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Facile fabrication of carbon microspheres decorated with $\text{B}(\text{OH})_3$ and $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles: superior microwave absorption

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Abstract

We demonstrate that novel three-dimensional (3D) $\text{B}(\text{OH})_3$ and $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles decorated carbon microspheres ($\text{B}(\text{OH})_3/\alpha\text{-Fe}_2\text{O}_3\text{-CMSs}$) can be fabricated *via* a facile thermal treatment process. The carbon microspheres with diameter of 1 to 3 μm and decorated $\text{B}(\text{OH})_3$ and $\alpha\text{-Fe}_2\text{O}_3$ nanoparticles with diameters of several to tens of nanometers are successfully fabricated. These novel 3D $\text{B}(\text{OH})_3/\alpha\text{-Fe}_2\text{O}_3\text{-CMS}$ composites exhibit enhanced microwave absorption with tunable strong absorption wavebands in the frequency range of 2–18 GHz. They have a minimum reflection loss (RL) value of -52.69 dB at a thickness of 3.0 mm, and the effective absorption bandwidth for RL less than -10 dB is as large as 5.64 GHz. The enhanced microwave absorption performance arises from the synergy of the impedance matching caused by the $\text{B}(\text{OH})_3$ nanoparticles, dielectric loss as well as the enhancement of multiple reflection

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among 3D α -Fe₂O₃ nanocrystals. These results provide a new strategy to tune electromagnetic properties and enhance the capacity of high-efficient microwave absorbers.

Keywords: Three-dimensional structure, B(OH)₃ and α -Fe₂O₃ nanoparticles, Carbon microspheres, Microwave absorption

1. Introduction

With the rapid development of the information technology, the massive usage of electrical and electronic devices has generated a new kind of pollution known as electromagnetic interference. This electromagnetic pollution is not only adversely affecting the operation of electronic devices but is also harmful to the health of human beings [1-4]. Therefore, there is a high demand for the high-performance microwave absorption (MA) materials with a good absorption capacity that can effectively eliminate adverse microwaves [5-13]. An ideal MA material is required to have not only a wide absorption frequency range and strong absorption properties, but also lightweight feature, low filler loading ratio, good corrosion resistance, and high thermal stability [14, 15]. Recently, carbon-based composites such as graphite [16], carbon nanotubes [17], graphene [18], carbon nanocoils [19], carbon foam [20], and carbon fibers [21] have attracted considerable attention as promising MA materials due to their light weight, high conductivity, good resistance against corrosion, excellent mechanical properties and low filler loading rate in comparison with traditional metal based materials. However, the microwave absorption properties of carbon materials still need improvement, because their mechanism of microwave absorption is usually dependent on the dielectric loss as well as their poor impedance matching characteristics [22].

The microwave absorption properties of MA materials are dominated by their dielectric properties (the complex permittivity; $\varepsilon_r = \varepsilon' - j\varepsilon''$), magnetic properties (the complex permeability; $\mu_r = \mu' - j\mu''$), and the electromagnetic impedance matching [23]. Therefore,

