

Multifunctional SiO₂-wrapped CNFs@NiFe₂O₄ nanocomposites for excellent electromagnetic wave absorption, hydrophobicity, and thermal insulation performance

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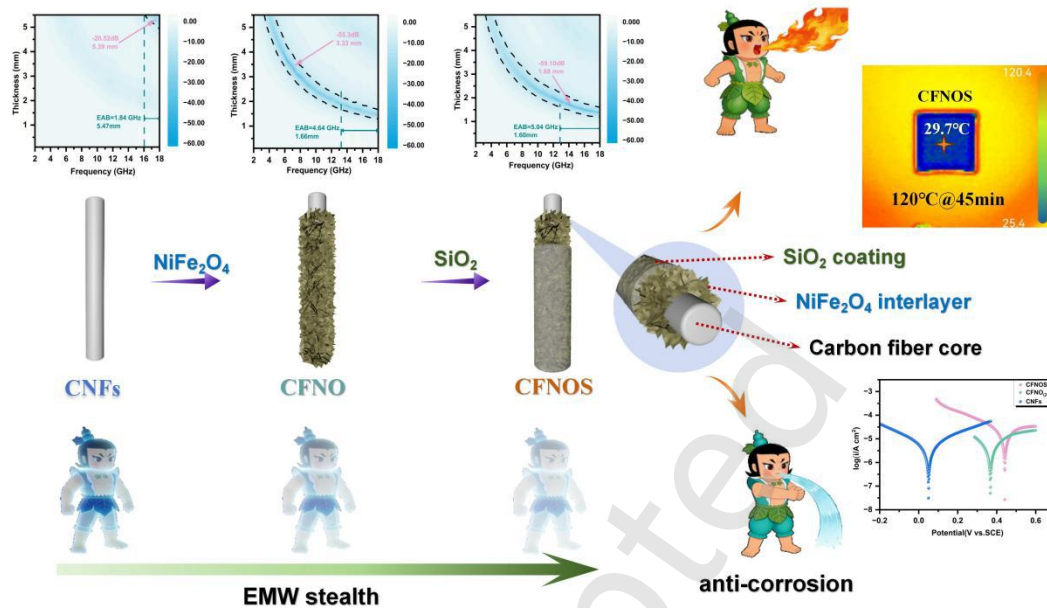
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This work rationally designs a carbon fiber-based ternary composite to realize the synergistic modulation of conductive loss and magnetic loss, achieving highly efficient electromagnetic wave absorption. Benefiting from the collaborative structural advantages, the as-fabricated composite integrates superior thermal insulation, infrared stealth, hydrophobicity and anti-corrosion capabilities, delivering a promising multifunctional solution for advanced intelligent devices and extreme-environment applications.

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ABSTRACT: The electromagnetic wave (EMW) absorbing materials used in harsh working environments are susceptible to thermal degradation and dielectric constant drift caused by humidity fluctuation, which severely restricts their practical applicability in aerospace, marine engineering and advanced electronics. Herein, a hierarchical nanoarchitecture consisting of silica-coated carbon nanofibers uniformly anchored with NiFe₂O₄ nanosheets (denoted as CFNOS) is fabricated via a combined electrospinning-hydrothermal-carbonization route. This ternary structural design circumvents the inherent drawbacks of pristine carbon nanofibers through synergistic dielectric and magnetic loss: NiFe₂O₄ introduces dominant magnetic loss, while the outer silica shell strengthens interfacial polarization to realize efficient EMW dissipation. The optimized CFNOS-2 delivers a minimum reflection loss (RL_{min}) of -59.10 dB at 1.68 mm, together with an effective absorption bandwidth (EAB) of 5.04 GHz at 1.60 mm, exceeding most previously reported carbon-based absorbers. Benefiting from the protective silica layer, CFNOS-2 possesses robust environmental adaptability, featuring a water contact angle of 139.1°, residual mass exceeding 80% upon heating to 800°C, as well as favorable thermal insulation and infrared stealth performance (surface temperature maintained at 120°C over 45min). This work provides a facile structural strategy toward high-performance EMW absorbers applicable for extreme environments, paving a feasible route for next-generation stealth devices and high-density electronic packaging.

KEYWORDS: electromagnetic wave absorption, carbon fibers, multifunctional, SiO₂, attenuation mechanism

1. Introduction

Advancements in electronics and communication technologies over the past decade have driven rapid proliferation of compact, miniaturized portable integrated devices. Despite remarkable improvements in daily life and industrial productivity, continuously intensified electromagnetic radiation from high-power, high-density electronics has aroused widespread concerns over electromagnetic pollution [1-4]. Accordingly, developing high-performance and practically applicable electromagnetic wave absorbers is critical to mitigate electromagnetic interference toward precision equipment and protect human health from related hazards [5-10].

Carbon-based absorbers including porous carbon, graphene, carbon nanotubes and carbon fibers feature high conductivity as well as tailorable dielectric permittivity and dielectric loss [11,12]. In alternating electromagnetic fields, interior polarization relaxation and interconnected conductive networks promote electromagnetic energy dissipation via heat conversion. Benefiting from low density, customizable microstructure, superior chemical stability and affordable raw cost, carbon materials are promising for lightweight EMW absorber

construction [13,14]. Nevertheless, their absorption is dominated merely by dielectric loss. Excessively high intrinsic conductivity further causes mismatched permittivity and permeability, deteriorating impedance matching and restricting their overall EMW absorption capacity [15-18].

Incorporating magnetic fillers into carbon substrates to fabricate binary composite absorbers has proven an effective optimization strategy [19, 20]. Such composite design overcomes the inherent drawbacks of carbon absorbers (single dielectric-loss dominance and unfavorable impedance matching) and meanwhile relieves the deficiencies of conventional magnetic absorbers, namely excessive bulk density and nanoparticle agglomeration [21].

Benefiting from synergistic magnetoelectric coupling, these hybrids realize boosted EMW energy dissipation and enhanced wideband absorption to satisfy the practical demands under complicated electromagnetic surroundings [22-26]. Among typical magnetic candidates, NiFe₂O₄ delivers favorable magnetic loss toward incoming electromagnetic waves. Distinct from metallic magnetic counterparts, its elevated electrical resistivity suppresses high-frequency eddy-current loss and facilitates wide-spectrum EMW attenuation [27-29]. Recent

investigations have verified the promising application potential of C@NiFe₂O₄ nanohybrids for high-performance EMW absorption [30, 31].

The incorporation of NiFe₂O₄ magnetic components can effectively improve the EMW absorption capacity of carbon-based materials. Nevertheless, NiFe₂O₄ suffers from poor weather resistance and structural stability, and fails to maintain stable absorption performance under harsh conditions including high temperature, acid and alkali environments [32-34]. Such inherent defects greatly restrict its practical applications in high-demand fields requiring extreme environmental adaptability, such as aerospace, marine engineering and advanced electronic devices [35-37]. In this work, a layered ternary nanostructure is rationally designed and fabricated through electrospinning, hydrothermal treatment and carbonization. The synergistic dielectric and magnetic loss mechanisms well compensate for the deficiencies of pure carbon fibers: NiFe₂O₄ provides efficient magnetic loss, while the outer SiO₂ coating introduces abundant interfacial polarization. More importantly, the SiO₂ layer endows the composite with multifunctional properties of hydrophobicity, thermal insulation and corrosion resistance, achieving efficient EMW attenuation and excellent environmental durability. This work offers a feasible strategy for the rational design of high-performance, multifunctional electromagnetic wave absorbers.

