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Microwave absorption properties of Fe_3O_4/PANi nanocomposites synthesized by sol-gel methods

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Abstract. The technological advances in the radar field are currently growing. So that it requires an absorbent material that has a high absorption capacity so that it can be applied as Radar Absorbent Material (RAM). The RAM made in this study is nanocomposite material which consists of magnetic material based on natural iron sand Fe_3O_4 and PANi conductive polymer. Fe_3O_4/PANi nanocomposite was made using the sol-gel method with a percentage combination of Fe_3O_4/PANi 30%, 40%, 50%, 60% and 70%. Fe_3O_4/PANi was characterized using XRD and VNA. At the culminate of XRD characterization, it was exposed that successfully synthesized Fe_3O_4/PANi nanocomposite using the sol-gel method. Fe_3O_4/PANi is a material that can be used as a microwave absorber, this is indicated by the results of characterization using VNA. In the C-Band (4-8 GHz) the maximum reflection loss is found in Fe_3O_4/PANi samples (40%) of -28,674 dB at a frequency of 6 GHz by a coefficient absorption of 0.0368 and absorption percentage of 96.32%. Whereas in the X Band (8-12 GHz) maximum reflection loss was obtained in the Fe_3O_4/PANi sample (60%) of -24.3533 dB at a frequency of 11.76 GHz, by a coefficient absorption is 0.0605 and a absorption percentage of 93.94%.

1. Introduction

At present, in the main defense fields in the field of military and governance, radar applications for the coordination of a target are increasing. In the world of military defense, radar is used to protect enemy targets such as stealth defense technology on aircraft and warships. Ship technology is a technology in which ships cannot be detected by radar. So this technology is very dependent on a lot of electromagnetic energy that is reflected back to the recipient by the target. Therefore we need a material that can absorb microwaves properly.

Microwave absorbent material can be used as a component in making RAM. Where this RAM is a material that can be used to detect the position of an object using microwaves or radio waves. Wave absorbent material must be material that has the ability to minimize reflections from electromagnetic waves, and must have good magnetic properties and electrical properties. One material that is a very good choice for use as a magnetic absorbent material in microwaves is iron sand [1].

Magnetite Fe_3O_4 nanoparticles are materials that have the ability to absorb microwaves [2]. Nano-size Fe_3O_4 has the advantage of responding to external magnetic fields compared to other compounds. Where on the nanoscale Fe_3O_4 has a small coercivity field resulting in a decrease in barrier energy. This is what causes the nanoscale Fe_3O_4 to absorb microwaves. Fe_3O_4 nanoparticles used in this study have been reported in previous studies [3, 4]. While research on Fe_3O_4 as a microwave absorber has been reported [5], by using a chemical oxidation polymerization method and mechanical nanocomposite manufacture.

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RAM can be made in various forms of material modification, such as nanocomposite. Nanocomposite material is a material consisting of two components, namely matrix and filler material that are less than 100 nm in size. Fe_3O_4 based nanocomposites have been previously reported in research [6, 7], that use PVDF polymers as nanocomposite matrices. To improve the quality of RAM, a conductive polymer material can be used which can be made in the form of polymer nanocomposite.

PANi is a conductive polymer material that can maximize absorption of electromagnetic waves consisting of electrical and magnetic fields [8]. PANi is obtained through aniline monomer polymerization process. PANi has unique properties, one of which is good thermal stability and very high conductivity at micro frequencies, so PANi is more conductive than other polymers. In addition, PANi is also an easy polymer to be synthesized both electrochemically and chemically [9, 5]. By combining Fe_3O_4 and PANi material as radar absorbent materials, it will be able to increase the material absorption power of radar waves. This is because PANi is a polymer that can reduce reflection and can absorb electromagnetic wave radiation.

Characterization tool that can be used to measure the amount of microwave absorption of a material in the frequency range 10 MHz to 110 GHz, namely Vector Network Analyzer (VNA). In this study the characterization of absorption of nanocomposite waves was measured in the frequency range 0 - 12 GHz. Where at frequency of 4-8 GHz is the C - Band range, while on the 8-12 GHz frequency area is the X-Band range. In general, each range has certain characteristics with different uses. The X-Band range is an area that can be used for microwave operations on aircraft in the military field.

2. Experimental

This type of research is experimental research. To make Fe_3O_4/PANi nanocomposite, the material used was Fe_3O_4 taken from natural iron sand, and the polymer used was PANi conductive polymer. Iron sand taken from nature was previously purified using permanent magnets by pulling 30 times, then washing and drying, this was previously reported in the study [3, 10, 11]. Next to make magnetite nanoparticles, iron sand is milled for 30 hours using the High Energy Milling Ellipse 3 Dimension (HEM-E3D) tool. Then the iron sand is ready to be milled, washed and dried again

