

Effect of Addition of Seaweed (*Sargassum sp*) Charcoal with Pure Graphite on the Electrical Properties of Graphene Oxide Synthesized by the Modified Hummer's Method

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ABSTRACT

Graphite is the primary raw material used to create graphene oxide. However, other biomass sources can also be used to create or combine the carbon source. Seaweed (*Sargassum sp.*), which has a decent carbon content, was used in this study as the biomass used as a carbon source for mixing. The objective of this research is to ensure how adding seaweed charcoal will affect the electrical characteristics of graphene oxide. The modified Hummer process was used to create the materials. There are four different compositions available: pure graphite (100%), 70:30 (graphite and seaweed), 60:40 (graphite and seaweed), and 50:50 (graphite and seaweed). The product of graphene oxide variations made from other compositions will be compared to those of 100% graphite composition. The result demonstrated that the addition of seaweed charcoal to the graphene oxide synthesis was successful. It can be seen from some of the characteristics of graphene oxide at an angle of 2θ by XRD testing. In the FTIR test, it was discovered that the graphene oxide sample contained C-O, C=O, C=C, and O-H functional groups, and in the SEM test, peeling was discovered on the sample surface as a result of the oxidation process. The addition of seaweed charcoal with pure graphite synthesized by the modified Hummers method affects the electrical properties of the resulting graphene oxide. The material's conductivity value and the area capacitance value of the graphene oxide material both drop in the LCR Meter characterization instrument.

Keywords : Graphite, Seaweed (*Sargassum sp.*), Graphene Oxide, Hummer method modification, electrical properties.



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I. INTRODUCTION

The Indonesian seas are rich in natural resources, one of which is seaweed. There are many types of seaweed in Indonesia, including *Sargassum* seaweed, whose economic value is quite high. *Sargassum sp.* is very abundant and widespread in Indonesian waters.

Brown seaweed *Sargassum sp.* has been widely researched and has been widely utilized. *Sargassum sp.* contains alginate and iodine ingredients used in the food, pharmaceutical, cosmetic and textile industries[1]. Over time the utilization of *Sargassum sp.* developed quite rapidly. This development cannot be separated from the bioactive compounds produced by *Sargassum sp.*, which can be used as food ingredients, fuels, cosmetics (moisturizing creams), medicines, pigments, and supplements[2].

Padang is a city located on the west coast of the Sumatra island. In the shallow water region across the city shore, there are many *Sargassum* can be found, especially the coast of Bungus Teluk Kabung district which is plentiful in number and wide-spread along the shoreline. However, the people of Bungus only consider

Sargassum sp. as a wild sea plants or mere just fish breeding ground and never been cultivated because the people rarely know its potential and benefits.

However, without human intervention to process it, *Sargassum* sp. itself can also provide other benefits. Macroalgae type *Sargassum* sp. is the macroalgae that absorb the most carbon in Karang Papak Garut Beach[3]. Previous research stated that *Sargassum* sp. can absorb 98 tons of CO₂/day on the coast of Indian Beach[4]. *Sargassum* sp. is one of the macroalgae that has the highest ability to capture and store carbon in the form of biomass in Sumbawa waters, which is 275.20 gC/m²[5].

The availability of *Sargassum* in the Bungus Teluk Kabung coast is abundant and it also can absorb carbon in large amount of biomass. Therefore, it can be used as alternative material to make Graphene Oxide and replaces graphite as the main carbon source which is expensive in cost. Graphene oxide is a monolayer form of graphite oxide[6] obtained from the process of exfoliating graphite oxide into sheets through a sonication or stirring process[7]. Graphene oxide is a functional class of highly reactive oxygen[8]. Graphene oxide has unique characteristics, good optical, electronic, and mechanical properties[7], so Graphene Oxide can be applied in various fields, including as sensors, polymer nanocomposites, energy safe equipment[9].

Graphene possesses an extensive surface area, rendering it effective for use as an electrode material in capacitors. The distribution of pores in a solid state does not significantly impact its value. This stands in contrast to supercapacitors constructed with activated carbon and carbon nanotubes [7]. Despite graphene oxide being a derivative of graphene, there exist slight differences in its electrical properties, where graphene oxide exhibits lower conductivity compared to pristine graphene. This discrepancy arises from graphene oxide being the oxidation product of graphene, resulting in carbon atom layers containing oxygen functional groups. Atomically, graphene oxide represents a thin layer of graphite oxide consisting of aliphatic six-membered rings containing –OH, epoxy, and conjugated C=C bonds. The carbon atom positions on graphene oxide, combined with the presence of –OH and –COOH groups, not only increase the distance between layers but also create a thick and hydrophilic atomic layer.