2. Experimental

2.1. Materials

Polyacrylonitrile (PAN) was purchased from Shanghai Maclin Biochemical Technology Co., Ltd. Ferric chloride (FeCl₃, ≥99%), nickel(II) chloride hexahydrate (NiCl₂·6H₂O, ≥98%), cetyltrimethylammonium bromide (CTAB, ≥99%), N,N-dimethylformamide (DMF, HPLC grade), tetraethyl orthosilicate (TEOS, ≥98%), urea (CH₄N₂O, ≥99%) and anhydrous ethanol (≥99.7%) were supplied by Shanghai Sinopharm Chemical Reagent Co., Ltd. Ammonium hydroxide solution (25-28%) was obtained from Yantai Far East Fine Chemical Co., Ltd. All chemicals were of analytical grade and used as received without further purification. Ultrapure water was produced via an SZ-93A water purification system for all experiments.

2.2. Synthesis of carbon fiber fabrics

A homogeneous precursor solution was prepared by dissolving 10 wt% polyacrylonitrile (PAN) in anhydrous N,N-dimethylformamide (DMF) under continuous magnetic stirring at 60°C for 2 h, forming a viscous, transparent yellow solution. Electrospinning was conducted with a programmable syringe pump fitted with a 21-gauge stainless steel needle at a feed rate of 0.8 mL/h and an applied voltage of 15 kV. The distance between the needle and grounded copper foil collector was fixed at 15 cm, with the collector temperature maintained at 25°C. The as-electrospun PAN nanofibers were pre-oxidized at 280°C for 1 hour in ambient air at a heating rate of 1°C/min. Subsequently, carbonization was carried out at 700°C for 2 hours under continuous high-purity argon flow at a heating rate of 3°C/min, yielding defect-mitigated, morphologically uniform continuous carbon nanofibers.

2.3 Hydrothermal Synthesis of NiFe₂O₄

Nanosheets on Carbon Nanofiber

NiFe₂O₄ nanosheet-decorated carbon nanofibers were fabricated via a facile and optimized hydrothermal strategy. Typically, carbon nanofibers were cut into 2 cm × 2 cm pieces, ultrasonically cleaned sequentially with deionized water and absolute ethanol, and vacuum-dried at 60 °C. The precursor solution was prepared by dissolving 2.5 mmol NiCl₂·6H₂O, different dosages of FeCl₃ (to achieve Ni:Fe molar ratios of 3:1, 2:1, 1:1 and 1:2), 2 mmol CTAB and 9 mmol urea in 40 mL of deionized water. After vigorous stirring for 30 min, a homogeneous ruby-red solution was obtained. The precleaned carbon nanofiber substrates were vertically fixed on the inner wall of a 100 mL Teflon-lined stainless steel autoclave, followed by slow injection of the precursor solution to guarantee uniform growth. The hydrothermal reaction was carried out at 100 °C for 12 h. The harvested composites were alternately rinsed with absolute ethanol and deionized water three times to eliminate residual impurities, and then vacuum-dried at 60 °C. Finally, thermal annealing was performed in a tubular furnace under continuous N₂ atmosphere. The temperature was increased to 350 °C at a heating rate of 3 °C/min and maintained for 2 h. The final samples were denoted as CFNO_(3:1), CFNO_(2:1), CFNO_(1:1) and CFNO_(1:2) according to their corresponding Ni:Fe stoichiometric ratios.

2.4 Fabrication of CNFs@NiFe₂O₄@SiO₂ Nanocomposite

CFNO samples were further coated with SiO₂ layers via a controllable sol-gel strategy. In a typical procedure, 160 mL of deionized water was added into a three-necked flask under continuous magnetic stirring, followed by the sequential addition of 120 mL tetraethyl orthosilicate (TEOS), 20 mL ammonia solution (28 wt%), and 100 mL absolute ethanol. The pre-treated CFNO samples were immersed in the homogeneous solution, and the reaction was maintained at room temperature for 1, 2, and 3 h to explore the effect of coating thickness on material performance. After completion of the sol-gel reaction, the composites were collected and vacuum-dried at 60 °C. The dried samples were subsequently calcined in a tube furnace: the temperature was raised to 700 °C at a heating rate of 3 °C/min under an argon atmosphere and held for 2 h. The final composites obtained with different reaction durations were designated as CFNOS-1, CFNOS-2, and CFNOS-3. The overall fabrication process of CFNOS nanocomposites is schematically illustrated in Figure 1a.

2.5 Characterization

The morphologies of the as-prepared composites were characterized via field-emission scanning electron microscopy (FE-SEM, Hitachi SU8010) and transmission electron microscopy (TEM, Hitachi H-8100). The crystal structures were analyzed by X-ray diffraction (XRD, Rigaku MiniFlex 600) at room temperature. Raman spectroscopy (Renishaw in Via) was utilized to evaluate the phase composition of different samples. X-ray photoelectron spectroscopy (XPS, Thermo Scientific ESCALAB 250XI with Al K α radiation) was employed to investigate the surface elemental chemical states.

Electrochemical performances were measured on a CHI 760E electrochemical workstation with a standard three-electrode system. Cyclic voltammetry (CV) and electrochemical

impedance spectroscopy (EIS) tests were carried out in 6 M KOH electrolyte. An infrared thermal imager (UTi260) and an electronic universal material testing system (TSE104B) were used to characterize the hydrophobic and thermal properties of the materials. The electromagnetic parameters were acquired using an Agilent N5230A vector network analyzer. The test specimens were prepared by uniformly mixing 10 wt% nanocomposites with 90 wt% paraffin wax and pressing the mixture into standard toroidal rings (inner diameter: 3.00 mm, outer diameter: 7.00 mm, thickness: 2.00 mm).

3. Results and discussion

3.1 Materials Preparation and Characterization

Figures 1c and S2a-b show that continuous and smooth CNFs interweave into a well-connected 3D conductive network, which serves as an ideal EMW absorption scaffold. The CNF matrix offers two prominent advantages for EMW attenuation. First, its high specific surface area provides sufficient active sites for the uniform growth of NiFe_2O_4 nanosheets, thereby maximizing material utilization efficiency. Second, the interconnected conductive network promotes rapid electron migration and inter-fiber hopping conduction, strengthening conductive loss via multiple scattering and interfacial polarization.

The hierarchical structure of CFNO composites is displayed in Figures 1d and S2c-d, where uniform petal-like NiFe_2O_4 nanosheets are densely anchored on CNF surfaces to form robust core-shell architectures. Notably, the insets in Figures 1c and 1d verify the excellent mechanical flexibility of the fabricated samples. Both pristine CNFs and CFNO composites maintain structural integrity even under bending deformation exceeding 80° , endowing them great potential for

flexible electronic devices. The statistical diameter distribution in Figure 1e indicates that the average diameter increases from 350 nm (pristine CNFs) to 585 nm after NiFe_2O_4 deposition. The XRD pattern in Figure 1f presents a broad diffraction peak at $2\theta = 25.8^\circ$, corresponding to amorphous carbon. By contrast, the CFNO composite exhibits a set of well-resolved diffraction peaks at 2θ values of 18.4° , 30.3° , 35.7° , 43.4° , 53.8° , 57.4° , 62.9° , 75.6° and 79.6° , which match well with the standard spinel NiFe_2O_4 phase (JCPDS No. 10-0325) and correspond to the (111), (220), (311), (400), (422), (511), (440), (622) and (444) crystal planes [37]. The sharp diffraction signals confirm the successful fabrication of high-crystallinity NiFe_2O_4 with a typical spinel structure.

Figure 1g shows the Raman spectra of pure CNFs and CFNO composites. The characteristic peaks located at 1356 cm^{-1} and 1575 cm^{-1} correspond to the D-band and G-band of carbon materials, which originate from lattice defects of sp^2 -hybridized carbon and in-plane stretching vibration of sp^2 carbon atoms, respectively [2, 38]. A distinct peak at 696 cm^{-1} is attributed to the A_{1g} vibration mode of inverse spinel NiFe_2O_4 , deriving from the symmetric stretching of Fe-O and Ni-O bonds [37].

