Microwave Absorption Analysis on Recycled Glass Doped with Ferum (II, III) Oxide (Fe₂O₃)

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Abstract— Composite consisting of recycled glass and Ferum (II, III) Oxide were prepared through a heating process in 1100°C for 4 hours. The prepared composite of recycled glass which is presented in cullet powder and Ferum (II, III) Oxide is an innovation in improving the absorption of microwave signal. Cullet has been found to possess a significant propensity for absorption microwave. In this research, the microwave absorbability of various ratios of cullet powder to Ferum (II, III) Oxide in the composite was investigated. Reflection coefficient (Γ) and transmission coefficient (T) are used to determine the absorbability of microwave in range of absorption coefficient of 1.2 GHz to 3.0 GHz. The results show that cullet powder containing Ferum (II, III) Oxide absorbed microwave efficiently from 2.5 GHz to 3.0 GHz. The comparison among the composite in different ratio of Ferum (II, III) Oxide is conducted to determine the best optimum of ratio in microwave absorption.

Index Term— Recycled glass, Ferum (II, III) Oxide, S-parameter, microwave absorption.

1. INTRODUCTION

At early of 20th century, the use of electronic devices has grown rapidly around the world. The concern on the effect of microwave radiation from electronic devices on health is rose. This effect has raised awareness regarding public exposure to microwave radiation emitted from electronic devices and the interaction between the radio frequency, microwave, and biological effect on human tissue, especially the human immune system and the brain. There is number of researches that prove that the microwave radiation increases the risk of health effects such as brain tumors, bloodbrain barriers (BBB) permeability function, sleep disorders, cognitive function, DNA damage, immune system function and stress response [1-4].

Recently, the application of the gigahertz range of microwave in electrical devices increase due to its hectic demand. Microwave interference has become one of the major issues in telecommunication. As a result, microwave absorption is one of the promising techniques to reduce microwave interference in electronic industry. Various materials used as microwave absorber were studied [5-9]. Ferrite composites [10-14], carbon foams [15-19], and foam/porous materials [20-21] with microwave absorption properties were reported, recycled glass that doped with conducting elements has been used as a conventional microwave absorbing materials. Glass is one of the material which can be recycled infinitely without losing strength, purity or quality. A glass bottle that is sent to a landfill can take up to a million years to be degenerated. They need to be recycled in order to reduce the waste pile up in landfill and keep our environment clean and safe. In this work, the behaviour of recycled glass from domestic glassware in microwave absorption due to dopant of Ferum (II, III) Oxide was observed. A new potential of recycled glass is explored as microwave absorbing material.

In this work, the recycled glass which has been prepared in powder was doped with Ferum (II, III) Oxide through sintering process by using furnace, in order to enhance microwave absorbing properties. Sample were
fabricated in rectangular pellet and heated at temperature of 1100°C. Microwave absorption test results show that increasing percentage of microwave absorption. Hence the Ferum (II, III) Oxide doped with glass powder can enhance microwave absorption properties. The sample was conducted analyses through scanning electron microscope (SEM) and X-ray diffraction (XRD) to establish the relationship between morphology of material and absorption.

Microwave absorption is a process in which the energy of microwaves is converted into other energy such as thermal energy that is not reflected or transmitted through the material. Figure 1 shows the general processes involved for incident microwave through a microwave absorption material. Three processes which are reflection, absorption and transmission were taken place when an incident microwave impinges on material. The absorption materials can be deduced from reflection and transmission. The insignificant of reflection and transmission implies that the absorption is occurred. This properties requires the material to exhibit strong magnetic or/and dielectric loss [22]. The doping metallic Oxide which is Fe$_3$O$_4$ with glass powder are going to enhance the material in magnetic properties and conductivity. Recycled waste materials for EM absorption have been carried out for several decades. Researches have been conducted to study the EM absorbing material since World War II. Since the demand on EM absorbing material increases, various types of absorbing material, e.g. Ferrite, carbon fiber, carbon-based nano-composite, highly conductive Polyethylene, and etc. were studied. A research is conducted using activated carbon fibers which have large specific surface area as a continuous reinforcement (35 vol.%) in a polymer–matrix composite to enhance the EMI shielding effectiveness of the composite due to multiple reflections. On the other hand, Tian et. al. [9] Study the absorbability of carbon fiber. Then, Liu et. al. [10] Compared between carbon black and carbon fiber and found that the coatings filled with carbon fiber shows higher complex permittivity than the coatings filled with carbon black if the content of carbon absorbed is same.

