ISSN (Print) : 2337-9030 ISSN (Online) : 2685-2608
PILLAR OF PHYSICS
JURNAL BERKALA ILMIAH FISIKA
Vol. 16 (2), 2023, page. 144-151
DOI : 10.24036/12996171074

ANALYS OF MICROWAVE ABSORPTION PROPERTIES OF GRAPHENE OXIDE FROM RICE HUSK WASTE

Andari Oktafiani[1], Ramli[1,2 *], Yenni Darvina[1,2], Gusnedi[1,2]

[1] *Department of Physics, Universitas Negeri Padang, Jl. Prof. Dr. Hamka Air Tawar Padang 25131, Indonesia*
[2] *Nanoscience and Nantotechnology Research Group, Universitas Negeri Padang, Jl. Prof. Dr. Hmka Air Tawar Padang 25131, Indonesia*

Corresponding author. Email: ramli@fmipa.unp.ac.id

ABSTRACT

Graphene Oxide (GO) was synthesized with variations in carbonization temperatures of 250 °C, 300 °C, and 350 °C using the modified Hummers method. 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. Therefore, in this report, GO uses rice husk waste instead of graphite as a carbon source because rice husk waste is abundant in nature and easy to obtain than graphite. In addition to reducing waste in Indonesia, this study aimed to determine the crystal size and GO functional groups and analyze the properties of microwave absorbers. X-ray diffraction (XRD) was used to determine the crystal size. The GO functional groups were determined using Fourier Transform Infrared (FTIR), and the microwave absorption characteristics of GO were analyzed using a Vector Network Analyzer (VNA). The XRD results show GO peaks between 26° peaks and 44° peaks. In the FTIR results, there are a collection of GO practices, in particular CO, C = C, and C = O. In the VNA results, the best microwave absorption properties are at an ignition temperature of 350 °C with the lowest reflection value - 39.95 dB, the highest absorption coefficient is 99%, and the absorption bandwidth is 0.06 GHz at a frequency of 8.5 GHz.

Keywords : Rice Husk; Modified Hummer Method; GO; Graphite; Microwave Absorbent

I. INTRODUCTION

Indonesia is a nation known as a country that has abundant fixed assets. One form of agricultural waste is husk, a "waste" of rice processing as shown in Figure 1. Since rice is the staple food of the Indonesian people, abundant rice production brings prosperity to people. However, with the abundance of rice production, the waste produced is also plentiful [1].

Fig 1. Rice husks

Rice husk contains 86.9-97.8% silica and 10-15% carbon [2]. The silica and carbon parts can be synthesized into a new material, graphene oxide (GO). This carbon from the processed rice husk will replace graphite as a carbon source used as the main ingredient for graphene oxide production. Previous research explained that burned waste can produce charcoal to be used as an active ingredient which is used as the main ingredient in the manufacture of Graphene Oxide (GO) [3].

Graphene Oxide (GO) is a monolayer graphite oxide acquired by stripping graphite oxide into sheets with sonication whether blending [4]. GO is a highly reactive functional group of oxygen [5]. GO has good optical, electronic, and mechanical properties, so it can be applied in various fields, including sensors, polymer nanocomposites, energy-safe equipment , electronics, and adsorption of pollutants [6]. GO was first researched by Brodie in 1859. Brodie examined the graphite structure by taking a gander at the animation of the graphite sheet. Brodie uses HNO3 and $KClO_3$ [7]. Subsequent investigations were conducted by Staudenmaier using materials, followed by the Staudenmaier mix and minor additions. Additions are made so that a robust oxidation process takes place. Hummers designed an elective technique in 1958 for integrating GO utilizing and as well as H_2SO_4, HNO_3, $KClO_3$, and $NaNO_3$ [8].

Techniques utilized for GO synthesis include the micromechanical exfoliation method, epitaxial growth method, Chemical Vapor Decomposition (CVD), Improved GO, and Hummer method [9]. Micromechanical shedding techniques are ineffective, while epitaxial and CVD development strategies cost colossal cash. Along these lines, the most proficient method utilized now is the Hummers technique. Among the upsides of the Hummers, the system is that the response cycle doesn't consume most of the day. The response interaction is exceptionally protected because it utilizes one that doesn't deliver dangerous materials (explosives), as produced from, using it as a substitute that can have acid mist H_2SO_4, ClO_3, HNO_3, $KClO_3$, $NaNO_3$ [10].

