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STRUCTURE ANALYSIS OF GRAPHENE MICRO OXIDE FROM OLD COCONUT SHELL WASTE

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ABSTRACT

Synthesis of graphene oxide from old coconut shell waste material. It has many applications, one of which is used as a microwave absorber. Old coconut shells are used as waste because the ingredients are easy to get. This study aims to analyze the microstructure of graphene oxide from old coconut shell waste. In the carbonization process using a furnace with temperature variations from 250˚C to 450˚C, the furnace time is 120 minutes. Making activated charcoal from old coconut shells using the modified hummers method with oxidizing agents KmnO2, H2SO4, and NaNO3. Characterization of graphene oxide from old coconut shell waste using FTIR, XRD and SEM. The test results using FTIR on GO samples show the formation of GO material where the sample contains functional groups containing bonds between carbon (C), Hydrogen (H) and Oxygen (O), the highest crystal size on XRD is 43.20800529 nm, and particle size the best SEM was obtained at GO 250˚C with a particle size of 65.701671 nm where the larger the particle size, the larger the surface pores of the sample, so that the better microwave absorbent was produced.

Keywords : GO; Old Coconut Shell; Functional Group; Crystal Size; Microstructure.

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I. INTRODUCTION

Graphene is a single layer of sp2 hybridized carbon atoms with a hexagonal lattice [1] . Graphene oxide has attracted a lot of attention because of its good thermal, mechanical, and electrical properties. Graphene oxide is currently widely used as an intermediate in the manufacture of graphene [2]. Graphene has a 2-dimensional structure with sp2 so it forms a very good hexagonal structure for thermal, electrical, and mechanical properties [3]. Graphene has a higher thermal conductivity than copper but has a lower material weight. The electrical conductivity of graphene is the same as that of copper. In addition, graphene has a very high surface area of 2,500m2/g so graphene is used for energy storage for supercapacitor, solar cell, and battery applications [4]. Reduced graphene oxide can be used to reduce oxygen atoms by using stirring and sonification methods [5].

In processing, the coconut flesh is taken to be processed into a finished product, while the coconut shell is considered as residual waste. In the coconut shell processing industry, it is generally discarded immediately. Coconut shell is organic waste, but this waste is decomposed by microorganisms due to its hard nature. Coconut shell waste can be reprocessed into charcoal and briquettes as alternative energy for daily activities. Old coconut shell has the main compounds, namely cellulose, lignin, and hemicellulose, and has atomic compounds with functional groups such as hydroxyl, alkane, kaboxy, carbonyl, and ester. The largest elements in the old coconut shell compound are carbon and oxygen [6].

The development of microstructure and the formation of pores in charcoal or biomass is determined from carbonization or heat treatment such as temperatures between 300-1400C causing the growth of crystallite carbon and the regularity of the microstructure in charcoal [7]. Using the heating temperature and reaction time (holding time) in the carbon material process to determine the relationship between the microstructure, chemical composition, and physical properties of the resulting material [8]

The manufacture of graphene uses several methods, namely chemical vapor decomposition and micromechanical exfoliation, but these two methods are very expensive. Therefore, using the modified hummers method by oxidizing the graphite by reacting graphite with KmnO4, NaNO3 in a sulfuric acid solution. The hummer method is better to use than the previous method because during the oxidation process it does not emit CIO2 gas. In addition, the oxide process proceeds rapidly at lower temperatures and uses materials that are more

readily available and less hazardous. [9] Experiments with graphene have attracted more interest in carbon materials. The use of graphene as new material has many advantages in its excellent mechanical, electrical, thermal, and magnetic properties [10].

In this research, the author wants to make graphene oxide to analyze the microstructure properties. Preparation of graphene oxide by varying the temperature from 250˚C to 450˚C. It is hoped that each temperature increase will affect the microstructure of the graphene oxide and can also obtain the optimum conditions. Graphene oxide applications include the manufacture of electrodes, supercapacitors, and lithium-ion batteries [11] to absorb contaminants, energy storage applications, and radio frequency electronics.