coupling dielectric/magnetic nanostructures with carbon materials is considered to be an effective approach to enhance the MA properties of carbon materials. Till now, diverse dielectric/magnetic nanostructure/carbon materials with good MA properties, such as silver coated graphite [24], FeCo particle/graphite nanoflake composites [25], core-shell nanostructured C/ZnO nanoparticles [26], rGO/NiO composite [27], rGO/ α -Fe₂O₃ composite hydrogel [28], Fe nanoparticle/carbon fibers [21], 3D Fe₃O₄ nanocrystals decorated carbon nanotubes [29], Fe₃O₄-Fe/graphene [30], rGO- γ -Fe₂O₃ incorporated polyaniline composite [21], CoFe₂O₄/graphene oxide hybrids [31], have been fabricated. Among these nanostructures, iron oxide based carbon hybrids have grabbed wide great attention due to their good properties [32-35]. More important, coupling iron oxides with carbon materials has also been demonstrated to be an effective way to improve their MA properties, because iron oxides possess low cost, good antioxidant capability and strong absorption characteristics after the hybridization [21, 28, 36]. For example, Zhang *et al.* have synthesized rGO/ α -Fe₂O₃ composite hydrogel which exhibits excellent MA property [28]. This rGO/ α -Fe₂O₃ composite shows a maximum reflection loss (RL) of -33.5 dB at 7.12 GHz with the absorber thickness of 5.0 mm and a bandwidth of 6.4 GHz corresponding to RL less than -10 dB with the absorber thickness of 3.0 mm [28]. Singh *et al.* have fabricated γ -Fe₂O₃-rGO-polyaniline core-shell tubes which possess high shielding effectiveness of about -51 dB at a critical thickness of 2.5 mm [21]. Chen *et al.* have prepared graphene-Fe₃O₄ nanohybrids which have a minimum RL up to -40.36 dB with 20 wt. % loading rate in the matrix at a thickness of 5.0 mm [37]. However, there are still some great challenge remaining for designing and fabricating such iron oxide/carbon materials with strong absorption properties, such as the high loading amount in the matrix [28, 38, 39], low absorption capacity [28, 38, 40], high cost and complicate functionalization steps [21, 39].

Recently, Chen *et al.* fabricate a novel dielectric–magnetic nanostructure by hybridizing three-dimensional Fe_3O_4 nanocrystals and multi-walled carbon nanotubes (3D Fe_3O_4 -MWCNTs) through a simple co-precipitation route [29]. These novel 3D Fe_3O_4 -MWCNT composites exhibit enhanced microwave absorption with tunable strong-absorption wavebands in the frequency range of 2–18 GHz [29]. Moreover, double-band microwave absorption can be observed for these composites in the investigated frequency range and at various thicknesses [29]. And the minimum RL loss values at 20 wt% loading of -23.0 dB and -52.8 dB are obtained at 4.1 GHz and 12.8 GHz [29], respectively. These results are superior to those of pure MWCNTs as well as some other hybrids of Fe_3O_4 as mentioned above. Chen *et al.* attribute this enhanced absorption capacity to the synergy of dielectric loss, magnetic loss, and the enhancement of multiple reflection among 3D Fe_3O_4 nanocrystals [29]. This research provides a new strategy to tune the electromagnetic properties of CNTs by creating 3D Fe_3O_4 nanocrystals thus enhancing the MA performance of CNTs. Additionally, in previous research, Francois *et al.* have studied the sound absorption performance of $\text{B}(\text{OH})_3$ in seawater at low frequency [41]. They find that $\text{B}(\text{OH})_3$ plays an important role in the absorption of sound in seawater [41]. This finding suggests that $\text{B}(\text{OH})_3$ can also absorb some waves with certain frequency range.

Herein, in this report, we developed a facile approach for large-scale fabrication of novel 3D $\text{B}(\text{OH})_3$ and $\alpha\text{-Fe}_2\text{O}_3$ nanoparticle decorated carbon microspheres (3D $\text{B}(\text{OH})_3/\alpha\text{-Fe}_2\text{O}_3$ -CMSs) *via* a facile thermal treatment process. The structure and composition of the as-obtained products are characterized by X-ray diffraction (XRD), scanning electron microscope (SEM), transmission electron microscope (TEM), X-ray photoelectron spectroscopy (XPS). The MA properties of these novel 3D $\text{B}(\text{OH})_3/\alpha\text{-Fe}_2\text{O}_3$ -CMS based absorbers are systematically investigated with different absorber thickness in the frequency range of 2-18 GHz. Furthermore,

the absorption mechanism for the enhancement of MA performance of these novel 3D B(OH)₃/α-Fe₂O₃-CMSs were also discussed in detail in terms of the characterization on their structure, composition, and electromagnetic parameters

