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**Research article** 

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# Complex permittivity and power loss characteristics of $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/ polycaprolactone (PCL) nanocomposites: effect of recycled $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller



Helivon

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# ABSTRACT

The development of microwave absorbing materials based on recycled hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) nanoparticles and polycaprolactone (PCL) was the main focus of this study.  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> was recycled from mill scale and reduced to nanoparticles through high energy ball milling in order to improve its complex permittivity properties. Different compositions (5% wt., 10% wt., 15% wt. and 20% wt.) of the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles were melt-blended with PCL using a twin screw extruder to fabricate recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites. The samples were characterized for their microstructural properties through X - ray diffraction (XRD) and high resolution transmission electron microscopy (HRTEM). The complex permittivity and microwave absorption properties were respectively measured using the open ended coaxial (OEC) probe and a microstrip in connection with a vector network analyzer in the 1–4 GHz frequency range. An average  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticle size of 16.2 nm was obtained with a maximum imaginary ( $\varepsilon$ ") part of permittivity value of 0.54 at 4 GHz. The complex permittivity and power loss values of the nanocomposites were between 13.3 dB and 14.4 dB and at 3.4 GHz, a maximum value of 16.37 dB was achieved for the 20 % wt. nanocomposite. The recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites have the potential for use in noise reduction applications in the 1–4 GHz range.

#### 1. Introduction

The fast advancement of electronics innovation in recent years has given rise to the ever increasing use of electronic gadgets operating at the lower microwave frequencies. These electronic gadgets can cause extreme electromagnetic interference (EMI) that needs to be suppressed using microwave absorption materials which are typically required to be thin, low cost, low density, with strong absorption properties and broad absorption bandwidth. In recent years, various forms of ferrites with different morphologies have been utilized in the development of microwave absorbing materials due to their superior magnetic properties. To date, one type of ferrites which has been less extensively utilized for such applications is  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>.  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> is a corundum-type iron oxide with a high chemical and thermal stability under ambient conditions and has been considerably used in several applications including chemical sensors [1], pigments [2] and anodes for lithium-ion batteries [3].  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> can be prepared through a variety of techniques such as chemical synthesis, gas-phase synthesis and template-based synthesis [4] which can be multi-staged with occasional complicated procedures. Recently, a non-chemical, less complicated and low-cost synthesis of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> using industrial mill scale waste materials was reported [5, 6, 7]. Mill scale is a waste product generated by the steel industries, and is magnetic in nature and generally composed of iron oxides (Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub> and FeO) with a small amount of impurity elements. Recycling  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> from mill scale can be of immense benefit to the environment while reducing the cost of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>-based applications such as polymeric microwave absorbing

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<b>Table 1.</b> Composition of $(\alpha - Fe_2O_3)_x$ (PCL) <sub>100-x</sub> samples.							
α – Fe <sub>2</sub> O <sub>3</sub> Nanopowder		PCL		Total Mass			
(x %)	$Mass \pm 0.0005 \ g$	(100-x)%	$Mass \pm 0.0005 \ g$	±0.0005 g			
5.0	1.2500	95.0	23.7500	25.0000			
10.0	2.5000	90.0	22.5000	25.0000			
15.0	3.7500	85.0	21.2500	25.0000			
20.0	5.0000	80.0	20.0000	25.0000			