Quantitative analysis delivers I_D/I_G ratios of 1.240 for pristine CNFs and 1.101 for CFNO, demonstrating the high graphitization degree of both samples. The slight difference in I_D/I_G values indicates that the decoration of NiFe_2O_4 nanosheets introduces negligible structural defects to the CNF matrix, well maintaining the integrity of the carbon framework. Such structural stability provides a reliable foundation for subsequent performance exploration. The high-resolution Ni 2p spectrum of CFNO (Figure 1h) exhibits typical spin-orbit doublets at 855.95 eV ($2p_{3/2}$) and 873.7 eV ($2p_{1/2}$), along with satellite peaks at 862.05 eV and 880.05 eV.

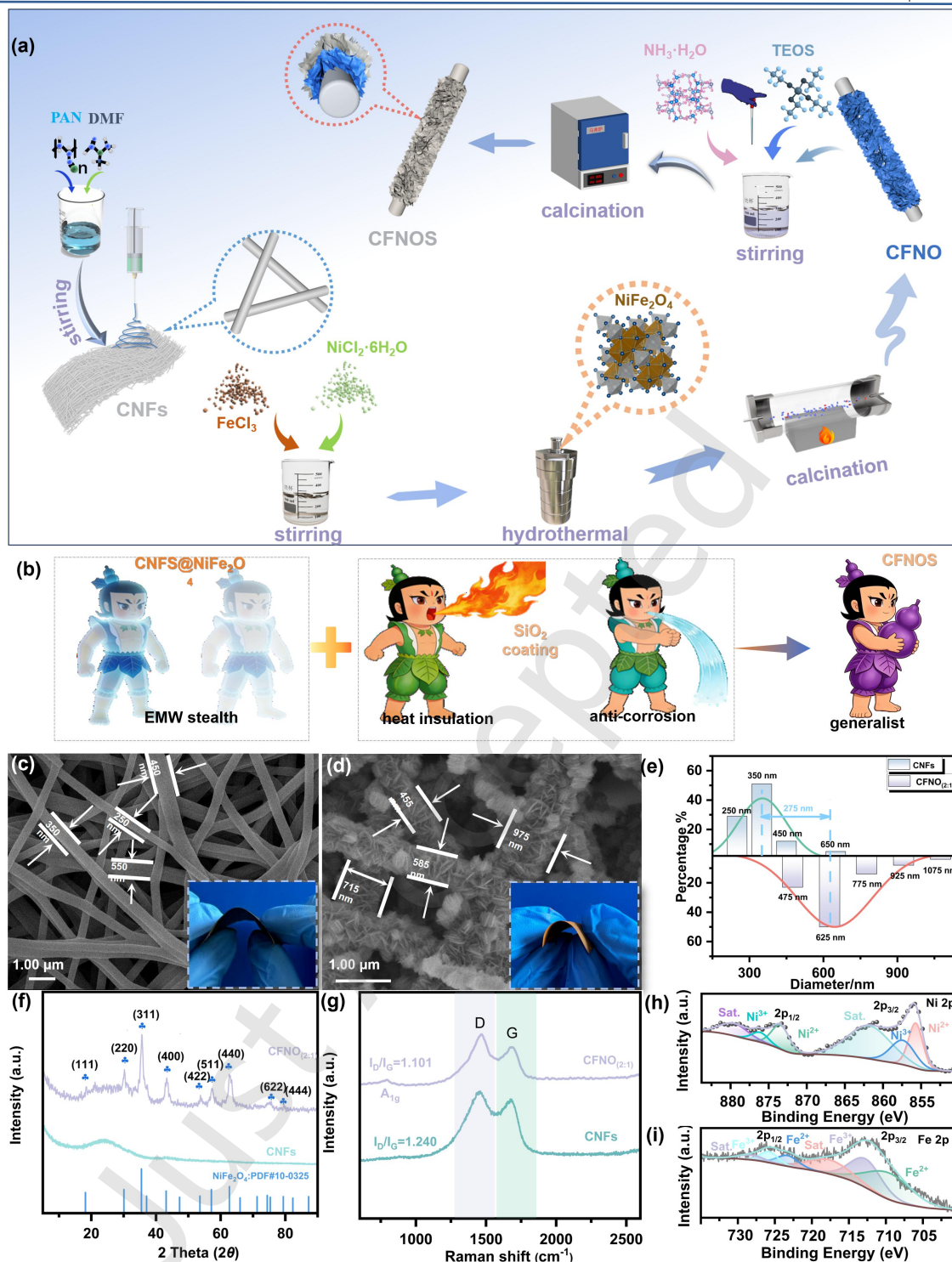


Fig. 1. a) Schematic illustration of the synthesis process for CFNO nanocomposites. b) Schematic illustration of the multifunctional composite properties of silica. c) SEM image of CNF. d) SEM image of typical CFNO sample. e) Diameter distribution profiles of CNF and CFNO samples. f) XRD patterns of carbon nanofiber and CFNO samples. g) Raman spectra of CFNO and CNF samples. h) XPS spectrum of Ni 2p. i) XPS spectrum of Fe 2p.

Gaussian-Lorentzian peak deconvolution resolves the Ni $2p^{3/2}$ peak into two sub-peaks at 855.75 eV and 857.6 eV, which are assigned to Ni^{2+} and Ni^{3+} , respectively [31]. Similarly, the Ni $2p^{1/2}$ spectrum is split into two components at 873.6 eV (Ni^{2+}) and 876.35 eV (Ni^{3+}), confirming the coexistence of bivalent and trivalent nickel ions and enriching the electronic structural characteristics of CFNO. The high-resolution Fe 2p spectrum (Figure 1i) presents six distinguishable spectral signals. Peak fitting results verify two groups of spin-orbit doublets corresponding to different iron valence states: Fe^{2+} (710.4 eV for $2p^{3/2}$, 723.45 eV for $2p^{1/2}$) and Fe^{3+} (713.25 eV for $2p^{3/2}$, 725.95

eV for $2p^{1/2}$). The satellite peaks at 718.5 eV and 728.55 eV further validate the coexistence of Fe^{2+}/Fe^{3+} , and all measured binding energies are consistent with previously reported iron oxide systems. The high-resolution C 1s and O 1s XPS spectra (Figure S4) confirm the existence of multiple functional groups, including O-C=O, C-O, C-N, C-C/C=C, -OH, oxygen vacancies and metal-oxygen (M-O) bonds. These diverse functional groups and defect sites act as polarization centers under alternating electromagnetic fields, inducing strong dipole polarization and additional EMW attenuation, which effectively optimize the wave-absorbing performance [15, 28].

3.2 Electromagnetic Characterization and evaluation of microwave absorption properties

Figures 2a-d and S6 illustrate the frequency- and thickness-dependent reflection loss behaviors of pristine CNFs and diverse CFNO nanocomposites. Pure CNFs deliver a minimum reflection loss (RL) of -20.52 dB at a thickness of 5.39 mm and an effective absorption bandwidth (EAB) of 1.84 GHz at 5.47 mm, revealing the inferior microwave absorption capacity of pristine carbon fiber substrates. The decoration of magnetic NiFe_2O_4 nanosheets substantially optimizes the RL and EAB of CFNO composites with different Ni/Fe ratios. Specifically, the $\text{CFNO}_{(2:1)}$ sample achieves a prominent RL of -55.30 dB at 3.33 mm and a maximum EAB of 4.64 GHz at 1.66 mm. As summarized in Figure 2e, $\text{CFNO}_{(2:1)}$ exhibits stronger absorption intensity, broader effective bandwidth and thinner matching thickness compared with pure CNFs and other CFNO samples, demonstrating superior microwave absorption efficiency. Apart from the magnetic loss contributed by NiFe_2O_4 , the interconnected CNF network endows the composite with efficient conductive loss. Meanwhile, the unique petal-like NiFe_2O_4 architecture induces abundant multiple reflections and scattering of incident electromagnetic waves. Such synergistic dielectric-magnetic loss mechanisms collectively realize efficient EMW attenuation [34, 39].