The resulting GO will be characterized by using FTIR to see the functional groups possessed by the sample. where this FTIR data will later support SEM data to analyze the microstructure properties of graphene oxide. It is important to analyze the microstructure because the microstructure of the particle size affects the absorption of microwaves. XRD was used to see the crystal structure formed, and SEM was used to observe the surface morphology of the sample and its particle size. Based on the description above, the researcher wants to do research using the hummer method with the title "Graphene Micro-Structure Analysis of Old Coconut Shell Waste"

II. METHOD

The first sample preparation will be cleaned on the old coconut shell from the coir until only the old coconut shell remains. Then the old coconut shell is dried for 3 days in the sun, after 3 days of drying the old coconut shell is cut into small pieces. Furthermore, remove the water content in the old coconut shell in the oven by using a temperature of 100˚C for 1 hour. After the old coconut shell is finished in the oven, then the Furnace process is carried out for 2 hours with a temperature variation of 250˚C to 450˚C. After the Old Coconut Shell has become charcoal, it is pounded with a mortar or pestle to produce charcoal powder and sieved using a 125-mesh size sieve.

After the old coconut shell becomes powder, the next step is to activate the carbon contained in the old coconut shell by mixing the old coconut shell powder with NaOH. First, the synthesis of NaOH solution with a temperature concentration of 250˚C to 450˚C was carried out in a fume hood. The solid NaOH will be dissolved using 100 mL of distilled water which has been measured using a measuring flask. Next, add 8 g of solid NaOH into the volumetric flask using a spatula, until the solid NaOH dissolves and the solution becomes homogeneous. Next, provide a 250 mL beaker, put 8 grams of coconut shell charcoal powder and add 100 mL of NaOH solution into a beaker that has been filled with old coconut shell charcoal powder so that the charcoal powder is submerged in the NaOH solution. Immersion of this mixture was carried out for 24 hours [12]. After the mixture was submerged, a precipitate formed at the bottom of the beaker. This precipitate is then filtered using ordinary filter paper and using a Buchner funnel to facilitate the filtering process. The filtering process is carried out by cutting filter paper and then placing it on a Buchner funnel. Next, wet the filter paper using distilled water. Then, strain the coconut shell mixture and the NaOH solution until the old coconut shell powder is completely separated from the liquid. After the old coconut shell powder is filtered, transfer the old coconut shell powder into a steamer cup. Then, the activated coconut shell powder was dried using an oven at a temperature of 105°C for 180 minutes [13].

GO synthesis was carried out using the modified Hummers method. The first stage of the synthesis process was to weigh 1.5 g of coconut shell charcoal powder with activated carbon and 0.75 g of NaNO 3. Next, put a 250 mL Erlenmeyer that has a magnetic stirrer inserted in it and add coconut shell and weighed NaNO 3 The second step is to add 34.5 mL of H 2 SO 4 98% into the Erlenmeyer earlier. The mixture was then stirred for 20 minutes at a temperature of 0-5°C with a constant speed of 250 rpm and the solution will turn black because of the carbon content from the old coconut shell charcoal. [14] Next, put the Erlenmeyer in an ice bath and stir for 2 hours on a hot plate. Then, add 4.5 g of KmnO 4 powder slowly to keep the temperature below 20°C. This process is carried out carefully so that the mixture does not explode and the synthesized coconut shell powder does not decrease. After adding KmnO 4 to the mixture, remove the ice bath from the hot plate and stir the mixture at 35°C for 30 minutes [15] so that the oxidation process can take place completely. This process is carried out until the color of the solution becomes pale brown. Use a thermometer to check whether the temperature of the mixture has reached 35°C or not. Then, add 69 mL of distilled water slowly using a dropper and stirring for 20 minutes. In this process, the temperature of the solution will increase because when adding distilled water an endothermic reaction occurs, which causes the temperature of the mixture to rise. At the time of adding this distilled water, the temperature was kept below 50°C to see the oxidation process, the mixture will turn dark brown with bubbles appearing. Add 100 mL of water followed by the addition of 1.5 mL of 30% H 2 O 2 . The addition of H 2 O 2 was carried out to remove the residue from KMnO 4 or to stop the reaction and the solution finally turned yellow which indicated the presence of graphene oxide [16]. The last step is to add 50 mL of distilled water to the mixture and form GO [17].

