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Microwave Absorption Properties of Fe_3O_4/Graphene Oxide Nanocomposites Synthesized from Coconut Shell Waste

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Abstract: Electromagnetic wave absorption technology is one of the technologies that need to be developed to control the problems caused by Electromagnetic Interference (EMI). This technology has created a material called Radar Absorbing Material (RAM) that Making RAM can be formed with material modifications such as nanocomposite materials. The manufacture of Fe_3O_4 nanocomposites and Graphene Oxide (GO) synthesized from coconut shell waste is one of the studies that need to be studied and developed at this time. Variations in the composition of Fe_3O_4 and GO used can determine the ability of microwave absorption. This study aims to analyze the microwave absorbing properties of Fe_3O_4/GO nanocomposites from coconut shell waste. The materials used to make this nanocomposite are magnetite (Fe_3O_4) and GO synthesized from coconut shell waste. This research uses experimental methods, with variations in the composition ratio between Fe_3O_4 and GO, namely 20% Fe_3O_4: 80% GO, 30% Fe_3O_4: 70% GO, and 40% Fe_3O_4: 60% GO. Characterization was carried out using a Vector Network Analyzer (VNA) to determine the nature of microwave absorption. The VNA characterization results found the influence of variations between Fe_3O_4 and GO composites. Based on the research results, the lowest absorption coefficient percentage was 95.50% at a frequency of 8.38 GHz for the composition variation of 20% Fe_3O_4 and 80% GO. The highest absorption coefficient was 96.43% at a frequency of 8.42 GHz for the composition of 40% Fe_3O_4 and 60% GO. The addition of Fe_3O_4 can make the absorption value of the material to microwaves higher.

Keywords: Graphene Oxide; Magnetite; Microwave Absorber; Nanocomposite; RADAR.

1. Introduction

RADAR *(Radio Detection and Ranging)* is a tool or facility to detect, measure distance and map objects such as aircraft, motor vehicles and weather information using electromagnetic wave

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emission. With the development of RADAR technology, the electromagnetic radiation generated increasingly affects the operational functions of electronic devices. In the military field, it requires the development of technology systems to reduce RADAR detection. Stealth technology systems need to be developed. Stealth technology is a system that is usually implemented on fighter aircraft that has the ability to avoid detection [1]. The technology has given birth to a material, namely RAM *(Radar Absorbing Material)*. RAM can absorb electromagnetic energy and convert it into heat energy [2]. RAM is a coating method applied to reduce the energy of Electromagnetic waves received by the target. RADAR operating frequencies range from 3 MHz to 300 GHz. The main groups for remote surveillance, military, aircraft, and space applications include L band (1-2 GHz), S band (2-4 GHz), C band (4-8 GHz), X band (8-12 GHz), Ku band (12-18 GHz), K band (18-24 GHz), Ka band (24-40 GHz) [3]. RAM has been made in various forms of material modifications such as nanocomposite materials. Nanocomposite material consists of two components, namely matrix and nano-sized filler.

Graphene Oxide (GO) is one of the basic materials used as RAM. Graphene Oxide has proven to be a good precursor material for the manufacture of graphene-based materials in large quantities due to its cheap and easily scalable synthesis process [4]. Graphene Oxide can be synthesized using coconut shell waste [5]. According to data from Indonesia's Central Bureau of Statistics (BPS) in 2022, coconut production in Indonesia amounted to 2.87 million tons. Coconuts produce meat and waste in the form of coconut shells. Coconut shell has lignin, cellulose and hemicellulose, so coconut shell can be utilized to create activated carbon for absorption capacity [6]. The method used to make graphene oxide is the modified hummer method. The modified Hummers method is one of the most popular methods of Graphene Oxide synthesis stages used because it provides advantages in its use, namely it can increase the oxidation rate. The advantages of the modified hummers method are that during the oxidation process it does not emit ClO_2 gas, the oxidation process is faster with lower temperatures and the materials used in this method are easily available and less dangerous than other methods [7].