As nonmagnetic materials, pure CNFs display real permeability (μ') close to 1 and imaginary permeability (μ'') near 0, resulting in negligible magnetic loss. Therefore, the dielectric loss differences between CNFs and CFNO composites serve as the primary analytical focus. Figures 2f and 2g present the frequency-dependent permittivity real part (ϵ') and imaginary part (ϵ'') of CNFs and various CFNO samples. Compared with pristine CNFs, CFNO nanocomposites deliver higher ϵ' and ϵ'' values, verifying that the introduction of NiFe_2O_4 simultaneously enhances the electromagnetic energy storage and dissipation capability of the composites. Varying the Ni/Fe molar ratio effectively regulates the dielectric permittivity, and $\text{CFNO}_{(2:1)}$ exhibits the optimal permittivity performance among all samples. In addition, $\text{CFNO}_{(2:1)}$ shows unique frequency-responsive dielectric behavior: both ϵ' and ϵ'' gradually decrease from 2 to 12 GHz and generate distinct resonance peaks within 12-18 GHz, which originates from the strong interfacial polarization at the $\text{NiFe}_2\text{O}_4/\text{CNF}$ heterogeneous interfaces.

As nonmagnetic materials, CNFs exhibit permeability real (μ') and imaginary (μ'') components hovering near 1 and 0 respectively, yielding negligible magnetic loss. The analysis was focused on comparative dielectric loss characteristics between CNFs and CFNO nanocomposites. Figures 2f and 2g show the variation trends of the real part (ϵ') and the imaginary part (ϵ'') of the permittivity recorded from CNFs and the CFNO samples. The CFNO nanocomposites exhibit enhanced ϵ' and ϵ'' values

compared to CNFs, demonstrating the dual capacity of NiFe_2O_4 components to improve electromagnetic energy storage and dissipation. Changing the magnetic content significantly modulates permittivity, with the $\text{CFNO}_{(2:1)}$ nanocomposite demonstrating superior permittivity values among the series. The optimized $\text{CFNO}_{(2:1)}$ nanocomposite exhibits distinct frequency-dependent dielectric responses: progressive decline of ϵ' and ϵ'' within 2-12 GHz transitions to marked resonance peaks at 12-18 GHz, attributable to interfacial polarization at heterogeneous $\text{NiFe}_2\text{O}_4/\text{CNF}$ interfaces. In the frequency range of 2-18 GHz, the real and imaginary permeability of CFNO samples exhibit modest variations. Nevertheless, this observation does not imply that the electromagnetic wave attenuation contributed by magnetic nanoparticles can be neglected, as these nanoparticles still consume wave energy via hysteresis loss and eddy current loss. For CFNO, magnetic loss mainly originates from direct contact between magnetic nanoparticles and carbon fibers. Accordingly, despite the similar variation trends of permeability, the silica shell induces distinct magnetic loss mechanisms between the two samples. The optimal electromagnetic wave absorbing capacity of CFNO can be attributed to optimized impedance matching and the introduction of extra dielectric loss. Meanwhile, it is noteworthy that the negative μ'' values observed for the CFNO sample imply that electromagnetic wave energy radiates out of the absorber, which is closely related to the internal electromagnetic interactions and microstructures of the sample. Benefiting from the interwoven network constructed by carbon fibers and randomly dispersed NiFe_2O_4 magnetic nanoparticles, electromagnetic waves experience multiple reflections, refractions and scatterings while propagating within CFNO. Interactions among the NiFe_2O_4 magnetic particles modify the direction and intensity of magnetic fields and may trigger electromagnetic field coupling and interference. This results in an abnormal distribution of magnetic field energy in local regions, consequently giving rise to negative imaginary permeability. Consistent with the permittivity evolution, the Cole-Cole semicircles (Figures 2h-l) confirm enhanced Debye relaxation behavior in $\text{CFNO}_{(2:1)}$, which is attributed to the intensified interfacial polarization effect [19, 22]. Moreover, $\text{CFNO}_{(2:1)}$ possesses a larger effective surface area ($|\Delta| < 0.4$) than pure CNFs and other CFNO counterparts (Figure S9), implying superior impedance matching that enables efficient penetration of incident electromagnetic waves into the absorber interior. The quarter-wavelength matching curves in Figures 2n and S10 further demonstrate that $\text{CFNO}_{(2:1)}$ achieves better matching between the RL position and the corresponding thickness. Such optimized matching strengthens the destructive interference effect and thus greatly reduces electromagnetic wave reflection.

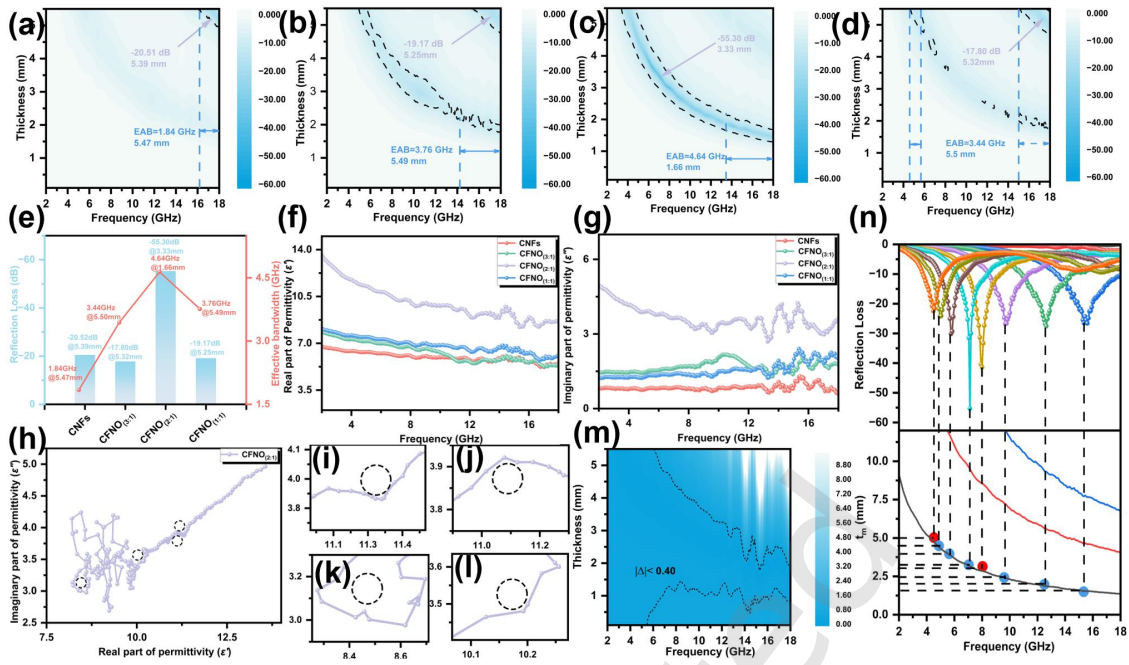


Fig. 2. a-d) RL_{min} values versus frequency for CNFs, CFNO_(3:1), CFNO_(2:1), CFNO_(1:1), e) Histograms and dot line plots of performance captured from CNFs and various CFNO samples, f) Real part curves of permittivity, g) Imaginary part curves of permittivity, h) Cole-Cole curve of optimized CFNO_(2:1) sample, i-l) Partial enlarged drawing of Fig. 2h, m) Delta curve of CFNO_(2:1) sample, n) One-quarter wavelength curve of CFNO_(2:1) sample

3.3 Structure and Characterization

SEM images (Figures 3a-c and S11) show that uniform SiO₂ coatings are anchored on the surface of NiFe₂O₄ nanosheets. Shorter reaction time (CFNOS-1) induce the formation of densely packed SiO₂ nanospheres, while longer reaction time (CFNOS-3) produce a continuous worm-like SiO₂ morphology. This structural variation increases the specific surface area and overall diameter of the composites, as verified by the corresponding inset statistics. The XRD patterns of CFNOS composites (Figure 3d) display characteristic diffraction peaks of NiFe₂O₄ crystallographic planes, alongside a distinct amorphous hump near 20° that originates from the SiO₂ coating. With increased TEOS dosage, the intensity of the amorphous SiO₂ peak gradually rises, whereas the diffraction peak intensity of NiFe₂O₄ decreases. This phenomenon is attributed to the gradual thickening of the silica coating with increasing SiO₂ loading [30, 35].