2. Materials and methods

2.1. Sample Preparation

In current work, 3D B(OH)₃/α-Fe₂O₃-CMSs were fabricated *via* a facile thermal treatment of ferrocene and ammonia borane mixture under argon atmosphere. The fabrication process was presented in Fig. 1. The precursor of ammonia borane was synthesized according to the method described in the literature [42]. Typically, the fabrication process for the 3D B(OH)₃/α-Fe₂O₃-CMSs was carried out as follows: Firstly, ammonium formate (94.5 g) and sodium borohydride (37.9 g) were slowly added into dioxane (1 L) at 40 °C following vigorously stirred for 12 h. The as-obtained mixture was filtered by vacuum pump to get colourless and transparent solution, and about 600 ml of ammonia borane/dioxane solution was obtained after filtration. Then, 12 g of ferrocene was dissolved into such as-obtained solution under stirring and clearly reddish brown solution was formed. Thirdly, the solution was stood for 24 h following dried at 80 °C in water bath. Finally, the collected powders were pressed into round shape blocks and transferred into a tube furnace for thermal treatment at 1200 °C with a heating rate of 5 °C min⁻¹ under argon atmosphere (100 ml/min). After 1 h of thermal treatment, the sample was cooled down to room temperature naturally. Then, the blocks were ground into powders for further characterization after the thermal treatment.

2.2. Sample Characterization

The crystalline nature of 3D B(OH)₃/α-Fe₂O₃-CMSs was confirmed by XRD studies carried out on MSAL-XD2 X-ray diffractometer with Cu K radiation ($\lambda=0.154178\text{nm}$) in the scattering range (2θ) of 10 - 80° with a scan rate of 0.02°/s. The morphology of 3D B(OH)₃/α-Fe₂O₃-CMSs were examined on TESCAN VEGA II SEM equipped with an energy dispersive X-ray spectroscopy (EDS). The SEM samples were prepared by dispersing the powder in ethanol using ultrasonication and placing small drops of the suspension on silicon wafers. The interior structure of 3D B(OH)₃/α-Fe₂O₃-CMSs was characterized by FEI Tecnai G2 F30 TEM. The TEM samples were prepared by dipping the holey-carbon coated copper grids into the above-mentioned dispersion followed by drying at room temperature. The composition and bonding information of 3D B(OH)₃/α-Fe₂O₃-CMSs were investigated by XPS performed on PHI 5700 spectrometer with Al K α excitation radiation (1486.6 eV) and the binding energy was referenced to C_{1s} at 284.6 eV.

2.3. Electromagnetic Parameter Measurements

The microwave absorption performance of 3D B(OH)₃/α-Fe₂O₃-CMSs was evaluated by a vector network analyzer (VNA; Agilent N5245A) in transmission-reflection mode in the frequency range of 2-18 GHz. The electromagnetic parameters of the 3D B(OH)₃/α-Fe₂O₃-CMSs were calculated *via* HP85071 software. The 3D B(OH)₃/α-Fe₂O₃-CMS/paraffin absorbers were prepared by uniformly mixing of 3D B(OH)₃/α-Fe₂O₃-CMSs and paraffin with a weight ratio of 1:4 (20 wt.%). Then, the mixtures were pressed into toroidal shaped samples with inner diameter of 3.0 mm, outer diameter of 7.0 mm, and thickness of 3.0 mm, respectively.

3 Results and Discussion

Fig. 2 shows the representative SEM images and corresponding EDS spectrum of the as-obtained products. As shown in the low magnification SEM image of Fig. 2a, a large amount of

microspheres are obtained. The diameter of the microspheres is in the range of 1 to 3 μm as observed in the high magnification SEM image (Fig. 2b). In addition, a large number of nanoparticles with diameter of tens of nanometers can also be clearly seen anchoring on the surfaces of the microspheres (Fig. 2b). Fig. 2c shows the EDS spectrum of the as-obtained products which is collected from the center part of Fig. 2b, revealing that the as-obtained products are composed of B, C, O, Si and Fe elements. Here, the peak of Si element is also observed that arises from the Si wafer used as substrate for SEM analysis.