composites where recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> from mill scale has rarely been applied. Generally,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> has a low imaginary part of permittivity [8] and exhibits weak ferromagnetic properties [9] which restrict its use particularly as a filler in non-conducting polymer composites for microwave absorption applications. However, the imaginary permittivity limitation can be effectively reduced by modifying the particle sizes while retaining the magnetic properties, since the microstructure and morphology of ferrites are known to influence their complex permittivity properties [10]. Recycled α-Fe<sub>2</sub>O<sub>3</sub> nanoparticles with enhanced permittivity can be reinforced with non-conducting polymer matrices such as polycaprolactone (PCL) since as a thermoplastic polymer, it can easily be melt-blended with metal oxides [11, 12] to fabricate novel absorbers that could possess promising attenuation properties critical for electromagnetic interference (EMI) suppression and radar-signature reduction. This application can be further supported by the magnetic nature of the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles coupled with their high interfacial density due to their compactness. In this current study, the particle sizes of recycled a-Fe<sub>2</sub>O<sub>3</sub> were reduced to nanosize in order to improve the dielectric permittivity properties. Recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites containing a total of 5-20 % wt. of the nanoparticles were fabricated using the melt-blend technique. The effects of the nanofiller loadings on the complex permittivity and power loss (dB) due to absorption by the nanocomposites were then investigated in the 1-4 GHz frequency range. The finite element method (FEM) was also applied to simulate the x-component of the electric field (V/m) distributions of the microstrip shielded by the nanocomposites.

#### 2. Experimental

#### 2.1. $\alpha$ – Fe<sub>2</sub>O<sub>3</sub> nanoparticles synthesis from mill scale

Mill scale shavings (Perwaja Sdn. Bhd. Terengganu, Malaysia) were crushed and purified to obtain wustite (FeO) sludge using methods described in previous studies [6, 7]. The FeO sludge was dried before oxidizing in air with a Protherm furnace operating at a temperature of 600 °C with a holding time of 6 h to produce the  $\alpha$  – Fe<sub>2</sub>O<sub>3</sub> particles. A high energy ball mill (SPEX Sample Prep 8000D, SPEX SamplePrep LLC, Metuchen, N.J., U.S.A.) fitted with a 1425 rpm, 50 Hz motor which operated at a clamp speed of 875 cycles/minute was used for 12 h to mill the  $\alpha$  – Fe<sub>2</sub>O<sub>3</sub> particles into nanoparticles. A powder-to-ball ratio of 1:5 was applied and the steel vials of the mill were kept open for 2 min after every 50 min of continuous milling. This was to ensure the refreshment of the vials' atmosphere in order to avoid the transformation of  $\alpha$  – Fe<sub>2</sub>O<sub>3</sub> to Fe<sub>3</sub>O<sub>4</sub> as previously reported [13].

#### 2.2. Fabrication of $\alpha$ – Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites

Polycaprolactone (Sigma-Aldrich, St. Louis, MO, U.S.A.) of 1.146 g/ cm<sup>3</sup> density and 97.0% purity was melt-blended with 5% wt., 10% wt., 15% wt. and 20% wt. recycled hematite nanoparticles using the Brabender twin screw extruder (Brabender GmbH & Co. KG, Duisburg, Germany) operating at 65 °C to fabricate the nanocomposites. The total mass of each nanocomposite was 25g. Using a pressure of  $110 \text{ kg/cm}^2$ , the 5% wt., 10% wt., 15% wt. and 20% wt. recycled α-Fe<sub>2</sub>O<sub>3</sub>/Polycaprolactone nanocomposites were subsequently hot pressed into 6 cm  $\times$ 3.6 cm x 0.70 cm pellets for characterization. Table 1 shows the material composition for the fabrication of the nanocomposites based on total sample mass of 25 g. PCL was used in this work due to its high dielectric properties, easy formability and blend-compatibility, biodegradability, lightweight, and good mechanical properties. Moreover, other polymeric materials having the appropriate balance of these properties can also be utilized provided the fabricated nanocomposites would possess high imaginary part of relative complex permittivity which relates to high microwave absorption.





#### Figure 1. Complex permittivity measurement set up. OEC, open ended coaxial.



The microstructure structure and phase composition characteristics of the samples were investigated through X-ray diffraction (XRD) using a



Figure 2. Measurement of the transmission and reflection coefficients of the nanocomposites.



Figure 3. XRD patterns of recycled  $\alpha$  – Fe\_2O\_3 and  $\alpha$  – Fe\_2O\_3/PCL nanocomposites.