Researchers often use the modified hummers method to obtain graphene oxide (GO). The Hummers method is not dangerous, the ingredients are easy to get, and it is better than the previous method because it does not emit ClO_2 gas during the oxide process. In the Hummers method, the graphite is oxidized with potassium permanganate ($KMnO_4$) and sodium nitrate ($NaNO_3$) in a solution of sulfuric acid (H_2SO_4)[11].

This study will discuss rice husk waste as a base material for graphene oxide for the synthesis of microwave absorpstion using the modified Hummer's method discuss rice husk waste as a base material for will be properties that will be detailed later. Microwaves are one type of electromagnetic energy that can convert energy into heat because of the cooperation between the parts that produce electric waves, and charged particles of the materials used [12]. Microwaves are usually electromagnetic waves with a frequency of 1m - 1 mm and a range of 0.3300 GHz. The test results were obtained from XRD, FTIR, and VNA tools [13].

II. METHOD

The type of research is experimental research. The tools used in this research are hot plate and magnetic bar, oven, furnace, digital scale, mortar and pestle, Erlenmeyer, sieve, evaporating dish, measuring flask, beaker, aluminum foil, spatula, centrifuge, Buchner funnel, fume hood, measuring cup pipettes, volume pipettes, dropper pipettes, thermometers, ultrasonic, pH paper, and concrete on it. The ingredient used were rice husk, H_2SO_4, 98%, $NaNO_3$ powder, $KMnO_4$ powder, equates, H_2O_2 30%, and HF. Implement characterization needed are X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR), and Vector Network Analyzer (VNA).

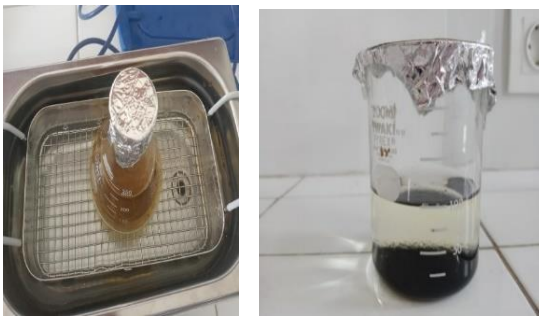
The rice husk material is processed into charcoal in the first stage, activating the rice husk charcoal. The rice husks that will be used are rice husks obtained from the waste of Kuranji Subdistrict, Padang City. Rice husks are collected and then dried for two days in the sun. Then, husk rice in the oven for 60 minutes at a temperature of 100 °C. Then dive into the range at temperatures of 250°C, 300°C, and 350°C for 120 minutes to turn the rice husks. Rice husks turned into charcoal are mashed with lumping and pestle, then sifted with a sieve size of 120 mesh. After that, silica release is carried out with carbon activities using a 30 ml HF solution of 40%, where the HF solution itself has become an activator. Rice husk charcoal powder is dissolved with HF, then heated to a temperature of 200°C until the white froth foam disappears, which indicates the absence of silica content [3].

The next stage is the GO synthesis stage. GO synthesis is performed utilizing the modified Hummers method. The synthesis process begins with weighing 2 g of activated carbon of rice husk charcoal powder and 1 g of $NaNO_3$. Then put it into a 250 mL Erlenmeyer which has a magnetic stirrer inserted. Then enter 46 mL of 98% H_2SO_4 into the Erlenmeyer flask. The mixture was then stirred at a speed of 300 rpm for 20 minutes at 05 °C. Place the Erlenmeyer flask in an ice bath and stirrer on a hot plate for 2 hours. Next, slowly add 4 g of powder to keep the temperature below 20°C $KMnO_4$, as shown in Figure 2.

Fig 2. GO Synthesis stage.

As shown in Figure 2, this process was carried out carefully so that the mixture did not explode and the synthesized rice husk powder did not decrease. After adding $KMnO_4$ to the mix, remove the ice bath from the hot plate and stir the mixture at 35°C for 30 minutes [4] so that in the end, the oxidation process can occur. Add 92 ml of distilled aquades slowly using a pipette and stir for 20 minutes [4]. Add 134 mL of distilled aquades, then add 2 mL of 30% H_2O_2. The addition of H_2O_2 was carried out to remove the remaining $KMnO_4$ or to stop the reaction, and the solution finally turned yellow, indicating the presence of graphene oxide [14]. Finally, add 67 mL of distilled water to the mixture, and Graphene Oxide (GO) will be formed [15].

