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Structure and morphology absorber material base on Iron Sand with SiO_2 fortification from water hyacinth

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ABSTRACT

Electromagnetic radar technology has become integral in various innovations, such as military, air navigation, and weather monitoring. However, the ability of radar to detect objects accurately creates vulnerability to detection by other parties, raising security and confidentiality issues. Therefore, the development of electromagnetic absorber material technology is becoming increasingly important, especially in the military. One promising effort is using smart magnetic pigments as electromagnetic wave absorber materials. This material can be synthesized from metal waste and iron sand, which have high permeability and permittivity. However, synthesizing effective and economical smart magnetic materials is still challenging. Iron sand is one of the potentially abundant material solutions. This study aims to synthesize and characterize smart magnetic pigments (Fe_2O_3) from iron sand and silica (SiO_2) from water hyacinth ash as electromagnetic wave absorber materials. The methods used include the extraction of silica from water hyacinth by a slow heating method at high temperatures and the synthesis of magnetite from iron sand by the coprecipitation method. The resulting material was then composited into an Unsaturated Polyester Resin (UPR) matrix and tested for electromagnetic wave absorption. The developed composite material has a porous structure (3.63 µm, porosity 15.746%) with synergistic properties between SiO_2 dielectric and Fe_2O_3 ferromagnetic. The Si-O-Si and Fe-O functional groups (FTIR) and the crystal phases SiO_2 , Cristobalite, and Butlerite (XRD) strengthen the material interactions. This combination of characteristics proves that the composite material can absorb and dampen electromagnetic waves.

Keywords:

Absorber; Iron Sand Fe_2O_3 ; Magnetic Pigment; Silica SiO_2 ; Water Hyacinth.

Introduction

Radar technology has become an integral part of the development of various innovations, including military, air navigation, weather monitoring, etc (Saragi et al., 2023; Taryana et al., 2019). However, the ability to detect with high accuracy through radar also means that our objects, especially military objects, will be vulnerable to being detected (Putri et al., 2022). It means that it will create new problems regarding the security and confidentiality of an object, especially military objects. Therefore, the development of electromagnetic wave absorber material technology is becoming increasingly important to maintain confidentiality and security in various fields, especially military access (Saragi et al., 2023; Imammuddin et al., 2018).

One of the promising efforts for developing military object coating materials for electromagnetic wave absorbers is using magnetic pigments. The use of magnetic materials for electromagnetic wave absorber materials is becoming very popular, especially in electronic equipment (Sebleku et al., 2017). Electromagnetic wave absorber materials must have high permeability and permittivity (Imastuti, 2019; Yunasfi et al., 2018). Magnetic pigment is one of

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the materials with quite good permeability and permittivity, which can be synthesized from metal waste and iron sand. Thus, magnetic pigment can be used as a radar electromagnetic wave absorber material.

However, synthesizing effective and economical smart magnetic materials is still challenging. One of the promising materials in the manufacture of magnetics is iron sand (Madesa et al., 2017; Sunaryono et al., 2013). Until now, iron sandstone has been abundant in nature and rarely utilized optimally (Mayestika, 2020). In addition, according to research conducted by Hutomo in 2017, iron sand has high permeability and permittivity properties, making it suitable for electromagnetic wave absorber materials.

In addition to iron sand, other materials are needed to synthesize electromagnetic wave absorber materials, namely silica (SiO_2). One natural material containing much silica is ash from water hyacinth (Junaudi et al., 2022). Water hyacinth is an aquatic plant with a relatively rapid growth rate; water hyacinth was chosen because it is abundant in nature and sometimes causes serious ecosystem problems (Kalsum, 2015). Excessive water hyacinth growth will block sunlight from entering the water; this causes a decrease in dissolved oxygen in the water. Water hyacinth that grows uncontrollably will also cause the shallowing of the aquatic ecosystem faster due to the sedimentation of dead water hyacinths; this will disrupt the aquatic ecosystem (Ratnani et al., 2024; Zevriawan et al., 2023). Eradication without further utilization will also cause new problems later, such as a complicated disposal process, many eradication costs, etc. Therefore, innovation is needed in using water hyacinths for more appropriate purposes. Water hyacinth can be used as a raw material to synthesize silica materials.

