



# Article **Microwave Absorption of** $\alpha$ **-Fe**<sub>2</sub>**O**<sub>3</sub>**@diatomite Composites**

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**Abstract:** A neoteric round sieve diatomite (De) decorated with sea-urchin-like alpha-type iron trioxide ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) synthetics was prepared by the hydrothermal method and further calcination. The results of the electromagnetic (EM) parameters of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>-decorated De ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D) showed that the minimum reflection loss (RL<sub>min</sub>) of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D could reach -54.2 dB at 11.52 GHz and the matched absorber thickness was 3 mm. The frequency bandwidth corresponding to the microwave RL value below -20 dB was up to 8.24 GHz (9.76–18 GHz). This indicates that  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D composite can be a lightweight and stable material; because of the low density of De (1.9–2.3 g/cm<sup>3</sup>), the density of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D composite material is lower than that of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (5.18 g/cm<sup>3</sup>). We found that the combination of the magnetic loss of sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and the dielectric loss of De has the most dominant role in electromagnetic wave absorption and loss. We focused on comparing the absorbing properties before and after the formation of sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and explain in detail the effects of the structure and crystal shape of this novel composite on the absorbing properties.

Keywords: diatomite; α-Fe<sub>2</sub>O<sub>3</sub>; microwave absorption

### 1. Introduction

At present, with the widespread application and rapid development of radio equipment, electromagnetic (EM) interference, electromagnetic radiation and electromagnetic compatibility have become an important issue. On the one hand, they endanger the health of human and electronic equipment, On the other hand, they affect the development of the modern military [1–6]. Electromagnetic wave-absorbing materials (EWAM) have been extensively used in many areas of social life, for example in reducing electromagnetic radiation. In the future, the EWAM should have the characteristics of excellent electromagnetic absorption performance, thin thickness, a wide, effective absorbing bandwidth (EAB) and being of light weight to satisfy different application requirements. It is well known that the combination of different compounds which have excellent microwave properties has led to new composite materials which have earned great technological interest in recent years. The addition of a second phase can significantly improve the electronic properties of the resulting composite materials: Co<sub>0.5</sub>Ni<sub>0.5</sub>Ga<sub>0.01</sub>Gd<sub>0.01</sub>Fe<sub>1.98</sub>O<sub>4</sub>/ZnFe<sub>2</sub>O<sub>4</sub> spinel ferrite nanocomposites [7] and carbon nanotubes/BaFe<sub>12-x</sub>Ga<sub>x</sub>O<sub>19</sub>/epoxy composites [8]. There are various types of ferrites based on their crystal structure, including Fe<sub>3</sub>O<sub>4</sub> [9], gamma- $Fe_2O_3$  ( $\gamma$ - $Fe_2O_3$ ) [10],  $\alpha$ - $Fe_2O_3$  [11] and so on. Due to its stable chemical properties [12], environment-friendly features [13], and controllable morphology [14],  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> can aid in the design of EWAMs. However, Fe<sub>3</sub>O<sub>4</sub> and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> are easy to reunite, resulting in weak RL performance [15,16]. Any synthesis for massive production should provide a large batch of the primary material of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> in this particular case [17]. There are techniques providing large batches, such as the Polyol Method [18]. In the case of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, one of



Citation: Zhang, C.; Wang, D.; Dong, L.; Li, K.; Zhang, Y.; Yang, P.; Yi, S.; Dai, X.; Yin, C.; Du, Z.; et al. Microwave Absorption of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@diatomite Composites. *Int. J. Mol. Sci.* **2022**, 23, 9362. https:// doi.org/10.3390/ijms23169362

