



THE EFFECT OF THE ADDITION OF SEAWEED CHARCOAL (*Sargasum sp.*) WITH PURE GRAPHITE ON THE ABSORPTIVE PROPERTIES OF GRAPHENE OXIDE SYNTHESSES USING THE HUMMER MODIFICATION METHOD

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ABSTRACT

Graphene Oxide or Graphene Oxide (GO) Is a graphite oxide in the form of a monolayer obtained from exfoliating graphite oxide into sheets through a sonication or stirring process. In this study, the material used was a mixture of pure graphite and seaweed (*Sargasum sp.*). Seaweed (*Sargasum sp.*) is used as a mixture of pure graphite because the carbon content in seaweed (*Sargasum sp.*) is good enough to reduce the use of pure graphite as the main ingredient in making graphene oxide. There are 4 variations of composition with the aim of seeing the best results from these five compositions, namely the composition between Graphite and *Sargasum sp.*, namely 70%: 30%, 60% ; 40%, and 50% : 50%. The research results obtained showed that graphene oxide had been successfully synthesized from the addition of seaweed charcoal because of some of the typical properties of graphene oxide. By X-ray diffractogram at an angle of 2, it was found to contain C-O, C=O, C=C, and O-H functional groups. In the VNA test, it obtained high reflection loss and adsorption coefficient values in a mixture of 50% pure graphite: 50% seaweed with a reflection loss value of -7.40 dB and an adsorption coefficient of 0.57342.

Keywords : Graphene Oxide, Pure Graphite, Seaweed (*Sargasum sp.*), Modified Hummer Method, Microwave Absorbent Properties.



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I. INTRODUCTION

Lately, the development of technology in the world has grown rapidly. The more technology develops, the more people's needs regarding this microwave-absorbing technology will certainly increase. The more technology develops, the more people's needs regarding this microwave-absorbing technology will certainly increase. Initially, microwave technology was used for telecommunications and radar needs during the Second World War. Over time, this technology has the potential to become an efficient heating device, and Japan is developing it for a food heater that is more flexible and can reach microwave heating speeds for research, information technology, medical devices, industry, polymer synthesis, and organic synthesis [1]. Microwave-absorbing materials have an important role in the development of anti-radar technology, anti-electromagnetic interference, and wireless communication [2].

Microwaves are electromagnetic waves with associated electric and magnetic fields. One of the factors that influences the interaction of microwaves with materials is the dielectric property, whose magnitude can be shown through the permittivity value [3]. Microwave frequencies are between 300 MHz and 300 GHz and have wavelengths of 1–300 mm.

Graphite is a variety of minerals formed from the element carbon, which is very important in the industrial world because it has many uses, including several new and developing technologies such as lithium-ion batteries, nuclear power, solar power, semiconductors, or even graphene [4].

Indonesia is known as a country with abundant natural wealth, including marine products, plantation products, agricultural products, mining products, and other natural wealth products. There are various abundant marine products in Indonesia, including sea cucumbers, pearls, and seaweed. Since 2007-2010, seaweed (sea weeds) in Indonesia has increased to reach 57% [5]. Based on data from the Ministry of Maritime Affairs and Fisheries in 2007, seaweed production in Indonesia reached 1.7 million tons and increased to 3.9 million tons in 2010. By 2019, national seaweed production had reached 9.9 million tons.

Seaweed, better known as seaweed, is one of the most abundant biological resources in Indonesian waters. The diversity of seaweed in Indonesia is the largest compared to other countries. However, the use of seaweed in Indonesia, especially for industrial purposes, is still not optimal [6]. Carbon absorption in macroalgae is necessary for growth and development as well as for their ability to bind carbon through a process of photosynthesis. This process has the potential to reduce global warming [7]. *Sargassum* sp macroalgae is the macroalgae that absorbs the most carbon in Karang Papak Garut Beach [8]. Other research states that *Sargassum* sp. has a carbon content ranging from $29.5\% \pm 1.32$, while in the substrate it is 1.98% to 0.90, and the estimated average value of stored carbon is 4.6 kg/m^2 [9].