The next stage is GO sonication and balance of GO. After the solution changes to yellow, which indicates the presence of graphene oxide, the mixture is sonicated for 2 hours to exfoliate the graphite into graphene [14], as shown in Figure 3.



(a) (b)

Fig 3. (a) Sonication and (b) GO penetrating.

The solution was precipitated for 16 hours until a liquid and solid phase was formed [16]. Then, a centrifugation cycle was carried out involving a speed of 4000 rpm for 15 minutes. The following process is the manual neutralization of GO, namely by precipitating GO powder and then changing the distilled water repeatedly until a neutral pH of 7. After a neutral pH is obtained, GO is baked at 110 ° C for 2 hours [16].

III. RESULTS AND DISCUSSION

GO testing using XRD was carried out to identify the emerging phase and determine the crystal structure. The results of GO characterization using XRD, which showed the relationship between diffraction peaks and intensity, are shown in Figure 4.

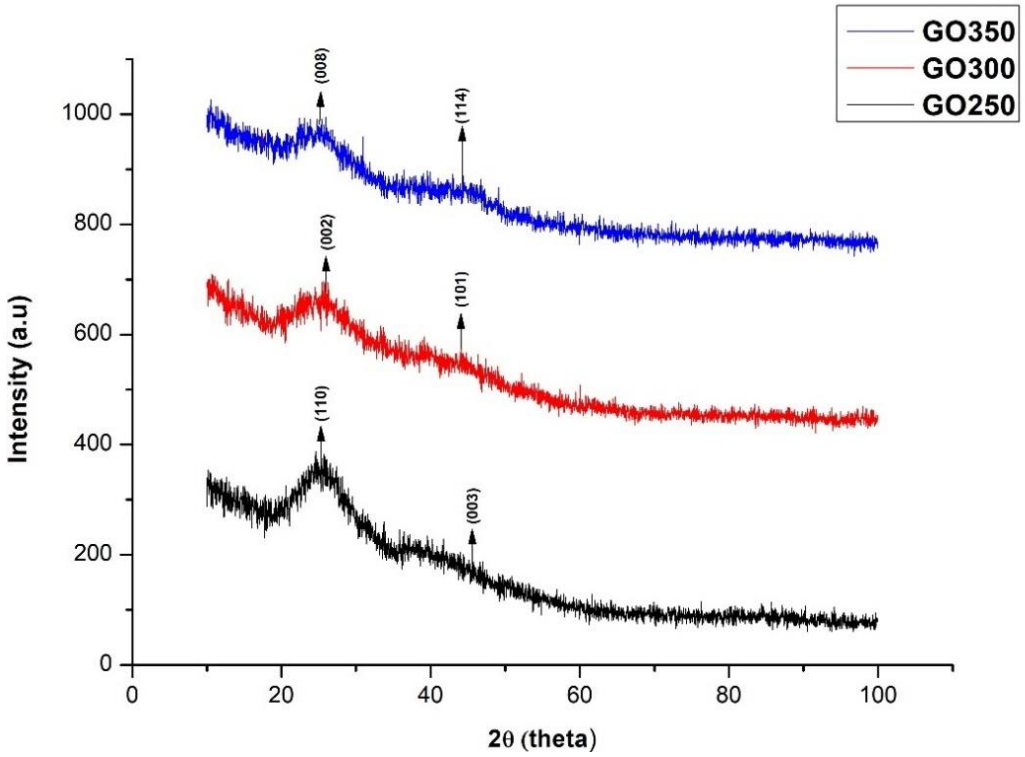


Fig 4. XRD result data from GO250, GO300, and GO350 samples

As can be seen in Figure 4, GO250 sample, it can be seen that there are two peaks at diffraction peaks of 25,798° and peaks 44,740° with d-spacing values obtained, namely 3.45060 and 2.02400, as well as the Miller index values contained in the peaks as associated with the emerging phase, namely (110) and (200). At the GO300 sample, it can be seen that there are two peaks at diffraction peaks of 25,689° and peaks of 44,281°, with the d-spacing values obtained are 3.46500 and 2.04390, and the Miller index values found on the peaks associated with the emerging phase are (002) and (101). At GO350 sample, it was seen that there were two peaks at diffraction peaks of 24.572° and peaks of 44.285° with the d-spacing values obtained, namely 3.62000 and 2.04370, and the Miller index values contained in the peaks associated with the emerging phase, namely (008) and (114). In the results of GO characterization using XRD, GO250 sample, GO300 sample, and GO350 sample, there is a desired phase, namely the GO phase, which refers to the previous study, namely GO made using graphite from waste found at the peak of 26.60° [8] and according to [5] explained that the GO obtained at the peak was around 44°. So, each variation of combustion temperature gives rise to different phases, which indicate that the combustion temperature affects the resulting GO.

GO testing using FTIR was carried out to find functional groups formed on GO. The results of FTIR characterization showed the relationship between wavenumber and transmittance, as shown in Figure 5.