This study chose the coprecipitation method as the primary technique for synthesizing magnetic materials from iron sand. Coprecipitation is an effective method for producing nanoparticles with uniform sizes (Safitri et al., 2021). This process is based on the precipitation of metal ions in the form of hydroxides or basic carbonates through a specific pH setting, which is then followed by a heating process to change the precipitate into an oxide form. One of the advantages of this method is its ability to work under normal environmental conditions, making it more practical and energy-efficient. The coprecipitation method is expected to produce materials Fe_2O_3 with even distribution (Prayoga, 2022). In addition, this method has the advantage of a shorter synthesis time than other methods, such as sol-gel, so it is more efficient to apply in this study.

Therefore, this study aims to determine the structure and morphology of $SiO_2/Fe_2O_3/UPR$ composite materials as an anti-electromagnetic radar material. By utilizing the existing iron sand, this research will produce a coating material that can reduce the reflection of electromagnetic waves, raise cheap local materials, and reduce the negative impacts caused by excessive water hyacinth growth.

This research focuses on the structure and morphology of composite materials $SiO_2/Fe_2O_3/UPR$ as an electromagnetic wave absorber material. Testing was carried out in the laboratory using characterization techniques such as Fourier Transform Infra-Red (FTIR) to identify functional groups and chemical bonds, Scanning Electron Microscopy (SEM), and Energy Dispersive X-ray (EDX) to analyze surface morphology and element composition. X-ray Diffraction (XRD) to determine the crystal structure and phase of the material formed. However, this study did not include absorbance testing using real radar due to limited access to radar facilities and the focus on analyzing the composite material's structure, morphology, and basic properties. This limitation was set to provide an initial understanding of the material's potential as an electromagnetic wave absorber based on laboratory parameters.

Methods

Synthesis of Silica SiO₂ from water hyacinth

Freshly harvested water hyacinth is cleaned of impurities and dried under the hot sun. The dried water hyacinth is then burned using conventional methods to obtain the ash. Next, add 150 ml of citric acid solution ($C_6H_8O_7$) to 48 grams of water hyacinth ash. Soaking is done for several minutes with a total mixture temperature of around 50° C using a stirrer heater. Water hyacinth soaked in a citric acid solution is then washed with hot water 3 times to remove its citric acid content. The residue from washing in the form of water hyacinth ash is then oven-dried at a temperature of 100° C for 25 minutes. The next step is the purification of SiO_2 using a furnace above 800° C (Hutomo, 2017). The material that has been successfully synthesized is then used as a filler material in the electromagnetic wave-absorbing composite that is made.

Synthesis of Fe_2O_3 *from iron sand (smart magnetic)*

Iron sand is the primary raw material used in synthesizing smart magnetic pigments, and the synthesis is carried out using the coprecipitation method. Before the coprecipitation process, iron sand is cleaned and separated from impurities not used in the synthesis process using a bar magnet and washing. Coprecipitation is done by soaking iron sand in a concentrated acid solution. Iron sand cleaned from various impurities is weighed as much as 30 grams and put into a concentrated HCL solution with a volume of 100 ml. Mixing iron sand and concentrated HCL solution is done by heating at a temperature of 80 $^{\circ}$ C.

Furthermore, iron sand is filtered and washed to clean from the HCL solution, and NaOH solution is added to the iron sand precipitate. Before adding the NaOH solution, iron sand is heated on a hotplate. After the soaking process for some time, it will produce a black precipitate, which we call smart magnetic pigment. The following process is washing the black precipitate, which is suspected to be a smart magnetic compound; washing is done using hot water. The purpose of using hot water is to remove impurities such as chloride ions. The smart magnetic pigments successfully synthesized will be used in the next stage (Hutomo, 2017).

Synthesis of UPR/ SiO_2/Fe_2O_3 Composite Material

The results of the silica and magnetic materials synthesis will then be used as composite materials based on Unsaturated Polyester Resin (UPR). Material Fe_2O_3/SiO_2 with a predetermined concentration was mixed with the UPR matrix by the curing method, adding a catalyst to the mixture so the composite could quickly harden. To determine the characterization of the synthesized $Fe_2O_3/SiO_2/UPR$ composite material, FTIR, SEM-EDX, XRD, and Colorimeter tests were carried out (Hutomo, 2017).

