Synthesis and Characterization of Composite UPR/Fe$_3$O$_4$ for Its Use as Electromagnetic Wave Absorber

Yusmaniar$^1$, W A Adi$^2$, Y Taryana$^3$, R Muzaki$^4$

$^1$,$^4$ Department of Chemistry, Faculty of Mathematics and Natural Science, Universitas Negeri Jakarta, Jl. Pemuda No 10, Rawamangun 13220, Jakarta, Indonesia
$^2$ Centre for Science and Technology of Advanced Materials, National Nuclear Energy Agency, Kawasan Puspiptek Serpong, Tanggerang Selatan, Banten, Indonesia
$^3$ Centre for Research of Telecommunication and Electronic, Indonesia Institute of Science, Komplek LIPI Sangkuriang, Bandung, Jawa Barat, Indonesia

E-mail: ys_maniar@yahoo.com

Abstract. Synthesis and characterization of UPR/Fe$_3$O$_4$ composite were performed to evaluate its potency as a electromagnetic absorbent. The composite was prepared from the mixture of unsaturated polyester resin and magnetite powder of iron oxide. Fe$_3$O$_4$ was used as filler and unsaturated polyester resins (UPR) was used as a matrix. Fe$_3$O$_4$ magnetite was synthesized from iron objects using electro synthesis method. The raw material was blended in the beaker glass for 120 min and then pressed at temperature of 60 °C for 30 min. The composite is in a semi-crystalline form that consists of amorphous matrix and the various crystalline fillers. The functional group’s analysis of the composite showed that crosslink (bridges) was formed between the chains of individual polymer and between Fe$_3$O$_4$ magnetite filler and UPR matrix. The performance of microwave absorbent measured by VNA method showed the highest RL at the frequency of 10.44 GHz and 11.74 dB. This value was achieved with a composition of 10wt% Fe$_3$O$_4$ and 90wt% UPR. We concluded that the composite of UPR/Fe$_3$O$_4$ has been successfully demonstrated as an electromagnetic wave absorber.

1. Introduction

In the past years, the use of wireless electronic devices grows rapidly. The wireless technology is based on transmission of energy through electromagnetic wave, which eventually may cause tissue damage from the radiation above the certain threshold. This danger can actually be avoided upon use of materials that can counteract the excess of electromagnetic wave radiation [1]. This material is called electromagnetic wave absorber.

Electromagnetic wave absorber can counteract transverse waves, which is the electromagnetic wave that propagates in a vacuum state. The wave can be transmitted, reflected and absorbed. The material absorber should be able to absorb the electromagnetic wave radiation, thereby it must have an optimum permeability ($\mu$), permittivity ($\varepsilon$), and resistivity, and low coercivity (Hc) [2].

One of the materials used as an electromagnetic wave absorbent is magnetite or iron (II)(III) oxide (Fe$_3$O$_4$) [3][4]. The magnetite acts as a filler or filler matrix in the composite. It has also been used as catalyst, gas sensor, and data storage device materials. Magnetite is derived from iron objects by an electrochemical method [5], which is the most effective methods to improve the ability of a material to absorb wave. Magnetite has good permeability. Therefore, it is an excellent candidate for electromagnetic wave absorbent. In this study, the magnetite was derived from the iron nail, an iron...
object that is commonly used in construction works. The leftovers, wasted, and used iron nails can be utilized as a cheap source of good magnetic and solid structure materials for magnetite.

The other component of composite is the matrix polymer, which serves as the binder. In this research, Unsaturated Polyester Resin (UPR) was selected because it has good mechanical properties, i.e. dielectric constant, making it an optimum supporting material for the composite. Characterization of the composite was carried out with the electromagnetic wave type X band around the frequency of 8-12 GHz. This frequency was selected because it is used in applications such as RADAR, telecommunication, and wireless electronic device.