Fig. 3 presents the XRD pattern of the as-obtained products. As shown in Fig. 3, the diffraction peaks centered at $2\theta = 26.7^\circ$ and 44.7° correspond to the (002) and (100) reflections of graphite (JCPDS card No.41-1487), respectively. The diffraction peaks centered at $2\theta = 14.7^\circ$, 22.9° , 28.1° , 30.3° , 31.7° and 40.4° agree well with the known values for B(OH)_3 and can be indexed to be the crystal planes of (010), (101), (002), (200), $(\bar{2}21)$ and $(\bar{1}31)$ reflections of B(OH)_3 (JCPDS Card No.73-2158), respectively. The other diffraction peaks at $2\theta = 24.3^\circ$, 32.9° , 35.8° , 43.5° , 56.7° , 58.2° and 62.9° can be respectively assigned to the reflections of (012), (104), (110), (202), (211), (018) and (214) crystal planes of $\alpha\text{-Fe}_2\text{O}_3$ (Rhomb-centered structure, space group $R\bar{3}c$, JCPDS card No. 84-0310). The XRD results indicate that the as-obtained products contain crystalline carbon, B(OH)_3 and $\alpha\text{-Fe}_2\text{O}_3$ phases.

The morphology and interior structure of the as-obtained products were further investigated by the TEM. As shown in Fig. 4a, numerous nanoparticles with diameter of tens of nanometers are randomly anchored on the surface of a microsphere with a diameter of about 1 μm . These attached nanoparticles tightly grow together to form a 3D hierarchical structure. This result matches well with that observed by the SEM. Fig. 4b presents the HRTEM image of the as-obtained products in which parallel lattice fringes with different d-spacing can be clearly

observed. This observation clearly indicates the high crystallinity of the anchored nanoparticles. The d-spacing values of about 0.48, 0.32, 0.37 and 0.27 nm can be calculated between the adjacent fringes, corresponding to the (0 $\bar{1}1$), (002) plane d-spacing of B(OH)₃ and (012), (104) plane d-spacing of α -Fe₂O₃, respectively.

In order to get more information about the composition and bonding state of the as-obtained products, XPS analysis was performed. The survey XPS spectrum indicates that the as-obtained products are composed of five elements of C, B, N, Fe and O as shown in Fig. 5a. Fig. 5b shows the fine spectrum of C_{1s} which is deconvolved into three peaks at 284.7, 285.8 and 288.9 eV, corresponding to C-C, C-N and C-R bonds [43, 44]. The fine spectrum of B_{1s} is shown in Fig. 5c, and only one peak at 193.1 can be deconvolved corresponding to the B-O bond [44]. The fine spectrum of Fe_{2p} is shown in Fig. 5d, and the core level binding energies at 711.4 and 724.8 eV can be assigned to the characteristic doublets of Fe 2p_{3/2} and 2p_{1/2} core-level spectra of Fe₂O₃ [28], respectively. The N_{1s} peak as shown in Fig. 5e can be deconvolved into two peaks centered at 399.4 and 401.2 eV, respectively, referring to C-N and graphite N/absorbed nitrogen [43, 45], respectively. The deconvolved O_{1s} spectrum with the peak positions at the binding energies of 530.3 eV and 532.4 eV are attributed to O-Fe bond of α -Fe₂O₃ and O-B bond [28, 45], respectively. Herein, combining all the results of XRD, EDS, SEM, TEM, HRTEM and XPS, it can be concluded that 3D B(OH)₃/ α -Fe₂O₃-CMSs are obtained.