The high availability of *Sargassum* sp. in the Bungus waters of Kabung Bay and the use of resources that have not been maximized mean that *Sargassum* sp., which has the ability to absorb carbon in a large amount of biomass, can be used as a mixed material in the production of graphene oxide, which reduces the role of graphite as the main source of carbon.

Graphene is a nanomaterial in a single atomic, two-dimensional form consisting of a single layer of graphite. Graphene is considered the world's thinnest material; it is composed of sp^2 -bonded sheets of carbon atoms in a 2D hexagonal lattice. Compared to steel, graphene is very thin and stronger because the covalent bonds between carbons are strong enough to make it difficult to stretch [10]. Graphene can be synthesized by processing graphite into a monolith through various processes to obtain graphite oxide. Graphene oxide (GO) can be synthesized by various methods, such as mechanical exfoliation, reduction of graphene oxide (Hummers), liquid dispersion (Hanns Peter Boehm), and epitaxy (Claire Berger). The Hummers method usually uses a combination of potassium permanganate and sulfuric acid as the oxidant.

Graphene oxide, or Graphene Oxide (GO), is a monolayer form of graphite oxide [11], obtained from the exfoliation process of graphite oxide into sheets through the sonication or stirring process [12]. GO has unique characteristics and good optical, electronic, and mechanical properties [12], so it can be applied in various fields, including as sensors, polymer nanocomposites, energy-safe equipment, electronics, and the adsorption of pollutant substances [13]. GO, then the exfoliation process from graphite must use a strong oxidation reaction using carbon material [14]. The process of exfoliating graphite into graphene can be done physically or chemically [15].

The method to be used is the "Modified Hummer" method. One of the advantages of the Modified Hummer method is that the reaction process does not take a long time, and the reaction process is very safe because it uses $KMnO_4$, which does not produce explosive materials (explosives), such as ClO_2 , which is produced from $KClO_3$, and uses $NaNO_3$ instead of HNO_3 , which can produce acid mist [15].

In this study, microwave absorbing properties are the properties to be analyzed. *Sargassum* sp. has the ability to absorb carbon in a large amount of biomass, so it can be used as a mixed material in the production of graphene oxide which reduces the role of graphite as the main source, and graphene oxide carbon has good optical, mechanical and electronic properties, so it can be applied in various fields, one of which is the field of electronics as a material for making electronic components. Therefore, this research will be carried out with the title "Effect of the addition of Seaweed Charcoal (*Sargassum* sp.) with Pure Graphite on the Microwave Absorbing Properties of Graphene Oxide synthesized using the Modified Hummer Method".

II. METHOD

Samples of seaweed (*Sargassum* sp.) were taken from the shallow waters of Teluk Kabung, Bungus District, Padang City. The method that will be used for this research is the modified hummer method. One of the advantages of the Hummers method is that the reaction process does not take a long time or only a few hours, the reaction process is very safe because it uses $KMnO_4$ and does not produce explosives (dynamite), such as ClO_2

produced from $KClO_3$. In previous studies, using $NaNO_3$ instead of HNO_3 produced acid mist [15]. Samples were washed and dried in the sun for 1 day, then dried in an oven at $105^\circ C$ for 1 hour for the dehydration process. The roasted seaweed (*Sargassum sp*) was then oven-treated at $300^\circ C$ for 30 minutes to turn the seaweed (*Sargassum sp*) into seaweed charcoal (*Sargassum sp*) charcoal, which was then ground into charcoal powder with a mortar and pestle and sieved through a 170 mesh sieve.

In this study, carbon activation was carried out by chemical activation using KOH solution. KOH activation improves the performance of biocarbons due to improved physico-chemical properties (surface area, microporosity, pore volume, and damaged sites). Therefore, it was found that chemical activation is required for better performance [16]. After the activation process, the solution was washed with distilled water and dried in an oven at $105^\circ C$ for 3 hours. then it can be continued at the graphene oxide synthesis stage using a modified Hummer method.