Academic Editor: Ana María Díez-Pascual

Received: 1 July 2022 Accepted: 10 August 2022 Published: 19 August 2022

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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). the most successful techniques is the laser target evaporation, for which both structural and magnetic properties, including microwave characterization techniques, have been carefully studied [19,20].  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> molecules are quite heavy (5.18 g/cm<sup>3</sup>) as traditional EWAMs. In order to solve this problem, a method of loading EWAMs on the surface of low-density materials is proposed. The porous structure of diatoms gives them a light mass  $(1.9-2.3 \text{ g/cm}^3)$ . This porous structure has a huge advantage. It has been reported that core/shell structured nanocomposites based on magnetic nanomaterials exhibit low reflection loss [21–27]. However, the effective absorption bandwidth of core/shell structure materials is not wide enough [28–30]. Therefore, on the basis of the existing research, the light and porous structure of diatomite (De) is used to load a rough sea urchin-like structure of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. Hollow micro-spheres and micro-organisms were used as templates to prepare ferrite particles decorated with De through the hydrothermal method [31-37]. De has the excellent characteristics of biological diatoms and a unique pore structure [38]. Therefore, as a low-density material, De with a hollow double-shell structure is considered to become an excellent material for ferrite particle loading. Lv et al. [39] reported the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@CoFe<sub>2</sub>O<sub>4</sub> core–shell composites. When the thickness of the coating is 2 mm, the  $RL_{min}$  of the composite is -60 dB at 16.5 GHz. The frequency bandwidth corresponding to the microwave RL value below -10 dB was up to 5 GHz (13–18 GHz). Guo et al. [40] reported Mg<sub>x</sub>Zn<sub>1-x</sub> ferrite/diatomite composites. The RL<sub>min</sub> of the composite is -7.23 dB at 15.4 GHz. In this work, a  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanorod composite was synthesized by the hydrothermal method and further calcination. The results showed that the RL<sub>min</sub> of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>-decorated De ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D) could reach -54.24 dB at 11.52 GHz and the matched absorber thickness was 3 mm. The frequency bandwidth corresponding to the microwave RL value below -20 dB was up to 8.24 GHz (9.76–18 GHz).

#### 2. Results and Discussion

The surface appearance of prepared MnO<sub>2</sub>@D, FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D is showed in Figure 1. Energy dispersive spectrometer (EDS) mapping (see Supporting information, Figure S2) of MnO<sub>2</sub>@D determined that the MnO<sub>2</sub> nanosheets were successfully prepared by the one-step hydrothermal method. The picture in Figure 1a–c indicates the welldistributed growth of MnO<sub>2</sub> nanosheets on the De. MnO<sub>2</sub> nanosheets were decorated on De and connected with each other to form a highly uniform surface morphology. SEM images in Figure 1d–f and its EDS mapping (see Supporting information, Figure S3) results further demonstrated that MnO<sub>2</sub> nanosheets have been completely transferred into seaurchin-like FeOOHs [41]. The picture in Figure 1g–i indicates that the uniform growth of sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> is similar to FeOOH. EDS (see Supporting information, Figure S4) mapping results further showed that the chemical elements of sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> are mainly made up of O, Fe and K, proving that the sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> specimens were prepared successfully.

Figure 2a presents the chemical constitution of the De and MnO<sub>2</sub>@D. The four characteristic diffraction peaks at 21.8°, 28.2°, 31°, 36° and 44.4° in De are attributed to the (101), (111), (102), (112) and (202) planes of SiO<sub>2</sub>, respectively, with a quartzite structure (JCPDS card no. 82-0512, a = 4.997 Å, b = 4.973 Å, c = 7.070 Å), which affirms a high crystallization grade. From the XRD patterns of MnO<sub>2</sub>@D, the diffraction peaks at 12.5°, 25.2°, and 37° nearly conform to the normal XRD patterns of birnessite-type manganese oxide crystal (JCPDS card no.80-1098, a = 5.149 Å, b = 2.843 Å, c = 7.176 Å). In particular, the high diffraction peaks of De at 36.1 became wide. The X-ray diffraction patterns in Figure 2a indicate the crystal texture and chemical constitution of FeOOH@D. The obvious broad peaks centered at 33.2°, 34.7°, 36.6°, 40°, 41.2°, 53.2°, 58.9°, and 61.3° correspond to the (130), (021), (111), (121), (140), (221), (151) and (002) of goethite FeOOH, respectively (JCPDS No. 81-0463, a = 4.616 Å, b = 9.955 Å, c = 3.023 Å). In Figure 2a, diffraction peaks of (012), (104), (110), (113), (024), (116), (214), and (300) are similar to the hematite phase (JCPDS card no. 33-0664). This suggests that the FeOOH had been completely transferred into  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>.