Fig. 6 presents the MA properties of the as-obtained 3D B(OH)₃/ α -Fe₂O₃-CMSs based absorber with a weight percentage of 20 wt.% in the absorber in the frequency range of 2-18 GHz. Here, the MA properties of the sample are calculated according to the following equations [20]:

$$RL(dB) = 20 \log \left| \frac{Z_{in} - 1}{Z_{in} + 1} \right|$$

where the normalized input impedance (Z_{in}) can be calculated by the following equation:

$$Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh \left[j \left(\frac{2\pi f d}{c} \right) \sqrt{\mu_r \epsilon_r} \right]$$

where d , f , c , Z_0 and Z_{in} represent the thickness of the absorber, the frequency of the electromagnetic microwaves, the velocity of light, the impedance of free space and input impedance of the absorber [46], respectively. As shown in Fig. 6, the minimum RL values move to low frequency region with the increase of the absorber thickness from 2.0 mm to 5.0 mm. This results indicate that the MA frequency of the as-obtained 3D B(OH)₃/α-Fe₂O₃-CMS based absorbers can be conveniently tuned by adjusting the thickness of the absorbers. The minimum RL values of the as-obtained 3D B(OH)₃/α-Fe₂O₃-CMS based absorbers with different thickness are outlined in Table 1. Obviously, when the absorber thickness is in the range of 2.0 to 5.0 mm, the RL values below -10 dB (90% microwave absorption) can be obtained in the frequency range of 4.68 -18.0 GHz, revealing that the as-obtained 3D B(OH)₃/α-Fe₂O₃-CMS based absorbers have superior electromagnetic MA performance [47]. The minimum RL value can reach -52.69 dB at 11.12 GHz with absorber thickness of 3.0 mm, and the RL value below -10 dB is in the frequency range of 8.44-13.80 GHz. In addition, the widest frequency range where RL is lower than -10 dB is between 11.32 and 16.96 GHz when the thickness of the absorber decreases to 2.5 mm. Compared to the representative iron oxide/carbon based nanocomposites published recently as summarized in Table 2, the as-obtained 3D B(OH)₃/α-Fe₂O₃-CMSs exhibit an excellent ability to absorb electromagnetic microwave. Generally, a MA material with a RL value less than -30 dB is regarded as an excellent absorber, because this value corresponds to 99.999% RL or

absorption of microwaves [48]. Therefore, these novel 3D B(OH)₃/α-Fe₂O₃-CMSs are promising candidate for practical applications.

The electromagnetic parameters, including relative complex permittivity and relatively complex permeability were measured at room temperature for the study of MA properties of 3D B(OH)₃/α-Fe₂O₃-CMS based absorbers which are shown in Fig. 7. The estimated real permittivity (ϵ') and real permeability (μ') are directly associated with the amount of polarization occurring in the material which symbolizes the storage ability of the electric and magnetic energy [21]. The imaginary permittivity (ϵ'') and imaginary permeability (μ'') are in regards to the dissipation of electric and magnetic energy [21]. As shown in Fig. 7a, the initial value for ϵ' is 9.73 at 2 GHz, and slowly decreases to 4.60 from 2 to 18 GHz, while the initial value for ϵ'' is 3.93 at 2 GHz, and slightly decreases to 2.61 from 2 to 14.56 GHz, then slightly increases to 3.14 from 14.56 to 18 GHz. As well known that, the ϵ' is probably associated with the polarizability of a material, which consists of electric polarization and dipolar polarization at microwave frequencies [58]. A large amount of interfaces are formed among the 3D B(OH)₃ and α-Fe₂O₃ nanoparticles and carbon microspheres (Fig. 8b), and different crystal planes along the interface are normally different which will cause many defects and dangling bonds for charge accumulation [59, 60], thus enhancing the polarization among the interfaces. Another factor of ϵ'' is associated with the conductivity of a material. The excellent conductivity of carbon microspheres (Fig. 8b) in 3D B(OH)₃/α-Fe₂O₃-CMSs will also benefit the attenuation of the microwave. Fig. 7b shows the frequency dependence of the real part (μ') and imaginary part (μ'') of the complex permeability of 3D B(OH)₃/α-Fe₂O₃-CMSs. The values of μ' (~ 1.1) and μ'' (~ 0) almost have no change in the frequency range of 2-18 GHz, except the slight resonance between 8.80 and 10.0 GHz. The values of magnetic loss ($\tan\delta\mu = \mu''/\mu'$) are almost all near or less than