Magnetic materials are very complex materials. Magnetic can be applied in various criteria such as electrical, magnetic and optical properties [8]. Iron oxides such as magnetite Fe_3O_4 are one of the nanometer-sized magnetic particles, the smaller the grain size, the Fe_3O_4 will have a high magnetic responsiveness. Particle size, surface properties and magnetic properties are the advantages of magnetite nanoparticles so that they are very easily modified with other materials [9]. The advantages of magnetite are that it is environmentally friendly, cheap, easy to obtain, and can increase adsorption power because magnetite also has good adsorption power [10].

Some research has been done to make microwave-absorbing composite materials or RAM. In the research of Ma, *et al.* 2013, Fe_3O_4 particle coating on graphene can improve impedance match characteristics, reduce surface reflection and benefit absorption. In Linda's 2012 research, combining Fe_3O_4 with PAni as a microwave absorbing material. In Ardianti's 2022 research, it shows that graphene oxide from coconut shell waste is used as a material that has absorption of microwaves, graphene oxide when it is at 350°C has a small reflection loss value of -24.30 dB, a large absorption coefficient of 93.83%. Based on research conducted by Mashuri 2012 on microwave absorbing materials using Fe_3O_4. Fe_3O_4 material as a typical magnetic interference absorber is most often researched mainly because of its excellent magnetic properties, natural soft metal, large magnetic anisotropy, good biocompatibility, and low toxicity.

Until now, the use of stealth technology on fighter aircraft is still not perfect, this is because fighter aircraft are still detected by enemy RADAR and the development of Magnetic Graphene Oxide (GO-Fe_3O_4) nanocomposite materials is still very limited because they generally use synthetic graphite powder which requires very expensive costs. Therefore, it is necessary to develop low cost adsorbents based on natural materials [11]. The mixing of Fe_3O_4 nanocomposites and graphene oxide seen from several research sources will be able to produce materials that have a large absorption ability to microwaves. In this study, graphene oxide and Fe_3O_4 materials were used from research conducted by Damayani, et al 2024 [12]. which was in accordance with the requirements. The graphene oxide used forms a diffraction peak at an angle of 2θ which is 24.94° Then in the Fe_3O_4/Graphene Oxide nanocomposite sample the formation of several diffraction peaks at angles of 30.03°, 35.40°, 43.01°, 53.45°, 56.95° and 62.52° with miller indices (220), (311), (400), (422), (511) and (440) This XRD pattern shows all Fe_3O_4 nanoparticle peaks as reported in the literature [13]. In FTIR testing, the graphene oxide used has formed peaks with O-H, C-H, C=O, C=C and C-O functional groups. In addition, the Fe_3O_4/Graphene Oxide nanocomposite sample used formed a new peak with the Fe-O functional group with a wave number of 559.99 cm^{-1}. The Fe-O functional group indicates that there is Fe_3O_4 in the nanocomposite sample. The FTIR spectrum for iron oxide compounds is located in the low frequency region of 1000-500 cm^{-1} [14]. Based on the above description, this article aims to discuss the microwave absorption properties of Fe_3O_4 composites with Graphene Oxide synthesized from coconut shell waste.

2. Materials and Method

The method used in this research was the experimental method with literature study. The research was conducted at the Physics Laboratory and Chemistry Laboratory of Universitas Negeri Padang, LLDIKTI Region X Padang Laboratory and Telecommunication Laboratory at Politeknik Negeri Padang. This study uses independent variables, namely Fe_3O_4 and Graphene Oxide from old coconut shell waste, with variations in the ratio of 20%: 80%; 30% : 70%; and 40%: 60%. In this study using control variables, namely Fe_3O_4 and graphene oxide synthesized using the modified hummer method and mixing of Fe_3O_4/Graphene Oxide composites was carried out using a ball mill with a speed of 300 rpm for 30 minutes. Graphene oxide was synthesized with a furnace temperature of 350 ° C with a time of 120 minutes. The process of making graphene oxide consists of several stages, namely the preparation of coconut shell samples, carbon activation stage, graphene oxide synthesis stage, sonication and neutralization of graphene oxide. The dependent variable in this study is the microwave absorption of Fe_3O_4/Graphene Oxide nanocomposite. Graphene oxide is henceforth given the initials GO.

