers method. The synthesis process begins by weighing 1.5 g of activated coconut shell charcoal powder and 0.75 g of $NaNO_3$. Coconut shell charcoal that has been activated is added with $NaNO_3$ and then stirred. After that, add 34.5 mL of H_2SO_4 . then stirred for 20 minutes at a temperature of 0-5°C with a constant speed of 250 rpm. Next, slowly add 4.5 g of powder to keep the temperature below 20°C. This process is done carefully so that the mixture does not explode and the coconut shell charcoal powder does not reduce. After $KMnO_4$ was added to the mixture, remove the ice bath from the hot plate and stir the mixture at 35°C for 30 minutes so that the oxidation process can take place completely. This

process was carried out until the color of the solution became pale brown. Add 69 mL of distilled water slowly using a dropper and stirring for 20 minutes. The temperature is kept below 50°C when adding distilled water, in order to see the oxidation process, the mixture will turn dark brown in color accompanied by the appearance of bubbles. Add 100 mL of water followed by the addition of H₂O₂ as much as 1.5 mL. The addition of H₂O₂ is done to remove the remaining KMnO₄ or to stop the reaction and the solution finally turns yellow which indicates the presence of graphene oxide.

2.3. Characterization of GO

GO characterization used X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR), and Vector Network Analyzer (VNA) to determine the phase, functional groups, and microwave absorbing properties of GO, respectively.

Based on characterization using XRD, a graph of the relationship between 2θ and intensity will be obtained, then the data will be analyzed for lattice parameters and crystal systems using High Score Plus software. In addition, the grain size of the sample can also be determined by the Scherer formula [10]. In addition, the crystal grain size can be found by the Scherer formula,

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

where D = crystal size (nm), k = constant (~0.9), λ = Cu wavelength (0.15406 nm), β = FWHM (rad), θ = diffraction angle (°). Meanwhile, based on the characterization data using FTIR, a graph of the relationship between the transmission percentage and the wave number will be obtained, then the data obtained can be analyzed by conducting a literature review.

The VNA used is the KEYSIGHT E5071 ENA Series Network Analyzer with a frequency of 300 kHz – 20 GHz. Based on the characterization data using VNA, the Reflection Loss (RL) value of graphene oxide will be known, then the value of the electromagnetic wave absorption coefficient will be found using the equation (2) [11] in order to determine the absorption properties of microwaves.

$$R_L(dB) = 20 \log_{10} \left| \frac{Z_{in}-1}{Z_{in}+1} \right| \quad (2)$$

where Z_{in} is the normalized input impedance of layer the microwave absorption.

III. RESULTS AND DISCUSSION

The crystal structure formed by the synthesis process using XRD. The results of the structural function test as outlined in the graph using temperatures of 250°C, 300°C, 350°, 400°C and 450°C can be seen in Figure 1. Based on the Figure 1, the diffraction pattern at all temperature variation has a peak at $2\theta = 25.68^\circ$ associated with Miller index (002) (ICSD card number: 01-077-7164). This peak having a d-spacing 3.47 Å. Based on the results of the characterization using XRD, the crystal sizes were obtained from five temperature variations of 250 °C, 300 °C, 350 °C, 400 °C and 450 °C, namely 43.2 nm, 39.9 nm, 29.5 nm, 39.9 nm and 28.8 nm, respectively.