zero, meaning that there is no significant magnetic loss for the as-obtained 3D B(OH)₃/α-Fe₂O₃-CMSs. Therefore, compared to the values of complex permittivity, the complex permeability values of both μ' and μ'' are relatively low, indicating that the main contribution to RL is complex permittivity.

In addition, there are another two factors except dielectric loss and magnetic loss contributing to the enhancement of the MA performance of 3D B(OH)₃/α-Fe₂O₃-CMSs. One is the multiple reflection of the microwave as illustrated in Fig. 8. The microwave propagation path in the 3D B(OH)₃/α-Fe₂O₃-CMSs based absorbers extends and the microwave reflects many times in the absorbers (Fig. 8a). The TEM images show that 3D B(OH)₃ and α-Fe₂O₃ nanoparticles randomly distributing on the surfaces of carbon microspheres. This structure can be simplified as the islanding model where every 3D B(OH)₃ and α-Fe₂O₃ nanoparticle can be regarded as an isolated island [48]. As a result, the RL of microwave occurs not only among plenty of carbon microspheres in the absorber but also within these 3D B(OH)₃ and α-Fe₂O₃ islands (Fig. 8c). These 3D B(OH)₃ and α-Fe₂O₃ islands can effectively increase the frequency of multiple reflections, which corresponds to the extension of microwave propagation path, thus enhancing the scattering loss of microwaves. The other factor is the proper electromagnetic impedance matching caused by the 3D B(OH)₃ nanoparticles. The existence of 3D B(OH)₃ nanoparticles can guide more electromagnetic waves into the interior of carbon microspheres leading to more conductivity loss (Fig. 8b). Therefore, the enhanced MA capacity with tunable strong-absorption wavebands of 3D B(OH)₃/α-Fe₂O₃-CMSs can be attributed to the synergy of dielectric loss, multiple reflection, as well as proper electromagnetic impedance matching.

4. Summary

In summary, novel 3D B(OH)₃/α-Fe₂O₃-CMSs with carbon spheres in diameter of 1 to 3 μm and decorated B(OH)₃ and α-Fe₂O₃ nanoparticles in diameter of several to tens of nanometers can be massively fabricated through a facile thermal treatment process. The MA investigation in the frequency range of 2–18 GHz demonstrates that 3D B(OH)₃/α-Fe₂O₃-CMSs exhibit tunable strong MA performance. The minimum RL value of 3D B(OH)₃/α-Fe₂O₃-CMS based absorber can reach -52.69 dB with absorber thickness of 3.0 mm at 11.12 GHz. Additionally, it is convenient to tune the MA properties of the 3D B(OH)₃/α-Fe₂O₃-CMS based absorbers by varying the absorber thickness. These excellent MA properties of 3D B(OH)₃/α-Fe₂O₃-CMSs are superior to those of pure micro/nano carbon nanoparticles/nanotubes as well as other hybrids of iron oxides. The enhanced MA performance can be attributed to the synergy of dielectric loss, multiple reflection among 3D α-Fe₂O₃ nanocrystals, as well as proper electromagnetic impedance matching caused by the 3D B(OH)₃ nanoparticles. These findings open up a new strategy to develop outstanding microwave absorbers.

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Table 1 The MA properties of 3D B(OH)₃/α-Fe₂O₃-CMSs based absorbers with different thickness.