However, graphene oxide holds considerable potential for electrode fabrication due to its ability to not only exist as individually coated sheets but also, when bound, readily expose itself to electrolytes, offering the prospect of a high surface area. According to research conducted by Park et al., graphene oxide exhibits electrical conductivity up to $3,7 \times 10^{-5}$ S/m [9]. This value may be further enhanced as graphene oxide proves to be a highly intriguing material with unique characteristics, allowing for the modification of its electrical properties through alterations in its chemical structure. Graphene oxide demonstrates the capability to undergo changes in resistance by the addition or removal of oxygen-containing bonds [8].

Methods used for the synthesis of Graphene Oxide include the micromechanical exfoliation method, epitaxial growth method, Chemical Vapor Decomposition (CVD)[10], Improved GO[11], and the Hummers method[12]. Micromechanical exfoliation methods are inefficient to use, while epitaxial and CVD growth methods cost quite a lot. While the Hummers method has advantages such as the reaction process does not take a long time, the reaction process is very safe because it uses KMnO₄, which does not produce explosive materials (explosives), such as ClO₂ produced from KClO₃, using NaNO₃ as a substitute for HNO₃ which can produce acid fog[13]. So, the most efficient method used now is the Hummers method.

Research on the synthesis of Graphene Oxide previously used graphite as a carbon source, as has been done by Marcano et al., 2010. The use of biomass as a carbon source is important to do. In addition to reducing the amount of waste in Indonesia, which is quite a lot, and reducing environmental pollution from waste materials, the use of organic waste as a carbon source for the synthesis of Graphene Oxide is also considered cheaper than the use of graphite and its raw materials are also abundantly available in nature[14]. So, the use of organic biomass from *Sargassum* sp as a carbon source and as a mixing material is considered to be efficiently used for the synthesis of Graphene Oxide. This research will help find new solutions for the manufacture of graphene oxide from biomass which will later be used for the manufacture of electrical components.

In this study, the electrical properties that will be analyzed are material resistivity, area capacitance and material conductivity. Therefore, a study was conducted with the title: " Effect of Addition of Seaweed (*Sargassum* sp.) Charcoal with Pure Graphite on the Electrical Properties of Graphene Oxide Synthesized by the Modified Hummer's Method."

II. METHOD

Numerous methods can be employed for the synthesis of graphene oxide, including the Hummers method, chemical vapor deposition (CVD), and sonication utilizing ultrasonic waves. The CVD method yields graphene of high quality and purity; however, it is afflicted by drawbacks such as elevated production costs and limitations in scalability for generating substantial quantities of graphene oxide. The Hummers method employs chemical compounds to exfoliate the graphite layers. This method is relatively straightforward, utilizing readily available, cost-effective chemical compounds, thus facilitating the production of graphene oxide in substantial quantities. However, drawbacks of this method include the generation of noise during production, resulting in GO with relatively low purity. Moreover, the utilization of NaNO_3 for the oxidation process leads to the emission of toxic gases. Among the various methods, the Hummers method stands out as the most efficient for GO synthesis due to its utilization of easily obtainable materials and a simple procedure. Nevertheless, modifications are essential to address the issue of toxic gas generation associated with the original Hummers method.

In this research seaweed samples (*Sargassum* sp) were taken from the shallow sea waters of Teluk Kabung, Bungus Teluk Kabung District, Padang City. The samples were washed and then dried in the sun for one day, then dried in the oven for 3 hours at 105°C for the dehydration process. Then, the seaweed (*Sargassum* sp) that has been ventilated was then carried out a furnace process for 30 minutes at a temperature of 300°C so that the Seaweed (*Sargassum* sp) turns into Seaweed charcoal (*Sargassum* sp), which has turned into charcoal, then mashed with mortar and pestle to produce charcoal powder and sifting with a sieve size of 170 mesh.

The activation process can be done in two ways, namely, chemical activation and physical activation. In chemical activation, immersion in carbon is carried out with chemical compounds, including H_3PO_4 , HF, NH_4Cl , AlCl_3 , HNO_3 , KOH, NaOH, KMnO_4 , SO_3 , H_2SO_4 , and K_2S . At the same time, physical activation is the activation of carbon physically by using weak oxidation, e.g., water vapor, CO_2 , N_2 , O_2 gases, and other oxidizing gases[15].