X'pert High Pro Panalytical (PW3040/60 MPD, Amsterdam, Netherlands) with Cu-Kα radiation ( $2\theta = 10-80^{\circ}$ ; scanning speed =  $2^{\circ}$ /min; operating voltage = 40.0 kV; current = 40.0 mA; wavelength = 1.5405 Å). Rietveld analysis was performed on all data with PANalytic X'Pert Highscore Plus v3.0 software (PANalytical B.V., Almelo, The Netherlands). The Inorganic Crystal Structure Database (ICSD) was used for the identification of the recycled α-Fe<sub>2</sub>O<sub>3</sub> nanoparticles by comparing the diffraction peaks with the database.

The size and shape of the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles were examined using high resolution transmission electron microscopy (HRTEM). Measurements were executed on a JEM-2100F (JEOL, Tokyo, Japan) and the size distribution of the particles was acquired by processing the HRTEM images using the particle analyzer of the ImageJ software (Version 1.50i, NIH, University of Wisconsin, Madison, WI, USA, 2016) for 100 particles. The recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticle dispersion in the PCL matrix was investigated with a JEOL JSM-7600 (JEOL, Tokyo, Japan) Field Emission Scanning Electron Microscope (FESEM).

#### 2.3.2. Complex permittivity

The complex permittivity properties of the samples were measured using the Agilent 85070B open ended coaxial (OEC) probe in conjunction with the Agilent N5230A PNA-L Vector Network Analyzer (Agilent Technologies, California, U.S.A.) in the 1–4 GHz range at room temperature. The OEC technique is suitable for the determination of the complex permittivity of solids, semi-solids and liquids. A one-port standard calibration (short – air – water) was performed after which unfilled polytetrafluoroethylene (standard reference material) was characterized to confirm the precision of the calibration. Measurements were taken by placing the OEC probe on the smooth and flat surfaces of the samples while avoiding an air gap between the surfaces in contact. The recycled hematite nanopowder was compacted into a cup-shaped sample holder (top diameter = 2.5 cm, base diameter = 2 cm, height = 3 cm) for the measurements. The instrumental set up of the measurements is as depicted in Figure 1.

### 2.3.3. Microwave absorption

The transmission/reflection line measurement technique was utilized for the determination of the magnitudes of the reflection coefficient (S<sub>11</sub>) and transmission coefficient (S<sub>21</sub>) of the nanocomposites in 1–4 GHz range. As illustrated in Figure 2, the measurement system comprised of an Anritsu MS 2024B VNA Master (Anritsu Corporation, Kanagawa, Japan) connected to a rectangular RT/duriod 5880 substrate microstrip of length 6.0 cm, width 5.0 cm and thickness 0.15 cm, having a transmission line (length = 6.0 cm, width = 1.5 mm) engraved on the surface along its broader side.

A full two-port Short-Open-Load calibration was performed to standardize the instruments for measurements. The samples were then placed flat onto the surface of the microstrip while ensuring air-gap free contact between the surfaces. The power loss (dB) due to absorption by the nanocomposites was subsequently calculated using the measured reflection and transmission coefficient magnitudes. The finite element method (FEM) was used to simulate the electric field distribution based on the microstrip model geometry covered by the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites. The simulations were executed on COMSOL Multiphysics software version 3.5 (COMSOL AB, Stockholm, Sweden, 2008) in the 1-4 GHz range using the permittivity values measured for the nanocomposites as inputs. The model geometry was created and the physical parameters and boundary conditions were set. The geometry was then discretized into finite elements which were then solved after applying RF electromagnetic waves with the harmonic propagation configuration. Parametric analysis was subsequently performed to obtain the x-component of electric field (V/m) distribution patterns.



Figure 4. TEM micrograph and size distribution of recycled α-Fe<sub>2</sub>O<sub>3</sub> nanoparticles.



Figure 5. FESEM images of recycled α-Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites with (a) 5% wt. (b) 10% wt. (c) 15% wt. (d) 20% wt. recycled α-Fe<sub>2</sub>O<sub>3</sub> filler content.