Results and Discussions



Figure 1. $SiO_2/Fe_2O_3/UPR$ Composite

The electromagnetic wave absorber composite material has been successfully synthesized, and the composite material has been prepared (Figure 1). Several characterizations were carried out, including Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX), and X-ray diffraction (XRD) to determine the success of the synthesis of SiO_2 and Fe_2O_3 elements, and Colorimeter to determine the level of material absorbance to electromagnetic waves.

Characterization of Composite Materials

FTIR Analysis

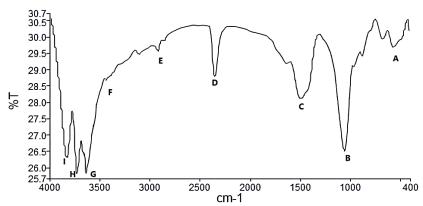


Figure 2. FTIR spectrum of SiO_2 and Fe_2O_3

Table 1	L. Functional	l Groups of	f SiO_2 and	Fe_2O_3

Code	Wave Number	Transmittance (%)	Bond
A	562,0155	29.75	Fe-0
В	1049.5896		
C	1487.6881	26.51	Si-O-Si
D		28.14	C-C
E	2355,677	28.85	0-Н
_	2932	29.2	C-H2
F	3355.29	28.94	О-Н
G	3463.2961	25.5	О-Н
Н	3742.25	25.7	O-H
I	3848.26	26.23	O-H

The FTIR test method can be used to determine the success of synthesizing SiO_2 and Fe_2O_3 from water hyacinth and iron sand. Figure 2 and Table 1 show the presence of several different functional group spectrum niches. Both are owned by silica, iron sand, and several other impurities. The peak of the spectrum number $562.0155\ cm^{-1}$ is the bending vibration of the Fe-0 functional group bond (Ahangaran et al., 2013). The wave number $1049.5896\ cm^{-1}$ is the stretching vibration of the Si-O-Si bond, which is associated with the movement of oxygen (Yulianingsih, 2016; Dippong et al., 2020). The wave number $1487.6881\ cm^{-1}$ is a spectrum niche of the C-C bond, which indicates the carbonization process in water hyacinth during the ashing process (Ashby et al., 2014). The Peaks niche $2355.677\ cm^{-1}$ is related to the strongly bound hydrogen OH group (Emara, AM, & Yousef, ES, 2018). The wave number $3643.296\ cm^{-1}$ indicates a strong bond of the O-H group, this indicates two possibilities. Peak E, with wave number $2932\ cm^{-1}$, indicates the presence of a C-H compound bond (Saren et al., 2021). The F; H; I peaks indicate the presence of O-H compounds with a stretching vibration pattern (Banerjee et al., 2023;

Kumar et al., 2011; Medlej et al., 2021). First, because the sample drying process is not perfect, the water content in the sample is still there. Second, the possibility of the O-H group comes from water vapor in the air, which is then absorbed by the Si element; this shows that the nature of the composite material is Hydrophilic (Kalinkin et al., 2005).

SEM-EDX Analysis

Scanning Electron Microscope-Energy Dispersive X-ray (SEM-EDX) characterization is used to determine the morphological structure of the composite material that has been synthesized. By carrying out SEM-EDX characterization, it can be used to predict the size of the pores, the level of porosity, and the constituent elements of the material. In addition, SEM-EDX characterization can predict the type of bond between elements in the composite material that has been successfully made. SEM scanning was carried out at a magnification of 1500 times, with an electron beam energy of 15kV.

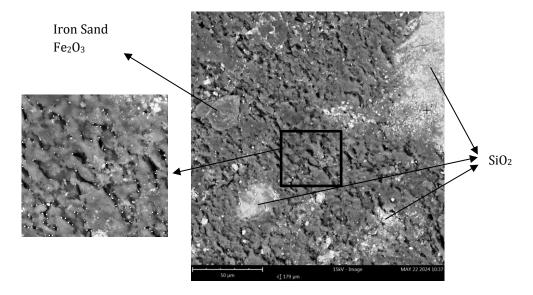


Figure 3. SEM SiO_2 /UPR/ Fe_2O_3

Manual calculations were carried out at 61 points in an area of $2773,925 \ um^2$ to determine the average pore size in the material. The average pore diameter size value was obtained at $3.63 \ um$, with a distance between pores of $16.79 \ um$.