In this study, carbon activation was carried out by chemical activation using a KOH solution. Carbon that has been finely weighed as much as 8 grams, KOH compounds are also weighed as much as 8 grams. Then the KOH compound is dissolved by mixing KOH into 100 ml of Aquades. After the KOH solution is completely dissolved, it is mixed into carbon that has been in a 250 ml beaker. The solution is stirred for 15 minutes until fully mixed, then let stand for 24 hours so that a precipitate of activated carbon is formed.

The sample precipitate is then dried by filtering it using filter paper. The perfectly activated filtered sample is then oven-dried at 105°C for 3 hours.

Activated seaweed carbon and graphite samples were weighed as much as the composition ratio had been established. Then it is synthesized with reducing compounds using the method of modified hummer. For the first mixing stirring process, a mixture of activated carbon and graphite is put into a beaker glass, and then add a solution of sulfuric acid (H_2SO_4) of 34.5 ml and NaNO_3 of 0.75 ml. The solution is placed on a hot plate and stirred for 20 minutes. Furthermore, it is treated by stirring in the ice bath for 2 hours. Next, add 4.5 grams of potassium permanganate (KMnO_4) slowly and then stir again for 30 minutes without an ice bath.

In the second mixed stirring process, the stirring is carried out in a fume hood. First, add 69 mL of aquadest slowly, then stir again for 20 minutes. Next, add another 100mL of aquadest, then the solution is mixed with 1.5 mL of peroxide acid (H_2O_2) to reduce the bubbles resulting from mixing KMnO_4 . Next, add aquades 50 ml.

After the visible color changed to yellow in the solution, the synthesis process was continued with the graphene oxide dispersion process by sonication for 2 hours to exfoliate the Graphene Oxide into a thin layer. The sonication process was carried out using an ultrasonic bath at room temperature for two h then the solution was precipitated for 24 h until a solid phase and liquid phase was formed. Furthermore, the centrifugation process is carried out using a microcentrifuge at a speed of 2500 rpm for 15 minutes. Furthermore, GO neutralization is carried out manually by precipitating the mixture until it is formed into a solid phase and a liquid phase. Then the liquid phase is replaced with aquadest repeatedly until a neutral pH of 7 is obtained. After obtaining a neutral pH, Graphene oxide is filtered and separated from the liquid phase and dried using an oven with a temperature of 60°C for 12 hours.

The final stage is the graphene oxide characterization stage. X-Ray Diffraction (XRD) is used to determine the crystal structure of graphene oxide samples. Fourier Transform Infra-Red (FTIR) is used to determine the functional groups contained in graphene oxide. Scanning Electron Microscopy (SEM) is used to determine the microstructure of Graphene Oxide samples. And based on the characterization data using the LCR Meter, a graph of the conductivity of the material (Graphene Oxide) will be obtained with the composition and capacitance of the material area (Graphene Oxide) with the composition.

III. RESULTS AND DISCUSSION

Graphene Oxide Testing using XRD is performed to identify emerging phases and determine the structure/system and crystal size. The value plot of the diffraction pattern of Graphene Oxide was obtained using the Origin application. While the characterization results from XRD are processed using the High Score Plus application. The data obtained from the characterization results using XRD are compared with the data contained in the ICDD database. The results of graphene oxide analysis data using XRD with four composition variations, namely 100% graphite, 70%: 30%, 60%: 40%, and 50%: 50%, can be seen in Figure 1 below.