Raman spectra display the D-band (1363 cm⁻¹) and G-band (1581 cm⁻¹) of carbon structures. The I_D/I_G values of CFNOS samples are 1.127, 1.139 and 1.105, indicating that SiO₂ coating hardly alters the graphitization and defect features of CNFs. The hydrothermal growth of NiFe₂O₄ nanosheets and the subsequent sol-gel coating of SiO₂ involve alkaline conditions and high-temperature calcination (700°C). This process introduces interfacial stress between the carbon matrix and the coated layers. Additionally, the formation of M-O-C (M=Ni, Fe) and Si-O-C bonds at the interfaces disrupts the local sp² carbon

lattice, converting some sp² carbon into sp³ defects, thereby increasing the D-band intensity. Partial Surface Oxidation: During the hydrothermal and sol-gel processes, despite the inert atmosphere protection during calcination, the surface of the carbon fibers is inevitably slightly oxidized by the oxygen-containing precursors (like TEOS and urea), introducing oxygen-containing functional groups (e.g., C-O, C=O) that act as structural defects. Regarding the variation in the A_{1g} peak intensity of NiFe₂O₄ despite similar initial loading amounts: this is a well-known phenomenon in Raman spectroscopy of core-shell nanostructures. As the SiO₂ coating thickness increases (from CFNOS-1 to CFNOS-3), the laser penetration depth of the Raman spectrometer is attenuated by the outermost SiO₂ layer. Because SiO₂ is optically transparent but physically scatters the incident laser, the signal from the underlying NiFe₂O₄ nanosheets is progressively shielded and absorbed. Therefore, the decrease in A_{1g} peak intensity is not due to a decrease in the NiFe₂O₄ content, but rather due to the physical shielding and scattering effects of the progressively thicker SiO₂ coating. The 685 cm⁻¹ and 474 cm⁻¹ Raman peaks correspond to the metal-oxygen stretching of NiFe₂O₄ and Si-O-Si vibration of silica, respectively. The unique Si-O signal in Figure 3f confirms successful SiO₂ modification. XPS results verify that the SiO₂ layer only replaces partial surface -OH groups with Si-O bonds without changing the intrinsic chemical states of C, Fe and Ni, thus maintaining the original EMW attenuation performance of the composites (Figure S12).

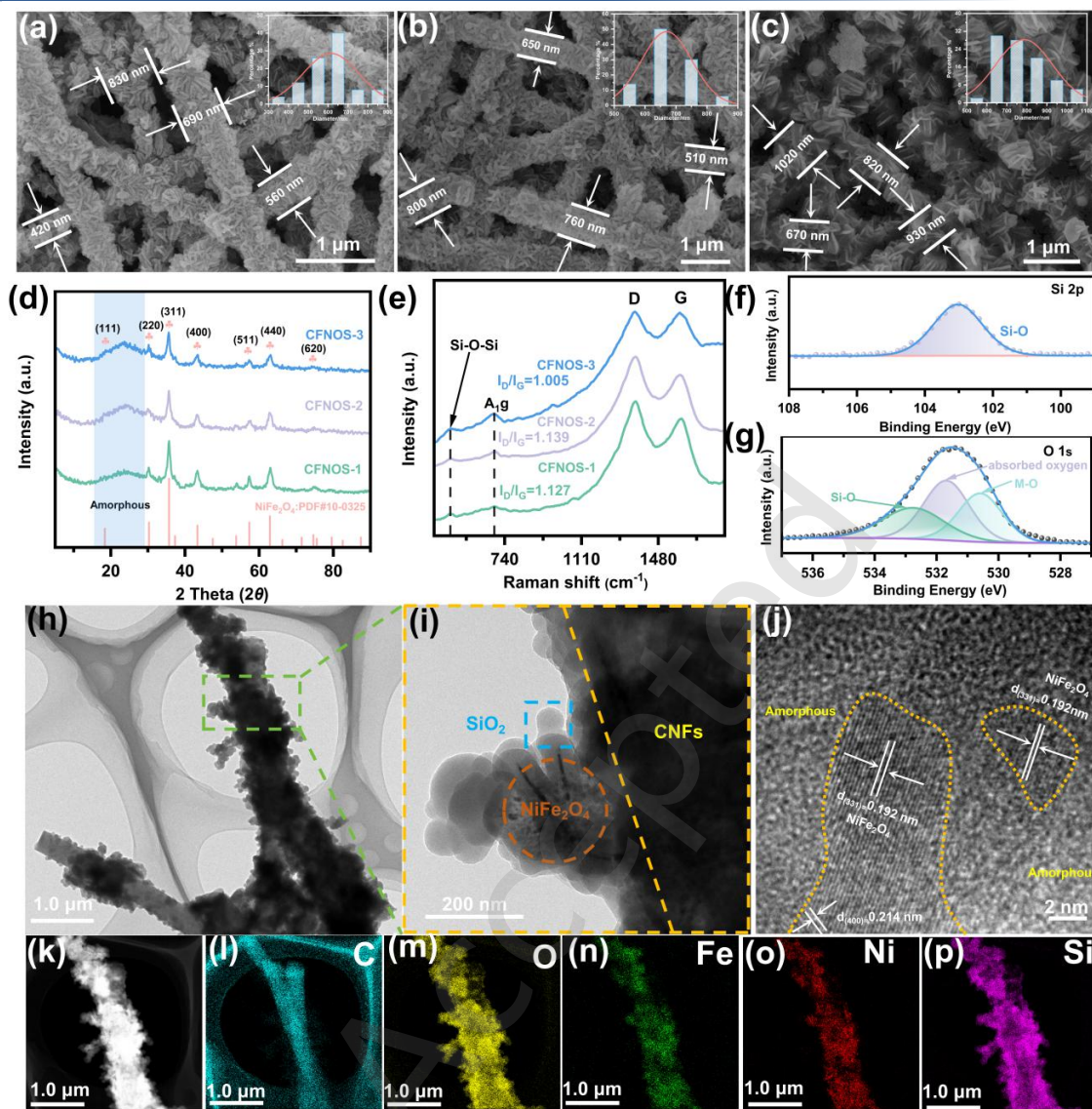


Fig. 3. SEM images and diameter distribution charts of a) CFNOS-1, b) CFNOS-2 and c) CFNOS-3, d) XRD patterns and e) Raman patterns of various CFNOS samples, f) Representative Si 2p, and g) O 1s spectra of CFNOS nanocomposite, h) Low-magnification and i) high-magnification TEM image, j) HRTEM image captured from orange dotted circular area, k-p) C, O, Fe, Ni, Si element mappings of CFNOS-2, respectively.