After the solution changed to yellow which indicated the presence of graphene oxide, the mixture was sonicated for 2 hours to exfoliate the graphite into graphene [18]. Then, the solution was precipitated for 1 day until liquid and solid phases were formed [19]. Then, the centrifugation process was carried out using a microcentrifuge by setting a speed of 4000 rpm for 15 minutes to separate the solid and liquid phases. The centrifugation process was followed by a manual GO neutralization process, namely by precipitating the GO powder and then changing the distilled water repeatedly until a neutral pH was obtained. After obtaining a neutral pH, the GO was baked at a temperature of 60°C for 12 hours [20].

III. RESULTS AND DISCUSSION

In the test to see the functional groups formed during the synthesis process using FTIR. Tests using FTIR with a wavelength of 600-4000 cm-1. The results of the functional group FTIR test are contained in a graph plot using a temperature of 250˚C to 450˚C can be seen in Figure 1:

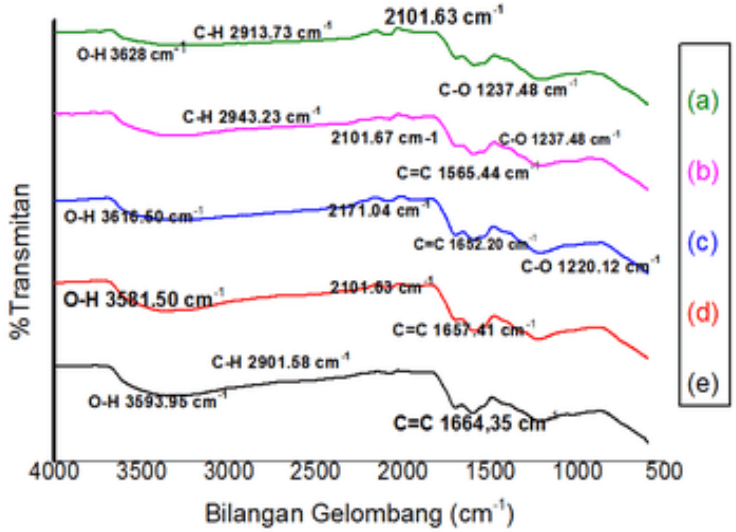
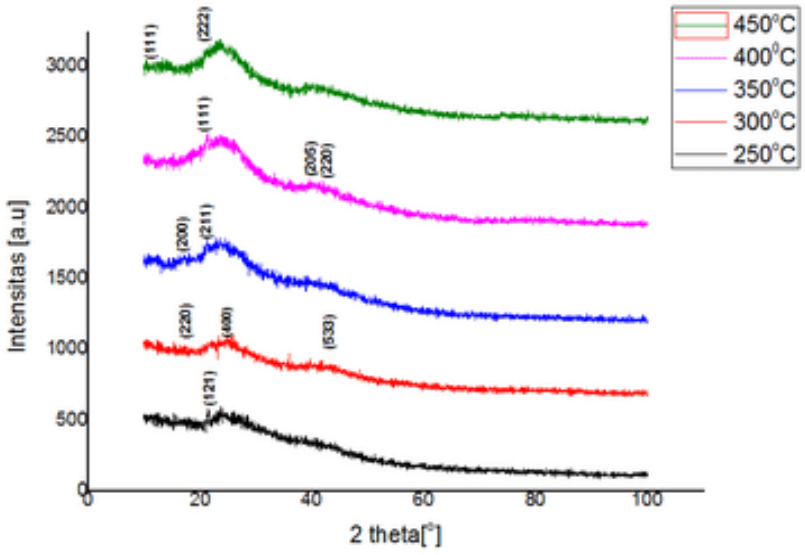


Fig 1. FTIR Test Result Data Processing (a) GO 250˚C, (b) GO 300˚C, (c) GO 350˚C, (d) GO 400˚C and (e) GO 450˚C

Based on Figure 1, graphene oxide FTIR test from five temperature variations using a furnace, we can see the presence of OH, CO, and CH compounds, this indicates that graphene oxide has formed where graphene oxide contains carbon (C), hydrogen (H) bonds, and graphene oxide. Oxygen (O).

The results of graphene oxide analysis using XRD with five temperature variations, namely 250˚C to 450˚C using origin software can be seen in Figure 2:



a

Fig 2. XRD GO test results 250˚C, 300˚C, 350˚C, 400˚C and 450˚C

Figure 2 shows the diffraction pattern of graphene oxide at temperatures of 250˚C, 300˚C, 350˚C, 400˚C and 450˚C using origin software. At a temperature of 250˚C, it can be seen that the deposition results from a peak with an angle of 21.379˚C with a peak Millers' index related to the phase (121). Based on the results of the diffractogram pattern, it can be used to determine the average crystal size using the Scherer equation obtained from the peak value of FWHM. The crystal size calculated by the Scherer equation is 43.20800529 nm.