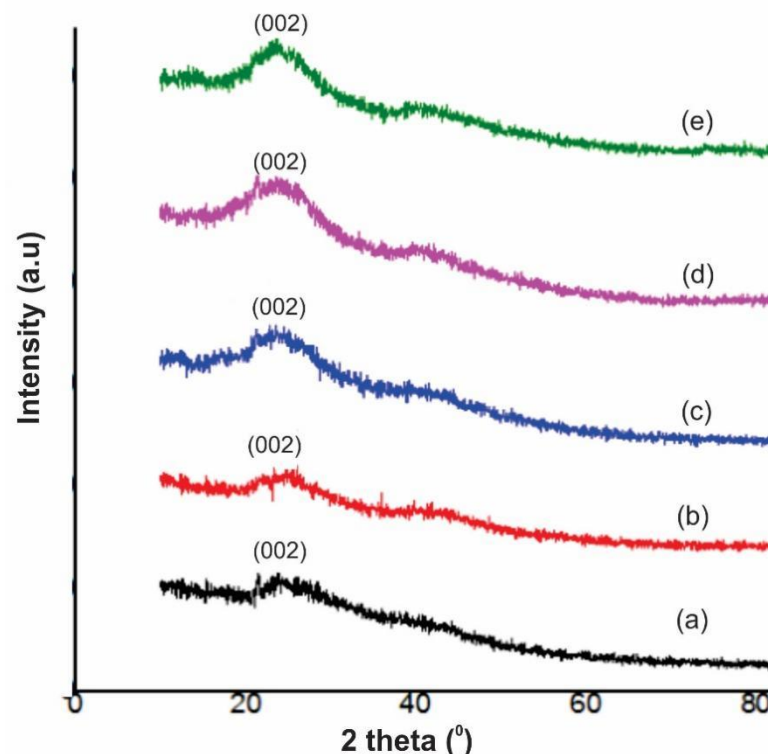


Fig 1. XRD characterization results from GO for (a) 250°C (b) 300°C (c) 350°C (d) 400°C (e) 450°C

GO testing using FTIR was carried out to determine the functional groups formed in GO. The results of the FTIR characterization are displayed on the Origin software and show the relationship between the wavenumber (cm^{-1}) on the X-axis and transmittance (%) on the Y-axis. The IR spectrum of GO can be observed in the wave numbers 600-4000 cm^{-1} , can be seen in Figure 2.

Based on Figure 2 on Tests using FTIR analysis with a wavelength range of 600-4000 cm^{-1} . The results of the FTIR functional group test are shown in a graph plot. In the picture you can see graphene oxide graph for a temperature of 250°C, the gun at a wavelength of 3358.91 shows the peak of the OH bond, the gun with a wavelength of 1702.26 cm^{-1} shows the peak of the C=O bond, the wavelength of the gun at 1605.39 cm^{-1} [7]. Fourier transform infrared (FTIR) is one of the important analytical techniques for researchers. This type of analysis can be used for characterizing samples in the forms of liquids, solutions, pastes, powders, films, fibers, and gases. This analysis is also possible for analyzing the material on the surfaces of the substrate. Fourier transform infrared spectroscopy (FTIR) has facilitated many different IR sampling techniques, including attenuated total reflection and diffuses reflectance infrared Fourier transform (DRIFT) spectroscopy. It has dramatically improved the quality of infrared spectra and minimized the time required to obtain data. The increased speed and higher ratio of signal-to-noise of FTIR relative to dispersion infrared have led to a substantially greater number of applications of infrared in natural fibers research [12].

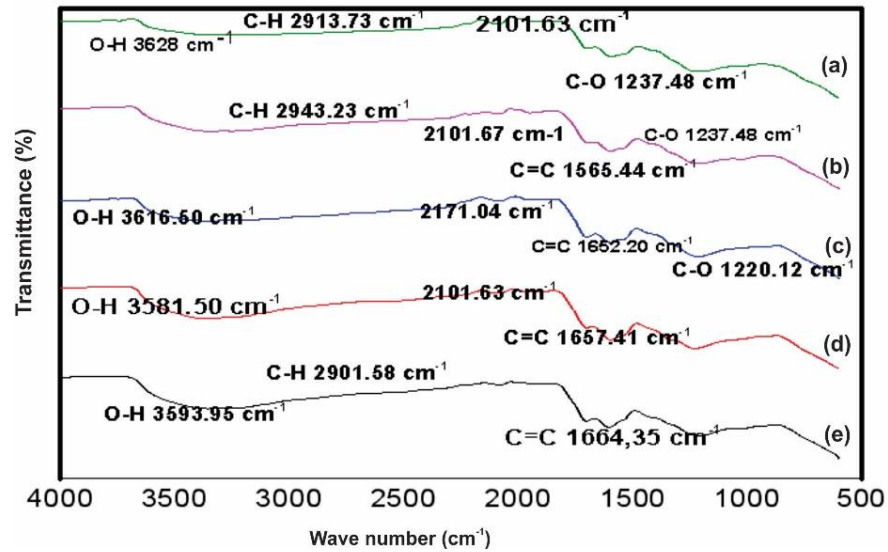


Fig 2. IR spectra of GO for (a) 250°C (b) 300°C (c) 350°C (d) 400°C (e) 450°C

GO testing using VNA was carried out to determine the absorption properties of material against microwaves. The graph of GO test results using VNA shows the relationship between Reflection Loss (RL) on the Y-axis and frequency (f) on the X-axis. The frequency values used are in the 8-12 GHz range (X-band frequency) can be seen in figure 3.