2. Materials and Method
Magnetite powder was made by electro-synthesis. At first, two iron nails prepared in a beaker filled with saline solution. Salt solution used is 0.9% NaCl solution. Iron nail that was submerged in a saline solution and then connect it to flow through the cathode and anode copper wire that fed the DC current from a battery or power supply. Electrolysis iron nails take about three hours to get precipitate magnetite (Fe$_3$O$_4$) blackish brown. After completion of the electrolysis process, the precipitate is filtered with Whatman paper 41. The precipitate has been filtered and after that allowed for about 15 minutes at the room temperature. The precipitate is then injected into the furnace at a temperature of approximately 400 °C for one hour. The next step was made of the composite between UPR and Fe$_3$O$_4$ with a weight ratio of 50: 50; 60: 40; 70: 30; 80: 20 and 90: 10. UPR as a matrix resin, hardener, and Fe$_3$O$_4$ were stirred for 10 minutes. After that in printing on a round plate with a thickness of 2 mm. Characterization of Fe$_3$O$_4$ synthesized is FTIR, SEM-EDS, and VSM. Phase composition analysis in the data used x-ray diffraction with a range 10-80° angle measurements; each specimen was analyzed by Vector Network Analyzer (VNA) to determine the reflection loss of the electromagnetic waves in the frequency range of 8 -12 GHZ.

3. Results and Discuss
Elemental and chemical property synthesized Fe$_3$O$_4$ was analyzed with EDS while the morphology was evaluated under the electron microscope.

![Figure1. The result of SEM-EDS from sample Fe$_3$O$_4$](image)

The EDS spectra indicated the presence of Fe and O in the synthesized material. Under the microscope, the material appeared as a crystalline (Figure 1). This result suggests that the material is Fe$_3$O$_4$. However further analysis must be performed to confirm that hypothesis by means FTIR and XRD.

The identity of the synthesized material as to contain Fe$_3$O$_4$ was shown by the IR spectra (Figure 2). The peak at 576.64 cm$^{-1}$ is characteristic for a Fe-O stretching [6]. Further confirmation of the material identity was provided by the X-ray diffraction experiment.
Figure 2. IR Spectra of the synthesized material Fe3O4 sample

Figure 3. The refinement results of X-ray diffraction pattern Fe3O4 sample

Figure 4. The curve Hysteresis of Fe3O4 sample

Figure 5. IR Spectra of the composite UPR/magnetite

The magnetic characteristic of Fe3O4 in the synthesized material was shown by the remanent magnetization (Mr), saturated magnetization (Ms), coercivity field (Hc) respective of 15.57 emu/g, 35.5 emu/g, and 90 Gauss. These values showed the Fe3O4 ferromagnetic properties. Thus the Fe3O4 in the material has successfully been identified and characterized.

Figure 5 shows an IR spectral of the UPR. In the spectral characteristics of UPR compounds with functional groups at wave numbers of 742 cm⁻¹, 1122.57 cm⁻¹, and 1284.59 cm⁻¹, respective for benzene, polyester, and ester, while stretching -CH bond at wave number of 1722 cm⁻¹ and 2964.59 cm⁻¹ [8-10].
The composites with the UPR: Fe$_3$O$_4$ ratio of 90:10, 80:20, 70:30, 60:40, and 50:50, were analyzed with VNA method to determine the electromagnetic wave absorption power (Figure 6). Apparently, the proportion of 10-50% Fe$_3$O$_4$ to the UPR has no significant influence on the absorption capacity. The best absorption performance was achieved at proportion of 10% Fe$_3$O$_4$, reaching -11.73 dB at the frequency of 10.44 GHz.

4. Conclusion
We have successfully synthesized Fe3O4 and produced the composite that contains the magnetite and UPR. The identity of the composite was confirmed with SEM-EDS, FTIR, VSM, and XRD. The composite was also successfully produced with the proportion of 10% Fe$_3$O$_4$ to UPR to give the best electromagnetic wave absorption performance.

References
[1] Wu W 2008 *Nanoscale Res Lett.* 3 397
[2] Cabrera L 2008 *Electrochimica Acta.* 53 3436
[3] Jazirehpour M 2015 *Journal of Alloys and Compounds* 638 188
[4] Cao J and Zhitomirsky I 2005 *Materials Chemistry and Physics* 96 289
[5] Donya Ramimoghadam S B 2014 *Journal of Magnetism and Magnetic Materials* 368 207
[6] Patil R M 2014 *Royal Society of Chemistry* 4 4515
[7] Xiaotai Zhang J K 2013 *Journal of Crystal Growth* 372 170
[8] Adeodu A O, Anyaechio C O and Oluwole O O 2014 *J. of Advancement in Engineering and Technology* 1
[9] Nariyal R K, Kothari P and Bisht B 2014 *Chemical Science Transactions* 3 1064
[10] Darmawan A, Smart S, Julbe A and Diniz da Costa 2011 *J.C. Iron oxide silica derived from sol-gel synthesis. Materials* 4 448