At a temperature of 300˚C, it can be seen that the deposition results formed as many as three peaks with angles of 17.577˚C, 24,957˚C and 41.492˚C with the peak Millers index related to the phase (220), (400) and (533). Based on the results of the diffractogram pattern, it can be used to determine the average crystal size using the Scherer equation obtained from the peak value of FWHM. The crystal size calculated by the Scherer equation is 39.94232897 nm.

At a temperature of 350˚C, it can be seen that the deposition results formed as many as two peaks with an angle of 17.956˚C and 21.203˚C with a peak Millers' index related to the phases (200) and (211). Based on the results of the diffractogram pattern, it can be used to determine the average crystal size using the Scherer equation obtained from the peak value of FWHM. The crystal size calculated by the Scherer equation 29.53680616 nm

At a temperature of 400˚C, it can be seen that the deposition results formed as many as three peaks with angles of 21.552˚C, 39.492˚C and 40.227˚C with the peak Millers index related to the phase (111), (205) and (220). Based on the results of the diffractogram pattern, it can be used to determine the average crystal size using the Scherer equation obtained from the peak value of FWHM. The crystal size calculated by the Scherer equation is 39.90178875 nm.

At a temperature of 450˚C, it can be seen that the deposition results formed as many as two peaks with an angle of 10.905˚C, and 21.911˚C with a peak Millers' index related to the phases (111) and (222). Based on the results of the diffractogram pattern, it can be used to determine the average crystal size using the Scherer equation obtained from the peak value of FWHM. The crystal size calculated by the Scherer equation is 28.78889168 nm.

Based on the results of GO using XRD where the XRD spectrum has a relationship between the scattering angle (2θ) and the intensity (I). It shows that when the number of counts of an intensity hits a compound, the atoms cause the diffraction count to be received by the detector, which is even greater so that the intensity has a sharp and sharp peak.

The results of SEM processing through Image-J digital processing software at a temperature of 250˚C can be seen in Figure 3:

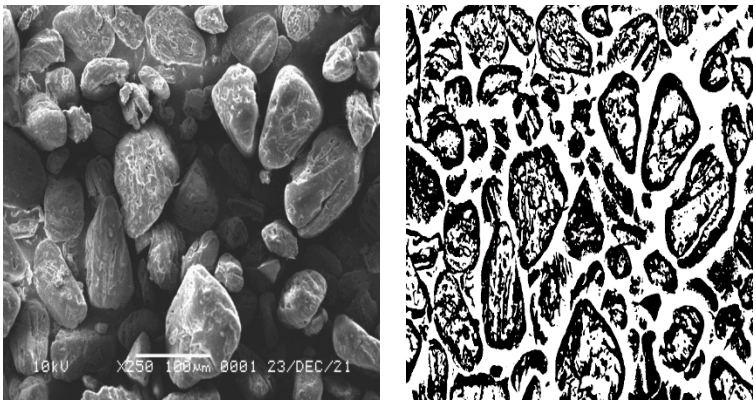


Fig 3. SEM GO 250˚C Test Results 250x Magnification

Based on figure 3 SEM characterization analysis graphene oxide material has a layered shape and looks thick. The thickness of the graphene oxide morphology is due to the presence of functional oxygen groups that affect it, so to find out the graphene oxide in the image using the image j application with data process using Origin software. Particle analysis using Image-J for particle size analysis by counting all parts of the image, resulting in an average particle diameter for GO 250˚ with 250x magnification of 65.701671 nm and at the origin the COD results are 0.99852 and the picture also shows shaped like a lump indicating the presence of graphene oxide and shaped like a particle.