The first stage of sample preparation started with cleaning the coconut shell from the husk. The coconut shells were dried in the sun for 3 days and cut into small pieces. Then, the old coconut shell was baked at 100˚C for 60 minutes, followed by a furnace process for 2 hours at 350˚C. After becoming charcoal, pounded with a mortar and pestle to produce charcoal powder and sieved using a 125 mesh sieve. In the second stage, the shell powder was dissolved with NaOH solution. NaOH solids are dissolved using 100 mL of distilled water and adding 8 g of NaOH solids into a volumetric flask, until the NaOH solids dissolve and the solution becomes homogeneous. Then, provide a 250 mL beaker and put 8 g of coconut shell charcoal powder and add NaOH solution. Soaking is done for 24 hours. After the soaking process, a precipitate is formed. This precipitate is then filtered using filter paper and use a Buchner funnel to facilitate the filtering process. After filtering, the resulting old coconut shell powder is transferred into a vaporizer cup. The activated coconut shell powder was dried using an oven at 105°C for 3 hours.

The third stage was the GO synthesis process using the modified Hummers method. Weighed 1.5 g of activated charcoal powder and 0.75 g of $NaNO_3$, then a 250 mL Erlenmeyer flask with a magnetic stirrer was prepared along with the charcoal powder and $NaNO_3$ Added H_2SO_4 98% as much as 34.5 mL. The mixture was stirred for 20 minutes at 0-5°C with a constant speed of 250 rpm. Placed the erlenmeyer in an ice bath and stirred for 2 hours on a hot plate. 4.5 g $KMnO_4$ powder was added slowly to prevent the mixture from exploding and the synthesized coconut shell powder from decreasing and to keep the temperature below 20°C. After adding $KMnO_4$ to the mixture, removing the ice bath and stirring the mixture at 35°C for 30 min. so that the oxidation process can take place completely. This process was carried out until the color of the solution became pale brown. Adding 69 mL of distilled water slowly using a dropper and stirring for 20 minutes. When adding distilled water, the temperature is kept below 50°C and the solution will be dark brown with bubbles. Adding 100 mL of water and followed by the addition of 30% H_2O_2 as much as 1.5 mL. The addition of H_2O_2 was done in order to remove the remaining $KMnO_4$ or to stop the reaction and the solution finally changed color to yellow which indicated the presence of GO. The last stage, adding 50 mL of distilled water. The stages of GO synthesis as shown in Figure 1.

(a) (b) (c)

Figure 1. (a) Sterring process of graphene oxide preparation (b) the result after KMnO4 addition (c) the synthesized GO

In Figure 1 above, it could be seen that the synthesis of GO was sterilized on a hot plate until the solution changed color to black because of the carbon content. After adding KMnO4 and stirring, the solution changed color to pale brown. Finally, the synthesis produced a solution that changed color to yellow, marked by the presence of GO. In the fourth stage, The GO solution was sonicated for 2 hours to exfoliate the graphite into graphene. Then, the solution was precipitated for 1 day so that the liquid and solid phases were formed. The centrifugation process used a microcentrifuge at 4000 rpm for 15 minutes to separate the solid and liquid phases. The centrifugation process was followed by a manual GO neutralization process, which involved precipitating GO powder and filling it with distilled water, then repeatedly replacing the distilled water until a neutral pH of 7 was obtained. After obtaining a neutral pH, graphene oxide was baked at 60°C for 12 hours. The stages of sonication and GO neutralization can be seen in Figure 2.

(a) (b) (c)

Figure 2. (a) Sonication process of GO solution (b) neutralization of the solution using distilled water (c) GO results after filtering

From Figure 2 above, it can be seen that GO is initially still in solution and then sonicated. After sonicating the solution precipitated and centrifuged. GO centrifugation results are neutralized using distilled water, after which it is dried in an oven so that GO is formed in powder form. The fifth stage, the preparation of Fe_3O_4/GO composites by mixing Fe_3O_4 and GO in the ratio of 20% Fe_3O_4 : 80% GO; 30% Fe_3O_4: 70% GO; and 40% Fe_3O_4: 60% GO. Mixing was done using a ball mill with a speed of 300 rpm for 30 minutes. The ball mill is used to homogenize the particle size. Fe_3O_4/GO nanocomposite material was characterized using a Vector Network Analyzer (VNA) to determine the value of material absorption to microwaves. The tool used is a KEYSIGHT E5071 ENA Series Network Analyzer with a frequency of 300 kHz - 20 GHz. The sample is inserted into a mold made of acrylic with a size of 3 cm x 1.5 cm with a thickness of 2 mm. The frequency value used is X-band frequency (8-12 GHz). Based on the VNA characterization data, the Reflection Loss (RL) value and frequency value will be known, then the electromagnetic wave absorption coefficient value will be sought using equations 1 and 2.