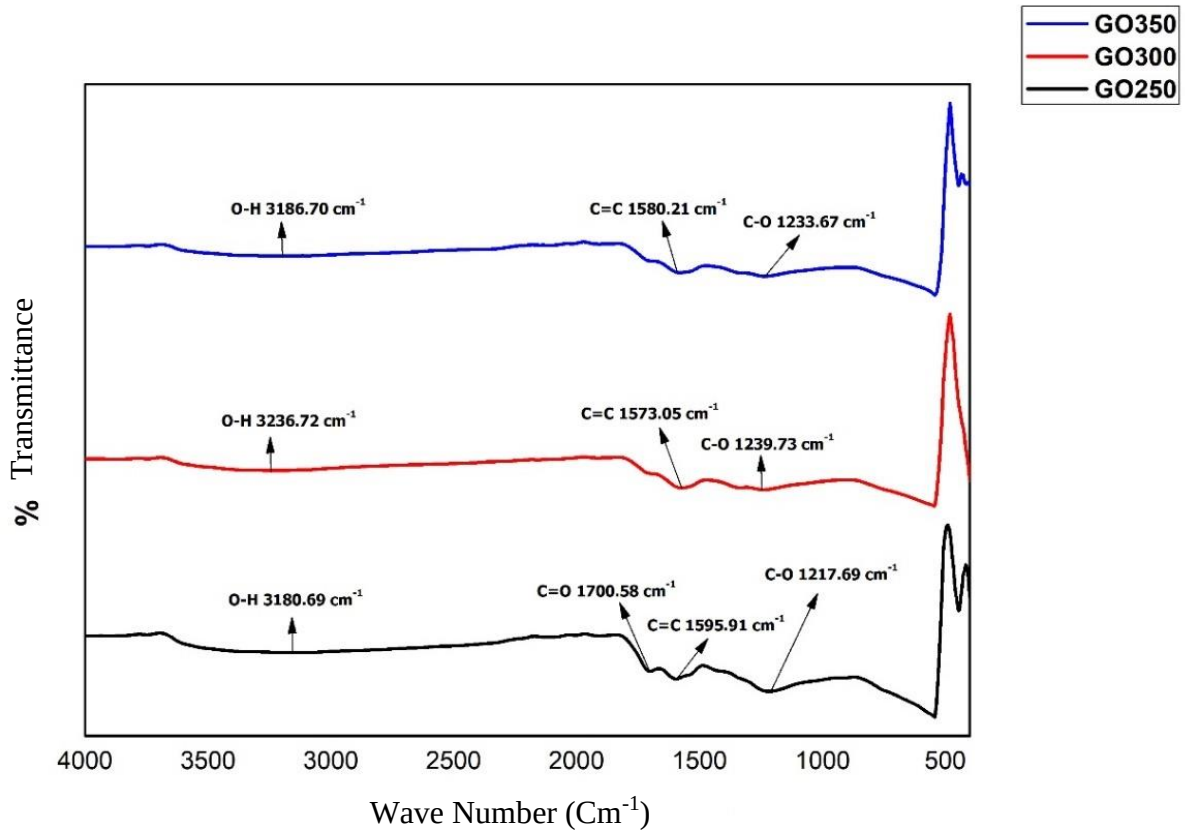


Fig 5. FTIR result data from GO250, GO300, and GO350 samples

As shown in Figure 5, the GO250 sample has four GO functional groups. The CO functional group with a wavenumber of 1217.69 cm-1, the C=C functional group with a wavenumber of 1595.91 cm-1, the OH functional group with 3180.69 cm-1, and the C=O functional group at a wavenumber of 1700,58 cm-1. GO300 sample has three GO functional groups. The CO functional group with a wavenumber of 1239.73 cm-1, the C=C functional group with 1573.05 cm1, and the OH functional group with 3236.72 cm1. In the GO350 sample, GO has three functional groups. The C-O functional group at wave number 1233.67 cm-1, the C=C functional group at wave number 1580.21 cm-1, and the O-H functional group at wave number 3186.70 cm-1. Therefore, in general, all-temperature changes include the GO functional group. The peak value of the spectrum at a specific wave number indicates the amount of material absorption. The lower and more prolonged the peak value of the spectrum, the higher the absorption value of the material at the wave number will be, and vice versa. The smaller the peak of the spectrum, the weaker the absorption of the material at that wave number [17].