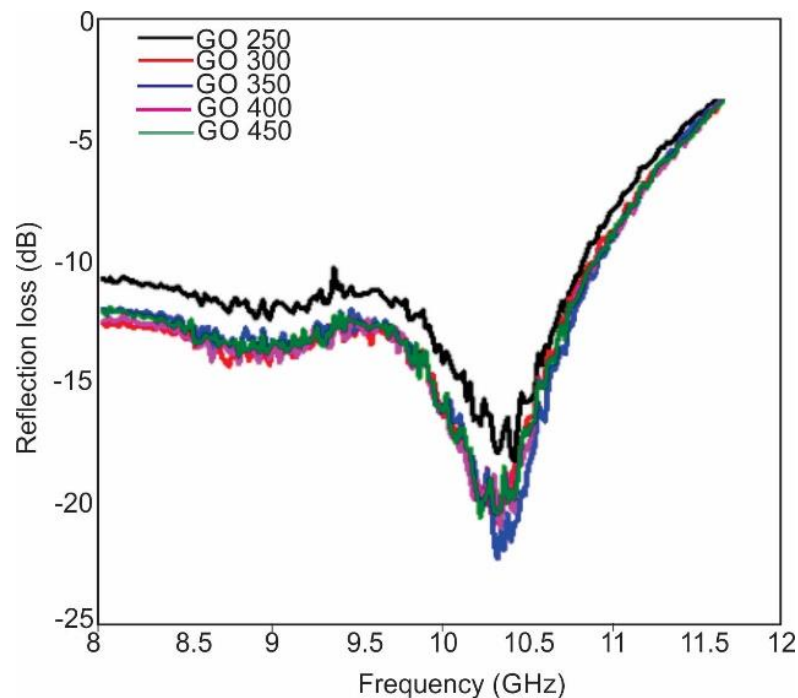


Fig 3. VNA Characterization of GO

Based on Figure 3, a good wave absorption test is shown by a deep curve valley and small reflection losses [13]. In it can be seen the value of the reflection losses of the five variations of the GO sintering temperature with a certain frequency.

The value of reflection loss is different for each variation of GO sintering temperature which indicates that the value of GO absorption for each variation is also different. The smallest reflection loss value is -24.40 dB in the GO 400 variation, the best absorption is GO 400. While the highest reflection loss value is -17.90 dB in the GO 250 variation, the highest absorption is obtained. low, namely, GO 250. Based on the obtained test, the coefficient of absorption of a GO for each variation of GO can be seen in table 1:

Table 1. The value of the GO absorption coefficient

GO	Frequency (GHz)	Reflection Loss (dB)	Absorption Coefficient (%)
250	10.20	-17.90	87.26%
300	10.30	-23.20	92.58%
350	10.30	-24.30	93.83%
400	10.40	-24.40	93.97%
450	10,40	-23.30	93.13%

Based on Table 1. it is explained that the smallest absorption coefficient value is found in the largest reflection loss value (-18.20 dB). The smallest absorption coefficient value is 87.26% in the GO 250 variation. While the largest absorption coefficient value is found in the smallest reflection loss value (-24.40 dB) in the GO 400 variation. The largest absorption coefficient value is 93.97% in the GO 400 variation. So, the relationship between reflection loss and the absorption coefficient is inversely proportional. The smaller the reflection loss value, the greater the absorption coefficient [14].

Based on the results of GO characterization using XRD, it is known that GO 350, 400, and 450 contain GO phase with a diffraction peak value of 23.967°C, Miller index value (201). while GO 250 and 300 contain GO phase with diffraction peak values of 23.821°C, and 17.956°C, Miller index values (221) and (200). The occurrence of changes in the peak position is influenced by the oxygen functional group [7] due to the oxidation process of GO 350,400 and 450 samples) and water molecules into a carbon layer structure [15].

Based on the results of GO characterization using FTIR, it was found that GO 250 has all GO functional groups [7], while for GO 400 and 450 C-O, C=C, and OH but with small absorbance values of OH. The small value of the absorbance of OH at GO 250 and 300 indicates that at GO 250 and 300, a GO reduction process occurs. The wavelength in the OH functional group region is related to the stretching and bending vibrations of the OH bond [18]. In addition, the OH GO 350,400 and 450 functional groups added absorbance values, so it can be concluded that the GO obtained was hydrophilic in line with research [7] which stated that GO was hydrophilic.

Based on the results of GO characterization using VNA, it was found that GO 400 has good microwave absorption because it has a small reflection loss value (-24.40 dB), a large absorption coefficient (93.97%) d. Reflection loss is a quantity that shows the amount of electromagnetic energy lost after hitting a material because the energy is absorbed by the material [13].