Thickness (mm)	Absorption peak (GHz)	RL (dB)	Absorption bandwidth (GHz, <-10dB)
1.0	18	-1.72	-
1.5	18	-7.95	-
2.0	17.04	-41.83	3.49 (14.51-18)
2.5	13.60	-30.95	5.64 (11.32-16.96)
3.0	11.12	-52.69	5.36 (8.44-13.80)
3.5	8.64	-25.96	4.60 (7.08-11.68)
4.0	7.36	-30.60	3.98 (6.14-10.12)
4.5	6.40	-30.41	2.93 (5.29-8.22)
5.0	5.60	-35.58	2.48 (4.70-7.18)

Table 2. Microwave absorption comparison of representative work.

Materials	wt. %	Minimum RL (dB)	Bandwidth (GHz < -10 dB)	Thickness (mm)	References
Fe ₃ O ₄ /Fe/rGO	50	-23.09	3.9	4.0	[14]
Aligned CNTs films	100	-47.66	4.4	2.0	[17]
Fe ₃ O ₄ /Al ₂ O ₃ /carbon nanocoils	25	-40.3	~2.0	3.1	[19]
rGO/a-Fe ₂ O ₃ composite hydrogel	82	-33.5	6.4	5.0	[28]
3D Fe ₃ O ₄ -MWCNTs.	20	-52.8	~2.0	6.8	[29]
Fe ₃ O ₄ -Fe nanoparticles/graphene	18	-58.0	1.6	4.6	[30]
Fe ₃ O ₄ -coated hollow glass spheres	50	-15.8	3.6	2.5	[40]
SWCNTs in SCPU	5	-21.9	2.6	2.0	[49]
γ -Fe ₂ O ₃ /MWNTs/PBO	12	-32.7	2.5	2.7	[50]
GO/CNT-Fe ₃ O ₄ nanohybrid	30	-37.2	1.0	5.0	[51]
MWCNT/Fe ₃ O ₄ @ZnO heterotrimers	50	-40.9	4.0	2.0-3.0	[52]
Fe ₃ O ₄ @C	66.7	-22.5	6.0	1.7	[53]
Graphene/Fe ₃ O ₄ @Fe/ZnO	20	-38.4	~3.0	5.0	[54]
SiO ₂ @Fe ₃ O ₄ core/shell nanorod	20	-31.9	~3.0	2.5	[55]
Fe ₃ O ₄ @TiO ₂ microspheres	20	-23.3	5.5	2.0	[56]
Fe ₃ O ₄ @ZnO nanohybrid	50	-22.7	5.8	3.5	[57]
3D B(OH) ₃ /α-Fe ₂ O ₃ -CMSs	20	-52.69	5.36	3.0	This work
3D B(OH) ₃ /α-Fe ₂ O ₃ -CMSs	20	-30.95	5.64	2.0	This work

Fig. 1 Procedure for the preparation of the 3D B(OH)₃/α-Fe₂O₃-CMSs.

Fig. 2 (a) low-magnification and (b) high-magnification SEM images, and (c) corresponding EDS spectrum of the as-obtained products.

Fig. 3 The XRD pattern of the as-obtained products.

Fig. 4 (a) TEM and (b) HRTEM images of the as-obtained products.

Fig. 5 The XPS spectra of the as-obtained products: (a) survey, (b) fitted C_{1s}, (c) fitted B_{1s}, (d) fitted Fe_{2p}, (e) fitted N_{1s}, and (f) fitted O_{1s} spectra, respectively.

Fig. 6 Frequency dependence of RL for the 3D B(OH)₃/α-Fe₂O₃-CMS based absorbers with different thickness in the frequency range of 2–18 GHz.

Fig. 7 Complex permittivity (a) and complex permeability (b) of 3D B(OH)₃/α-Fe₂O₃-CMS based absorbers in the frequency range of 2–18 GHz.

Fig. 8 Schematic description of possible microwave absorption mechanisms of the 3D B(OH)₃/α-Fe₂O₃-CMSs in a paraffin matrix.