Structural, magnetic and microwave absorption properties of natural iron sand

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Abstract. This paper presents the development and characterization of the natural iron sand as a microwave absorber. To develop a new microwave absorbing paints in the 7–13 GHz range, the iron sand sample was prepared through the mechanical milling process and characterized with X-ray diffraction (XRD), scanning electron microscopy (SEM), Permagraph and vector network analyzer (VNA). The reflection loss (RL) was calculated and simulated for different thicknesses of the material using the complex permittivity and permeability and found the broadband characteristics with minimum absorption. The results indicated that the natural iron sand is beneficial as potential candidates for military applications.

1. Introduction

Recently, with the effects of electronic and electrical devices in high frequency, such as electromagnetic (EM) radiation pollution and EM interference (EMI) have become a crucial issue in the world, not only degrading the device functions but also harmful for the human health and affecting the environmental problems [1–5]. Thus, highly effective microwave absorbers are an essential issue for overcoming unwanted EM waves caused by EM radiation in the gigahertz frequency. Researchers have investigated the development of microwave absorbers that can attenuate EM interference and convert the EM energies into thermal energies [6–10]. Ferrite materials are a promising candidate as microwave absorbers because of their magnetic and dielectric properties, such as the complex permeability and permittivity, respectively [11–14].

There are lots of research papers about microwave absorption of ferrite materials, such as BaFe$_{12}$O$_{19}$/Fe$_3$O$_4$ composite [15], flake carbonyl iron/epoxy resin composites [16], SiC@SiO$_2$@Fe$_3$O$_4$ hybrids [17], and Fe$_3$O$_4$/C composite [18]. From these reports, it can be concluded that the EM wave absorbers of Fe$_3$O$_4$-based materials are significantly able to enhance EM wave absorption performances. Therefore, in this work, natural iron sand and mechanical milling process were used to prepare Fe$_3$O$_4$ content, which is an important phase for determining the microwave absorption abilities of absorbers. Structural, magnetic, and microwave absorption properties of natural iron sand are studied in detail.
2. Experimental methods

The natural iron sand from Puger beach, Jember, Jawa Timur, Indonesia, was prepared by milling process to get smaller particle size using a vibration ball mill for 1 hour. Phase structures of the natural iron sand were investigated using a PANalytical X-ray diffractometer (XRD) with Cu-Kα radiation (λ = 0.15418 nm). The surface morphology of the sample was observed on a JEOL JSM 5310LV scanning electron microscope (SEM). Room temperature magnetic property measurement was performed on Permagraph Electromagnet EP3 with an applied external field of 1.0 T. EM parameters (reflected signal S_{11} and transmitted signal S_{21}) of the iron sand was measured by a Rohde-Schwarz ZVA 67 vector network analyzer (VNA) with the WR90 sample holder in the frequency range of 7 – 13 GHz.

3. Results and discussion

3.1. Structural identification

Figure 1a shows XRD patterns of the natural iron sand. The standard position and intensity of X-ray diffraction lines for Fe_{3}O_{4} based on the international crystal diffraction data (ICDD) #98-003-1156 (Fe_{3}O_{4}). The five diffraction peaks (2θ = 30.09, 35.41, 35.53, 43.12, 57.13 and 62.61°) of the natural iron sand correspond to the (112), (121), (220), (231), and (224) planes of Fe_{3}O_{4}, respectively. With orthorhombic of crystal systems and these d-spacing values for the crystal planes of Fe_{3}O_{4}, the lattice parameters (a=b=5.9038 Å, c=8.3947 Å) were calculated by the least-squares method using PANalytical High Score Plus software ver. 3.0e. The microstructure of the sample was analyzed using SEM. The SEM image of the natural iron sand sample is shown in Fig. 1b. A wide distribution of particle sizes (1 μm–10 μm) was observed in the sample.

![Figure 1](image-url)  
(a) XRD pattern and (b) SEM image of the natural iron sand.