The method used to make nanokomposite Fe_3O_4/PANi is the sol-gel method. The process of making Fe_3O_4/PANi nanokomposite is by making Fe3O4 sol-gel first. Fe_3O_4 sol-gel was made by mixing iron Fe_3O_4 which has been extracted as much as 3.48 gr into oxalic acid as much as 4.5 g and nitric acid as much as 42 ml, at a temperature of 110^0C for 15 minutes. Next ethylene glycol add to into solution and stirring it using a magnetic stirrer riffed alongside a hot plate. The stirrer process is carried out at a condition of 80^0C to 2 hours, so that a magnetite gel is formed. Then make PANi with aniline polymerization process by dissolving PANi as much as 0.93 gr and 1.15 ammonium persulfate into 100 ml nitric acid solution. Then it combines the percentage content of Fe_3O_4/PANi in the nanokomposite, by mixing magnetite gel and PANi using a magnetic stirrer until the solution is homogeneous. After being homogeneous, the nanokomposite was dried using an oven at 110^0C for 24 hours. Then in the furnace at 350^0C for 3.5 hours. The percentage variation of Fe_3O_4/PANi used in this study was as much as Fe_3O_4/PANi 30%, 40%, 50%, 60% and 70%. Fe_3O_4/PANi nanocomposite was characterized using the X-Ray Diffraction (XRD) and Vector Network Analyzer (VNA) tools for see the absorption characteristics from the microwaves.

3. Results and discussion

Based on the results from characterization using XRD, it was shown that Fe_3O_4/PANi nanokompsoite was successfully used using the sol gel method, this was shown by the presence of Fe_3O_4 and PANi peaks on the X-ray diffraction pattern in each variation of the addition of Fe_3O_4/PANi composition used. The results of XRD Characterization are displayed at Figure 1.

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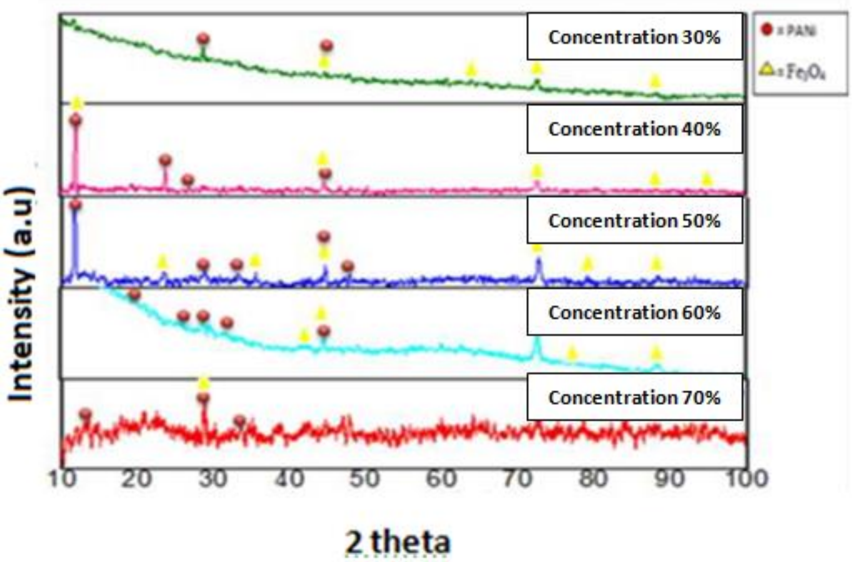

Figure 1. X-Ray Diffraction Patterning from Nanokomposite Fe_3O_4/PANi for Concentration Variations 30%, 40%, 50%, 60% and 70%

XRD diffraction peaks showed that the peak of Fe_3O_4/PANi was present in nanocomposite which was made using the sol-gel method in all variations of the concentration used. Where the results above also show that throes a change on the intensity of the crystal field orientation at Fe_3O_4/PANi nanocomposite caused by increasing the fraction of the concentration of nanocomposite used. From the XRD data above, we can know the size from the crystal diameter (D) of nanocomposite Fe_3O_4/PANi with applying the Scherrer equation.

$$D = K\frac{\lambda}{B\cos\theta_B} \qquad (1)$$

where λ (lamda) is a X-Ray wavelength, B is FWHM, θ_B is the Bragg diffraction angle and K is a constanta (~0,9). By using this value from FWHM, the size of the crystallite from a nanocomposite can be determined.

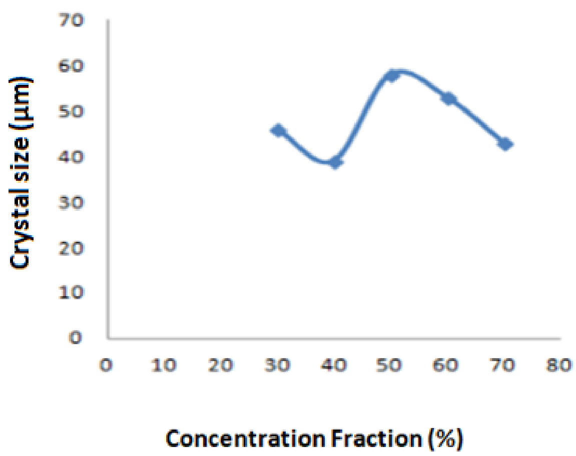

Figure 2. Graph of relationship between the fraction of Fe_3O_4/PANi nanocomposite concentration to the crystal size

Based on the Figure 2, it bottle be detect that in general throes a drop off at the size of the crystals by rising the percentage concentration of Fe_3O_4 in Fe_3O_4/PANi nanocomposite. From 30% to 40%, the size of Fe_3O_4/PANi nanocomposite crystals decreased from 46 nm to 39 nm. However, at a concentration of 50% the size of the crystallite has increased, and at 50%, 60% and 70%, the crystal size has been relatively continuously decreasing, which is 58 nm, 53 nm and 43 nm.