$$\mathrm{RL} = 20 \log |\mathrm{Z}| \quad (1)$$

$$\%\mathrm{absorpsi} = (1 - \mathrm{Z}) \times 100\% \quad (2)$$

Where, the RL (*Reflection Loss*) value is expressed in units of (dB) and Z is the normalized impedance.

3. Results and Discussion

Based on the implementation of the research that was carried out in accordance with the method used, the Fe_3O_4/GO nanocomposite sample was obtained with a variation of the ratio of 20% Fe_3O_4: 80% GO; 30% Fe_3O_4: 70% GO, and 40% Fe_3O_4: 60% GO. By characterizing with a VNA tool, the graph results showed the relationship between Reflection Loss (RL) on the Y axis and frequency on the X axis. The data obtained from testing the 20% Fe_3O_4: 80% GO nanocomposite sample can be seen in Figure 3.

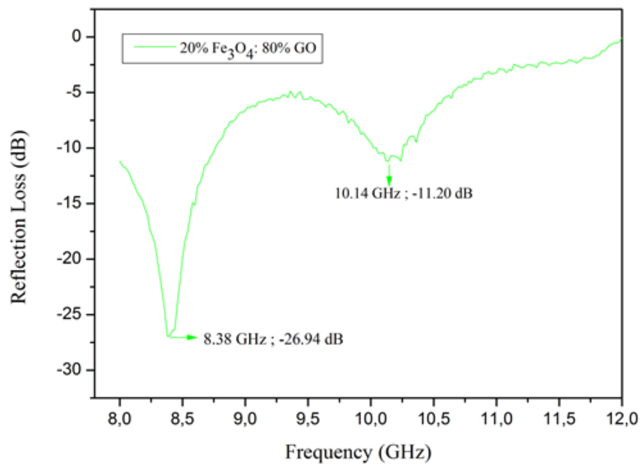

Figure 3. Graph of VNA characterization results of 20% Fe_3O_4 and 80% GO

Based on Figure 3 above, the VNA characterization results show that the use of Fe_3O_4/GO nanocomposite can produce RL value and frequency value. The test results show that there is absorption in two curve valleys with RL values of -26.94 dB at a frequency of 8.38 GHz and -11.20 dB at a frequency of 10.14 GHz. Analysis of the difference in RL values indicates the maximum absorption of microwave power at certain frequencies. At a frequency of 8.38 GHz there is maximum absorption of the sample characterized by the smallest RL value. Meanwhile, at a frequency of 10.14 GHz, the sample absorption of microwaves is less than maximum as seen from the results of a larger RL value. There are 3 possibilities that will occur when an incident wave hits the sample, namely the wave is absorbed by the absorber material, reflected (reflection) and transmitted (tranmission). The waves are all absorbed by the microwave absorbing material and none are transmitted. The percentage of waves absorbed by the material can be determined by calculating the RL value and will indicate the absorption ability of the material [15].