GO synthesis was carried out using a modified Hummer method, in which in the first stage 1.5 g of activated carbon in the form of pure graphite and seaweed (*Sargassum sp*) were weighed according to the variation in composition and 0.75 g of $NaNO_3$. Then plug it into the Erlenmeyer, which already has a bar magnet. After that, 34.5 mL of a 98% sulfuric acid solution (H_2SO_4) was added, followed by stirring at a constant speed of 250 rpm for 20 minutes, until the solution turned black due to the sawing of used charcoal. Place the Erlenmeyer in an ice bath and continue stirring on the hot plate for 2 hours. Next, slowly add 4.5 grams of $KMnO_4$ powder, keeping the temperature below $20^\circ C$. This process must be done carefully so that the mixture does not explode and the synthetic carbohydrates are not reduced. After adding $KMnO_4$ to the mixture, remove the ice bath from the hot plate and stir the mixture at $35^\circ C$ for 30 minutes. In order for the oxidation process to take place perfectly, this process is carried out until the solution changes color to a pale brown. A thermometer is used to determine if the temperature of the mixture reaches $35^\circ C$. Then add 68 mL of distilled water slowly, using a pipette, and stir for 20 minutes. During this process, the temperature of the solution will increase because an endothermic reaction occurs when distilled water is added, causing the temperature of the mixture to increase. When adding distilled water, the temperature must be kept below $50^\circ C$ so that the oxidation process can be seen as the mixture turns dark brown with the appearance of air bubbles. Then 100 mL of water was added, followed by 1.5 mL of 30% H_2O_2 . This aims to stop the reaction and remove the remaining $KMnO_4$ once the color of the solution turns yellow, indicating the presence of GO, the next step is to add 50 mL of distilled water to the mixture [17].

After observing the yellow color change in the solution, the synthesis process was continued with a GO dispersion process with sonication for 2 hours to exfoliate GO into thin layers. Sonication was carried out using an ultrasonic bath at room temperature for 2 hours, then the solution was precipitated for 24 hours to form solid and liquid phases. Furthermore, the centrifugation process was carried out at a speed of 2500 rpm for 15 minutes using microcentrifugation. Then, the GO was manually neutralized by precipitating the mixture to form solid and liquid phases. The liquid phase was then replaced repeatedly with distilled water until a neutral pH of 7 was reached. After obtaining a neutral pH, GO was filtered and separated from the liquid phase, then dried in an oven at $60^\circ C$ for 12 hours.

X-ray diffraction (XRD) was used to determine the crystal structure of graphene oxide samples. Fourier transform infrared (FTIR) is used to determine the functional groups present in graphene oxide. The Vector Network Analyzer (VNA) is used to determine the microwave absorption properties of graphene oxide, one of which is the *Reflection Loss* (RL) value.

III. RESULTS AND DISCUSSION

Testing of graphene oxide using XRD was carried out to identify the phases that appeared and determine the structure, system, and size of the crystals. A plot of the values of the diffraction patterns of graphene oxide obtained using the Origin application while the characterization results from XRD were processed using the High Score Plus application. The data obtained from the results of the characterization using XRD were compared with the data contained in the ICDD database. The results of graphene oxide analysis data using XRD with 4 composition variations, namely 100% graphite, 70%: 30%, 60%: 40%, and 50%: 50%, can be seen in Figure 1 below.