**Figure 1.** Magnification increases from left to right. SEM images of (**a**–**c**) MnO<sub>2</sub>@D; (**d**–**f**) FeOOH@D; (**g**–**i**) α-Fe<sub>2</sub>O<sub>3</sub>@D.



**Figure 2.** XRD patterns of De, MnO<sub>2</sub>@D, FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D. (a) The XPS of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D: survey; (b) O 1s; (c) Fe 2p; (d) (M–H) loops of MnO<sub>2</sub>@D, FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D; (e) Illustration: the relationship between magnetization and magnetic field of the sample is shown in an enlarged view.

To go further to determine the electronic structure, chemical bonding state and composition of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D nanocomposites, XPS analysis was performed. The XPS survey spectrum (Figure 2b) shows that O, Fe and Si elements coexist in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D. The XPS spectra of O1s (Figure 2c) shows three peaks, which were located at 529.7, 531.3 and 532.7 eV. These peaks were assigned as metal–oxygen bonds (Fe–O), silicon–oxygen bonds (Si–O–Si) and surface-adsorbed water (H<sub>2</sub>O), respectively [42–44]. There are two main peaks in XPS spectra of the Fe 2p region at 710.7 and 724.3 eV (Figure 2d), which reflect the Fe 2p<sub>3/2</sub> and Fe 2p<sub>1/2</sub> orbitals, respectively. Their satellite peaks are clearly distinguishable at 718.8 and 733.6 eV respectively. These peaks belong to Fe<sup>3+</sup> species [45,46]. The existence of Fe–O further illustrated the successful preparation of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. The detailed Rietveld refinement images of samples are shown in the image (See Supporting information, Figure S5). The cell parameters are exhibited in the table (See Supporting information, Table S1).

The magnetic hysteresis loops of the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D compound obtained by a vibrating sample magnetometer are demonstrated in Figure 2e. It displays the applied external field-dependent magnetization hysteresis (M-H) loops of the samples measured between -10 K and 10 K Oe at room temperature. It can be seen that all the samples show similar S-type hysteresis loops, indicating their ferromagnetic behavior. The saturation magnetizations (Ms) of MnO<sub>2</sub>@D, FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D are 0.19, 0.14, and 0.11 emu·g<sup>-1</sup>, respectively. The composite material is composed of a magnetic shell and a diamagnetic De core. Therefore, the decrease of the Ms value mainly depends on the change of ferrite content on the surface. At the same time, the coercivity (Hc) of samples fluctuated in the range of  $-7.08 \sim 60.75$ ,  $-1.74 \sim 60.39$  and  $-28.41 \sim 32.78$  Oe. The main reason for the Hc decrease may be that the average particle size increases with the increase of the specific surface area of De during the growth of surface metal oxides.

MnO<sub>2</sub> nanosheets are a dielectric-absorbing material, and their electromagnetic attenuation properties are mainly attributed to their unique lamellae and strip morphologies. However, due to the lamellae structure of pure MnO<sub>2</sub>, it is not suitable for electronic transmission; the real part of the dielectric constant of  $MnO_2(\varepsilon')$  is smaller than that of FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D. As antiferromagnetic materials, the magnetic loss of De and  $MnO_2$  is small enough to be ignored, so the real part ( $\mu'$ ) of the magnetic permeability of MnO<sub>2</sub>@D under alternating electric field is less than one. At the same time the imaginary parts of the dielectric constant ( $\varepsilon''$ ) represent the loss moduli of electric energy and permeability ( $\mu''$ ) represent the loss moduli of magnetic energy. The  $\varepsilon''$  and  $\mu''$  values of MnO<sub>2</sub> are low at low frequencies, but show a temporary upward trend at high frequencies, indicating that at low frequencies the electromagnetic wave absorption capacity of  $MnO_2$  is poor (Figure 3a). The values of  $\mu'$  and  $\mu''$  can be reflected in the ratio of saturated magnetization of non-magnetic composite particles. In contrast, the specific EM parameters of FeOOH@D are given in Figure 3d. After calcination at 350 °C, the impedance matching of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D was further improved, and the  $\varepsilon''$  was increased at a certain frequency (Figure 3j). This change may be caused by various aspects; the porous outer layer of De can be regarded as a micro-ring, while the radio-symmetric sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> can be regarded as numerous antennas that convert electromagnetic waves into vibrating microcurrents. Therefore, a micro current can be generated in the micro circuit, resulting in a dielectric resonance peak in the  $\varepsilon''$  curve.