Figures 3h-i illustrate the hierarchical architecture of the CFNOS-2 composite, where CNFs act as the structural matrix and are uniformly wrapped by SiO_2 -modified NiFe_2O_4 nanosheets. Figure 3h presents a typical fibrous structure with an average diameter of 760 nm, which is highly consistent with the SEM results in Figure 3b. Figure 3i shows the magnified view of the yellow squared region in Figure 3h. The distinct brightness contrast of the polycrystalline NiFe_2O_4 particles reflects diverse grain orientations, and the NiFe_2O_4 surface is densely covered with raspberry-like SiO_2 nanospheres. This well-designed hierarchical structure endows multiple advantages for electromagnetic wave absorption. The outer SiO_2 layer constructs a wave-transparent surface layer to optimize impedance matching, while abundant hetero-interfaces between SiO_2 and NiFe_2O_4 are created to induce interfacial polarization.

More importantly, the superior oxidation and corrosion resistance of SiO_2 compensates for the inherent structural instability of bare NiFe_2O_4 , enabling the composite to maintain stable microwave absorption performance under harsh service environments. The HRTEM image (Figure 3j) displays clear lattice fringes of NiFe_2O_4 . The measured interplanar spacings of 0.192 nm and 0.214 nm correspond to the (331) and (400) crystallographic planes of spinel NiFe_2O_4 , respectively [40]. Elemental mapping images (Figures 3l-p) verify the spatially distributed structure of CFNOS-2. Carbon is mainly concentrated in the central region originating from the CNF matrix, while Fe and Ni are uniformly distributed in the intermediate layer. By contrast, O and Si are enriched in the outer shell region. Such a distinctive elemental distribution is highly consistent with the dark-field morphological features in Figure 3k.

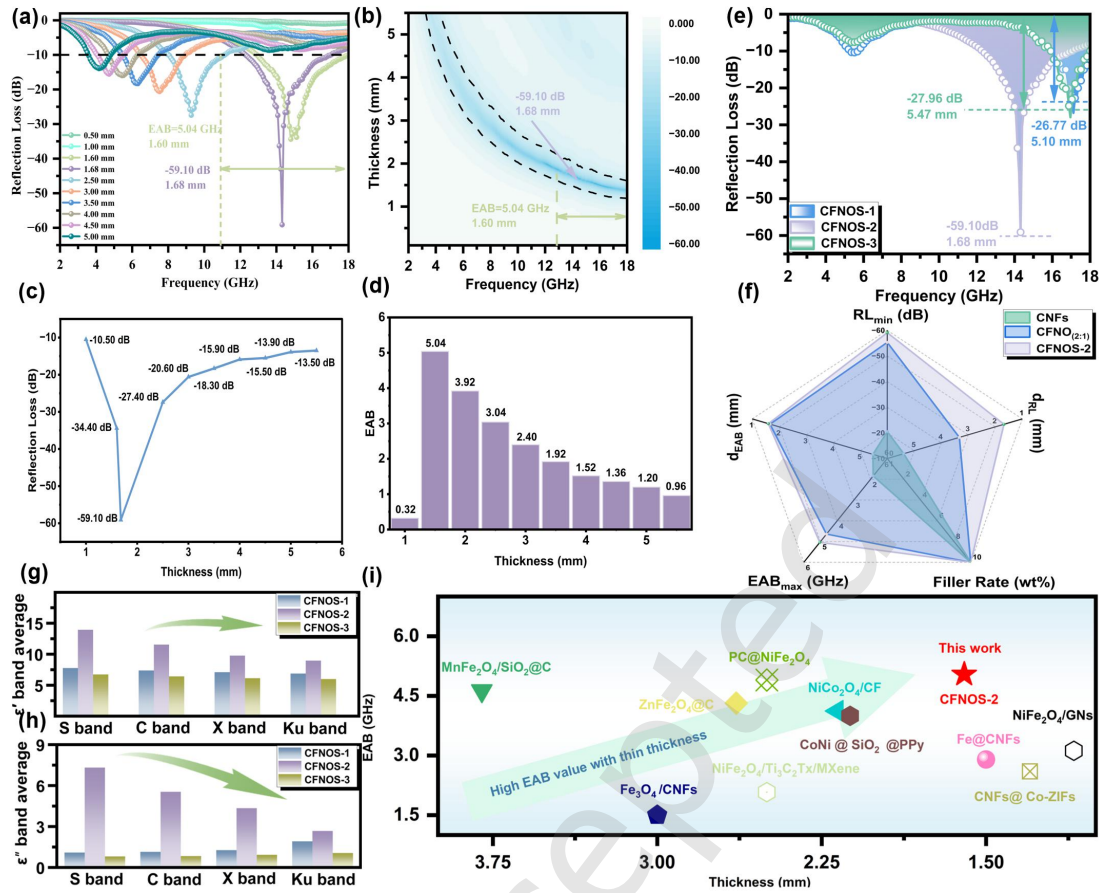


Fig. 4. a) The RL_{min} values of CFNOS-2 at various matching thickness; b) The 2D contour map of EAB range for optimal CFNOS-2 sample; c) Reflection loss diagrams of the CFNOS-2 sample at different thicknesses d) EAB diagrams of CFNOS-2 samples under different thicknesses e) Comparison of the RL_{min} values of different samples f) Comprehensive performance comparison radar chart of CNFs; Average values of g) real parts and h) imaginary parts of different CFNOS samples in different bands; i) EAB values versus thickness between the optimal sample (CFNOS-2) and the relevant samples reported in recent literature .

3.4 Performances and mechanism

As displayed in Figures 4a and S14, the frequency corresponding to the RL_{min} of CFNOS samples gradually shifts toward the low-frequency region from the Ku band to the S band as the matching thickness increases. This trend originates from dielectric dispersion, a typical and fundamental characteristic of microwave-absorbing materials. All CFNOS samples deliver optimal RL_{min} and EAB values within the Ku band. Notably, CFNOS-2 exhibits outstanding microwave absorption performance at all matching thicknesses, with substantially improved RL_{min} and EAB values compared with the other two

counterparts. As summarized in Figure 4e, CFNOS-2 achieves an optimal RL_{min} of -59.10 dB at 1.68 mm and a maximum EAB of 5.04 GHz at 1.60 mm, confirming that a well-regulated SiO₂ coating layer can effectively boost the electromagnetic wave attenuation capability of CFNO composites. Figure 4f compares the overall absorption performance of samples at different fabrication stages. The results clearly verify that the synergistic integration of optimal NiFe₂O₄ magnetic nanosheets and SiO₂ coating into the CNF network significantly enhances microwave absorption performance, enabling CFNOS-2 to realize high-efficiency electromagnetic wave attenuation at a thin matching thickness.