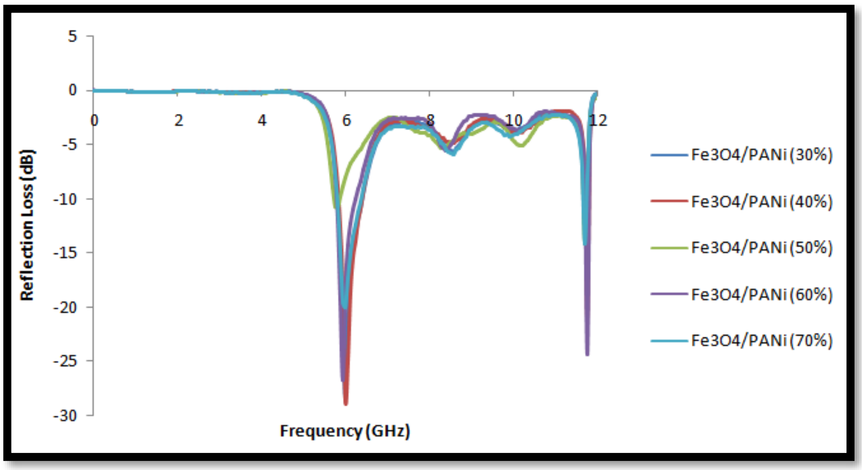

Figure 3. Results of Characterization VNA of Nanokomposite Fe_3O_4/PANi

Figure 3 shows the magnitude of the reflection loss value of Fe_3O_4/PANi nanokomposite 30%, 40%, 50%, 60% and 70% of the frequency in the range 0 - 12 GHz. Absorption that occurs is shown by the appearance of absorption valleys in the picture above. Where the depth and width of the absorption valley is formed, the better the absorption ability of the material.

Reflection loss is a quantity that defines the loss of electromagnetic waves after having hit a material (the amount of energy absorbed). A good radar wave absorbent material is a material that not only has a large reflection loss value, but also has a wide absorption band. In addition, the more absorption valleys are formed, the better the material is used as RAM material. This is because more and more frequency absorption ranges from the material.

Table 1. Reflection Loss (RL) maximum value of Fe_3O_4/PANi nanocomposite

Frequency	30%		40%		50%		60%		70%	
	RL Max (dB)	A (%)	RL Max (dB)	A (%)	RL Max (dB)	A (%)	RL Max (dB)	A (%)	RL Max (dB)	A (%)
C-Band (4 - 8 GHz)	-28.41	96.20	-28.67	96.32	-10.83	71.28	-26.74	95.39	-20.06	90.07
X-Band (8 - 12 GHz)	-12.56	76.46	-20.98	91.07	-14.14	80.38	-24.35	93.94	-14.11	80.31

From the Table 1, can see the maximum reflection loss value of each Fe_3O_4/PANi concentration. The maximum reflection value in the C-Band range was found in Fe_3O_4/PANi nanocomposite with a concentration of 40% at -28.6736 with a percentage of absorption of 96.32%. Whereas the maximum reflection loss range of X-Band obtained is -24.3533 dB in nanocomposite with 60% Fe_3O_4/PANi concentration, with a absorption percentage of 93.94%. This negative value on reflection loss indicates that the material is able to absorb microwaves. The greater the negative reflection loss value, the greater the absorption of the material against microwaves [12]. However, not all high reflection loss produces high absorption as well, this is because not all microwaves regarding the material are absorbed in their entirety, but there are also some that are reflected and transmitted [13].

In general, all of the results of VNA testing characteristics indicate that nanocomposite Fe_3O_4/PANi can be used as a microwave absorber. Both in the C-Band frequency and X-Band frequency ranges. Each particular range has its own characteristics. In the X-Band range, this is an area that can be used for microwave operations on aircraft in the military field. These results are in accordance with those previously reported for Fe_3O_4/PANi nanocomposite made by the coprecipitation method [5] and chemical oxidation polymerization [1].

4. Conclusion

The Fe_3O_4/PANi nanocomposite has been successfully made as a microwave absorber. Where the results of XRD and VNA characterization show that Fe_3O_4/PANi nanocomposite is a material that can be used as a microwave absorber, both in the C-Band frequency range and the X-Band frequency range. The maximum reflection value in the C-Band range was obtained at 40% Fe_3O_4/PANi nanocomposite, while in the X-Band range, the maximum reflection value was obtained at 60% Fe_3O_4/PANi nanocomposite, with a reflection loss value of -28.6736 and -24.3533 with absorption percentage of 96.32% and 93.94%.

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