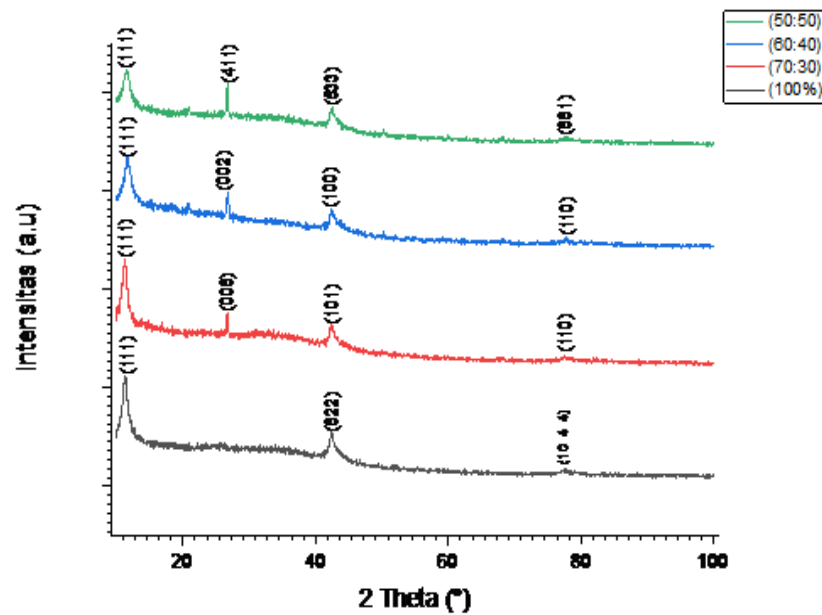


Fig 1. XRD characterization results data with a composition of 100% graphite, 70% : 30%, 60% : 40% and 50% : 50%.

Based on Figure 1, it shows the diffraction pattern of graphene oxide from a composition of 100% graphite, 70% : 30%, 60% : 40%, and 50% : 50% using Origin software. In the composition of 100% graphite, it can be seen that the deposition results in three peaks with an angle of 10.819°, 42.329°, and 77.415°, with peak Miller indices related to phases (111), (622), and (1044). Average lattice parameters on ICDD code 01-082-2261 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 the FWHM. The average size of the crystals calculated by the Scherer equation is 32.56 nm.

At a composition of 70% : 30%, it can be seen that the deposition results formed as many as four peaks with an angle of 10.905°, 26.69°, 44.23°, and 77.7741°, the peak Miller's index related to phases (111), (006), (10(101), and (110). The average lattice parameters on ICDD code 00-026-1076 are as follows: 10.905°, 26.6926°, 42.3316° and 77.4171° with the peak Miller's index related to phases (111), (006), (101) and (110). The average lattice parameters on ICDD code 00-026-1076. 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 the FWHM. The average size of the crystals calculated by the Scherer equation is 31.48 nm.

At a composition of 60% : 40%, it can be seen that the deposition results formed four peaks with an angle of 11.1208°, 26.7632°, 42.3739°, and 77.6805°, with the peak millers index related to phases (111), (002), (100), and (110). Average lattice parameters on ICDD code 01-071-3739. 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 the FWHM. The average size of the crystals calculated by the Scherer equation is 30.79 nm.

In the 50% : 50% composition it can be seen that the deposition results formed four peaks with an angle of 10.819°, 26.6982°, 42.3924° and 77.089° with peak millers indices related to phases (111), (411), (533) and (881). Average lattice parameters on ICDD code 01-079-1715 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 the FWHM. The average size of the crystals calculated by the Scherer equation is 29.93 nm.

Graphene Oxide Testing using FTIR was carried out to determine the functional groups formed in Graphene Oxide. FTIR characterization results are displayed in the Origin software and display the relationship between wave number (in cm-1) on the X axis and transmittance (%) on the Y axis. The IR spectrum of Graphene Oxide can be observed at wave numbers 500-4000 cm-1 as shown in Figure 2.

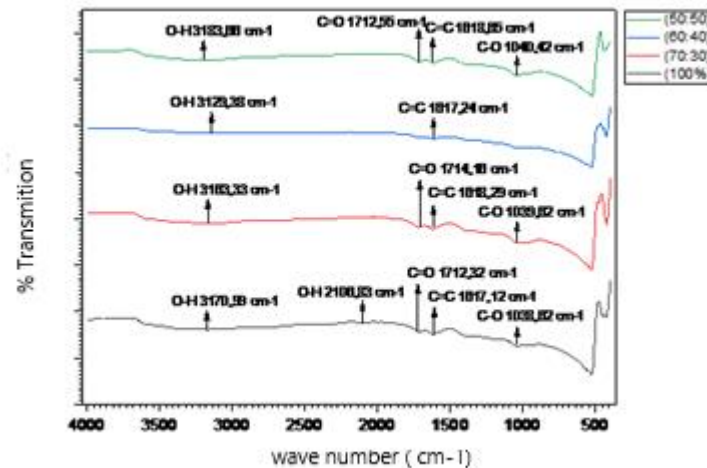


Fig 2. FTIR characterization results data with a composition of 100% graphite, 70% : 30%, 60% : 40% and 50% : 50%.