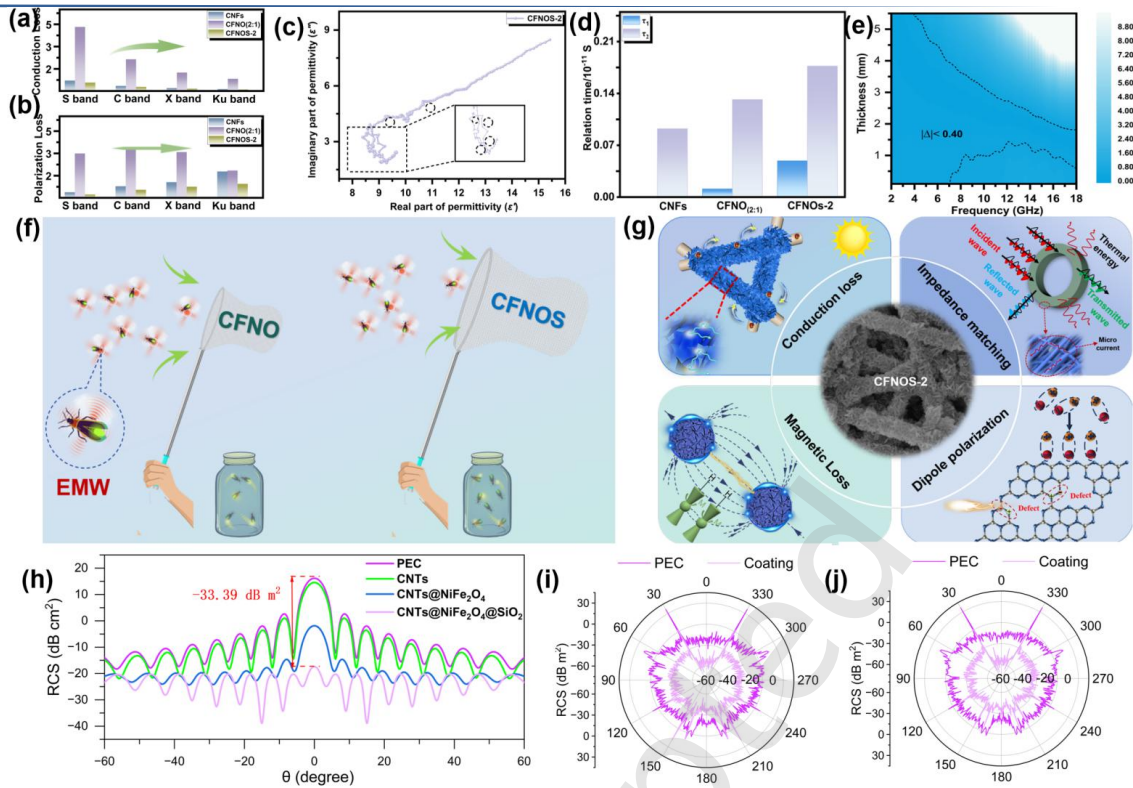


Fig. 5. The average of a) Conduction loss curve and b) Polarization loss curve of CNFs, CFNO(2:1) and CFNOS-2 samples, c) Cole-Cole curve of the CFNOS-2 nanocomposite, d) relaxation times of CNFs, CFNO(2:1) and CFNOS-2, e) CFNOS-2 for the sample delta, f) Schematic illustration of impedance matching improvement, g) Schematic illustration of the electromagnetic wave attenuation for the CFNOS-2 sample, h) Front view and i) Top view RCS profile of UAV covered by CFNOS-2. j) Top view RCS profile of UAV covered by CFNOS-2.

After SiO₂ modification, CFNOS-2 delivers a reduced saturation magnetization of 12.89 emu/g (Figure S13), which is notably lower than that of pristine CFNO. The weakened magnetic performance may partially impair the magnetic loss capacity for electromagnetic wave attenuation. Accordingly, the superior microwave absorption performance of CFNOS-2 is predominantly governed by its enhanced dielectric loss behavior. As shown in Figures 4g and S15, CFNOS-2 exhibits a higher average permittivity real part (ϵ') than CFNOS-1 and CFNOS-3 across the entire tested frequency range. For CFNOS-2, ϵ' decreases monotonically from the S band to the Ku band, and the performance gap between CFNOS-2 and the other two samples gradually narrows at higher frequencies. In comparison, CFNOS-1 and CFNOS-3 maintain relatively stable ϵ' values with negligible fluctuations throughout the measured frequency region. The imaginary permittivity (ϵ'') of the three samples presents a similar variation tendency (Figure 4h). Specifically, CFNOS-2 shows a more significant decline in ϵ'' with increasing frequency, and the discrepancy between its ϵ' and ϵ'' values gradually diminishes. This phenomenon verifies the strengthened electromagnetic wave attenuation ability and optimized impedance matching of CFNOS-2. Furthermore, the comparative analysis in Figure 4i reveals that CFNOS-2 achieves a more competitive $RL_{\min}$, broader EAB and thinner matching thickness than recently reported magnetic and SiO₂-based absorbers, demonstrating its great potential as a high-efficiency microwave absorption material.

3.5 Performances and mechanism

To clarify the underlying mechanisms responsible for the improved dielectric loss of CFNOS-2, the conductive loss and polarization loss behaviors of pure CNFs, CFNO(2:1) and CFNOS-2 composites were systematically investigated over 2-18 GHz (Figures 5a and 5b). Both CNFs and CFNO(2:1) exhibit much lower conductive loss values than CFNOS-2. In addition, the conductive loss of CNFs and CFNO(2:1) decreases slightly

with increasing frequency, whereas CFNOS-2 presents a sharp declining trend. This phenomenon suggests that the contribution of conductive loss to electromagnetic wave attenuation is greatly weakened in the high-frequency region.

The frequency-dependent polarization loss curves of CNFs, CFNO(2:1) and CFNOS-2 are displayed in Figure 5b. CFNOS-2 exhibits the most prominent polarization loss behavior in the high-frequency region. In the mid-to-high frequency region, although the absolute polarization loss value decreases, it surpasses the rapidly declining conductive loss, indicating that polarization loss becomes the dominant attenuation mechanism in this frequency band. Figure 5c presents the Cole-Cole curves of CFNOS-2, where semicircular profiles correspond to interfacial polarization between different composite components. The morphology and distribution of these semicircles intuitively reflect the intensity and effective frequency range of interfacial polarization. Notably, CFNOS-2 possesses six distinct ring-like structures, which are more abundant than those of CFNOS-1 and CFNOS-3 (Figure S17). Additionally, the linear tail at the end of the Cole-Cole curve further verifies the enhanced conductive loss capability of CFNOS-2. The presence of multiple Cole-Cole semicircles confirms the existence of diverse polarization relaxation behaviors when the absorber is exposed to alternating electromagnetic fields. The relaxation time can be quantified according to Equation 3 [38]:

$$\epsilon' = \epsilon_{\infty} + \frac{\Delta\epsilon}{1+(\omega T)^2} \quad (1)$$

$$\epsilon'' = \frac{\Delta\epsilon \cdot \omega T}{1+(\omega T)^2} \quad (2)$$

$$\epsilon' = \frac{1}{2\pi\tau} \cdot \frac{1}{f} + \epsilon_{\infty} \quad (3)$$

According to the theoretical formula, dielectric polarization loss corresponds to a linear relationship between ϵ' and ϵ''/f . Thus, the polarization relaxation time can be determined from the

slope of the ϵ' versus ϵ''/f fitting curve [39]. As shown in Figure 5d, the relaxation times of the obtained composite materials are 0.0924 for CNFs, 0.0111 and 0.1318 for CFNO_(2:1), and 0.0491 and 0.1769 for CFNOS-2. The relaxation time of the CNFs samples is 0 in the low-frequency region, while it shows a longer relaxation time in the high-frequency region. This result is due to the presence of numerous defects in the CNFs, which leads to interface polarization. Unfortunately, these losses contribute very little to the EMW attenuation. However, the CFNOS-2 samples show the longest relaxation time in both the low-frequency and high-frequency regions. The explanation for this phenomenon is that the magnetic nanosheets and SiO₂ have more heterogeneous interfaces. When EMW passes through the magnetic nanosheets and SiO₂, electrons have a greater chance to generate polarization and displacement, thereby enhancing interface polarization.