An analysis was carried out using Imagej software to determine the percentage of material porosity, and the material porosity was found to be 15.746%. In line with research conducted by Imammuddin et al. in 2018 and by Wulandari et al. (2024), these conditions provide a large surface area, thereby increasing interaction with electromagnetic waves and allowing for a damping effect through wave diffusion. The UPR matrix is heterogeneously combined with the elements SiO_2 and Fe_2O_3 ; this can be seen in Figure 3, which shows the distribution pattern of white minerals (SiO_2) only in certain spots. It is predicted to occur because the mixing process of elements in the preparation process is not perfect. While the Fe_2O_3 element was not detected, this is because of the precipitation process of the Fe_2O_3 element during the drying of the mixture. The density of Fe_2O_3 , which is greater than other constituent elements, makes this element easier to precipitate, so the distribution of the Fe_2O_3 element becomes uneven. EDX testing was carried out to ensure the presence of these elements, with the results as in Figure 4.

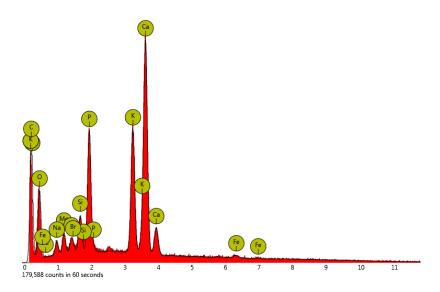


Figure 4. EDX spectrum of SiO_2 /UPR/ Fe_2O_3

The synthesized material was also subjected to EDX analysis to determine the composition of SiO_2 and Fe_2O_3 , as shown in Figure 4. Figure 4 shows that the composition of SiO_2 is close to the synthetic composition. In the synthesis, the composition of SiO_2 was made at 5% of the total mass. At the same time, the EDX analysis shows that the actual composition of SiO_2 is 4.96% (Table 2). Fe_2O_3 has a discrepancy with the synthetic composition, and the EDX analysis reads the composition of Fe_2O_3 at 1.35% (Table 2).

Table 2. EDX Test Results of Fe2O4/SiO2/UPR Composite Material

Element Symbol	Oxide Symbol	Stoich. wt Conc (%)
С		
0		
Ca		
K		
P		
Br		
Si	SiO_2	4,96
Mg		
Na		
Fe	Fe_2O_3	1,35

Meanwhile, in synthetic composition, the mass of Fe_2O_3 used in the synthesis process is 5% of the total mass of the mixture. The difference between synthetic composition calculations and EDX analysis is the precipitation of the Fe_2O_3 element during the preparation process, which causes the distribution of Fe_2O_3 to be uneven. Other elements were also obtained, such as the constituent elements of the UPR matrix and impurity compounds that have yet to be completely removed during purification.

XRD Analysis

The results of the synthesis of UPR/ Fe_2O_3/SiO_2 -based composite materials will be tested using an X-ray diffraction tool (target Cu, $\lambda K\alpha = 1.54060$ A°). Testing with this tool aims to determine the phase and crystal structure of the composite material sample.