Based on figure 2 of the FTIR test for graphene oxide from five composition variations, we can see the presence of C-O, C-C, C=O, and O-H bonds. This shows that graphene oxide has been formed, and graphene oxide contains carbon (C), hydrogen (H), and oxygen (O) bonds. Graphene oxide (GO) is a mixture of carbon (C), hydrogen (H), and oxygen (O) obtained through a strong oxidation process from graphite [18].

In 100% pure graphite, the wave number of 1038.62 cm⁻¹ indicates that there is a stretching of the C-O bond groups. At wave number 1617.62 cm⁻¹, it indicates the presence of an aromatic C–C bond. The C=O functional group is at wave number 1712.32 cm⁻¹ with the type of ester compound. The O-H functional group is at wave number 3170.99 cm⁻¹ with the type of alcohol compound.

At 70% pure graphite and 30% seaweed, the wave number of 1039.62 cm⁻¹ indicates that there is a stretching of the C-O bond groups. At wave number 1618.29 cm⁻¹, it indicates the presence of an aromatic C–C bond. The C=O functional group is at wave number 1714.16 cm⁻¹ with the type of ester compound. The O-H functional group is at wave number 3163.33 cm⁻¹ with the type of alcohol compound.

At 60% pure graphite and 40% seaweed, wave number 1617.24 cm⁻¹ indicates the presence of an aromatic C=C bond. The O-H functional group is at wave number 3129.38 cm⁻¹ with the type of alcohol compound.

At 50% pure graphite and 50% seaweed, the wave number of 1040.42 cm⁻¹ indicates that there is a stretching of the C-O bond groups. At wave number 1618.65 cm⁻¹, it indicates the presence of an aromatic C–C bond. The C=O functional group is at wave number 1712.55 cm⁻¹ with the type of ester compound. The O-H functional group is at wave number 3183.66 cm⁻¹ with the type of alcohol compound.

Based on the results of graphene oxide characterization using FTIR, it is found that it has all the functional groups of graphene oxide, namely C-O, C=O, C=C and –OH [14]. Based on the characterization data using FTIR that has been obtained, it can be said that graphene oxide has been formed based on the peaks of bonds containing C, H, O [19]. It can be said that graphene oxide contains a mixture of Carbon (C), Hydrogen (H) and Oxygen (O) compounds. GO has an O-H functional group which indicates the content of water molecules and hydroxyl functional groups, the C = O functional group indicates the presence of carbonyl functional groups and carboxyl groups at the end of the GO layer.

Graphene oxide testing using VNA was carried out to determine the value of *reflection loss* on graphene oxide. The results of the VNA characterization are displayed in the Origin software and display the relationship between the frequency (GHz) on the X axis and the reflection loss (dB) value on the Y axis. The frequency values used are the X-band frequencies (8–12 GHz).

In this test, an acrylic mold measuring 3 cm by 1.5 cm with a thickness of 2 mm was used. The output form of the VNA characterization test results is shown graphically in Figure 3, which was obtained using the origin software.

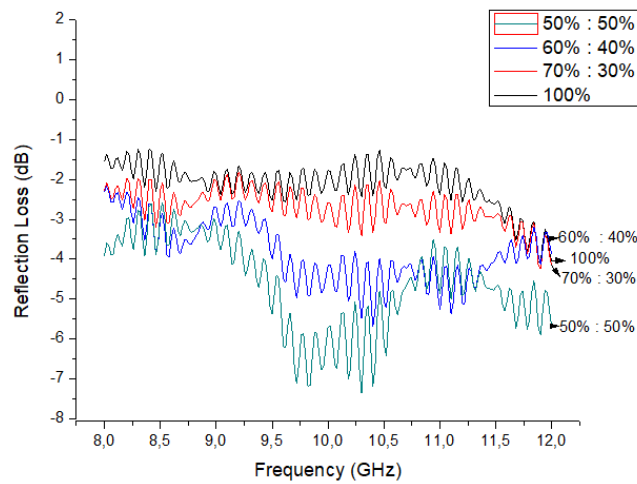


Fig 3. VNA characterization results data with a composition of 100% graphite, 70% : 30%, 60% : 40% and 50% : 50%.