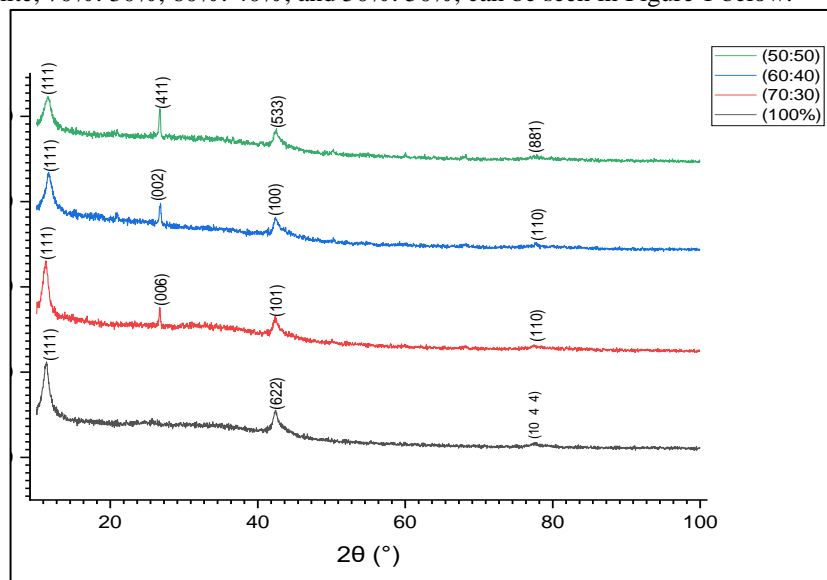


Fig. 1. XRD Results Data composition 100% graphite, 70%: 30%, 60%: 40% and 50%: 50%

Based on Figure 1 shows the diffraction pattern of Graphene Oxide from the composition of 100% graphite, 70%: 30%, 60%: 40%, and 50%: 50% using Origin software. At a 100% graphite composition, it is seen that the deposition result is formed three peaks with an angle of 11.074° ; 42.36° and 77.50° with peak miller indices relating to phases (111), (622), and (1044). The average grid parameter on ICDD code 01-082-2261. Based on the results of the diffractogram pattern, it can be used to determine the average of the crystal size using the Scherrer equation obtained from the peak value of FWHM. The average size of the crystal calculated by the Scherrer equation is 32.56 nm.

In the composition of 70%: 30%, it is seen that the deposition result is formed as many as four peaks with an angle of 11.25° ; 26.69° ; 42.33° and 77.41° with peak millers index relating to phases (111), (006), (101) and (110). The average grid parameter on ICDD code 00-026-1076. Based on the results of the diffractogram pattern, it can be used to determine the average of the crystal size using the Scherrer equation obtained from the peak value of FWHM. The average size of the crystal calculated by the Scherrer equation is 21.78 nm.

In a composition of 60%: 40%, it is seen that the deposition result is formed as many as four peaks with an angle of 11.12° ; 26.76° ; 42.37° and 77.68° with peak millers index relating to phases (111), (002), (100) and (110). The average grid parameter on ICDD code 01-071-3739. Based on the results of the diffractogram pattern, it can be used to determine the average of the crystal size using the Scherrer equation obtained from the peak value of FWHM. The average size of the crystal calculated by the Scherrer equation is 28.35 nm.

At 50%: 50%, it can be seen that the deposition result is formed by four peaks with an angle of 11.07° ; 26.69° ; 42.39° and 77.08° with peak millers index relating to phases (111), (411), (533) and (881). The average grid parameter on ICDD code 01-079-1715. Based on the results of the diffractogram pattern, it can be used to determine the average of the crystal size using the Scherrer equation obtained from the peak value of FWHM. The average size of the crystal calculated by the Scherrer equation is 29.93 nm. Xrd results show a crystal structure in the form of Graphene Oxide, namely Hexagonal.

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

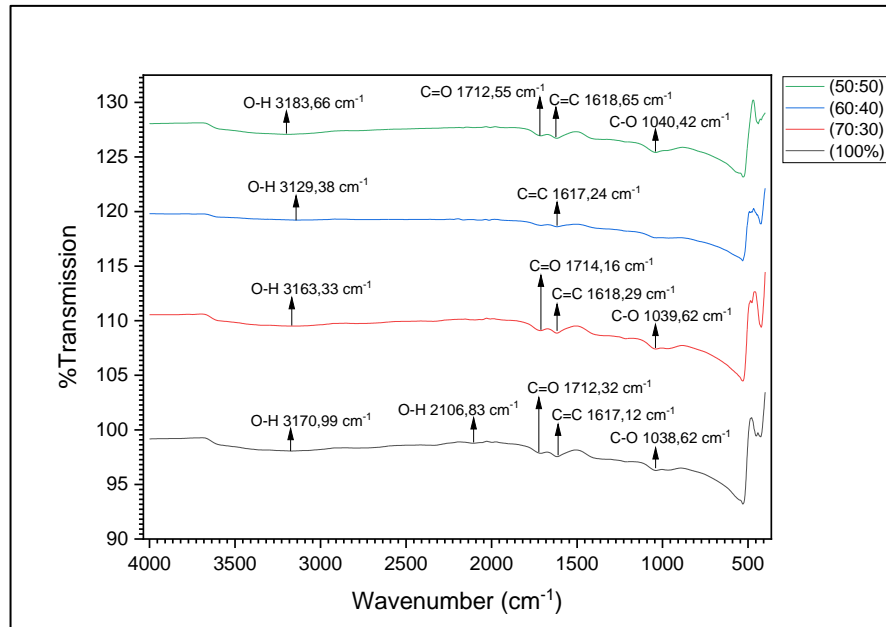


Fig. 2. FTIR Characterization Results Data composition 100% graphite, 70%: 30%, 60%: 40% and 50%: 50%