The matching software is used based on the analysis, as shown in Figure 5. It is known that several minerals have been formed. At angle 2 Theta (Θ) = 18.2684; 21.4658; 23.0146; 28.3603; 40.6505 is a diffraction pattern owned by the SiO₂ compound, with a trigonal crystal system, and the Crystallography Open Database (COD) id code: 4124055. The diffraction pattern shown at angle 2 Theta (θ) = 23.0146; 28.3603; 37.1533, is a diffraction pattern owned by Cristobalite (SiO₂) with a Cubic crystal system, and the COD id: 9008230. The SiO₂ compound has 2 crystal phases, namely SiO₂ crystals and Cristobalite crystals because the water hyacinth ash material forms 2 crystal phases during the sintering process. Based on research conducted by Putri et al. (2020) Cristobalite is formed in the 800 - 1000 C range. From the sintering process carried out at a temperature of 800, it is natural that cristobalite is formed unevenly due to the quantity of material used during the sintering process. Value 2 Theta (θ)= 15.3208; 182.684; 28.3603; 32.8068, is a diffraction pattern shown by crystals of iron sand, with the formula FeH₃O₇S (butlerite), which has a monoclinic crystal structure, id COD: 9000225. The emergence of FeH₃O₇S compounds is indicated because the iron element binds with other elements in the coprecipitation process. In this XRD test, the presence of Fe₂O₃ compounds has not been identified due to the software system's failure to identify the XRD graph with the existing database. However, the $\mathrm{Fe_2}\mathrm{O_3}$ existence has been proven using previous characterization tests.

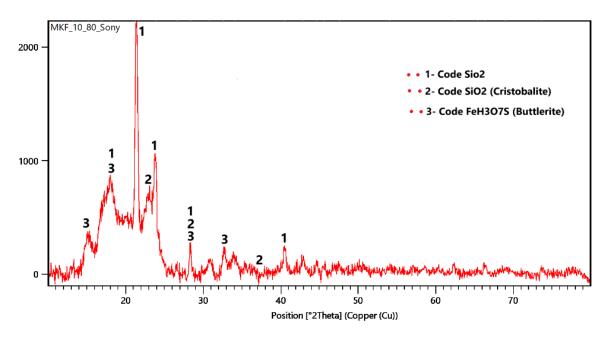


Figure 5. XRD Spectrum of $Fe_2O_3/SiO_2/UPR$ Composite Material

Color Characteristics

Electromagnetic wave absorber composite materials based on smart magnetic pigments, silica from water hyacinth, and UPR have been successfully synthesized. The sample was tested

using a colorimeter and obtained coordinate points L*: 29.8; a*: 3.8; b*: 1.7. The color coordinate points were then analyzed with the L*a*b website (nixsensor.com) producing the HEX4D444 color code which means the composite material has a charcoal gray color.

SEM characterization shows that the composite material has a significant porous structure, with an average pore size of $3.63~\mu m$ and a porosity level of 15.746%. It provides a large surface area that increases interaction with electromagnetic waves and allows for a damping effect through wave diffusion (Imammuddin et al., 2018; Wulandari et al., 2024). EDX analysis reveals the presence of SiO_2 as a dielectric material and Fe_2O_3 as a ferromagnetic material, creating a synergistic effect in wave absorption. The FTIR spectrum shows functional groups such as Si-O-Si and Fe-O, which strengthen the interaction of the composite with electromagnetic waves through the polarization mechanism (Hutomo, 2017). XRD characterization carried out on the material shows the presence of a crystal structure SiO_2 , Cristobalite, and (butlerite) FeH_3O_7S . The XRD diffraction pattern shows the degree of crystallinity of the compound, supporting the efficiency of magnetic and electromagnetic conductivity for wave damping.(Wardiyati et al., 2018). This combination of characteristics proves that the composite material can absorb and dampen electromagnetic waves.

Conclusion

Research shows that the composite material has a significant porous structure, the average pore size is 3.63 μ m, and a porosity level reaching 15.746%. EDX analysis reveals the presence of SiO_2 as a dielectric material and Fe_2O_3 as a ferromagnetic material, creating a synergistic effect in wave absorption. The FTIR spectrum shows functional groups such as Si-O-Si and Fe-O, which strengthen the interaction of the composite with electromagnetic waves through the polarization mechanism. XRD characterization carried out on the material shows the presence of a crystal structure SiO_2 , Cristobalite, and (butlerite) FeH_3O_7S . This combination of characteristics proves that the composite material can absorb and dampen electromagnetic waves. However, further research is recommended to involve absorbance performance testing using radar facilities to ensure effective materials in real applications. It is important to evaluate the material's capabilities under real operational conditions so that its development can better support industrial needs, especially in the military and electromagnetic technology fields.

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Conflicts of interest

The authors declare that there are no conflicts of interest.

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