The results of SEM processing through Image-J digital processing software at a temperature of 300˚C can be seen in Figure 4 :

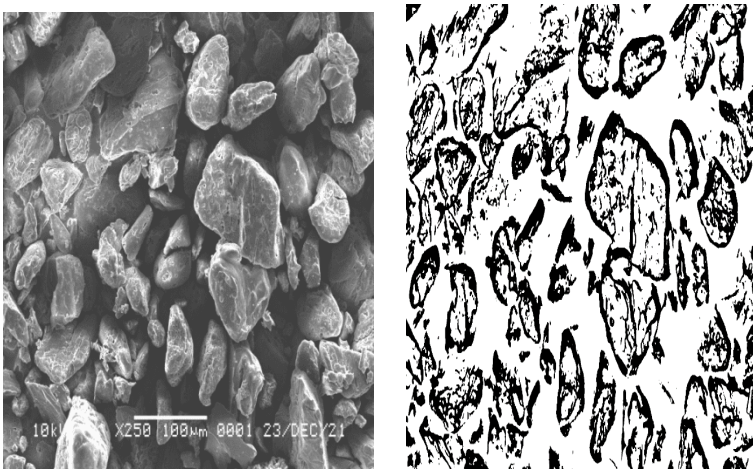


Fig 4. SEM GO 300˚C Test Result 250x Magnification

Based on Figure 4, analyzing the SEM characterization of graphene oxide material has a layered shape and looks thick. The thickness of the graphene oxide morphology is due to the presence of functional oxygen groups that affect it, so to find out the graphene oxide in the image using the image j application with data processing using Origin software. Particle analysis using Image-J for particle size analysis by counting all parts of the image after calculating the image j application, the data will come out entirely, resulting in an average particle diameter at GO 300˚ with 250x magnification of 47.924632 nm and at the origin the COD result is 0.99937, this proves that the data is accurate and the picture also looks like a lump which indicates the presence of graphene oxide and is shaped like a particle.

The results of SEM processing through Image-J digital processing software at a temperature of 350˚C can be seen in Figure 5:

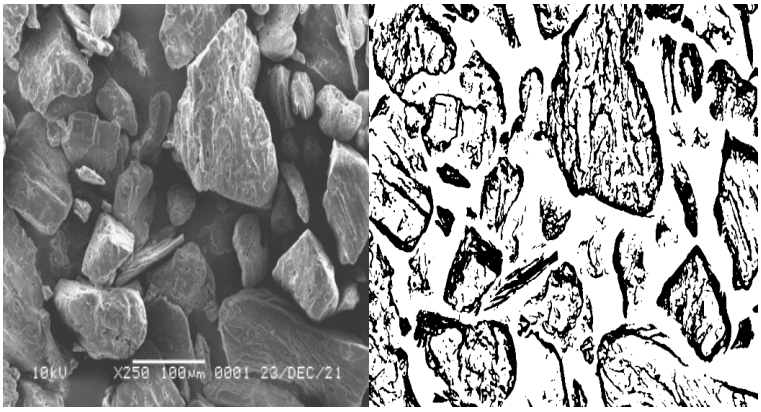


Fig 5. SEM GO 350˚C Test Result 250x Magnification

Based on Figure 5, the SEM characterization uses the image j application which in the image j application processing as a whole, after the image j application processes the entire data, the data will come out, the data will be processed using the origin application, then the particle size is obtained at a temperature of 350˚ with a speed of 250x of 32.579529 and the COD data of COD was 0.99886 and, in the image, it is shaped like a lump, this indicates the presence of graphene oxide.

The results of SEM processing through Image-J digital processing software at a temperature of 400˚C can be seen in Figure 6:

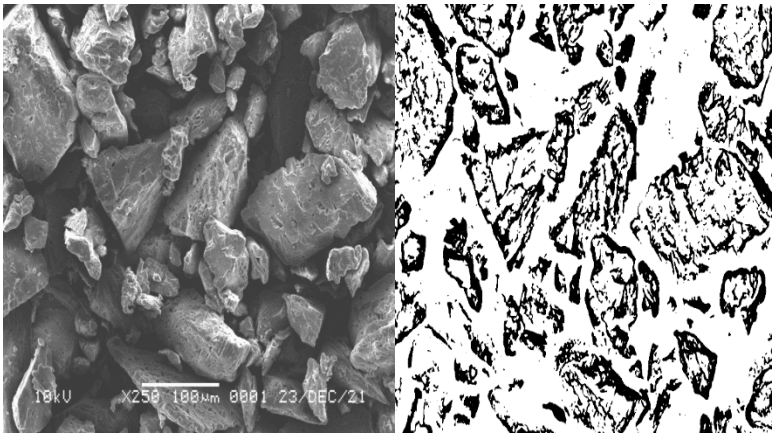


Fig 6. SEM GO 400˚C Test Result 250x Magnification

Based on Figure 6, the SEM characterization uses the image j application which in the image j application processing as a whole, after the image j application processes the whole data, the data will come out, the data will be processed using the origin application, then the particle size is obtained at a temperature of 400˚ with a speed of 250x of 39.796459 nm which in previous studies showed the presence of graphene oxide because it has a particle size range of 32 nm to 84 nm and COD data of COD was 0.99886. This also proves that the data obtained is correct, and the image is in the form of lumps then this indicates the presence of graphene oxide.