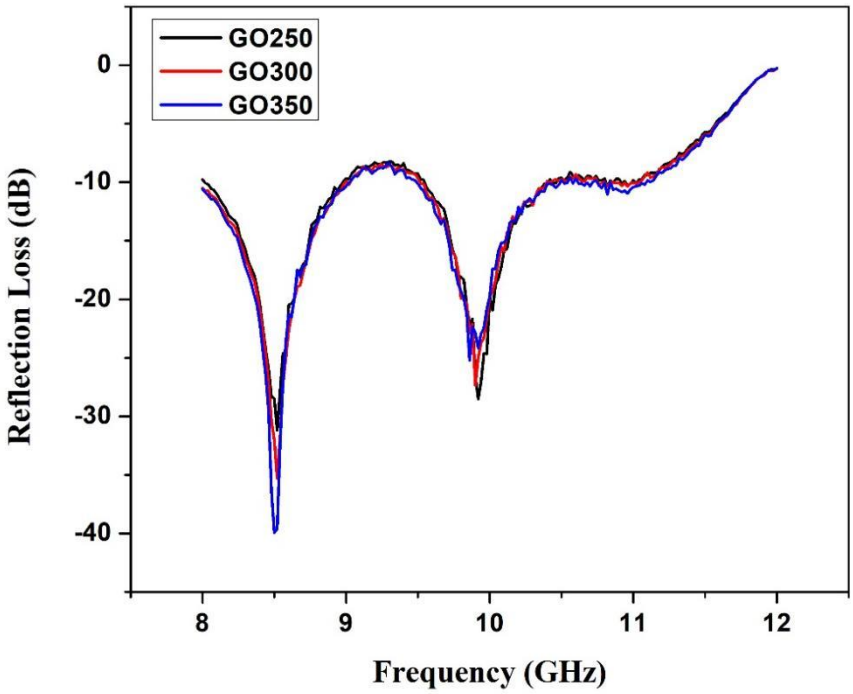


Fig 6. VNA result data from GO250, GO300, and GO350 samples

Figure 6 shows the reflection loss of sample GO250, GO300, and GO350. As shown in Figure 6, A deep valley indicates a good absorption on the curve and a small reflection attenuation value. The figure shows the reflected attenuation values for three changes in GO combustion temperature at specific frequencies. GO250 sample, the reflection loss value is 31.19 dB at a frequency of 8.52 GHz. For the GO300 sample, the reflection loss value is 35.28 dB at a frequency of 8.52 GHz. For the GO350 sample, the reflection loss value is 39.95 dB at a frequency of 8.5 GHz. The results obtained show that the reflection loss value and the adsorption value received vary depending on changes in the GO sintering temperature. The minimum reflection loss value is -39.95 dB for the GO350 sample, and the best absorption is the GO350 sample. The maximum reflection loss value is -31.19 dB for the GO250 sample, and the lowest absorption is the GO250 sample. The value of the coefficient absorption of each variation of GO can be seen in Table 1.