Apart from synthetic microwave absorber, waste

from natural product, e.g. agricultural product. Agricultural waste is organic material which is carbon-based material. Recently, many researchers have been conducted to study the potential of agricultural wastes (organic materials) as a microwave absorbing material. The typical agricultural waste that have been studied for its microwave absorption are including coconut shell [23-24], sugar bagasse [25], rice husk [26-27], coco peat [28] and etc. These carbon-based material exhibit the behaviour of microwave absorption. These performed very promising capability in microwave absorption. It is primarily due to the conductivity that presented in carbon where the conductivity is major parameter to increase the loss factor of a material. The agglomeration of carbon when the percentage of carbon in a material exceed percolation threshold will increase the conductivity of material. The conductivity become an enlightenment of this work.

2. METHODOLOGY

Sample preparation

Domestic glass bottle were collected and crushed into fine powder form and mixed with Fe$_3$O$_4$ powder in particular ratio. Hydraulic press is used to prepare the sample in rectangular pellet in conjunction with tailor-made mould. The ratio of Fe$_3$O$_4$ to glass was determined by weight. All of the ratio of compositions in samples are shown in Table 1. These mixture are placed in alumina crucible for heating process in furnace for 3 hours at temperature of 1100°C., in order to fuse the compositions in mixture.

Experimental measurement and characterization

Fig. 1. The general process of an incident microwave through an microwave absorption material [22].
Reflection and transmission measurement were conducted by using Mini-circuits PWR-6GHS+ Power Sensor. This power sensor was connected with Mini-circuits ZX95-2800+ Voltage Controlled Oscillator (VCO) and a pair of open-ended coaxial sensor as shown in Figure 2. Mini-Circuits ZGDC10-362HP+ directional coupler will be additionally used for transmission measurement.

Absorption coefficient was calculated through Equation (3). The magnitude of reflection and transmission coefficient is normalized power where reflected and transmitted power due to presence of sample is normalized by the reflected power without material under test (free space).

Reflection and transmission measurement is conducted with using Mini-circuits PWR-6GHS+ as shown in Figure 1. Absorption coefficient, $A$ is calculated as follows:

$$\Gamma = \frac{\text{reflected power/voltage}}{\text{power/voltage of incident wave}}$$

$$T = \frac{\text{transmitted power/voltage}}{\text{power/voltage of incident wave}}$$

Insertion absorption loss (absorption coefficient),

$$A = 1 - |\Gamma|^2 - |T|^2$$

where $\Gamma$ is reflection coefficient and $T$ is transmission coefficient.

3. RESULTS AND DISCUSSION

Effect of thickness on reflection and transmission

Figure 3 shows the variation of sample thickness with reflection coefficient, $\Gamma$ and transmission coefficient, $T$. The studied thickness is including 1 mm, 2 mm, 3 mm and 4 mm. The 4 mm thickness of sample exhibit highest $\Gamma$ for all percentages ratio and followed by 3 mm, 2 mm and 1 mm of sample thickness. Literally, $\Gamma$ increase due to sample of thickness increase. It can be explained by the wave equation where the increment of real part of propagation constant which cause wave attenuation is attributed to the increment of distance in a certain medium. Thicker sample provide longer travelling distance for wave propagation and hence yield to tremendous attenuation.

In Figure 3 (a), (c), and (e), it can be seen that the difference in term of $\Gamma$ is insignificant for all thicknesses. It might due to insignificant effect of thickness on reflection coefficient, since the reflection coefficient is primarily due to mismatch impedance occurred on interface of two different medias, i.e. waveguide and sample under test. For a particular ratio of sample, the samples exhibit same dielectric and magnetic properties. Hence, its mismatch impedance will be similar. For all percentages of cullet doped with $\text{Fe}_3\text{O}_4$ shows that the $\Gamma$ is relatively low within range from 2.45 GHz to 3 GHz. It is probably due to the less mismatch impedance between sample under test [Equation (4)] and waveguide within this range of frequency

$$Z = \sqrt{\frac{j\omega \mu}{\sigma + j\omega \epsilon}}$$

where $\omega$ is angular velocity, $\mu$ is complex permeability, $\epsilon$ is complex permittivity and $\sigma$ is conductivity. The impedance difference between waveguide and sample become less. It allows less wave reflection occur. These mismatch impedance can be explained through the discrepancy of impedance due to complex permittivity and complex permeability of sample under test from the free space, as impedance is function of dielectric constant, real
part of complex permeability ($\mu'$) and frequency. Meanwhile, $\Gamma$ was recorded within range from 0.6 to 0.95 for 1.2 GHz to 2.45 GHz. It is relatively high compared with $\Gamma$ found in frequency range from 2.45 GHz to 3 GHz. Thus, it can be inferred that degree of mismatch impedance is higher than at low frequency range from 1.2 GHz to 2.4 GHz. A similarity can be observed through Figure 3 (a),

(c), and (e) that the highest reflection coefficient was recorded at frequency 1.88 GHz, whereas the lowest reflection coefficient can be observed at 2.80 GHz.

