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FuYao Chen

17855371372@163.com

Zhejiang Sci-Tech University

Zhehong Lu

Henan University

Han Chen

Zhejiang Sci-Tech University

Dongqi Liu

Xi'an Modern Chemistry Research Institute

Jingshuai Liu

Zhejiang Sci-Tech University

Xuzhen Zhang

Zhejiang Sci-Tech University

Junmin Wan

Zhejiang Sci-Tech University

Yi Huang

Zhejiang Sci-Tech University

Juming Yao

Zhejiang Sci-Tech University

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Facile fabrication of ternary MoO₂/Fe₂O₃/CNFs nanofibers with efficient electromagnetic wave absorption performance

Fuyao Chen^{1,2*}, Zhehong Lu³, Han Chen¹, Dongqi Liu^{4*}, Jingshuai Liu¹, Xuzhen

Zhang¹, Junmin Wan¹, Yi Huang¹, Juming Yao^{1*}

1: School of Materials Science and Engineering, Zhejiang Sci-Tech University, Hangzhou, 311200, China.

2: Tongxiang Research Institute of Zhejiang Sci-Tech University, JiaXing, 314000, China.

3: College of Chemistry and Molecular Sciences, Henan University, Kaifeng, 475004, China.

4: Xi'an Modern Chemistry Research Institute, Xi'an 710065, China.

Corresponding author: Dr Fuyao Chen, Dr Dongqi Liu, Prof. Juming Yao.

Address: School of Materials Science and Engineering, Zhejiang Sci-Tech University, Hangzhou, 311200, China.

Xi'an Modern Chemistry Research Institute, Xi'an, 710000, China.

Tel./fax: Fuyao Chen, +86 17855371372; Dongqi Liu, +86 18691850785; Juming Yao, +86 13967196026

Email: 17855371372@163.com, ldqnjust1997@163.com, yaoj@zstu.edu.cn

Abstract

Carbon nanofibers (CNFs) integrated with multicomponent nanoparticles have shown great promise for enhancing electromagnetic wave (EMW) absorption performance. In this work, ternary MoO₂/Fe₂O₃/CNF composites were successfully fabricated through a facile electrospinning approach followed by high-temperature carbonization. Among the prepared samples, MoO₂/Fe₂O₃/CNF-2 sample achieved a minimum reflection loss (RL_{min}) value of -40.90 dB at 10.56 GHz with a thickness of 2.00 mm. Notably, when the absorber thickness was reduced to 1.50 mm, the effective absorption bandwidth (EAB) reached 5.76 GHz, covering nearly 96% of the Ku band.

In addition, CST simulations further demonstrated the promising practical applicability of the MoO₂/Fe₂O₃/CNF-2 sample in prospective high-frequency scenarios. The favorable EMW attenuation performance derived from the synergistic effect of prominent conductive loss, appropriate magnetic loss, abundant multipolar polarization, as well as optimal impedance matching, all arising from the unique composite architecture consisting composed nano MoO₂, nano Fe₂O₃ and CNFs.

Keywords: γ -Fe₂O₃; Carbon nanofibers; MoO₂; Wave-absorbing performance.

1. Introduction

With the rapid development of wireless communications technologies, electromagnetic waves (EMW) have become deeply integrated into modern daily life. However, undesirable electromagnetic waves not only interfere with the normal operation of electronic devices and precision instruments, but also poses potential risks to human health and the surrounding environment^[1-3]. Consequently, the development of high-performance electromagnetic absorption materials (EAMs) has become increasingly urgent for suppressing and attenuating excessive electromagnetic radiation. Ideally, advanced EAMs should simultaneously exhibit strong absorption capability, broad effective absorption bandwidth (EAB), lightweight characteristics, thin thickness, and environmental-friendliness^[4]. Nevertheless, conventional EAMs typically represented by ceramics and semiconductors, despite their effective EMW attenuation capability, suffer from inherent drawbacks such as high density, poor corrosion resistance, and inferior machinability, which severely limit their applications^[5]. Therefore, the development of novel EAMs featuring thinness, flexibility, easy processability, and excellent performance is of great practical significance.

Rational design of composition and structure is an effective strategy to enhance EMW absorption performance. Extensive studies have been devoted to dielectric loss materials (e.g., graphite^[6, 7], carbon nanotubes (CNTs)^[8], MXene^[9, 10]) and magnetic loss materials (e.g., Fe₃O₄^[11] and NiO^[12, 13]). It has well established that the hybridization of conductive and magnetic components can effectively construct high-performance absorbers, benefiting from the synergistic multiple electromagnetic loss mechanisms of the two components. In recent years, magnetic carbon nanofiber composites, such as Fe₃O₄/CNF^[14] and Fe₃C/CNF^[15], have been extensively investigated for EMW absorption. In contrast, Fe₂O₃/CNF composites have rarely been explored for EMW absorption to the best of our knowledge, this may due to the conventional recognition of Fe₂O₃ as a non-magnetic oxide. Nevertheless, the magnetic

properties of Fe_2O_3 among all its polymorphs, only the $\gamma\text{-Fe}_2\text{O}_3$ crystal phase exhibits intrinsic magnetic characteristics. Accordingly, this study is primarily focused on the fabrication and EMW absorbing properties of $\gamma\text{-Fe}_2\text{O}_3/\text{CNF}$.

MoO_2 has gained considerable attention in the field of catalysis^[16, 17], energy storage^[18] and batteries^[19] owing to its low cost, superior electrical conductivity, and excellent structural stability. Moreover, MoO_2 shows tremendous application prospects in the field of EMW absorption^[20, 21]. For example, Cheng et al.^[22] fabricated one-dimensional mesoporous MoO_2/C hetero-nanowires via a facile in situ synthesis process, achieving a minimum reflection loss ($\text{RL}_{\min}$) of -47.6 dB and an EAB of 3.8 GHz at a thickness of only 2 mm. In 2020, Xing et al.^[23] reported the synthesis of $\text{C}/\text{MoO}_2/\text{Co}_3\text{O}_4$ micro-flowers through a facile coordination-polymerization method, realizing an $\text{RL}_{\min}$ of -59.7 dB with a thickness of 2 mm. In 2025, Mo et al.^[24] constructed MoO_2/S -doped carbon nanofibers by integrating electrospinning, oxidative stabilization, and high-temperature carbonization. The optimized composite delivered an RL of -61.26 dB at 9.43 GHz and a broad EAB of 4.81 GHz at a matching thickness of 3.65 mm. The superior EMW absorption performance of MoO_2 -based composites originates from the enhanced conduction loss, multiple polarization effect and well-matched impedance. These representative studies demonstrate that MoO_2 , as a typical high-dielectric oxide, holds great potential for the fabrication of high-efficiency EMW absorbers.

Inspired by the above considerations, we rationally integrated Fe_2O_3 and MoO_2 nanoparticles in a polyvinyl alcohol (PVA) solution, and successfully fabricated $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ composites via electrospinning followed by subsequent high-temperature calcination. Among the as-prepared composites, the $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ -2 samples exhibited the most favorable EMW absorption performance, which is attributed to the synergistic coupling of prominent conductive loss, favorable magnetic loss, abundant multipolar polarization and optimal impedance matching. This work may provide a novel insight into the fabrication of EMW absorbers featuring ultra-thin and strong absorption characteristics.

2. Materials and Experimental method

2.1 Materials

Polyvinyl alcohol solution (>95 mass%) was obtained from Guangzhou Als New Material Co., Ltd. Nano MoO_2 (>98 mass%) was purchased from Ningbo Zhengzhuang

new material co., ltd. Nano γ -Fe₂O₃ (analytical grade) was purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. All reagents were used without further purification.