Based on Figure 2 FTIR Graphene Oxide tests from five compositional 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, where graphene oxide contains bonds of carbon (C), Hydrogen (H), and Oxygen (O). Graphene Oxide (GO) is a compound of a mixture of carbon (C), hydrogen (H), and oxygen (O) obtained through a strong oxidation process of graphite[16].

Wave numbers 890 – 1300 cm^{-1} indicate the presence of a C-O bond group tension. The wave numbers 1500 – 1675 cm^{-1} indicate the presence of aromatic C=C bonds. The function group C=O is at the wave number 1650 – 1900 cm^{-1} with the type of ester compound. The function group O-H is at wave number 3580– 3650 cm^{-1} with the type of Alcohol compound.

Based on the results of graphene oxide characterization using FTIR, it was found that it has all the functional groups of Graphene Oxide, namely C-O, C=O, C=C, and -OH[17]. Based on characterization data using FTIR that has been obtained, it can be said that graphene oxide has been formed based on the peaks of the bond containing C, H, and O according to the statement[18], which says that graphene oxide contains a mixture of compounds Carbon (C), Hydrogen (H) and Oxygen (O).

The results of characterization imaging using SEM Oxide Graphene of each variation of the composition of 100% graphite, 70%: 30%, 60%: 40%, and 50%: 50% can be seen in Figure 3, Figure 4, Figure 5, and Figure 6.



Fig. 3. Surface image of Graphene Oxide composition of 100% graphite composition with 1000X magnification

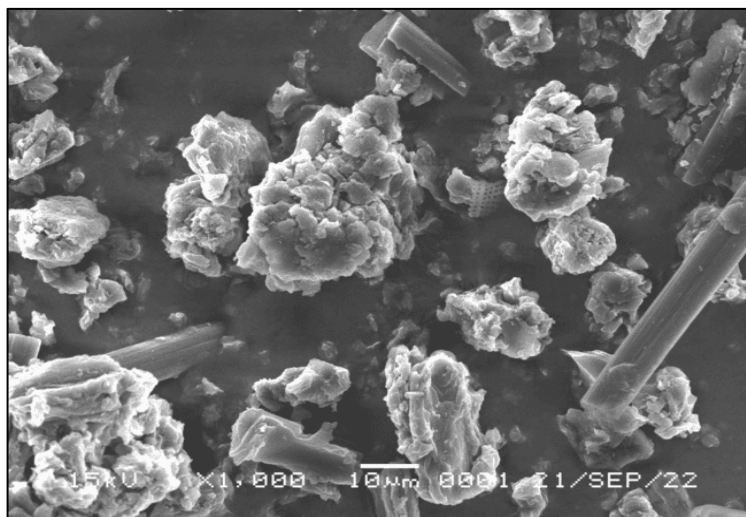


Fig. 4. Surface image of Graphene Oxide composition of 70% graphite with 30% seaweed with 1000X magnification

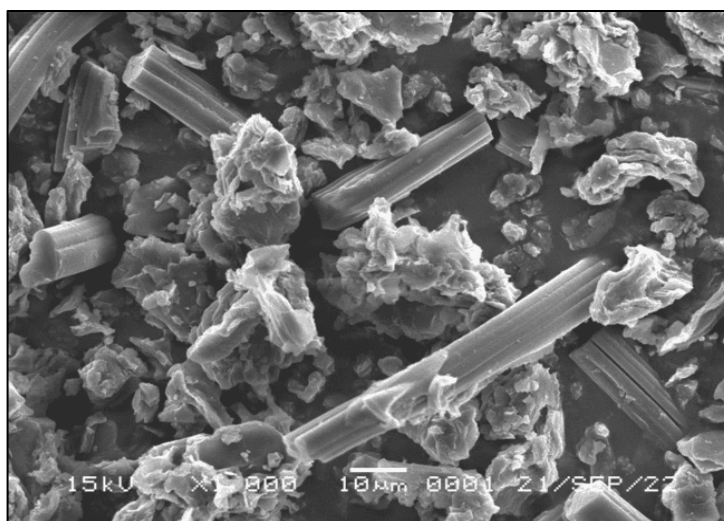


Fig. 5. Surface image of Graphene Oxide composition of 60% graphite with 40% seaweed with 1000 × magnification