**Figure 3.** Relevant EM parameters of MnO<sub>2</sub>@D; (**a**–**c**) FeOOH@D; (**d**–**f**) and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D; (**g**–**i**). Frequency dependence of  $\varepsilon'$ ,  $\varepsilon''$ ,  $\mu'$  and  $\mu''$  (**a**,**d**,**g**); dielectric and loss tangent (**b**,**e**,**h**); attenuation constant  $\alpha$  and  $\mu''(\mu')^{-2}f^{-1}$  values (**c**,**f**,**i**).

Due to the dielectric loss tangent  $(\varepsilon''/\varepsilon')$  and magnetic loss tangent  $(\mu''/\mu')$  value being calculated, the influence of dielectric loss and magnetic loss on the material was reflected (Figure 3b,e,h). As can be seen from the figure (Figure 3b,e,h), from top to bottom, the dielectric loss angle tangent and magnetic loss angle tangent of the three samples all show certain peaks at specific frequencies. The relatively high value of  $\tan \delta_{\varepsilon}$  indicates that sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> exhibits a strong dielectric loss, which is caused by dipole polarization, interfacial polarization and related relaxation phenomena. Meanwhile, the formation of new phases due to the synergistic interaction between different compounds is also helpful to improve the electromagnetic wave-absorption performance.

The calculation formula of RL [47,48]:

$$RL(dB) = 20\log|(Z_{in} - Z_0)/(Z_{in} + Z_0)|$$
(1)

$$Z_{in} = Z_0 \sqrt{\mu_r / \varepsilon_r} \tanh[j(2\pi f d/c) \sqrt{\mu_r \varepsilon_r}]$$
<sup>(2)</sup>

The thickness of the specimen is d, the input impedances of the absorbing material and air are  $Z_{in}$  and  $Z_0$ . The top to bottom rows represent the RL values of MnO<sub>2</sub>@D to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D, and the three columns on left are 1D plots of the RL values of MnO<sub>2</sub>@D to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D as a function of frequency and thickness; the middle of three columns is 2D and the three columns on right are 3D in Figure 4. MnO<sub>2</sub> is an impedance-matching material. If the value of MnO<sub>2</sub>@D cannot always be adjusted to -20 dB in the range of 2–18 GHz (Figure 4a–c), it indicates that MnO<sub>2</sub>@D does not have a good absorbing ability and is suit-

able for adjusting impedance matching materials. FeOOH@D has the potential to become an electromagnetic wave-absorbing material. When the thickness of FeOOH@D is 2.3 mm, the total absorption bandwidth of FeOOH@D is 4.2 GHz at high frequencies (13.8–18 GHz). However, for good absorbent materials, these are not enough. Its mediocre absorbing properties at low frequencies limit the application of FeOOH@D as an absorbing material. After calcination, the magnetic ferrite nanomaterial has a complex geometric structure, which is combined with De to form a double-shell structure. The outer shell is  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, and the inner shell is De. The enhanced microwave absorption performance is obtained. The electromagnetic absorption performance is gradually enhanced (Figure 4g-i). When the thickness of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D is 3 mm, the f<sub>E</sub> reaches 8.24 GHz (9.76–18 GHz); meanwhile, the ( $RL_{min}$ ) can reach -54.2 dB. The results show that the thickness of the material, the effective absorption bandwidth and excellent electromagnetic absorption performance allow  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D to satisfy the needs of a good-performance absorbing material. Rational material design and morphological construction endow  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D with a good adsorption performance. At the same time, the RL<sub>(min)</sub> and effective absorption bandwidth (RL < -20 dB) of MnO<sub>2</sub>@D, FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D are shownn in Table 1.



**Figure 4.** One—dimensional, two—dimensional, and three—dimensional picture of the RL values, which varies with frequency and thickness:  $(a-c) MnO_2@D$ , (d-f) FeOOH@D and  $(g-i) \alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D.