#### 3. Results and discussion

#### 3.1. Structure and composition

The XRD patterns of the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles and the nanocomposites are depicted in Figure 3. The Bragg peaks in the patterns for the nanoparticles were identified by comparing them with standard patterns of the Inorganic Crystal Structure Database (ICSD) which confirmed the crystal structure of the sample as single phase hexagonal (rhombohedral) of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, belonging to the R-3c space group and with ICSD reference number 98-001-2733. The patterns for the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites with different % wt. composition of α-Fe<sub>2</sub>O<sub>3</sub> nanopowder show two sharp Bragg peaks belonging to pure PCL located at  $2\theta$ angles of 21.36° and 23.78° (crystalline regions) while weak peaks were found at 22.0°, 40.26° and 44.36° (amorphous regions). The dominant peaks of the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller located at 33.12° and 35.51° can also be clearly identified in the nanocomposites, which became more pronounced as the nanofiller content increased. The absence of any new Bragg peaks or shift in the peak positions of the nanocomposites suggests that the recycled α-Fe<sub>2</sub>O<sub>3</sub> nanofiller did not interact chemically with the PCL matrix and that the mixture was physical in nature. Hence, the overall properties of the nanocomposites can be associated with the individual properties of the constituent materials.

The recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticle shape and size distribution, examined through high resolution transmission electron microscopy (HRTEM), are shown in Figure 4. The particles showed considerable agglomeration with aggregation, and the shapes were generally sphere-like. The agglomeration can be attributed to higher adhesive forces between particles due to increased specific surface area associated with the nanoparticles [14]. From the analysis of the micrographs using the ImageJ software, the particle size distribution was found to be in the range 10.3–24.5 nm with an average particle size of 16.2 nm.

The dispersal of the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles in the PCL matrix was examined from the FESEM micrographs of the nanocomposites shown in Figure 5. The surface morphologies of the nanocomposites

depict a uniformly dispersed recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles, seen as brighter (higher contrast) aggregates situated in the darker (lower contrast) background of PCL, which increased with the nanofiller loadings. The dispersion of recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> in the PCL matrix as discrete nanoparticles suggests their full incorporation, and could therefore provide the interfacial linkage needed for the enhancement of the dielectric properties of the nanocomposites.

#### 3.2. Dielectric characterization

The variation of the real ( $\varepsilon'$ ) and imaginary ( $\varepsilon''$ ) parts of relative complex permittivity,  $\varepsilon^* = \varepsilon' - j \varepsilon''$ , with frequency in the 1–4 GHz range for the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller (average particle size = 16.2 nm) and



Figure 6. Variation of  $\epsilon'$  with frequency for recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, PCL and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites.



**Figure 7.** Variation of  $\varepsilon''$  with frequency for recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, PCL and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites.

Table 2. Comparison of relative complex permittivity properties of α-Fe<sub>2</sub>O<sub>3</sub>.

Sample	ε′	ε"	Frequency (GHz)	Reference
α-Fe <sub>2</sub> O <sub>3</sub> (Recycled)	12.19	0.44	2.4	This study
α-Fe <sub>2</sub> O <sub>3</sub> (Alfa Aesar)	3.60	0.15	2.4	[ <mark>16</mark> ]
α-Fe <sub>2</sub> O <sub>3</sub> (Recycled)	11.94	0.40	10.0	[17]
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> (Alfa Aesar)	6.09	0.16	10.0	[18]