Table 1. GO Absorption Value

No.	GO Variations (°C)	Frequency (GHz)	Reflection Loss (dB)	Absorption coefficient (%)
1	250	8.52	-31.19	97.20
2	300	8.52	-35.28	98.28
3	350	8.50	-39.95	99.00

The smallest absorption coefficient value is the maximum reflection loss value (-31.19 dB). The minimum absorption coefficient value is 97.2% on the GO250 sample, while the largest is the minimum reflection loss value (-39.95 dB) on the GO350 sample. The highest absorption coefficient value is 99% on the GO350 sample. So, the relationship between reflection loss and the absorption coefficient is inversely proportional. The smaller the reflection loss value, the greater the absorption coefficient, and vice versa. The larger the reflection loss value, the smaller the absorption coefficient.

In addition to having a minimum reflection loss value and a deep curve valley, materials with good absorption power have a broad absorption band. The GO absorption bandwidth value for each variation can be seen in Table 2.

Table 2. GO Absorption Bandwidth

No.	GO Variations (°C)	Frequency (GHz)	Reflection loss (dB)	Absorption tape (GHz)	Absorption bandwidth (GHz)
1	250	8.52	-31.19	8.49-8.54	0.05
2	300	8.52	-35.28	8.49-8.54	0.05
3	350	8.50	-39.95	8.47-8.53	0.06

The minimum absorption band value is the maximum reflection loss value (-31.19 dB). The minimum absorption band value is 0.05 GHz. The minimum absorption band is 0.05 GHz on the GO 250 sample and GO300 sample. At the same time, the maximum absorption band is 0.06 GHz on the GO 350 sample. So, the relationship between the reflection loss value and the absorption bandwidth is inversely proportional. The smaller the reflection loss value, the wider the absorption band, and vice versa. The greater the reflection loss value, the minimum the absorption band. This is because a material will be able to absorb microwaves in a wide frequency range [16]. Not only the parameters of reflection loss values, absorption coefficients, and absorption band widths that affect the absorption of a material, but also the density and thickness of a material also affect its absorption [18]. The denser a material, the greater the absorption, because the distance between material grains is getting closer (the arrangement of grains is tight) so that there is no empty space that allows waves to be transmitted. Likewise, the thickness of the material will also affect its absorption so that the power transmitted will be smaller. Therefore, the density and thickness of the material must be the same so as not to affect its absorption [18].

IV. CONCLUSION

Based on the research results it was concluded that GO could be synthesized from rice husk waste using the modified Hummer method with changes in carbonization temperature at 250 °C, 300 °C, and 350 °C. XRD GO300 sample test results contain the GO phase with diffraction peaks of 25,689° and peaks of 44,281°, while for GO250 sample, it contains diffraction peaks of 25,798° and peaks of 44,740°, and the GO350 sample, it contains diffraction peaks of 24,572° and peaks 44,285°. FTIR test results, GO from rice husk has functional groups OH, CO, C = O, and C = C. The VNA test results which have the best microwave absorption characteristics are the GO350 sample because it has a bit reflection loss value (39.95dB), a significant absorption coefficient (99%), and a broad absorption band (0.06GHz).

REFERENCES

[1] P. Utomo and I. Yunita, "Sintesis Zeolit dari Abu Sekam Padi Pada Temperatur Kamar," pp. 1–39, 2014.

[2] S. Herodian, "Peluang dan tantangan industri berbasis hasil samping pengolahan padi," *Pangan*, no. 48, pp. 38–49, 2007.

[3] G. Supriyanto *et al.*, "Graphene oxide from Indonesian biomass: Synthesis and characterization," *BioResources*, vol. 13, no. 3, pp. 4832–4840, 2018, doi: 10.15376/biores.13.3.4832-4840.

[4] P. Ranjan, S. Agrawal, A. Sinha, T. R. Rao, J. Balakrishnan, and A. D. Thakur, "A Low-Cost Non-explosive Synthesis of Graphene Oxide for Scalable Applications," *Sci. Rep.*, vol. 8, no. 1, pp. 1–13, 2018, doi: 10.1038/s41598-018-30613-4.

[5] L. Stobinski *et al.*, "Graphene oxide and reduced graphene oxide studied by the XRD, TEM and electron spectroscopy methods," *J. Electron Spectros. Relat. Phenomena*, vol. 195, no. March 2018, pp. 145–154, 2014, doi: 10.1016/j.elspec.2014.07.003.