2.2 Preparation of MoO₂/Fe₂O₃/PVA

Typically, 0.03g MoO₂, 0.3g γ -Fe₂O₃, and 2.67g of PVA aqueous solution were added in a 30 mL bottle. The mixture was continuously stirred magnetically at 500 rpm for 8 h at room temperature to obtain a homogeneous suspension, which was then subjected to ultrasonication for 5 min to yield a uniform reddish spinning solution. Subsequently, the as-prepared solution was loaded into a 10 mL syringe equipped fitted with a 14G stainless steel needle and mounted on the electrospinning apparatus. Electrospinning was conducted under the following conditions: an applied voltage of 15 kV, a feeding rate of 0.3 mL/h, a roller rotation speed of 500 rpm, and a working distance of 15 cm between the needle tip and collector. After the electrospinning process, the as-spun MoO₂/Fe₂O₃/PVA composite was collected on silicone oil paper and designated as MoO₂/Fe₂O₃/PVA-1. To investigate the effect of MoO₂ content, two additional samples, denoted as MoO₂/Fe₂O₃/PVA -2 and MoO₂/Fe₂O₃/PVA -3, were prepared by varying the amount of MoO₂ while keeping other parameters constant. For comparison, Fe₂O₃/PVA and MoO₂/PVA fiber were prepared by the same process for comparison. The detailed compositions of all five samples are summarized in Table 1.

Table 1. Compositions of each component in different samples (g)

Samples	MoO ₂	Fe ₂ O ₃	PVA
MoO ₂ /CNF	0.30	-	2.70
Fe ₂ O ₃ /CNF	-	0.30	2.70
MoO ₂ /Fe ₂ O ₃ /PVA-1	0.03	0.30	2.67
MoO ₂ /Fe ₂ O ₃ /PVA-2	0.06	0.30	2.64
MoO ₂ /Fe ₂ O ₃ /PVA-3	0.12	0.30	2.58

2.3 Preparation of MoO₂/Fe₂O₃/CNF

Initially, the MoO₂/Fe₂O₃/PVA-1 precursor was dried at 70 °C for 6h in an oven to completely remove the residual surface moisture. Subsequently, the dried precursor was then transferred to a tube furnace and subjected to high-temperature carbonization under a pure N₂ atmosphere. The furnace was heated to 750 °C at a heating rate of 10°C/min and maintained held at this temperature for 2 h. The resulting carbonized product was denoted as MoO₂/Fe₂O₃/CNF-1. The remaining series samples were

fabricated via same drying and carbonization procedures. A schematic illustration of the overall experimental process was shown in Fig 1a.

2.4 Measurement and Characterizations

Powder X-ray diffraction (XRD) patterns were performed with a Bruker D8-Advanced diffractometer equipment with Cu K α radiation, and the sample was scanned in the 2θ range from 15° to 80° ($\lambda = 0.15406$ nm) operated at 40 kV and 40 mA. The morphology and microstructure of the samples were examined using Zeiss Gemini Sigma 300 VP field scanning electron microscopy (FE-SEM) and Thermo Fisher Scientific Talos F200s field emission transmission electron microscope (FE-TEM). N₂ adsorption/desorption isotherms were performed at -196°C using a Micromeritics ASAP 2460 adsorption instrument. The composition and graphitization degree of the samples were analyzed with an excitation wavelength of 532 nm by HORIBA HR Evolution Raman spectrometer. X-ray photoelectron spectroscopy (XPS, Thermo Scientific K-Alpha) was utilized to characterize surface chemical compositions and elemental valence states of the samples. The XPS were performed with monochromatic alkaliphile rays ($h\nu=1486.6$ eV) and the binding energy of C1s=284.8 eV as the energy standard for calibration. The magnetic hysteresis loop of the samples was determined by vibrating sample magnetometer Lakeshore-7404 equipment. The electromagnetic parameters of the samples were measured using a vector network analyzer (Agilent N5222A) in the frequency range of 2-18 GHz. The samples were mixed with paraffin at a weight ratio p of 6:4 and pressed into the coaxial ring-shaped composite specimens (U_{out}: 7.00 mm, U_{in}: 3.04 mm).

2.4 RCS simulation

Radar cross section (RCS) simulations were performed by using CST Studio Suite 2024. The simulation model consisted of an absorption layer and a perfect electric conductor (PEC) substrate. Both the absorber and the substrate were configured with in-plane dimensions of 200 mm $\times$ 200 mm, and thicknesses of 3.65 mm and 2 mm, respectively. The electric field was monitored at a fixed frequency of 10.26 GHz. The model was positioned on the XOY plane, with the incident electromagnetic wave propagating vertically along the Z-axis (i.e., normal incidence) Theta and Phi angle determined the direction of scattered EMW in the sphere coordinate.

3. Results and discussion

3.1 Structure and composition

The XRD patterns were employed to determine the physical phase composition of the different samples (Fig 1b). For the MoO_2/CNF composite, the diffraction peaks located at 26.03° , 37.03° , 41.35° , 53.57° , 60.20° and 66.36° belonged to the (-111) , (-211) , (210) , (-222) , (031) and (202) crystal planes of MoO_2 (PDF card 32-0671). The diffraction peaks of $\text{Fe}_2\text{O}_3/\text{CNF}$ at 26.15° , 30.24° , 35.63° , 43.27° , 53.73° , 57.11° and 62.76° corresponded to the $\gamma\text{-Fe}_2\text{O}_3$ standard card (211) , (220) , (311) , (400) , (-213) , (511) and (440) crystal planes (PDF card 39-1346), respectively. In the XRD patterns of three $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ samples, the characteristic peaks at around 35.50° corresponds to the (311) crystal face of Fe_2O_3 , while the peaks at around 26.02° and 36.9° attributed to (-111) and (-211) planes of MoO_2 , collectively confirming the successful preparation of the ternary $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ composites.