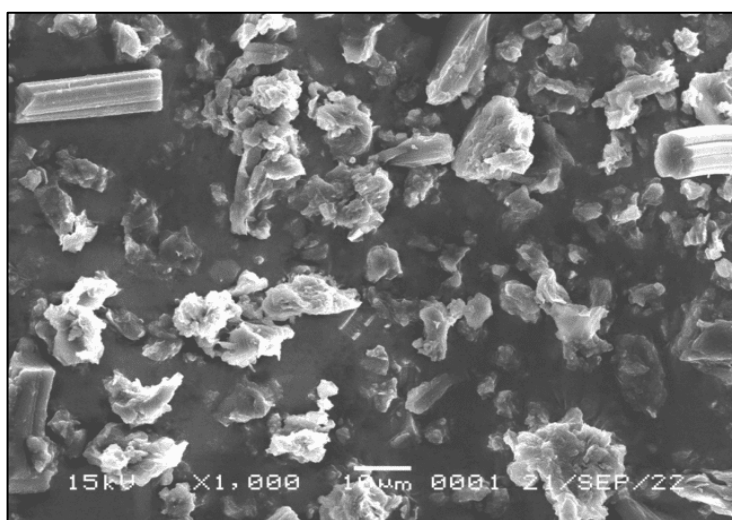


Fig. 6. Surface image of Graphene Oxide composition of 50% graphite with 50% seaweed with 1000X magnification

The results of SEM characterization on graphene oxide samples taken with a magnification of 1000x show the surface layers of Graphene Oxide due to exfoliation of oxidation results, which in Figure 6 parts are highlighted for clarity. This shows that the surface of Graphene Oxide is very thin in the nanometer order and folds due to the thermodynamic instability of the 2D material that forms folds to reduce its energy.

On this characterization, it can be seen that the surface of Graphene Oxide is very thin in the nanometer order and folds due to the thermodynamic instability of the 2D material that forms folds to reduce its energy. The surface folds of Graphene Oxide are also following the observations of Janowska et al. in 2010. In all the samples tested, there was no obvious morphological difference. Graphene Oxide Sheets consist of one layer and appear somewhat transparent from all the others. These Graphene Oxide sheets are arranged with each other forming an irregular round pattern with a thinner layer. In SEM characterization, it can be seen that the surface of Graphene Oxide is very thin in the nanometer order and folds due to the thermodynamic instability of the 2D material that forms folds to reduce its energy. The surface folds of Graphene Oxide are also following the observations of Janowska et al. in 2010. In all the samples tested, there was no obvious morphological difference. Graphene Oxide Sheets consist of one layer and appear somewhat transparent from all the others. These Graphene Oxide sheets are arranged with each other forming an irregular round pattern with a thinner layer.

The results of the characterization of Graphene Oxide using the LCR Meter obtained data in the form of capacitance values and resistance of Graphene Oxide materials, then processed to obtain the value of material conductivity and capacitance of the area affected by the sample size, while the sample used in this test was in the form of pellets with a diameter of 3 mm and a thickness of 2 mm so that the surface area of the pellets was known using pellets using the surface area of the tube, that is 33 mm^2 . The results of the characterization of Graphene Oxide using the LCR Meter obtained data in the form of capacitance values and resistance of Graphene Oxide materials, then processed to obtain the value of material conductivity and capacitance of the area affected by the sample size, while the sample used in this test was in the form of pellets with a diameter of 3 mm and a thickness of 2 mm so that the surface area of the pellets was known using pellets using the surface area of the tube, that is 33 mm^2 .

Based on the results of LCR Meter characterization, the value of material conductivity and capacitance of the material area can be observed with variations in the composition of the material. The two values can be compared at 100% graphite composition with a composition of 70%: 30%, 60%: 40%, and 50%: 50%.

The results of data processing to obtain conductivity data of the processed material from resistance data can be seen in Figure 7.