Based on the results obtained, GO 250, GO 300, GO 350, and GO 400 and 450 have reflection loss values, different absorption coefficients due to the influence of the sintering temperature [16]. This effect is indicated by a change in the value of reflection loss, absorption coefficient, and absorption bandwidth with each increase in the variation of the sintering temperature. The higher the sintering temperature, the greater the absorption coefficient and absorption bandwidth, while the smaller the reflection loss value. The GO temperature of 250 has the smallest reflection loss value, the largest absorption coefficient. A small reflection loss value will have a large absorption coefficient and a large absorption bandwidth, so GO 400 can be said to be a material that has good microwave absorption.

Based on the results of GO characterization using FTIR, it was found that GO 250 has all GO functional groups, namely CO, C=O, C=C and -OH [7], while for GO 400 and 450 CO, C= C, and OH but with a small absorbance value of OH. The small value of the absorbance of OH at GO 250 and 300 indicates that at GO 250 and 300, GO reduction occurs. The wavelength in the OH functional group area is related to the stretching and bending vibrations of the OH bond [17]. In addition, the OH GO 350,400 and 450 functional groups added absorbance values, so it can be concluded that the GO obtained was hydrophilic.

Based on the results of GO characterization using VNA, it was found that GO 400 has good microwave absorption, because it has a small reflection loss value (-24.40 dB), a large absorption coefficient (93.97%). Reflection loss is a quantity that shows the amount of electromagnetic energy that is lost after hitting a material, because the energy is absorbed by the material [13]. Graphene Oxide (GO) was synthesized from graphite and also produced from waste materials. The characterization of GO was with X-Ray diffraction (XRD) analysis showed the formation of the GO and graphite from various angles and revealed that the chemical reaction had an important in the formation of the GO particles. [18]. The use of activated charcoal as an ingredient absorbent, then the optimal conditions for make activated charcoal with quality The best of coconut wood is temperature 800°C activation and 3 hours of activation produce Parts missing on heating 950°C[18]. The results showed that the optimum activation condition to acquire activated charcoal with the best quality was at 800°C for 3 hours

duration. There is several factors that influence the absorption of activated charcoal, namely the properties of activated charcoal, the properties of the components it absorbs, the properties of solution and contact system. Absorption activated charcoal to the components in solution or gas caused by conditions surface and pore structure[19].

Based on previous studies, various methods have been developed for the synthesis of graphene as a method of peeling (scotch tape method), hydrothermal, chemically derived, and the growth substrate at CVD (Chemical Vapor Deposition). To get graphene with a pure C = C bond, it can only be done using the CVD method, which is relatively expensive and the process is difficult with equipment that is not easily accessible[20]. That it is possible to synthesis graphene oxide using coconut husk as a raw material. FT-IR spectrum confirmed that the GO had some oxygen functional groups. Graphene oxide is an important carbon-based two-dimensional nanomaterial. coconut husks is considered as an agro waste and evenly burned [21].

Based on the results obtained, GO 250, GO 300, GO 350, GO 400 and GO 450 have different reflection loss values, absorption coefficients. This is due to the influence of the sintering temperature [13]. This effect is indicated by a change in the value of reflection loss, absorption coefficient, at each increase in the variation of the sintering temperature. The higher the combustion temperature, the greater the absorption coefficient, while the smaller the reflection loss value. Sintering is the process of compaction of a collection of powders at high temperatures. It aims to compact the material that has been printed at high temperatures [12]. The sintering process will affect the formation of the crystal phase of the material and the crystal growth structure. The higher the sintering temperature, the faster the crystal formation process. GO temperature 250 has the smallest reflection loss value, the largest absorption coefficient. A small reflection loss value will have a large absorption coefficient, so GO 400 can be said to be a material that has good microwave absorption. However, in this study, the particle size factor did not have a significant effect. The oxidized form of graphene oxide hydrophilic in nature and it can be easily dispersed in water [21].

IV. CONCLUSION

Based on these results, it can be concluded that GO can be synthesized from Old Coconut Shell waste using the modified Hummers method. Through the results of the FTIR test, GO synthesized from the waste of Old Coconut Shell contains the functional groups C-O, C=O, C=C, and -OH, and through the results of the XRD test, the GO synthesized from the coconut shell waste has a GO phase. Variations in GO sintering temperature (250 °C, 300 °C, 350 °C, 400 °C, and 450 °C) affect the absorption properties of microwaves. GO which has the best microwave absorbing properties is GO 400 because it has a small reflection loss value (-24.40 dB), a large absorption coefficient (93.79%).

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