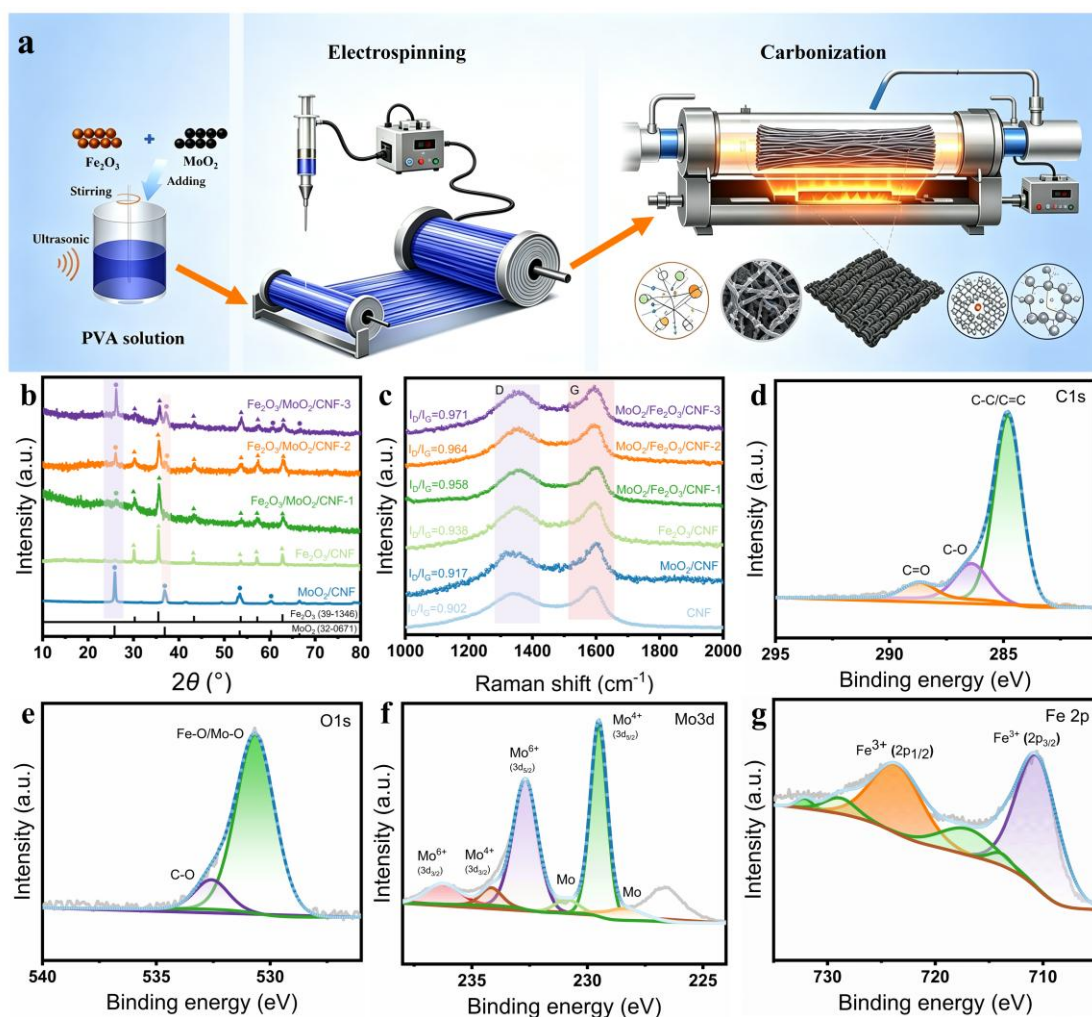


Fig 1. Preparation process of $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ composites (a), XRD (b) and Raman spectrum (c)

of different kinds of carbon fibers, the high resolution XPS spectra of C 1s (d), O 1s (e), Mo 3d (f), and Fe 2p (g) for the MoO₂/Fe₂O₃/CNF-2 composite.

Raman spectroscopy was further carried out for analyzing carbon structural features of the nanocarbon fiber samples (Fig 1c). The intensity ratio of the D band to the G band (I_D/I_G) serves as a critical indicator of lattice defects and the degree of graphitization degree of in carbon materials. Specifically, the D band correspond to disordered or amorphous carbon structures, while the G band originates from the in-plane vibration mode of sp²- hybridized of carbon atoms. A higher I_D/I_G ratio reflects a higher density of structure defects within carbon matrix. The I_D/I_G ratios for CNF, MoO₂/CNF, Fe₂O₃/CNF and MoO₂/Fe₂O₃/CNF-1, MoO₂/Fe₂O₃/CNF-2, MoO₂/Fe₂O₃/CNF-3 were 0.902, 0.917, and 0.936, 0.958, and 0.971, respectively. This progressive increase can be attributed to the partial carbothermal reduction of the embedded MoO₂ and Fe₂O₃ nanoparticles on the CNF surface during high-temperature carbonization, which leads to a decreased graphitization degree and an increased density of lattice defects. These defects can serve as polarization centers, thereby enhancing polarization loss and contributing to the overall EMW absorption performance.

XPS analysis was performed to unravel the surface chemical composition and the valence states of MoO₂/Fe₂O₃/CNF-2 composite. The survey spectrum (Fig. S1) confirmed the presence of C, O, Fe, and Mo, with characteristic photoelectron peaks at 284.8 eV (C 1s), 531.0 eV (O 1s), 712.08 eV and 726.08 eV (Fe 2p_{1/2} and Fe 2p_{3/2}) and 232.08 eV (Mo 3d). The high-resolution spectrum of C 1s (Fig. 1d) was deconvoluted into three distinct subpeaks: C-C/C=C at 284.8 eV, C-O at 286.4 eV, and C=O at 288.7 eV. The presence of the C=O peak is attributed to the decomposition of PVA during high-temperature carbonization^[25]. O 1s high-resolution spectrum (Fig. 1e) displayed two peaks at 532.58 and 530.65 eV, corresponding to Fe-O/Mo-O and C-O, respectively^[26]. The high-resolution spectrum of Mo 3d (Fig. 1f) matched well with multiple valence states, including Mo (228.39 eV and 230.97 eV), Mo⁴⁺ (229.51 eV and 234.35 eV) and Mo⁶⁺ (232.67 eV and 236.21 eV). The Mo⁴⁺ species originate from MoO₂, while the metallic Mo is generated via carbothermal reduction of MoO₂ by the carbon nanofiber matrix during high-temperature treatment, which strengthens the electronic coupling at the MoO₂/carbon interface. The present of Mo⁶⁺ on the surface of carbon fiber is ascribed to superficial oxidation of MoO₂ upon exposure to air^[24]. The Fe 2p high-resolution spectrum (Fig 1g) exhibited two primary peaks at 710.55 eV (Fe³⁺ 2p_{3/2})

and 723.58 eV ($\text{Fe}^{3+} 2p_{1/2}$), which are the typical characteristic peaks of Fe_2O_3 . the additional minor signals at 717.02 eV, 732.14 eV, 713.92 eV, 728.79 eV were attributed satellite peaks of Fe^{3+} and Fe^{2+} (produced by Fe^{3+} and carbon matrix)^[26].

3.2 Surface Morphology

The morphology and microstructure of the diverse CNF-based composites and their corresponding precursors were observed by SEM. Compared with the precursors (Fig 2a-b, Fig S2 and Fig S3), all CNF-based composites exhibited noticeable shrinkage and curling, which can be attributed to pyrolysis and chain fracture of PVA molecular chains during high-temperature thermal treatment. The MoO_2/CNF and its precursor (Fig S4a and Fig S5a) displayed a highly uniform fiber diameter distribution, with average diameters of 352.46 nm and 402.23 nm, respectively. In contrast, $\text{Fe}_2\text{O}_3/\text{CNF}$ and its precursor showed finer and more irregular fiber diameters, with average values of 132.00 nm and 177.59 nm (Fig S4b and Fig S5b). This pronounced disparity is presumably attributed to the inferior electrical conductivity of the $\text{Fe}_2\text{O}_3/\text{PVA}$ spinning solution. Correspondingly, the fiber diameters of the $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNFs}$ composites and their precursor gradually increased with rising MoO_2 content (Fig S4 c-e and Fig S5 c-e). Furthermore, the EDS elemental mapping distribution diagram (Fig 2c-f) confirmed the presence and homogeneous distribution of C, O, Fe, and Mo elements across the surface of $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ -3 sample.

The morphology and the structure of $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ -2 composites were further characterized by HRTEM technology with morphological details at varied magnifications. As shown in Fig 2g, the decoration of nano Fe_2O_3 and nano MoO_2 particles imparted a rough surface texture to the CNF matrix, decorated with abundant granular nanoparticles. High-resolution TEM images (Fig. 2h and 2k) revealed distinct lattice fringes with interplanar spacings of 1.70 nm and 3.41 nm, which were indexed to the (-222) crystal plane of MoO_2 and the (211) plane of Fe_2O_3 . Collectively, the SEM and HRTEM results confirmed the successful fabrication of the $\text{MoO}_2/\text{Fe}_2\text{O}_3/\text{CNF}$ composites. Notably, the homogeneous distribution of Fe_2O_3 and MoO_2 nanoparticles within carbon fiber matrix is expected to endow the composites with superior EMW attenuation capability.