Theoretically, the microwave transmission coefficient, $T$ should decrease with thickness. However, the high absorbability of sample due to addition of Fe$_3$O$_4$ enhance the transmission loss, regardless the thickness of sample. Therefore, it can be seen that the range of transmission coefficient is considerably low which is in between 0.01 to 0.25.

From Figure 3(c) and Figure 3(e), the sample with 4mm thickness exhibit the lowest T. Therefore, One of the factor to reduce T and enhancing microwave absorption is by thickening the sample thickness, in order to have greater loss due to microwave absorption in sample. The 50 wt% and 75 wt% of Fe$_3$O$_4$ which were doped in cullet heighthen conductivity of sample. It is subsequently rise the loss factor up which can be described by $\varepsilon''=\sigma/\omega$ and imaginary part of permeability ($\mu''$). These terms are the factors cause wave absorption in sample.

**Morphological Analysis through Scanning Electron Microscope (SEM)**

In order to investigate further about the relationship among inhomogeneity, mass ratio and the absorbability of sample, SEM analysis is conducted. By referring to image SEM as shown in Figure 4, it can be observed as inhomogeneous material. When the microwave propagate through the sample, the inhomogeneity in material lead to multiple reflection. The inhomogeneity is most probable due to presence of pore in sample. On the other
The image contains two graphs showing the relationship between frequency and reflection coefficient ($\Gamma$) as well as transmission coefficient $T$ for different thicknesses of materials. The graphs are labeled (c) and (d).

Graph (c) plots reflection coefficient $\Gamma$ against frequency for four different thicknesses: 0.8mm, 1.8mm, 2.5mm, and 3.6mm.

Graph (d) plots transmission coefficient $T$ against frequency for the same thicknesses as in graph (c).
Fig. 3. Variation of different thickness on (a) reflection coefficient and (b) transmission coefficient for ratio of 1 : 3 (Fe₃O₄ : glass); (c) reflection coefficient and (d) transmission coefficient for ratio of 1 : 1 (Fe₃O₄ : glass); (e) reflection coefficient and (f) transmission coefficient for ratio of 3 : 1 (Fe₃O₄ : glass)
Fig. 4. SEM images of glass powder doped with Ferum (II, III) Oxide: (a) 75 %wt Fe₃O₄ (3:1); (b) 50 %wt Fe₃O₄ (1:1); (c) 25 %wt Fe₃O₄ (1:3) absorption in sample.

hand, thick sample requires large quantities of cullet and Fe₃O₄ and it lead to increment of inhomogeneity in material. In addition, it can be observed that ratio of 3:1 has more porous structure compared to the other two. The larger mass ratio is, the more porous structure can be found. It comes to know that that increment of Ferum (II, III) Oxide generate high porosity or inhomogeneity in sample. The open pores existed in the pellet is determined by the quantity of Ferum (II, III) Oxide added. The irregular shape of cullet and dissimilar size of cullet and Ferum prepare pores in sample. It can be observed that high ratio of Ferum (II, III) Oxide to cullet which shown in Figure 4 (a) exhibit greater number of pore if compare with Figure 4(b) and (c). It can be deduced that the number pores increase with the quantity of Ferum (II, III) Oxide in sample. The SEM images in Figure 4(a) show that the activation stage produced extensive external surfaces with quite irregular cavities and more cracks or voids form to be pore. The pore can capture the electromagnetic wave to conduct multiple reflection which it will subsequently lead to severe multiple reflection loss. Hence, the absorption is occurred.
X-Ray Diffraction Analysis

Figure 5 shows XRD surface spectra of the 75 %wt Ferum (II, III) Oxide and 25 %wt of glass powder. The diffract grams generally reveal the glass and Ferum (II, III) Oxide composites to have an amorphous nature. However, the composition of the 75 %wt Ferum (II, III) Oxide and 25 %wt of glass powder exhibit small peaks of crystallinity that are possibly associated with Ferum (II, III) Oxide. In Figure 6, it is semi-crystalline. Thus, this sample can absorb radiation as it carries feature of amorphous. The nature of glass and the presence of pore in yield to this feature.