Figure 3 shows the relationship between frequency and reflection loss of graphene oxide in variations of pure graphite samples with seaweed, namely the compositions of 100% graphite, 70%: 30%, 60%: 40%, and 50%: 50%. Reflection Loss shows how much power absorbs microwaves in a material [20]. In the 100% pure graphite variation, the reflection loss value is -4.14 dB at a frequency of 11.9 GHz. In the 70% pure graphite variation with 30% seaweed, the lowest reflection loss value is -4.10 dB at a frequency of 11.9 GHz. In the 60% pure graphite variation, 40% seaweed, the lowest reflection loss value is -5.80 dB at a frequency of 10.4 GHz. In the 50% pure graphite variation and 50% seaweed, the lowest reflection loss value is -7.40 dB at a frequency of 10.3 GHz. The value of the absorption coefficient can be seen in Table 1. Reflection Loss required for good wave absorption is ≤ 10 dB for cost-effective production of radar wave absorbers [20].

Table 1. The absorption value of graphene oxide varies with composition: 100% graphite, 70% : 30%, 60% : 40%, and 50% : 50%.

Pure Graphite Blend Variation with Seaweed	Frequency (GHz)	Reflection Loss (dB)	Adsorption Coefficient	Bandwith (GHz)
100% Graphite	11.9	-4.14	0.37	0.02
70% : 30%	11.9	-4.10	0.37	0.02
60% : 40%	10.4	-5.80	0.48	0.02
50% : 50%	10.3	-7.40	0.57	0.02

Based on table 1, the composition of 100% graphite has a composition of 70% : 30%, 60% : 40%, and 50% : 50%. Seaweed has a reflection loss value and an absorption coefficient. This is influenced by the sintering temperature [20]. This effect is characterized by changes in the values of reflection loss and absorption coefficient at sintering temperature and a mixture of pure graphite and seaweed. The higher the sintering temperature and the percentage of pure graphite mixed with seaweed, the higher the absorption coefficient and the lower the reflection loss value. Sintering is the process of compacting a mass of powder at high temperatures. This is made to compress materials that have been printed at high temperatures [21]. The sintering process affects the formation of the material's crystalline phase and the structure of the crystal growth. The higher the sintering temperature, the faster the crystallization process. It has the smallest reflection loss value and the largest absorption coefficient in a mixture of 50% pure graphite and 50% seaweed with a temperature of seaweed when in the furnace that reaches 300°C. A small reflection loss value will also have a large absorption coefficient, so in this study, a mixture of 50% pure graphite and 50% seaweed can be said to be a material that has good microwave absorption. In the reference obtained, the two-layer absorber has a measured RL of -27.20 dB at 10.8 GHz and for the three-layer absorber, the RL reaches up to -32.58 dB at 11.2 GHz. The measured RL values agree quite well with the calculated ones, which demonstrates the effectiveness of the absorber for various practical EM wave absorption applications [22].

IV. CONCLUSION

Based on the research that has been done, Graphene Oxide synthesized by the modified Hummers method by mixing pure graphite with seaweed charcoal is an alternative material when there is scarcity or diminishing pure graphite and want to add seaweed in the mixture of pure graphite. It can be seen from some typical properties of Graphene Oxide at an angle of 2θ by XRD testing, found to contain C-O, C=O, C=C and O-H functional groups in FTIR testing. The addition of seaweed charcoal to pure graphite synthesized by a modified Hummers method also affects the value of the microwave absorbent properties of graphene oxide. This happens if the more pure graphite mixed with seaweed charcoal has been done, the higher the *Reflection Loss* value.

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