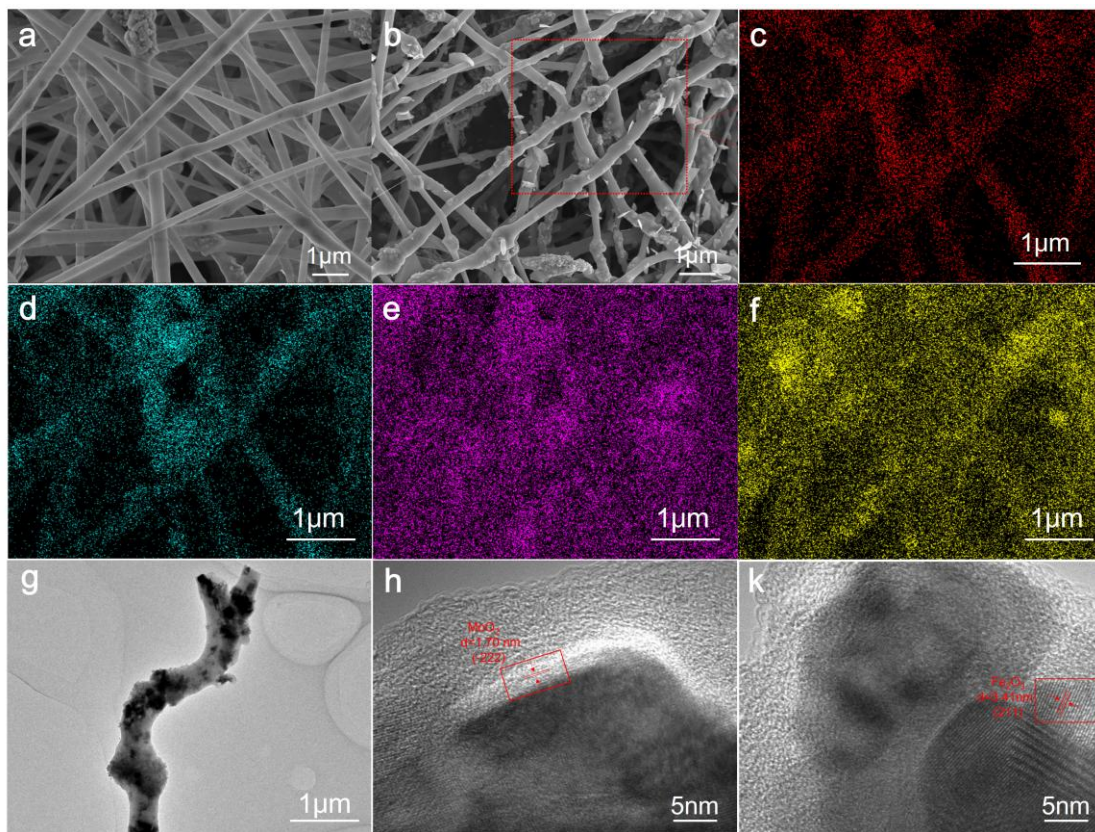


Fig 2. SEM images of MoO₂/Fe₂O₃/PVA-2 (a), MoO₂/Fe₂O₃/CNF-2 (b), along with the corresponding EDS mapping spectrum of C, O, Fe and Mo (c-f), and its HRTEM images at different magnification (g-k).

N₂ adsorption/desorption testing were performed to characterize the specific surface area and pore size of different CNF-based fibers. The specific surface areas of MoO₂/CNF, Fe₂O₃/CNF, MoO₂/Fe₂O₃/CNF-1, MoO₂/Fe₂O₃/CNF-2, and MoO₂/Fe₂O₃/CNF-3 were determined to be 44.51 m²/g, 101.14 m²/g, 91.66 m²/g, 75.06 m²/g, 44.40 m²/g, respectively (Fig 3a). The specific surface area of Fe₂O₃/CNF was higher than that of MoO₂/CNF, which is primarily attributed to the fewer inter-fiber gaps in both quantity and dimension in the MoO₂/CNF sample (Fig S3a and Fig S3b). Furthermore, the specific surface area of MoO₂/Fe₂O₃/CNF composites gradually declined with increasing MoO₂ loading content. Notably, the MoO₂/CNF sample exhibited an incompletely closed N₂ adsorption-desorption isotherm (Fig 3b). This unique behavior may arise from the collapse or contraction of the material microstructure during the desorption process as the pressure decreases, which restricts the release of internally absorbed nitrogen. In contrast, the Fe₂O₃/CNF sample displayed a distinct hysteresis loop with a relatively wide opening, indicating the presence of macropores within the material (Fig 3c). The three MoO₂/Fe₂O₃/CNF

samples displayed well-defined Type IV isotherms with characteristic curves (Fig 3d and Fig S6), suggesting a mesoporous structure resulting from the synergistic effect of MoO₂ and Fe₂O₃ incorporation.

The magnetisms of the different CNF-based composite fibers were determined by VSM, and the results are presented in Fig 3e and 3f. At the room temperature, the magnetic hysteresis loops of the samples containing Fe₂O₃ component exhibited S-type curve, unambiguously verifying their intrinsic ferromagnetic characteristics. Given that MoO₂ is an intrinsically nonmagnetic and displays a prominent diamagnetic response, the saturation magnetization (M_s) of MoO₂/CNF reached only 0.176 emu/g, which is drastically lower than of Fe₂O₃/CNF (42.524 emu/g). Consistent with this trend, the ternary composites delivered sequentially reduced M_s values: 37.956 emu/g for MoO₂/Fe₂O₃/CNF-1, 31.520 emu/g for MoO₂/Fe₂O₃/CNF-2 and 25.549 emu/g for MoO₂/Fe₂O₃/CNF-3. This monotonic decline in M_s value can be rationally ascribed to the increased loading of nonmagnetic MoO₂, which inevitably suppresses the overall magnetization of the Fe₂O₃/CNF composites.

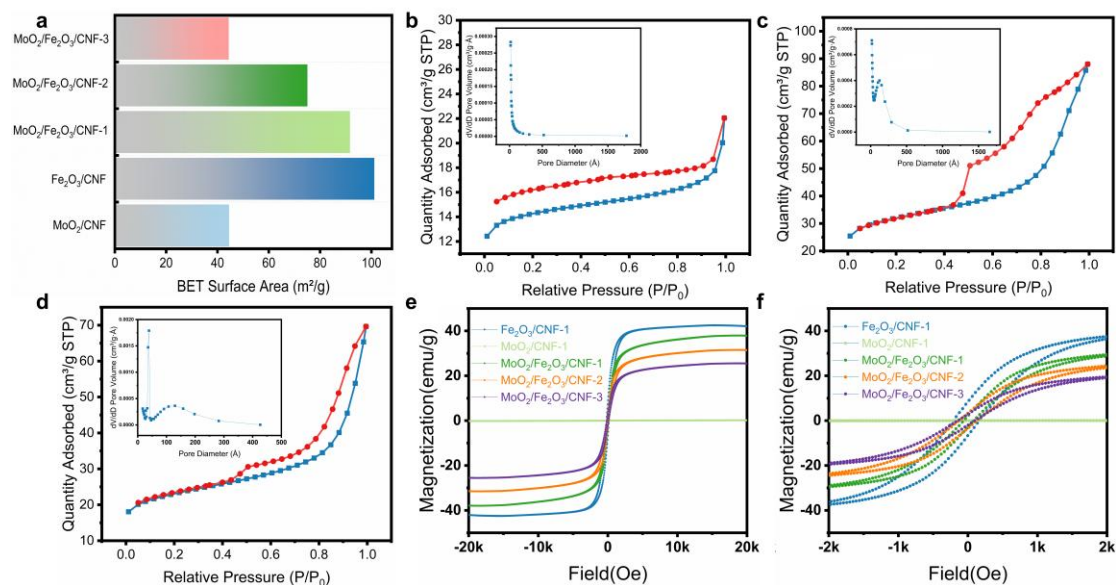


Fig 3. Specific surface area of different CNF fibers (a) and N₂ adsorption-desorption isotherms and pore size distribution curves of MoO₂/CNF (b), Fe₂O₃/CNF (c), MoO₂/Fe₂O₃/CNF-2 (d), and the magnetic hysteresis loops of different CNF fibers (e-f).