![Figure 5](image_url)

Fig. 5. XRD surface spectra of the 75 %wt Ferum (II, III) Oxide and 25 %wt of glass powder.

Effect of Ferum (II, III) Oxide (Fe₃O₄) doped with cullet powder on microwave absorption

Figure 6 show the performance of microwave absorption for different ratio of Fe₃O₄ to recycled glass (cullet) from 1.23 GHz to 2.99 GHz for various sample thicknesses. Percentages of microwave absorption increase as mass ratio increases. Evidently, the microwave absorption properties of 75 wt% Fe₃O₄ doped with 25 wt% glass powder exhibit the best absorption than the rest. Increment of percentages Fe₃O₄ could enhance the dielectric and magnetic properties. Theoretically, high microwave absorption can be risen by combination of electrical and magnetic properties [27]. The presence of Fe³⁺ due to chemical reaction that occurred during the heating process in 1100°C fuse with cullet to form new compound. The conductive element that presented in sample increase its conductivity. Likewise, the loss factor and loss tangent which determine the absorption increases.

Pure cullet which presented in pellet had proven to have potential of microwave absorption as shown in Figure 6. Absorption (%) of each sample increase with frequency. It might due to the coherence between relaxation frequency of sample and the operating frequency of microwave. It would causes increment of $\varepsilon''$ and $\mu''$, respectively which will results increment of loss tangent (tan $\delta_e = \varepsilon'' / \varepsilon'$ and tan $\delta_m = \mu'' / \mu'$) too. On top of that, the maximum of energy dissipation take place during the polarization and magnetization. The highest absorption (%) are consistently occurred at 2.80 GHz for all ratio and thickness.

Absorption (%) of pure cullet is act as a control. It comes to learn that the lowest absorption (%) which exhibited by pure cullet justify that the presence of Ferum (II, III) Oxide can strengthen the absorbability of cullet to microwave. The absorption (%) for pure cullet in within range from 5% to 25%. Meanwhile, the ratio of 1:1 and 3:1 (Fe₃O₄ to cullet power) of composites are able to exhibit >50% of absorption from 2.45GHz to 3.00 GHz especially
2.80 GHz. Absorption of at 2.80 GHz is appeared in between 90% to 100%.

On the other hand, the effect of thickness on absorption (%) is insignificant as the loss tangent or
Fig. 6. The performance of microwave absorption in percentage for different ratios of Fe$_3$O$_4$ doped with cullet in various range of thicknesses, i.e. (a) 0.7mm-1mm; (b) 1.6mm-1.8 mm; (c) 2mm-2.8mm; and (d) 3.5mm-3.6mm sample thickness.
conductivity dominant the mechanism of absorption in sample. Hence, the variation of thickness to absorption (%) is not visibly observed through Figure 6.

4. Conclusion

Pure recycled glass/cullet in powder show very low percentage of absorption over 1.23 GHz to 3 GHz. This implies that the cullet has potential to absorb electromagnetic radiation, but in insignificant amount. A dopant is needed to increase the conductivity of the glass, in order to enhance its absorbability to electromagnetic wave. Thus, Ferum (II, III) Oxide used as the dopant in this research. From 2.4 GHz to 3 GHz, sample with 75 %wt Ferum (II, III) Oxide and 25 %wt cullet (3:1) exhibit the lowest $\Gamma$ and considerably low of $\tau$. It causes highest absorption, i.e., $\approx 99\%$ compared with other ratio. This is due to larger quantity of Ferum (II, III) Oxide in ratio of composite/sample. Even though thicker sample exhibit high absorption (%), however, it is insignificant. This might due to the limitation of experimental setup in this work. Larger quantity of Ferum (II, III) Oxide which doped into cullet results to higher loss factor and loss tangent in terms of permittivity and permeability due to the presence of Ferum as conductive and magnetic component in sample. Subsequently, this ratio heighthen its absorbability within microwave range from 2.45GHz to 3GHz. Apart from this, porosity of sample can also be cause to the absorption, as the the pore in sample tend to capture the electromagnetic wave. The captured wave in pore is subjected to multiple reflection loss. The highest absorption (%) was recorded at 2.80 GHz.

REFERENCES