3.2. Magnetic properties

The hysteresis curve was measured with magnetic fields of 1 tesla for the natural iron sand sample using Permagraph techniques at room temperature is shown in Fig. 2a. It can be seen that the hysteresis loop of the sample is slightly and the same for Fe_{3}O_{4}[19], which indicates that the natural iron sand not only have Fe_{3}O_{4} as the main phase but also excellent soft magnetic performance (less than 10 kA/m) with low coercivity (H_c) and high saturation magnetization (M_s) values. This can be attributed to the EM wave absorbing properties of the natural iron sand.

EM characteristics of the sample can be characterized by its complex permeability (μ = μ' − jμ'') and permittivity (ε = ε' − jε'') that were calculated by using the Agilent Technologies Materials Measurement software with the S-parameters (S_{11} and S_{21}), which were simultaneously measured by VNA. The real (μ' and ε') and imaginary (μ'' and ε'') parts represent energy storage and energy dissipation or dielectric loss, respectively [10]. Fig. 2a shows the frequency dependence of two important EM variables of the natural iron sand measured in the frequency range of 7–13 GHz. The sample results the maximum
dielectric variables of 7.18 and 5.55 for $\varepsilon'$ and $\varepsilon''$, respectively. The $\varepsilon'$ decreases, and the $\varepsilon''$ with the increase of frequency. The values of the $\mu'$ and $\mu''$ of complex permeability of sample do not show an obvious dependence on frequency and relate to the magnetic energy loss of $\text{Fe}_3\text{O}_4$, respectively.

Figure 2. (a) Magnetic hysteresis loops and (b) complex permeability and permittivity of the natural iron sand.

3.3. Microwave absorption properties

The EM wave absorbing properties usually are associated with a reflection loss (RL) value of the sample, which can be resulted by calculating and simulating through the following equations $Z_{in} = \sqrt{\frac{\mu}{\varepsilon}} \tanh \left[\frac{\sqrt{-j2\pi f t}}{c}\sqrt{\mu\varepsilon}\right]$ and $\text{RL (dB)} = 20\log \left[\frac{(Z_{in}-1)}{(Z_{in}+1)}\right]$. Where $Z_{in}$ is the impedance of the sample, $f$ is a frequency in the gigahertz range, $c$ is the velocity of EM waves in free space, and $t$ is the thickness of the sample. The $\mu$ and $\varepsilon$ are the complex permeability and permittivity, respectively [20,21]. Fig. 3 shows the RL curves of the natural iron sand sample with various thicknesses (0.30, 0.35, 0.40, 0.45, and 0.50 mm). The minimum RL of $-15.68$ dB (96.8% absorption) is showed at 7.62 GHz of 0.40 mm thickness. The RL values less than $-10$ dB for the natural iron sand with 90% absorption were observed in the frequency range of 8.56–10.13 GHz, 7.29–9.94 GHz, 7.10–9.00 GHz and 7.08–7.87 GHz with a thickness of 3.0 mm, 0.35 mm, 0.40 mm, and 0.45 mm, respectively. According to the typical $\text{Fe}_3\text{O}_4$-based materials in recent literature [22–24], the natural iron sand is very promising EM-wave absorptive candidate ferrite powder used in the paint due to give the minimum RL and optimum frequency bandwidth with a smaller thickness of EM wave absorbers.

Figure 3. The calculated reflection loss of the natural iron sand with different thicknesses in the frequency range of 7–13 GHz.
4. Conclusions

In summary, the natural iron sand with 1–10 μm of particle sizes was prepared by using a vibration ball mill for 1 hour and also have Fe₂O₃ as the main phase. The sample exhibited an excellent soft magnetic material with a Hₑ value of less than 10 kA/m. The complex permittivity tended to constant, but permeability changed with the increase of frequency, which indicated that the natural iron sand had the microwave absorption properties. The natural iron sand has more excellent absorbing properties in the range of 7–13 GHz and can be recommended as candidates for the electromagnetic wave absorbing paint materials.

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