3.3 Electromagnetic and Microwave Absorption Performances

Electromagnetic parameters serve as the fundamental basis for investigating the EMW absorption characteristics of materials^[27]. Among them, the relative complex permittivity (ϵ_r) and relative complex permeability (μ_r) are the most critical parameters

determining the absorption performance, and are calculated using Equation (1) and (2).

$$\varepsilon_r = \varepsilon' - j\varepsilon'' \quad (1)$$

$$\mu_r = \mu' - j\mu'' \quad (2)$$

Where ε' and ε'' are the real and imaginary part of the dielectric constant, representing the storage capacity and loss capacity of electric energy, respectively; μ' and μ'' are the real and imaginary part of the permeability constant, representing the storage capacity and loss capacity of magnetic energy, respectively. The EMW parameter curves of MoO₂/CNF, Fe₂O₃/CNF and several MoO₂/Fe₂O₃/CNF composites are shown in Fig 4. With increasing EMW frequency from 2 to 18 GHz, the ε' and ε'' curves of all CNF-based composites all exhibited fluctuations in the middle and high frequency bands, evidencing the presence of polarization process (Fig 4a-b). Among all the fabricated samples, MoO₂/Fe₂O₃/CNF-2 achieved the highest ε' and ε'' values, manifesting its exceptional electromagnetic energy storage and dissipation capacity as a result of its prominent dielectric response. Furthermore, the dielectric loss tangent ($\tan\delta_\varepsilon$) (Fig 4c) of all the samples followed a trend analogous to that of ε'' , confirming the coexistence of diverse polarization relaxation processes within these materials. As illustrated in Fig 4d-e, owing to its inherent nonmagnetic feature, the μ' and μ'' values of MoO₂/CNF composite fluctuated near 1 and 0, respectively. In sharp contrast, the μ' and μ'' value of the Fe₂O₃/CNF constants were approximately 1.43 and 0.37, respectively, owing to its intrinsic magnetic properties, both of which declined monotonically with ascending frequency. For the three MoO₂/Fe₂O₃/CNF composites, their real and imaginary permeability fell below those of Fe₂O₃/CNF yet remained superior to those of MoO₂/CNF. Notably, elevated MoO₂ loading rendered the overall permeability of MoO₂/Fe₂O₃/CNF-3 nearly identical to that of MoO₂/CNF. This trend originates from the drastic reduction in the mass fraction of magnetic Fe₂O₃ as more MoO₂ is introduced, consequently imparting a subdued magnetic response to MoO₂/Fe₂O₃/CNF-3. The magnetic loss tangent ($\tan\delta_\mu$) of all five samples evolved consistently with μ'' (Fig 4e and Fig 4f), demonstrating that magnetic loss within these composites primarily derived from hysteresis loss and magnetic domain relaxation. A comparison of the $\tan\delta_\varepsilon$ and $\tan\delta_\mu$ curves for all five samples reveals that all carbon-fiber composites except Fe₂O₃/CNF are dominated by dielectric loss, with magnetic loss contributing only minor attenuation.

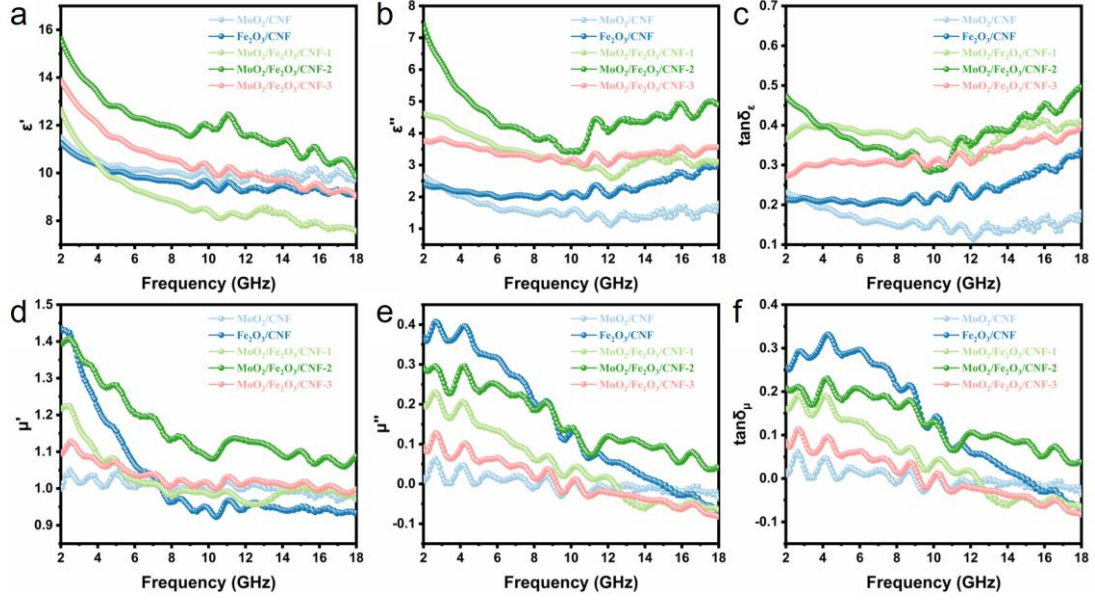


Fig 4. Electromagnetic characteristics of different carbon fiber composites: the real part (a) and imaginary part (b) of permittivity, the dielectric loss ($\tan \delta_\epsilon$) (c), the real part (d) and imaginary part (e) of permeability, and the magnetic loss ($\tan \delta_\mu$) (f).

According to transmission line theory, the microwave attenuation capacity of absorbers can be accessed via the reflection loss (RL), which is calculated using in Equations (3)-(5).

$$Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\epsilon_r}} \tanh \left| j \frac{2\pi f d}{c} \sqrt{\mu_r \epsilon_r} \right| \quad (3)$$

$$Z_0 = \frac{\mu_0}{\epsilon_0} \quad (4)$$

$$RL = 20 \lg \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \quad (5)$$

Where, Z_{in} represents the transmission impedance of electromagnetic waves in absorbers, Z_0 is the transmission impedance of electromagnetic wave in free space, The parameters d , c , and f correspond to the absorber thickness, speed of light, and testing frequency, respectively^[28, 29]. An RL value below -10 dB corresponds to over 90% EMW attenuation, and the matching frequency range over which this condition is satisfied is defined as the effective absorption bandwidth (EAB). Overall, high-performance microwave absorbers are featured by a low minimum RL, a wide EAB, and a small matching thickness, all of which collectively signify outstanding EMW attenuation performance. The 3D RL diagrams of various CNF-based composites are shown in Fig 5.