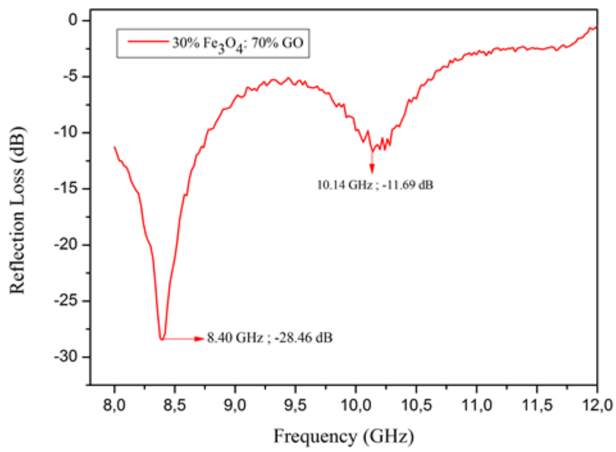

Figure 4. Graph of VNA characterization results of 30% Fe_3O_4 and 70% GO

Based on Figure 4 above, the VNA characterization results obtained 2 curve valleys, namely with an RL value of -28.46 dB with a frequency of 8.40 GHz and -11.69 dB at a frequency of 10.14 GHz. The analysis shows that the addition of Fe_3O_4 reduces the RL value of the sample. In the Fe_3O_4/GO nanocomposite sample with a ratio of 30% Fe_3O_4 and 70% GO which produces the maximum wave absorption at the reflection loss value of -28.46 dB at a frequency of 8.40 GHz. The change in RL value in this variation is quite significant compared to the variation of 20% Fe_3O_4 and 80% GO. Fe_3O_4 material can increase the minimum value of RL contained in the sample, so that the absorption ability will also increase.

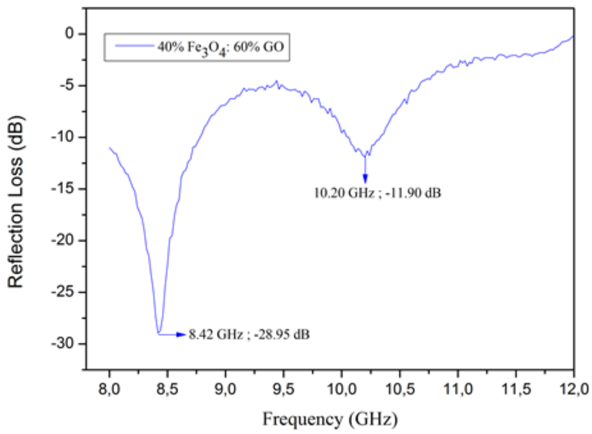

Figure 5. Graph of VNA characterization results of 40% Fe_3O_4 and 60% GO

Based on Figure 5 above, the VNA characterization results of Fe_3O_4/GO nanocomposites with a ratio of 40% Fe_3O_4 and 60% GO also show that the addition of Fe_3O_4 composition produces two curve valleys with RL values of -28.95 dB at a frequency of 8.42 GHz and -11.90 dB at a frequency of 10.20 GHz. Good absorption is characterized by low reflection values [16]. Comparative analysis of the RL values of the previous variation at 30% Fe_3O_4 and 70% GO does not differ too much. The addition of Fe_3O_4 linearly decreases the RL value and increases the frequency value. On the other hand, the reduction of GO in these results also shows a decrease in RL value and an increase in frequency value. The RL value is a value that shows the amount of electromagnetic energy lost after hitting a material, because the energy is absorbed by the material [16].

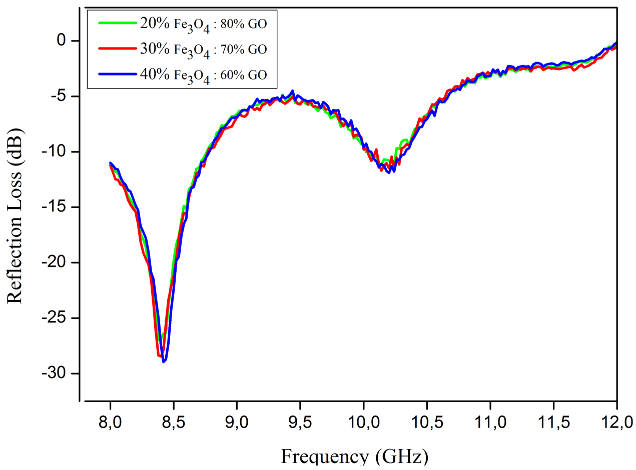

Figure 6. VNA characterization combined graph of Fe_3O_4/GO nanocomposite 20% Fe_3O_4 : 80% GO, 30% Fe_3O_4: 70% GO and 40% Fe_3O_4 : 60% GO