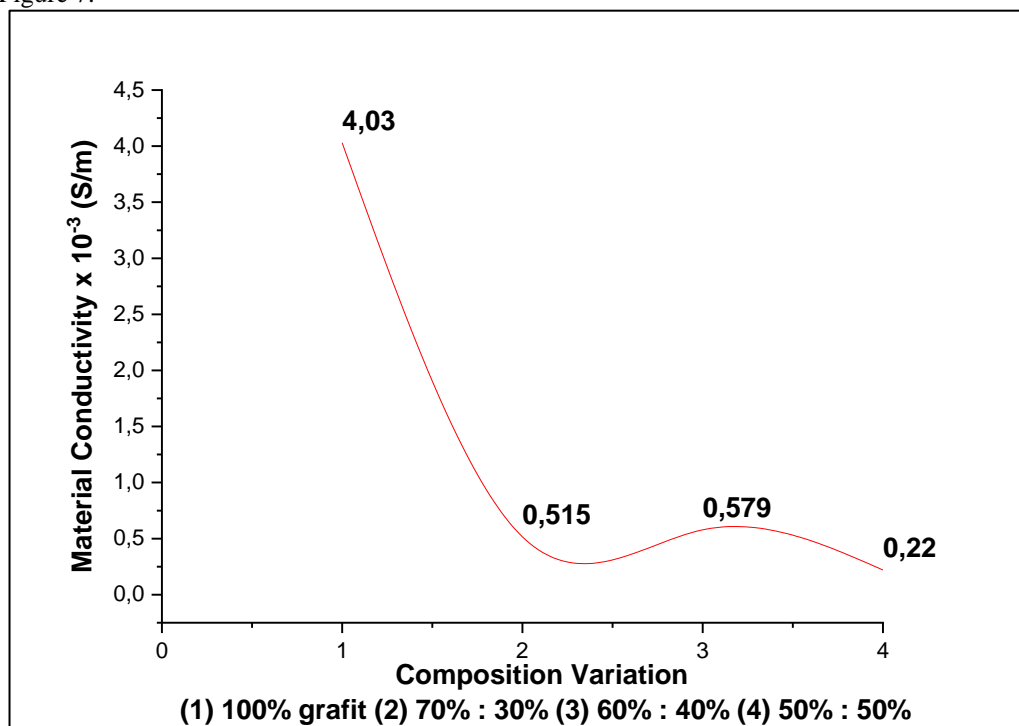


Fig. 7. The relationship of composition variations with the conductivity of materials

From the graph in Figure 7 can be seen the conductivity value of each variation of the composition. For a 100% graphite composition, the average material conductivity value is obtained by $4.03 \times 10^{-3} \text{ S/m}$. At the composition of 70% graphite with 30% seaweed, the average material conductivity value is $0.515 \times 10^{-3} \text{ S/m}$. At

a composition of 60% graphite with 40% seaweed, the average material conductivity value is 0.579×10^{-3} S/m. And at the composition of 50% graphite with 50% seaweed obtained, the average material conductivity value is 0.22×10^{-3} S/m.

This shows a decrease in the conductivity value of Graphene Oxide material after pure graphite is mixed with seaweed. It is clear that the difference in value is from 4.03×10^{-3} S/m to 0.515×10^{-3} S/m, but with the other two variations, it is not too big to see the difference in value. This has not achieved the conductivity corresponding to the research conducted by Park et.al where the conductivity of the material obtained is as large as $3,7 \times 10^{-5}$ S/m [7]. Based on the conductivity value of the material, Graphene Oxide is included in semiconductor materials, where semiconductor materials have conductivities ranging from 10^{-8} to 10^3 S/m. There is a change in the conductivity of Graphene Oxide due to the provision of energy in the Graphene Oxide synthesis process which causes the break in the interaction of van der Waals on carbon so that it can peel off graphite into sheet shape. Coupled with the content of O and H functional groups which can affect the mobility of electrons contained in Graphene Oxide [7].

The results of data processing to obtain area capacitance data can be seen in Figure 8.