The MoO₂/CNF composites delivered an RL_{min} of -53.64 dB at a frequency of 16.40 GHz with a thickness of 4.4 mm, albeit with a narrow EAB of only 2.16 GHz (Fig 5a). As for the RL_{min} of Fe₂O₃/CNF composites, its RL_{min} reached -45.55 dB at 8.48 GHz with a thickness of 2.9 mm, yet the maximum attainable EAB was restricted to merely 3.20 GHz (Fig 5b). In sharp contrast to MoO₂/CNF and Fe₂O₃/CNF, the MoO₂/Fe₂O₃/CNF-1 composite exhibited a slightly elevated RL_{min} of -41.69 dB at a frequency of 9.20 GHz with a thickness of 2.9 mm; nevertheless, it realized a substantially expanded EAB of 4.4 GHz at an ultrathin thickness of 1.8 mm (Fig 5c). Remarkably, the MoO₂/Fe₂O₃/CNF-2 composite displayed an RL_{min} of -40.90 dB at a frequency of 10.56 GHz with a thickness of 2.0 mm, and its optimized EAB was broadened to 5.76 GHz at 1.5 mm, covering over 96% of the Ku band (Fig 5d). By comparison, the MoO₂/Fe₂O₃/CNF-3 sample only achieved an EAB of 4.08 GHz at 1.6 mm, while its RL_{min} value of -43.90 dB was emerged at 4.64 GHz with an undesirably large thickness of 4.7 mm (Fig 5e). Collectively, this indicated that superfluous MoO₂ excessive MoO₂ addition to the Fe₂O₃/CNF system weakens the EMW absorption capacity of the materials.

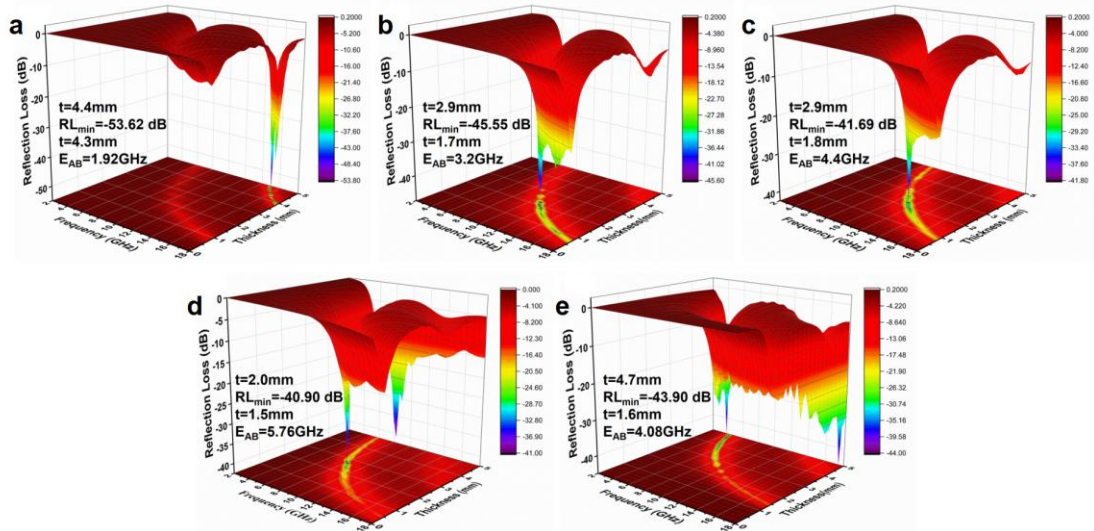


Fig 5. The 3D reflection loss diagram of MoO₂/CNF (a), Fe₂O₃/CNF (b), MoO₂/Fe₂O₃/CNF-1 (c) MoO₂/Fe₂O₃/CNF-2 (d) and MoO₂/Fe₂O₃/CNF-3 (e).

The shift of the matching frequency toward lower frequencies with increasing absorber thickness can be intuitively explained by the quarter-wavelength matching model. In this case, the relationship between the peak frequency and the absorber thickness is presented by Equation (6)^[30].

$$t_m = \frac{n\lambda}{4} = \frac{nc}{4f_m \sqrt{|\mu_r| |\epsilon_r|}} \quad (6)$$

where t_m represents the corresponding coating thickness; λ is the wavelength of microwave; $|\mu_r|$ and $|\varepsilon_r|$ are modules of μ_r and ε_r respectively; c is the speed of light; f_m is frequency and n is the uneven number. When t_m and f_m satisfy the above equation, destructive interference of EMW occurs within the material. Fig S7 shows the relationship between t_m and f_m for the different CNF-based composites. When the thickness of MoO₂/CNF were 4.4 mm and 5 mm, the absorption curve exhibited two peaks, corresponding to $\lambda/4$ and $3\lambda/4$ curve respectively (Fig S7a). For the other four composites, the thickness corresponding to the best performance of the other four carbon fiber composites was highly consistent with the $\lambda/4$ curve, which accords well with the quarter-wavelength model. These results demonstrate that Fe₂O₃/CNF and three different MoO₂/Fe₂O₃/CNF composites can achieve destructive interference of EMW and dissipate electromagnetic energy.

According to Debye's theory, the dielectric loss mainly includes polarization relaxation and conduction loss, which can be specifically expressed using the Cole–Cole curve (Equation7)^[31]. When ε' and ε'' satisfy the above equation, then the plotted ε' – ε'' curve yields a semicircle, which is defined as Cole–Cole polarization.

$$\left(\varepsilon' - \frac{\varepsilon_s + \varepsilon_\infty}{2}\right)^2 + \left(\varepsilon'' - \frac{\sigma}{\varepsilon_0 \omega}\right)^2 = \left(\frac{\varepsilon_s - \varepsilon_\infty}{2}\right)^2 \quad (7)$$

As shown in Fig 6, all the samples showed several semicircles, revealing the existence of multiple polarization relaxation processes. Among them, the MoO₂/Fe₂O₃/CNF-1 composites displayed the largest Cole–Cole semicircle, demonstrating its abundant polarization relaxation behavior. Benefiting from the accumulation of space charges, abundant interfacial polarization loss emerges at the heterogeneous interfaces among Fe₂O₃, MoO₂ and CNF within composite nanofibers. In addition, the linear tail of the Cole–Cole curve is associated with conductive loss, verifying that the conductive network constructed by interwoven nanofibers also participated in the conductive loss pathway of the composite system. Overall, the synergistic dielectric loss mechanisms, consisting of interfacial polarization, dipole polarization and conductive loss, dominate the EMW absorption performance of the composites.

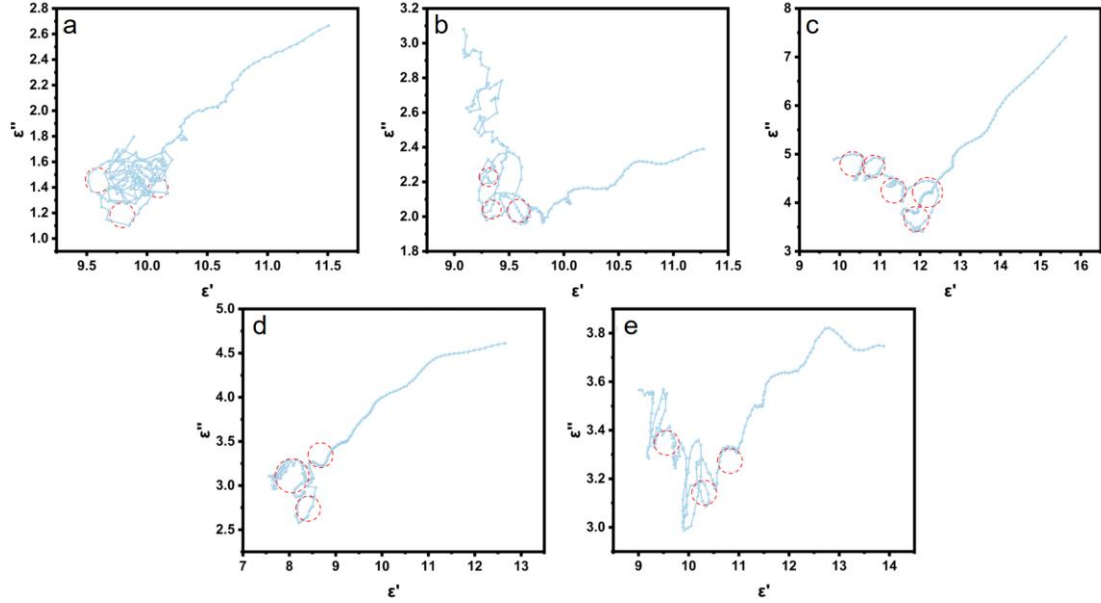


Fig 6. Cole-Cole images of MoO₂/CNF (a), Fe₂O₃/CNF (b), MoO₂/Fe₂O₃/CNF-1 (c), MoO₂/Fe₂O₃/CNF-2 (d) and MoO₂/Fe₂O₃/CNF-3 (e).