[6] N. I. Zaaba, K. L. Foo, U. Hashim, S. J. Tan, W. W. Liu, and C. H. Voon, "Synthesis of Graphene Oxide using Modified Hummers Method: Solvent Influence," *Procedia Eng.*, vol. 184, pp. 469–477, 2017, doi: 10.1016/j.proeng.2017.04.118.

[7] S. N. Alam, N. Sharma, and L. Kumar, "Synthesis of Graphene Oxide (GO) by Modified Hummers Method and Its Thermal Reduction to Obtain Reduced Graphene Oxide (rGO)*," *Graphene*, vol. 06, no. 01, pp. 1–18, 2017, doi: 10.4236/graphene.2017.61001.

[8] Y. Zhu *et al.*, "Graphene and graphene oxide: Synthesis, properties, and applications," *Adv. Mater.*, vol. 22, no. 35, pp. 3906–3924, 2010, doi: 10.1002/adma.201001068.

[9] Y. Rafitasari, H. Suhendar, N. Imani, F. Luciana, H. Radean, and I. Santoso, "Sintesis Graphene Oxide and Reduced Graphene Oxide," 2016, doi: 10.21009/0305020218.

[10] L. T. Sutayasa, "Karakterisasi Graphene Arang Ampas Tebu Berbasis X-Rd Dan Tem," *UNESA J.*

Chem., vol. 5, no. 3, pp. 23–27, 2016.

[11] N. Syakir, R. Nurlina, S. Anam, A. Aprilia, and S. Hidayat, "Kajian Pembuatan Oksida Grafit untuk Produksi Oksida Grafena dalam Jumlah Besar," *J. Fis. Indones.*, vol. XIX, no. November, pp. 26–29, 2015.

[12] I. Wandira, P. Karo Karo, and W. A. Adi, "Material Absorber Gelombang Elektromagnetik Berbasis (La0.8Ba0.2)(Mn(1-x)/2ZnxFe(1-x)/2)O3(x = 0 – 0,6)," *J. Teor. dan Apl. Fis.*, vol. 06, no. 01, pp. 1–66, 2018.

[13] N. Ardianti, Y. Darvina, F. U. Jhora, and R. Ramli, "Microwave Absorption Properties of Graphene Oxide Derived from Coconut Shell Waste by Modified Hummer ' s Method," vol. 15, no. 2, pp. 88–95, 2022.

[14] M. Ikhsan and R. Ramli, "Measurements and analysis of crystal structures of activated carbon of empty fruit bunch from oil palm biomass waste," *J. Phys. Conf. Ser.*, vol. 1528, no. 1, pp. 0–4, 2020, doi: 10.1088/1742-6596/1528/1/012031.

[15] M. Hapsari, A. H. Cahyana, S. H. Oktavia, and A. R. Liandi, "Synthesis of spirooxindole-pyrrolizidine compounds using fe3o4-go catalyst and their bioactivity assays," *Rasayan J. Chem.*, vol. 13, no. 4, pp. 2317–2324, 2020, doi: 10.31788/RJC.2020.1345583.

[16] J. Guerrero-Contreras and F. Caballero-Briones, "Graphene oxide powders with different oxidation degree, prepared by synthesis variations of the Hummers method," *Mater. Chem. Phys.*, vol. 153, pp. 209–220, 2015, doi: 10.1016/j.matchemphys.2015.01.005.

[17] O. Gustira, Y. Darvina, and Ramli, "Pengaruh Komposisi $MnFe_2O_4$ Terhadap Sifat Listrik Nanokomposit MnFe2O4 / PVDF yang Disintesis dengan Metode Spin Coating," *Pillar Phys.*, vol. 13, no. April, pp. 59–66, 2020.

[18] M. I. Ramadhan, W. Widanarto, and S. Sunardi, "Pengaruh Temperatur Sintering Terhadap Struktur dan Sifat Magnetik Ni2+- Barium Ferit sebagai Penyerap Gelombang Mikro," *J. Teras Fis.*, vol. 1, no. 1, p. 23, 2018, doi: 10.20884/1.jtf.2018.1.1.567.