the nanocomposites are presented in Figures 6 and 7 respectively. The profiles of the  $\varepsilon'$  and  $\varepsilon''$  values displayed in the figures appear rippled, and this can be attributed to the impedance mismatch at the interface between the surface of the samples and open ended coaxial probe. The  $\varepsilon^\prime$ values for the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller shown in Figure 6 depict a decreasing trend with frequency, a behavior consistent with ferrites and supported by the Maxwell-Wenger polarization configuration [15]. However, the  $\varepsilon''$  pattern for the nanofiller demonstrated in Figure 7 describes an increasing trend with frequency, which conforms to the open ended coaxial probe's calibration profile for water in the 1–4 GHz range. It can be deduced from Figure 7 that the  $\varepsilon$ " value at 1 GHz was 0.40 which increased to 0.54 at 4 GHz, while the  $\varepsilon'$  values shown in Figure 6 were 12.75 and 12.04 at 1 GHz and 4 GHz respectively. Table 2 shows a comparison of the  $\epsilon'$  and  $\epsilon''$  values at 2.4 GHz and 10. 0 GHz for 16.2 nm recycled α-Fe<sub>2</sub>O<sub>3</sub> and 0.46 µm commercial α-Fe<sub>2</sub>O<sub>3</sub> measured using the open ended coaxial probe.

It can be observed at the specified frequencies that the values for the recycled α-Fe<sub>2</sub>O<sub>3</sub> nanoparticles are significantly higher than the recently reported values for the commercial  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. The higher  $\varepsilon''$  value is very significant since it corresponds to enhanced absorption and therefore lower transmission of electromagnetic waves through the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> as compared to the commercial  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. These results can be linked to the mechanical milling of the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> to produce nanoparticles in which there was compactness and less air gaps which gave rise to the formation of effective contact between neighboring particles and an increase in interfacial density. This contributed to the increase in the interfacial polarization resulting in the observed improved complex permittivity properties. The effect of recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller incorporation in PCL matrix, shown in Figures 6 and 7, indicates that the complex permittivity values of the 5% wt., 10% wt., 15% wt. and 20% wt.  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites followed the profile the nanofiller and increased with its % wt. content due to enhanced interfacial polarization, conductivity in the nanocomposite materials and hopping exchange of charges between localized states [19]. At 1 GHz, the  $\epsilon'$  and  $\epsilon''$  values of the 5% wt., 10% wt., 15% wt. and 20% wt. α-Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites were respectively 3.49, 3.68, 3.88, 4.07 and 0.30, 0.32, 0.35, 0.37 while the corresponding values at 4 GHz were



Figure 8. Power loss (dB) of the 5–20% wt. recycled  $\alpha\text{-}Fe_2O_3/\text{PCL}$  nanocomposites.

3.21, 3.34, 3.49, 3.64 and 0.31, 0.34, 0.36 and 0.39. It is evident from these results that the 16.2 nm recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller has been effective in enhancing the complex permittivity of the PCL matrix using low loadings (1.25 g–5 g as shown in Table 1) and can as well support the light-weight requirement of microwave absorbers.

#### 3.3. Microwave absorption

The ability of a material to attenuate an electromagnetic signal can be determined by calculating the energy dissipated or power loss (dB) due to material absorption using power ratios related to reflection coefficient (*R*), transmission coefficient (*T*) and absorption (*A*) expressed as:  $P_a/P_i = A$ ;  $P_o/P_i = T = |S_{21}|^2$ ; and  $P_r/P_i = R = |S_{11}|^2$ , where [20];

$$P_i = P_a + P_r + P_a \tag{1}$$

 $P_b$ ,  $P_a$ ,  $P_r$  and  $P_o$  are respectively the input power, absorbed power, power reflected at the input interface and output power. The input power can further be presented as;

$$1 = (P_a + P_r + P_o) / P_i = A + |S_{11}|^2 + |S_{21}|^2$$
(2)

Absorption (A) can be perceived as the power loss or the energy dissipated in the electromagnetic wave and may be stated from Eq. (2) as;

Power loss(dB) = 
$$10 \log(1 - |S_{11}|^2 - |S_{21}|^2)$$
 (3)

The difference between the power loss (dB) of each nanocomposite and open microstrip power loss (dB) characterizes the material absorption. Figure 8 shows the variation in power loss for the 5% wt., 10% wt., 15% wt. and 20% wt. recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites in the 1–4



Figure 9. Variation in power loss with nanofiller content at specified frequencies.