The impedance matching ($Z_m=Z_{in}/Z_0$) is a crucial index for assessing the EMW absorption capability of materials. Severe impedance mismatch causes most incident electromagnetic waves to be reflected from the material surface rather than penetrating into the absorbers. The Impedance matching of the different CNF-based composites is shown in Fig 7.

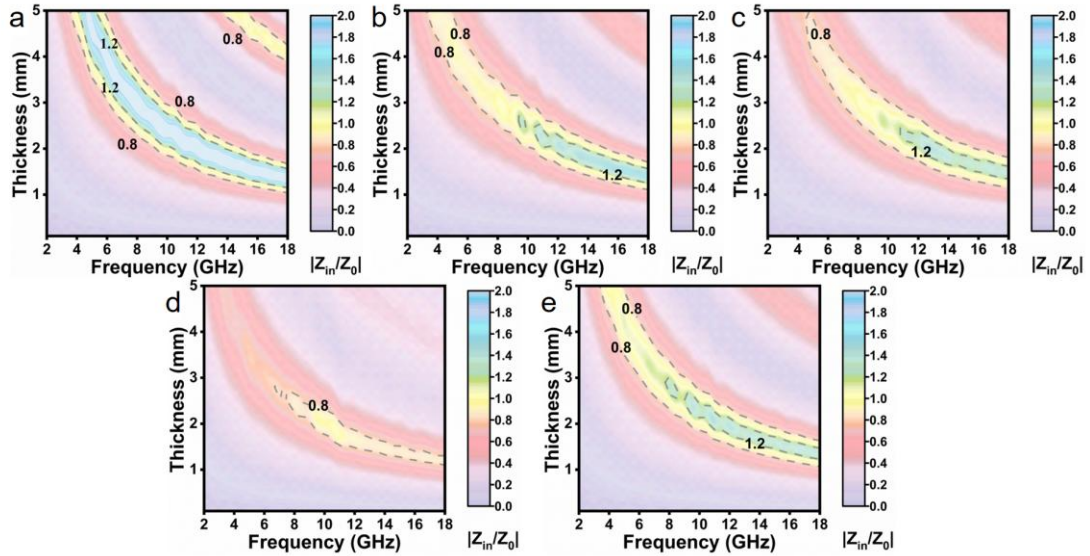


Fig 7. Impedance matching of MoO₂/CNF (a), Fe₂O₃/CNF (b), MoO₂/Fe₂O₃/CNF-1 (c), MoO₂/Fe₂O₃/CNF-2 (d) and MoO₂/Fe₂O₃/CNF-3 (e).

The MoO₂/Fe₂O₃/CNF-2 composite delivered the optimal impedance matching within the frequency range of 8-18 GHz at a thickness of 1-2.5 mm (Fig 7d); followed

by Fe₂O₃/CNF and MoO₂/Fe₂O₃/CNF-1 (Fig 7b-c). In comparison, MoO₂/CNF and MoO₂/Fe₂O₃/CNF-3 exhibited inferior impedance matching behavior under the same thickness conditions (Fig 7a and Fig 7e).

The attenuation constant (α) represents the ability of absorbing materials to dissipate electromagnetic waves and is calculated using Equation (8).

$$\alpha = \frac{\sqrt{2}\pi f}{c} \sqrt{\mu''\varepsilon'' - \mu'\varepsilon' + \sqrt{(\mu''\varepsilon'' - \mu'\varepsilon')^2 + (\mu'\varepsilon'' + \mu''\varepsilon')^2}} \quad (8)$$

A higher α value corresponded to a stronger electromagnetic wave attenuation capacity of the absorbers^[32]. As shown in Fig 8a, the α value of MoO₂/Fe₂O₃/CNF-2 was much higher than the other four CNF composites across the entire 2-18 GHz frequency range, revealing its superior electromagnetic energy dissipation performance. Generally speaking, high attenuation constant and excellent impedance matching are mutually exclusive, making it difficult to achieve both characteristics simultaneously. Nevertheless, through reasonable composition control, the MoO₂/Fe₂O₃/CNF composites can achieve an optimal balance between impedance matching and attenuation constant, which serves as the core reason for their efficient EMW absorption performance.

Magnetic loss mainly consists of eddy current loss, natural resonance, exchange resonance, domain wall resonance loss and hysteresis loss. Within the measured frequency range of 2-18 GHz, the latter two terms can be neglected. The eddy current loss effect can be evaluated by C_0 value (Equation (9)).

$$C_0 = \mu'' \times (\mu')^{-2} f^{-1} \quad (9)$$

When magnetic loss is governed by eddy current loss, the C_0 value remains nearly constant. In contrast, remarkable fluctuations in C_0 are observed if natural resonance acts as the primary magnetic loss mechanism. As depicted in Figure 8b, the C_0 curves of all carbon fiber composites fluctuate drastically within 2-10 GHz, confirming that natural resonance governs magnetic loss in this low-frequency region. By comparison, C_0 gradually levels off from 12 to 18 GHz, revealing that eddy current loss becomes the dominant loss pathway at high frequencies.

To evaluate the practical application potential of the MoO₂/Fe₂O₃/CNFs composites, their far-field absorption characteristics were investigated via radar cross-section (RCS) simulation (Fig 8c-d and Fig S8). RCS is an essential parameter for assessing the radar signal reflection intensity of targets under electromagnetic irradiation, and is calculated

using Equation (10)^[33].

$$\sigma(\text{dBm}^2) = 10 \log \left(\frac{4\pi S}{\lambda^2} \left| \frac{E_s}{E_i} \right| \right)^2 \quad (10)$$

where σ represents to the RCS value, S is the area of the target model, λ corresponds to the wavelength of the incident wave, and E_s and E_i are the electric field intensity of scattered waves and incident waves, respectively. In general, a lower RCS value indicates superior electromagnetic wave absorption capacity. The PEC coated with MoO₂/Fe₂O₃/CNF-2 demonstrated the lowest reflected wave intensity compared with bare PEC and other four materials, confirming its superior far-field EMW absorption performance. At a detection angle of 0°, the RCS values of bare PEC, the PEC coated MoO₂/Fe₂O₃/CNF-2 were 13.98 dBm² and -24.55 dBm², respectively; moreover, the latter remained consistently below -19 dBm² across detection angles from -90° to 90°, manifesting the prominent RCS reduction ability of the composite at the resonant frequency of 10.56 GHz (Fig 8e). These findings demonstrated that the MoO₂/Fe₂O₃/CNF-2 composite holds promising practical application prospects for EMW absorption.