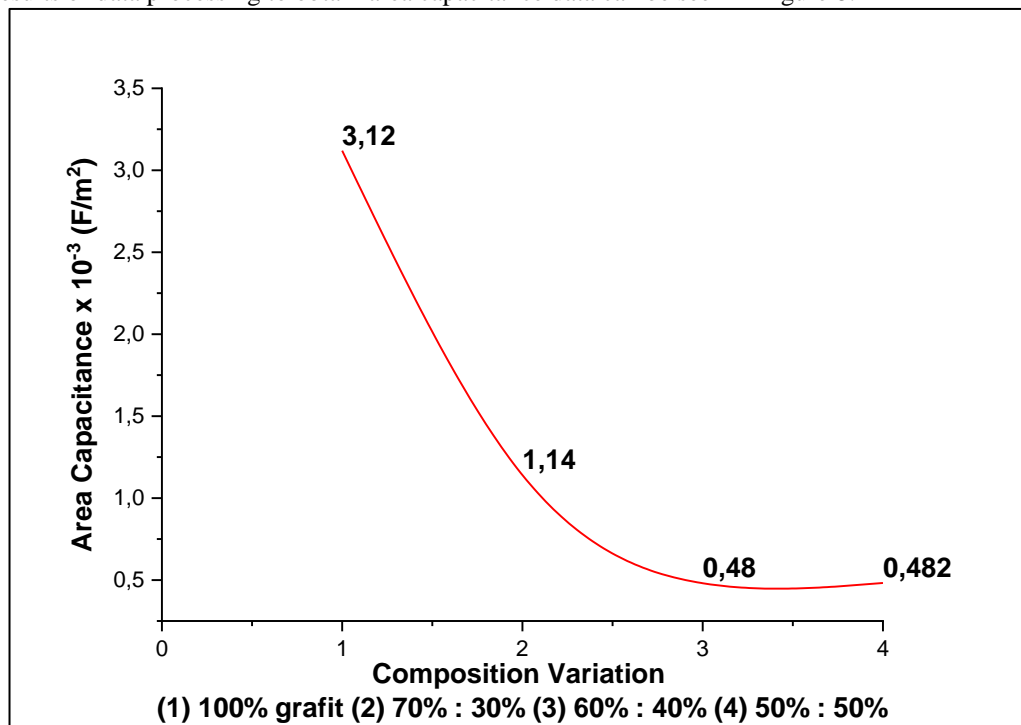


Fig. 8. The relationship of composition variation with area capacitance

For a 100% graphite composition, the average area capacitance value is obtained by 3.12×10^{-3} F/m². In the composition of 70% graphite with 30% seaweed, the average area capacitance value is 1.14×10^{-3} F/m². At a composition of 60% graphite with 40% seaweed, the average area capacitance value was 0.48×10^{-3} F/m². And at the composition of 50% graphite with 50% seaweed, the average area capacitance value is 0.482×10^{-3} F/m².

It also shows a decrease in the capacitance value of the graphene oxide material area after pure graphite is mixed with seaweed. It is clear that the difference in values is from 3.12×10^{-3} F/m² to 1.14×10^{-3} F/m², but with the other two variations, there is still a difference in values that fall to 0.48×10^{-3} F/m². At variations of 60%: 40% and 50%: 50% the value is relatively the same.

Based on research conducted by Cahyani, the capacitance of the Graphene Oxide area was obtained which was exfoliated using aquades, which was as large as $1,29 \times 10^{-11}$ F/cm² or $1,29 \times 10^{-7}$ F/m² [19] when compared to the capacitance obtained by the author is quite large, namely 0.48×10^{-3} F/m² to 3.12×10^{-3} F/m². But when compared with the research conducted by Amelia et al, the reduced capacitance of Graphene Oxide using Cu can achieve $6,15 \times 10^{-3}$ F/cm² [20] where this value is greater when compared to that obtained by the author.

From the two values above, namely the conductivity value of the material and the area capacitance value that has been compared between 100% graphite with other compositions, it can be seen that the effect of adding seaweed charcoal with pure graphite on the electrical properties of Graphene Oxide is to further reduce the quality of the electrical properties of Graphene Oxide.

IV. CONCLUSION

Based on the research that has been carried out, Graphene Oxide synthesized by the modified Hummers method by mixing pure graphite with seaweed charcoal can be seen from several distinctive properties of Graphene Oxide at an angle of 2θ with XRD testing. It was found to contain functional groups C-O, C=O, C=C, and O-H in FTIR testing and the discovery of exfoliation on the sample surface due to the oxidation process in the SEM test. The addition of seaweed charcoal with pure graphite synthesized by the modified Hummers method affects the electrical properties of the Graphene Oxide produced. In the LCR Meter characterization tool, there is a decrease in the conductivity value of the material and the capacitance value of the graphene oxide material area. This is due to the provision of energy in the Graphene Oxide synthesis process which causes the break in the interaction of van der Waals walls on carbon so that it can peel off graphite into sheet shape. Coupled with the content of O and H functional groups which can affect the mobility of electrons contained in Graphene Oxide.

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