**Table 1.** RL(min) and effective absorption bandwidths (RL < -20 dB) of MnO2@D, FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D.

Sample Name	RL <sub>(min)</sub>	Effective Absorption Bandwidth (RL < $-20 \text{ dB}$ )	
MnO <sub>2</sub> @D	-7.9 dB	0	
FeOOH@D	−17.8 dB	0	
α-Fe <sub>2</sub> O <sub>3</sub> @D	-54.2 dB	8.24 GHz	

Specifically, good material design should include the following aspects. First, the electric and magnetic energy can be availably removed in the electromagnetic field, because of the synergistic action of the magnetic loss and the dielectric loss. In this work, the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> was essential to the magnetic loss component. The influence of the eddy current loss on the absorption results can be roughly analyzed by calculations. The following equation is usually used to judge this effect [41]:

$$\mu'' = 2\pi\mu_0 (\mu')^2 \sigma d^2 f$$
(3)

The right-hand side of the equation above is a constant. If the magnetic permeability parameter of the material meets the above formula in the frequency range, the magnetic loss is eddy current loss only. The results show that the value of  $\mu''(\mu')^{-2}f^{-1}$  fluctuates up and down, not horizontally. Therefore, it can be judged that there are other important reasons such as hysteresis loss, not just eddy current loss. In addition to magnetic loss, dielectric loss is also an important reason for the good absorbing performance of materials. When an electromagnetic wave is incident on a dielectric material, the first microscopic mechanism encountered is the polarization of the medium, which is quite different from that of a conductor with free electrons. In De, the electrons are in bound state, under the action of alternating electric fields. Due to the layered structure of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> molecules, the electromagnetic wave produces dielectric polarization phenomena; the center position of the positive and negative charges from superposition into separation generates rotating torque, that is, the electric dipole moment. At the same time, a weak electric field is formed. Therefore, the dielectric loss of the material mainly comes from the rotation and orientation of the dipole during the polarization process, and when the frequency of the applied electric field is consistent with the frequency of the thermal vibration of the molecule, it mainly comes from the resonance.

The loss tangent and attention constant ( $\alpha$ ) can be calculated directly from the EM parameters. The attention constant can characterize the ability of the absorber to attenuate electromagnetic waves. The higher the value, the stronger the ability of material to dissipate electromagnetic waves. The specific calculation formula is as follows [49]:

$$\alpha = \left(\sqrt{2}\pi f/c\right) \times \sqrt{\left(\mu'' \varepsilon'' - \mu' \varepsilon'\right) + \sqrt{\left(\mu'' \varepsilon'' - \mu' \varepsilon'\right)^2 + \left(\mu' \varepsilon'' + \mu'' \varepsilon'\right)^2}$$
(4)

The velocity of the electromagnetic wave in vacuum is *c*, The frequency of the incident electromagnetic wave is *f*. As you can see from the Figure 3c, *f*, *i*,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D reaches a peak value at about 6 GHz, followed by repeated fluctuations at high frequencies, indicating that there is a good microwave absorption effect at 6 GHz, while there is absorption instability at high frequencies, possibly because the batch stability of De needs to be further improved.

The microwave absorption mechanism of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D composites is shown in Figure 5. At low frequencies, ferrites including  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> exhibit domain wall resonance with increasing magnetic loss. The defects of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> can induce local dipole polarization. Electron migrate in  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. At the same time, the constancy in the values of  $\varepsilon'$  indicates the existence of a polarization process, that is, the oscillations of the electric dipole moment coincide or are slightly out of phase with the microwave frequency. The most possible mechanism in this frequency range is orientational polarization [50,51]. First, there are abundant  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>-De interfaces in the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D core–shell structure that promote the aggregation and transfer of free electrons to the interface. This accumulation leads to interfacial polarization relaxation, which is favorable for microwave dissipation into other energy forms. Second, due to the closed current lines formed on the surface of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, the induced current in the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D core–shell composite material leads to energy loss, and eddy current loss occurs. The interaction between  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and De contributes to the formation of natural resonance and exchange resonance, which is also one of the reasons for the occurrence of magnetic loss. Finally, the abundant sea-urchin-like  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> on the surface of De can scatter electromagnetic waves and deflect the direction of the



incident electromagnetic waves in the same direction, resulting in electromagnetic wave energy offset.