Based on Figure 6 above, a good wave absorption test is indicated by a deep curve valley and a small RL [16]. It can be seen the reflection loss value of the three variations of Fe_3O_4/GO nanocomposite with a certain frequency. In the comparison of 20% Fe_3O_4 and 80% graphene oxide, the lowest reflection loss value is -26.94 dB. In the comparison of 30% Fe_3O_4 and 70% GO, the lowest reflection loss value is -28.46 dB. Then in the comparison of 40% Fe_3O_4 and 60% GO the lowest reflection loss value is -28.95 dB. The addition of Fe_3O_4 affects the change in RL value. The greater the negative value of RL, the greater the value of microwave absorption material that impinges on it [17]. With the RL value, the absorption coefficient can be determined using equations 1 and 2. The absorption value of Fe_3O_4/GO with a ratio of 20%: 80%, 30% : 70% and 40%: 60% can be seen in Table 1.

Table 1. The absorption value of Fe_3O_4/GO with the ratio of 20%: 80%, 30% : 70% and 40% : 60%

Variation of Fe_3O_4 and GO (%)	Frequenzy (*GHz*)	Reflection Loss (*dB*)	Absorbing Coefficient (%)
20% : 80%	8.38	-26.94	95.50
30% : 70%	8.40	-28.46	96.22
40% : 60%	8.42	-28.95	96.43

Based on Table 1 above, the lowest microwave absorbing coefficient value is at a large reflection loss, namely in the variation of the ratio of 20% : 80% with an absorption coefficient of 95.50% and the highest wave absorbing coefficient is at the smallest RL value, namely in the variation of 40% Fe_3O_4 : 60% GO with an absorption coefficient of 96.43%, this result is better than the research conducted by Ardianti, et al 2022, which produced an absorption coefficient of 93.83% with GO samples from coconut shells without additional Fe_3O_4 [5]. From the VNA test results, there is a decrease in the reflection loss value when adding Fe_3O_4, thus making the absorption coefficient value also increase. The advantages of Fe_3O_4 can increase the adsorption power because Fe3O4 also has good adsorption power [9]. The addition of Fe_3O_4 in the variation of 30% Fe_3O_4 : 70% GO compared to 40% Fe_3O_4 : 60% GO, the effect of the addition is not too significant, the reflection loss value is almost close to -28.46 dB and -28.95 dB. This can be influenced by the composition of GO which is also reduced.

The absorbency of a material is not only influenced by the parameters of RL value, absorption coefficient, and absorption bandwidth. However, the density and thickness of a material also affect its absorbency. The denser a material is, the greater its absorption capacity will be, due to the closer distance between the material grains (tight grain arrangement) so that there is no empty space that allows waves to be transmitted. Conversely, the more tenuous the arrangement of a material, the smaller the absorption capacity will be. Thickness also affects microwave absorption where the thicker the absorbent material can reduce the matching frequency value and increase absorption which is characterized by the smaller the maximum reflection loss value [18]. Based on the data above, it can be seen that each addition of the composition of Fe_3O_4 makes the RL value of a sample smaller, thus making the microwave absorption ability of a sample also greater. This can be seen from the increase in the absorption coefficient value of each sample. In the three variations of the composition ratio of Fe_3O_4 and GO, it can be said that all materials can absorb waves well. When the RL is less than -10 dB, microwave absorbing materials can absorb 90% of electromagnetic waves, which meets the requirements for practical applications [19]. The results of this study show that the addition of Fe_3O_4 as a GO composite material makes the microwave absorption value higher.

4. Conclusion

Based on the findings, it can be concluded that Fe_3O_4/GO nanocomposites exhibit excellent microwave absorption properties, making them suitable for practical applications in stealth technology. The incorporation of Fe_3O_4 into the GO matrix significantly influences the microwave absorption characteristics, where increasing the Fe_3O_4 content leads to a reduction in reflection loss and, consequently, an enhancement in absorption efficiency. The addition of Fe_3O_4 as a composite component in the GO matrix notably improves the microwave absorption performance. The optimal absorption was achieved in the nanocomposite sample with a composition of 40% Fe_3O_4 and 60% graphene oxide, reaching an absorption capacity of 96.43%.

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