The microwave absorption properties of MoO₂/Fe₂O₃/CNF-2 composite compared with other similar materials are shown in Fig 8f. The micro-wave absorbing performance was enhanced by designing multi-components or heterojunction structures between carbon materials with single metal oxide (Fe₂O₃)^[34-39]. For example, Hou et al^[40] prepared γ -Fe₂O₃@N-rGO/MWCNTs by an in situ anisotropic-oriented growth strategy, showing that the values of RL_{min} and EAB were -59.20 dB and 3.40 GHz when the matching thickness is 4.98 mm and 3.50 mm, respectively. Wang et al^[41] prepared Fe₂O₃(Co₃O₄)/CNT by hydrothermal method and heat treatment way, the sample exhibited the values of RL_{min} and EAB were -53.50 dB and 3.50 GHz when the matching thickness is 2.500 mm. Compare with these work (Fig 8f), MoO₂/Fe₂O₃/CNF-2 composites has obvious advantages in EAB and matching thickness.

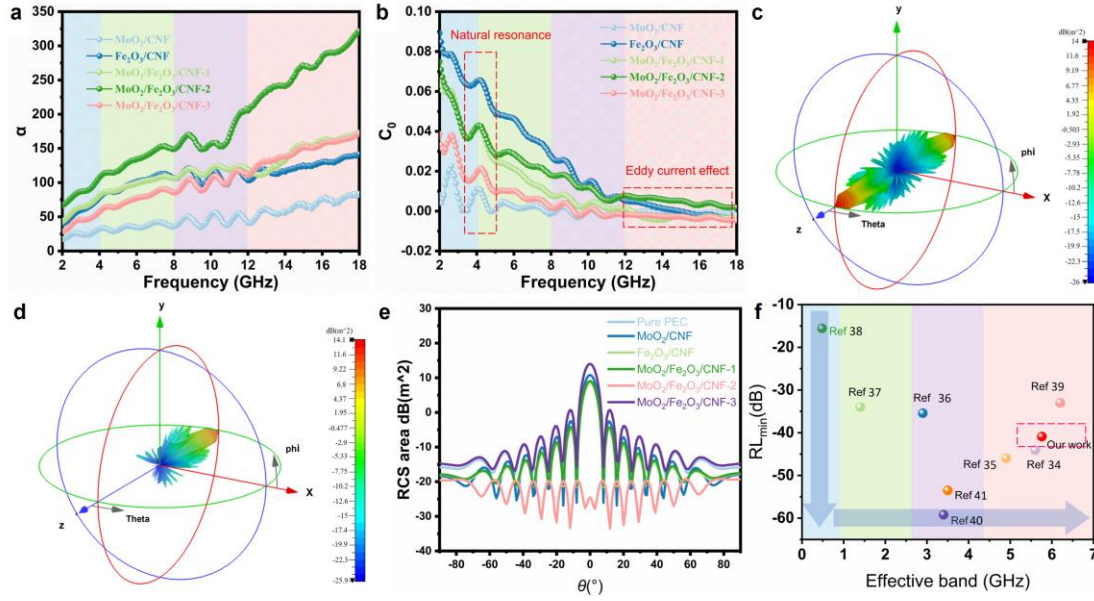


Fig 8 Attenuation constants (a) and C_0 curve (b) of the different carbon fibers composites; CST far-field simulation at 10.56 GHz of PEC (c) and PEC coated with MoO₂/Fe₂O₃/CNF-2 (d); RCS values of pure PEC and PEC coated different carbon fibers composites (e); comparison of MoO₂/Fe₂O₃/CNF-2 with other similar materials (f).

The electromagnetic wave absorption mechanism of MoO₂/Fe₂O₃/CNF is schematically illustrated in Fig 9. The crosslinked network constructed by CNFs, nano MoO₂ and nano Fe₂O₃ possesses a high specific surface area and abundant pores, which facilitated interfacial polarization as well as multiple reflection and scattering of electromagnetic waves. The incorporation of MoO₂ improves the electrical conductivity of CNFs^[24], accelerates the migration and hopping of electrons throughout the network, and thereby boosts the conductive loss of the composite. Meanwhile, Fe₂O₃ nanoparticles introduce magnetic loss pathways to the system, further elevating the composite's capacity for electromagnetic wave attenuation. In summary, the MoO₂/Fe₂O₃/CNFs composites feature abundant heterogeneous interfaces and dielectric-magnetic coupling networks that establish a favorable architecture for EMW absorption. Synergistic interactions among the multiple components enable the coupling of diverse loss mechanisms. Combined with favorable impedance matching, these factors collectively endow the material with outstanding electromagnetic wave absorption performance.

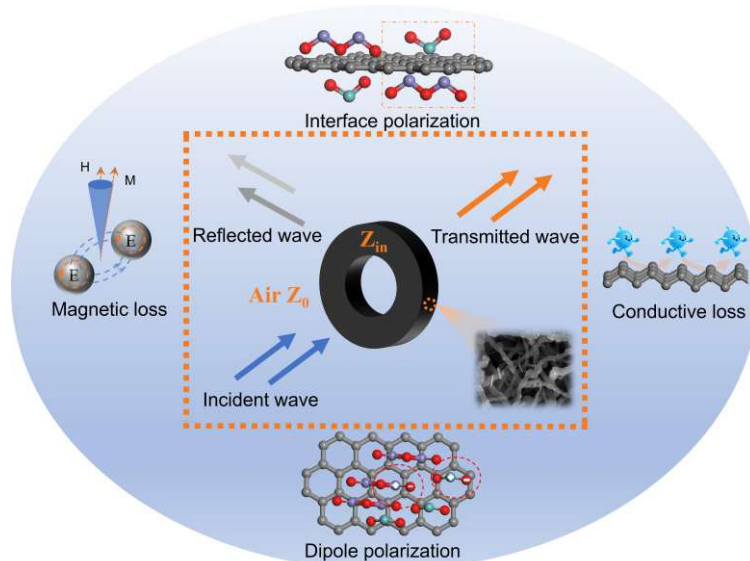


Fig 9 Schematic illustration of the microwave absorption mechanism of MoO₂/Fe₂O₃/CNF composites.

Conclusions

In summary, ternary MoO₂/Fe₂O₃/CNFs composites were successfully fabricated via electrospinning followed by high-temperature carbonization. MoO₂ and Fe₂O₃ nanoparticles were uniformly distributed in carbon nanofibers matrix, contributing synergistically to the enhancement of microwave absorption performance. The optimized MoO₂/Fe₂O₃/CNF-2 sample exhibited a minimum reflection loss of -40.90 dB at 10.56 GHz with a matching thickness of 2 mm, and achieved an effective absorption bandwidth of 5.76 GHz at a reduced thickness of 1.5 mm, covering nearly 96% of the Ku band. CST simulation results further confirmed its outstanding radar-wave attenuation capability and effective suppression of electromagnetic scattering, as evidenced by a significant reduction in radar cross section from 13.98 dBm² for bare PEC to -24.55 dBm² for the PEC substrate coated with the MoO₂/Fe₂O₃/CNF-2 absorber at a detection angle of 0°. The superior microwave absorption performance is mainly attributed to the synergistic effect of conductive loss, magnetic loss, interfacial/dipolar polarization, and favorable impedance matching, which derive from the rational integration of dielectric MoO₂, magnetic Fe₂O₃, and conductive CNFs within a heterogeneous fibrous architecture. Overall, this study offers a promising route for constructing thin EMW absorbers with strong absorption, and may provide useful guidance for the design of next-generation high-performance electromagnetic protection materials.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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