Figure 10. X-component of the electric field (V/m) distribution patterns for the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites (a) 5% wt.  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (b) 10% wt.  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (c) 15% wt.  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, and (d) 20% wt.  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>.

GHz range. Generally, the profiles follow the behavior of the  $\varepsilon$ " values exhibited by the nanocomposites in Figure 7, while the values were observed to be increasing with nanofiller composition and varied from 11.25 (dB) at 1 GHz to a maximum of 16.37 (dB) at about 3.4 GHz.

The increase in power loss with nanofiller composition can be attributed to the dependence of material absorption on  $\varepsilon''$  values.  $\varepsilon''$  is largely associated with the loss of electromagnetic energy in materials and as a result, increasing the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller content in the nanocomposites enhanced the  $\varepsilon''$  leading to reduced transmission of the electromagnetic wave due to higher absorption. Figure 9 suggests a linear relationship between the power loss (dB) and recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller content for all the samples at the specified frequencies, where the strongest correlation was located at 2.4 GHz. It also indicates that increasing the nanofiller content beyond 20% wt. could lead to further increases in the absorption properties of the nanocomposites. The power loss values at 2.4 GHz for the 5% wt., 10% wt., 15% wt. and 20% wt. recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites were respectively 13.3 dB, 13.7 dB, 14.1 dB and 14.4 dB.

The visualization of the FEM simulated electric field (V/m) distribution (x-component) at 4 GHz using the microstrip model geometry covered by the nanocomposites is as shown in Figure 10. The electric field (V/m) distribution patterns were based on the permittivity values of the nanocomposites which were used as inputs for the simulation. Generally, there was a noticeable shift in the maximum and minimum values of the electric field distribution and a decrease in the intensity of the propagating wave from the input to the output port as the nanofiller content increased due to material absorption. As expected, a material with a higher  $\varepsilon^{"}$  possesses a higher absorption properties and can effectively attenuate the propagation of electromagnetic waves through it. Therefore the higher the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanofiller content, the higher is the absorption loss leading to the lowering of the intensity of the propagating wave. These results are consistent with the  $\varepsilon^{"}$  and power loss (dB) results for the nanocomposites.

#### 4. Conclusion

Recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites were fabricated using the melt-blend technique and their complex permittivity and power loss

characteristics investigated in the 1-4 GHz frequency range. The recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> was synthesized from mill scale shavings and the particle size reduced to an average value of 16.2 nm via high energy ball milling for 12 h. This reduction to nanosize enabled an improvement in the complex permittivity property of the nanofiller which influenced the absorption loss properties of the nanocomposites. From the presented results, the nanocomposites with higher recycled α-Fe<sub>2</sub>O<sub>3</sub> nanofiller content showed greater complex permittivity properties and the corresponding power loss (dB) values were also higher and varied from 11.25 (dB) to 16.37 (dB) in the 1–4 GHz frequency range. The recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>/PCL nanocomposites are therefore promising alternatives for microwave noise reduction materials in the 1–4 GHz range in view of their low cost, low density, biodegradability and attractive absorption behavior. Additionally, the recycled  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanoparticles exhibited superior complex permittivity properties to commercial (Alpha Aesar) α-Fe<sub>2</sub>O<sub>3</sub> in the 1-4 GHz and 8-12 GHz ranges and thus have the potential for use as fillers in other polymeric composites since their application can reduce the cost of ferrite-based microwave absorbing materials significantly without compromising the absorption efficiency of the materials.

#### Declarations

#### Author contribution statement

Ebenezer Ekow Mensah: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

Zulkifly Abbas: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Raba'ah Syahidah Azis: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Nor Azowa Ibrahim: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data.

Ahmad Mamoun Khamis, Daw Mohammad Abdalhadi: Performed the experiments; Analyzed and interpreted the data.

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#### Data availability statement

Data will be made available on request.

#### Declaration of interests statement

The authors declare no conflict of interest.

#### Additional information

No additional information is available for this paper.

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