Figure 5. Microwave absorption mechanism of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D composite materials.

In Figure 6, the values of  $Z_r = \sqrt{\mu_r/\varepsilon_r}$  increase and then decrease as frequency increases. This may be because  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D creates the right eddies when electromagnetic waves reach the surface. This matches the multiple reflection of De and enhances the incidence and absorption of electromagnetic waves. Due to the skin effect of dense  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D, the impedance-matching performance decreases, and the overflow eddy current breaks the equilibrium.



**Figure 6.** Frequency dependence of impedance matching Zr of the MnO<sub>2</sub>@D, FeOOH@D and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D composite material.

Compared with other De-based EWAM, the fabricated  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D in this work contained two major advantages. Firstly, De has the characteristics of size control; De has an excellent size, otherwise it is not conducive to the adhesion of other load materials, and if De is not uniform, it will affect the uniformity of composite materials. Additionally, the double-shell structure is connected by tight chemical bonds, which ensures the safety of the double-shell structure. Therefore, five samples with De-based were selected to compare their absorbing properties with our samples, as shown in Table 2. The consequences indicated that the RL<sub>min</sub> of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D is -54.2 dB. The wide effective absorption bandwidth (8.24 GHz) at a thinner thickness (3 mm) demonstrated that the EWAM has the characteristics of being lightweight and of high efficiency, so its application prospect is very broad in the future.

Table 2. Comparison with other De basic materials.

Sample Name	Percentage (wt.%)	RL <sub>min</sub> (dB)	Absorberthickness (mm)	EAB(RL < -10  dB)  (GHz)	Reference
$Fe_2O_4/\alpha$ - $Fe_2O_3$	20	-52.69	3	5.36	[52]
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> -graphene	15	-30.6	4	5.5	[53]
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> /OPEFB fiber/PCL	25	-38	6	3	[54]
$RGO/PANI/\alpha$ -Fe <sub>2</sub> O <sub>3</sub> @SiO <sub>2</sub> with 1:4	16.7	-31.06	5	2	[55]
$RGO/PANI/\alpha$ -Fe <sub>2</sub> O <sub>3</sub> @SiO <sub>2</sub> with 1:6	16.7	-25.88	4	3	[55]
$\alpha$ -Fe <sub>2</sub> O <sub>3</sub> @D	20	-54.2	3	8.24	this work

## 3. Methods and Materials

 $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D, the De, potassium hypermanganate [KMnO<sub>4</sub>], ferrous sulfate [FeSO<sub>4</sub>·7H<sub>2</sub>O], and ethylene glycol [HOCH<sub>2</sub>—CH<sub>2</sub>OH] were purchased from Aladdin. In this work, all the lab supplies used were analytically pure and used without further purification.

## 3.1. Synthesis of MnO<sub>2</sub> Nanosheets on De

The one-step hydrothermal method was used to prepare  $MnO_2$ -decorated De ( $MnO_2@D$ ). Typically, 100 mg of De and  $KMnO_4$  homogeneous solution (70 mL, 0.05 M) was put into a 100 mL autoclave. The Teflon-lined stainless-steel autoclave was subsequently sealed and maintained in a homogeneous reactor at 160 °C for 24 h. The solid precipitates were washed with distilled water and alcohol, and dried in atmosphere at 60 °C for 12 h.

# 3.2. Preparation of Sea-Urchin-Like FeOOH on De

The sea-urchin-like FeOOH on De was achieved via a hydrothermal method. An amount of 110 mg FeSO<sub>4</sub>·7H<sub>2</sub>O was dissolved into 70 mL of solvent including distilled water and ethylene glycol (V<sub>distilled water</sub>/V<sub>ethylene glycol</sub> = 7/1), then the mixed solvent was stirred for about 10 min. Next, 80 mg MnO<sub>2</sub>@D and 70 mL FeSO<sub>4</sub>·7H<sub>2</sub>O mixed solution were transferred into a 100 mL Teflon-lined stainless-steel autoclave and maintained at 120 °C for 2 h in a homogeneous reactor. The autoclave was cooled to 25 °C, after the hydrothermal reaction finished. The samples were washed with distilled water and alcohol; after that, the washed samples were dried in atmosphere at 60 °C for 12 h.