The results of SEM processing through Image-J digital processing software at a temperature of 400˚C can be seen in Figure 7:

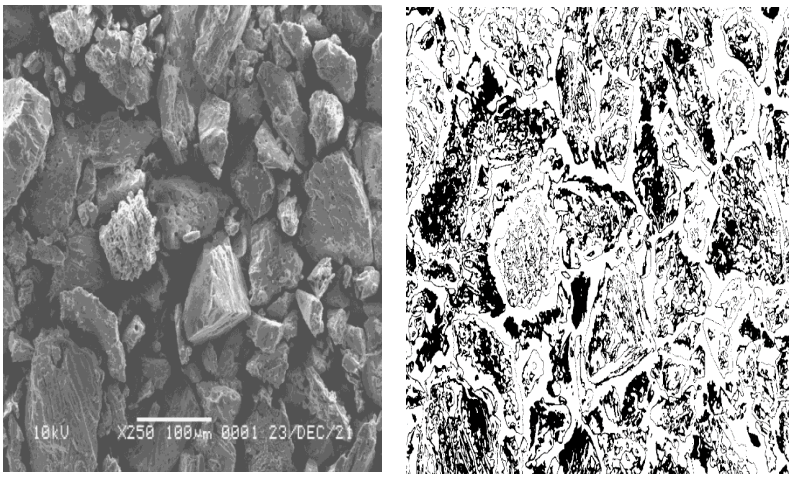


Fig 7. SEM GO 450˚C The Result 250x Magnification

Based on Figure 7, the SEM characterization uses the image j application which in the image j application processing as a whole, after the image j application processes the entire data, the data will come out, the data will be processed using the origin application, then the particle size is obtained at a temperature of 450˚ with a speed of 250x of 35.233888 nm, which in previous studies shows the presence of graphene oxide because it has a particle size range of 32 nm to 84 nm and COD data of COD was 0.99886. This also proves that the data obtained is correct, and the image is in the form of such as lumps as well as sheets then this indicates the presence of Graphene oxide and chemical peeling.

Based on the SEM analysis of the average particle diameter and surface morphology of five variations of temperature with different particle diameter sizes, the best results were found in one temperature variation. The best Graphene oxide material is at a temperature of 250˚C where at that temperature the particle size is greater than the four temperature variations, namely 65.701671 nm. This has succeeded in showing the formation of GO particles which are located in the size of 32 nm to 84 nm [21]. These results also showed the presence of nano-particles in the size of 1 nm to 100 nm. Because the larger the particle size, the larger the surface pores of the sample, so the better microwave absorbers are produced [22]. GO 250˚C has a structure that tends to be like chunks. This is in accordance with research [23] which states that the morphological structure of GO is like

chunks. When the pores and particles become large based on the morphology of the particles obtained, this shows that the particles formed are graphene. Previous research has shown that the lump-like shape has thin particles and the presence of an inhomogeneous clumping part that stacks irregularly [24]. The shape of GO is shaped like chunks and sheets because at each temperature variation there is chemical peeling [25]. This is evident at GO 250˚C which has the largest and increasing particles than the other four temperatures and has a structure that tends to be like lumps.

IV. CONCLUSION

Based on the research that has been done regarding the analysis of the microstructure of graphene oxide from old coconut shell waste using the modified hummers method, it can be concluded that graphene oxide in old coconut shell waste in FTIR contains bonds between Carbon(C), Hydrogen(H), and Oxygen(O), With the best results obtained at GO 250˚C where the sample has a crystal size of 43.20800529 nm and a particle size of 65.701671 nm where the larger the particle size causes the surface pores of the sample to also be larger so that the microwave absorber is better. produced and has a structure that tends to be like chunks.

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