# 3.3. Preparation of $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D

 $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D was obtained by calcination of FeOOH@D at 350 °C for 2 h under O<sub>2</sub> atmosphere. For the preparation flow chart of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D, (see Supporting information, Figure S1)

# 3.4. Characterization of Material

The nanostructures and surface appearance of the material were characterized by scanning electron microscopy (FIB/SEM, ZEISS AURIGA). The crystal texture and chemical constitution of the nanostructures were confirmed by X-ray diffraction (XRD, PANalytical X'Pert Powder), and the data of XRD results were analyzed with the JADE 6.0 software. The data of X-ray photoelectron spectroscopy (XPS) spectra results were obtained on a spectrometer (ESCALAB 250Xi). To measure the permittivity and permeability of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D, the compounds were prepared by mixing the as-prepared  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D with

melted paraffin according to the mass fractions of 20%, then each mixture was pressed to form a cylindrical toroidal with an outer diameter of 7.0 mm, inner diameter of 3.04 mm and thickness of 3.0 mm. The relative complex permeability ( $\varepsilon_r = \varepsilon' - j\varepsilon''$ ) and permittivity ( $\mu_r = \mu' - j\mu''$ ) were resulted by the coaxial line method with an Agilent 85071E vector network analyzer in the frequency range of 2.0–18.0 GHz.

#### 4. Conclusions

The conclusion of the paper is updated as follows: in summary, a series of three samples with a double hull configuration were systematically designed by the hydrothermal method. By decorating De with  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> nanowires, the electrical conductivity, dielectric properties and impedance matching of the De were improved effectively. Therefore,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D exhibits enhanced EM wave-absorption performance thanks to its optimal dielectric performance and impedance matching. The RL<sub>min</sub> of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D could reach –54.2 dB at 11.52 GHz and the matched absorber thickness was 3 mm. The frequency bandwidth corresponding to the microwave RL value below –20 dB was up to 8.24 GHz (9.76–18 GHz).  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>@D synthesized in this paper has been applied to the electromagnetic wave absorption field for the first time, providing an important reference for eliminating the influence of electromagnetic interference on the human body and equipment.

**Supplementary Materials:** The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/ijms23169362/s1.

**Author Contributions:** Conceptualization, C.Z. and D.W.; data curation, C.Z., D.W. and K.L.; formal analysis, K.L. and Z.D.; funding sacquisition, Y.Z. (Yuxin Zhang); investigation, C.Z., D.W., K.L., Q.Z., Z.Y., C.Y., Y.Z. (Yifan Zhang) and P.Y.; methodology, C.Z., D.W. and Y.Z. (Yuxin Zhang); project administration, Y.Z. (Yuxin Zhang); software, L.D.; validation, S.Y. and X.D.; visualization, Z.D.; writing—original draft preparation, C.Z. and D.W.; writing—review and editing, P.Y., J.R. and X.Z. All authors have read and agreed to the published version of the manuscript.

**Funding:** The authors gratefully acknowledge the financial support provided by the National Natural Science Foundation of China (Grant No. 51908092), Projects (No. 2020CDJXZ001, 2021CDJJMRH-005 and SKLMT-ZZKT-2021M04) supported by the Fundamental Research Funds for the Central Universities, the Joint Funds of the National Natural Science Foundation of China-Guangdong (Grant No. U1801254), the project funded by Chongqing Special Postdoctoral Science Foundation (XmT2018043), the Chongqing Research Program of Basic Research and Frontier Technology (cstc2017jcyjBX0080), Natural Science Foundation Project of Chongqing for Post-doctor (cstc2019jcyjbsh0079, cstc2019jcyjbshX0085), Technological projects of Chongqing Municipal Education Commission (KJZDK201800801), the Innovative Research Team of Chongqing (CXTDG201602014) and the Innovative technology of New materials and metallurgy (2019CDXYCL0031). The authors also thank the Electron Microscopy Center of Chongqing University for materials characterizations.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data of the compounds are available from the authors.

Conflicts of Interest: The authors declare no conflict of interest.

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