CFNOS-2 exhibits a broader effective region with $|\Delta| < 0.4$ in the impedance matching contour plot (Figure 5e) than CNFs and CFNO_(2:1), demonstrating optimized impedance matching. Meanwhile, compared with CNFs and CFNO samples, CFNOS-2 (Figure S19) exhibits optimal impedance matching performance, and its matching thickness precisely satisfies the condition of odd multiples of the quarter wavelength. This confirms that CFNOS-2 also strictly follows the destructive interference mechanism. The key difference lies in the matching thickness. Due to the SiO₂ coating altering the effective electromagnetic parameters, CFNOS-2 requires a significantly reduced matching thickness to achieve its RL_{min} peak. On the one hand, CFNOS-2 can effectively suppress electromagnetic wave reflection at the medium interface and raise the fraction of electromagnetic waves penetrating into the absorber, thereby broadening the effective absorption bandwidth. On the other hand, the attenuation curves further verify that CFNOS-2 possesses stronger electromagnetic wave attenuation capability, contributing to its superior overall wave-absorbing performance. This feature enables maximum penetration of incident electromagnetic waves into the absorber interior, which is a prerequisite for high-efficiency microwave attenuation. As intuitively illustrated by the firefly-capturing analogy in Figure 5f, the superior impedance matching of CFNOS-2 functions similarly to a large-aperture net, greatly improving electromagnetic wave capture efficiency. Based on the above analysis, the outstanding microwave absorption performance of CFNOS-2 originates from multiple synergistic loss mechanisms [40-44]. First, the interconnected CNF framework constructs a continuous 3D conductive network, providing efficient pathways for electron migration and hopping, thereby enhancing conductive loss. This cross-linked network also induces abundant multiple scattering and reflection of incident electromagnetic waves, further extending the attenuation path. Second, rich surface defects and functional groups in CFNOS-2 trigger strong dipole polarization, while abundant heterogeneous interfaces between CNFs/NiFe₂O₄ and NiFe₂O₄/SiO₂ generate intense interfacial polarization. The combined dipole and interfacial polarization effects hinder the synchronous response of internal dipole moments with alternating electromagnetic fields, significantly boosting polarization dissipation. Third, the magnetic NiFe₂O₄ nanosheets contribute additional magnetic loss via eddy current effects and natural resonance behavior, further optimizing impedance matching and strengthening the overall attenuation capability of the carbon-based matrix. Radar cross-section (RCS) simulation results further verify the excellent absorption performance of CFNOS-2. The maximum RCS attenuation of CFNOS-2 reaches -33.39 dB m². As shown in Figures 5h-j and S20, the aircraft model coated with CFNOS-

2 presents substantially weaker reflected electromagnetic signals than the bare PEC substrate at the optimal absorption frequency, strongly confirming the superior wave-absorbing and radar stealth potential of the composite.

3.6 Multifunctional Properties

To evaluate the corrosion resistance of the prepared composites, electrochemical tests including electrochemical impedance spectroscopy (EIS) and Tafel polarization curves were performed in three typical corrosive environments: 3.5 wt% NaCl solution (neutral), pH 13.6 NaOH solution (alkaline), and pH 1.3 HCl solution (acidic) [45-48]. The EIS results of the three samples under neutral, acidic, and alkaline conditions are displayed in Figures 6a, S21a, and S22a, respectively. A larger semicircle radius of the EIS spectrum corresponds to higher charge transfer resistance, which reflects superior corrosion protection performance. The tested samples exhibit consistently enhanced impedance responses across neutral, acidic, and alkaline environments, with CFNOS-2 delivering the highest charge transfer resistance among all groups. The high-frequency phase angle is another critical indicator for evaluating coating barrier performance. As presented in Figures 6b, S21b, and S22b, a higher coating resistance corresponds to a larger phase angle between voltage and current in all corrosive media. The prominently enlarged phase angle of CFNOS-2 confirms its excellent capacitive behavior, which enables it to act as a reliable physical barrier against corrosive media. Tafel polarization curves of the composites under three different conditions are shown in Figures 6c, S21c and S22c. Corrosion potential (E_{corr}) and corrosion current density (I_{corr}) are core parameters for assessing coating stability and corrosion resistance. In general, a more positive E_{corr} and a lower I_{corr} represent better anti-corrosion capability. A larger semicircular radius corresponds to higher charge transfer resistance (R_{ct}) of CFNOS-2 reflects weaker charge transfer capability of the samples, which is primarily attributed to its dense and intact SiO₂ shell. This compact coating effectively blocks the penetration of water, oxygen, and corrosive ions, preventing the internal carbon fiber substrate from corrosion. Additionally, the rigid CNF framework improves the mechanical robustness of the composite coating, extending its service durability and protective stability.

Impedance statistical results in Figures 6d, S20d and S21d further verify that CFNOS-2 possesses substantially higher impedance than the other samples in all corrosive solutions, which is well consistent with its excellent anti-corrosion performance. Equivalent circuit models before and after corrosive medium penetration to the substrate are established in Figures 6e-g, further demonstrating that the SiO₂ coating efficiently suppresses the electrochemical corrosion behavior of the substrate in simulated seawater and other harsh environments. These results comprehensively confirm the excellent and stable corrosion resistance of CFNOS-2 under acidic, neutral, and alkaline conditions.

The hydrophobicity of CNFs, CFNO_(2:1), and CFNOS-2 was evaluated via water contact angle tests (Figures 6h-j). CFNOS-2 exhibits the strongest hydrophobic performance with a high water contact angle of 139.1°, which is substantially higher than those of pure CNFs and CFNO_(2:1). The superior hydrophobicity originates from the rough surface microstructure of the SiO₂ coating, which reduces the solid-liquid contact area [49-51]. Such excellent hydrophobic behavior further improves the anti-corrosion capability of the composite in humid and

corrosive environments [52]. Thermogravimetric analysis was performed to assess the thermal stability of CFNOS-2. As displayed in Figure 6p, the TGA curves present distinct differences in mass loss below 400 °C for CNFs, CFNO and CFNOS samples. For pristine CNFs, the prominent weight loss below 400 °C mainly originates from the volatilization of adsorbed water and the decomposition of residual oxygen-containing surface functional groups generated during electrospinning and stabilization. Bare carbon fibers are prone to early oxidative degradation. For CFNO, anchored NiFe_2O_4 nanosheets serve as a protective layer covering surface defects of carbon fibers. Furthermore, the hydrothermal treatment and subsequent annealing partially eliminate or stabilize the most labile oxygen-containing groups on CNFs. Consequently, CFNO delivers enhanced thermal stability below 400 °C with negligible mass loss. In contrast, the SiO_2 coating of CFNOS is fabricated via the sol-gel route using TEOS. Despite drying at 60 °C and calcination, abundant silanol groups (Si-OH) and unhydrolyzed ethoxy residues persist within the SiO_2 network. Upon heating from ambient temperature to 400 °C, Si-OH undergoes condensation reactions accompanied by water release, together with the decomposition of residual organic fragments. Such physicochemical evolution inside the SiO_2 coating accounts for the greater weight loss of CFNOS relative to CFNO in this low-temperature range.

CFNOS-2 achieves its maximum thermal decomposition rate at 543.5 °C under a nitrogen atmosphere, with a residual mass retention of 79.1% at 800 °C, demonstrating outstanding thermal stability. Beyond its superior corrosion resistance and hydrophobicity, CFNOS-2 also possesses excellent thermal insulation performance [53]. The thermal insulation capability was tested by placing the composite on heating platforms at 60 °C and 120 °C for 15 min and 45 min, respectively. The surface temperature variation was recorded in real time using an infrared thermal imager (Figures 6k-m and S23). After continuous heating at 120 °C for 45 min, the central surface temperatures of CNFs, CFNO_(2:1), and CFNOS-2 reach 91.4 °C, 82.4 °C, and 32.7 °C, respectively. The remarkably low surface temperature of CFNOS-2 verifies the effective thermal insulation effect introduced by SiO_2 modification [13, 54-58]. Auxiliary thermal insulation verification experiments further confirm the excellent heat-resistance performance of CFNOS-2 (Figure 6o). When CFNOS-2 was applied as a thermal insulation layer beneath fresh flowers, the flowers remained intact without withering after 120 s of heating, intuitively demonstrating the prominent thermal insulation advantage of CFNOS-2 for high-temperature service scenarios. The reliable thermal insulation performance of CFNOS-2 is governed by two structural factors: (1) The unique 3D network structure of CFNOS-2 reduces the interfacial contact between nanofibers, thereby suppressing solid-phase thermal conduction; (2) The densely packed SiO_2 particles confine air molecules within the composite framework, effectively weakening gas-phase thermal convection.

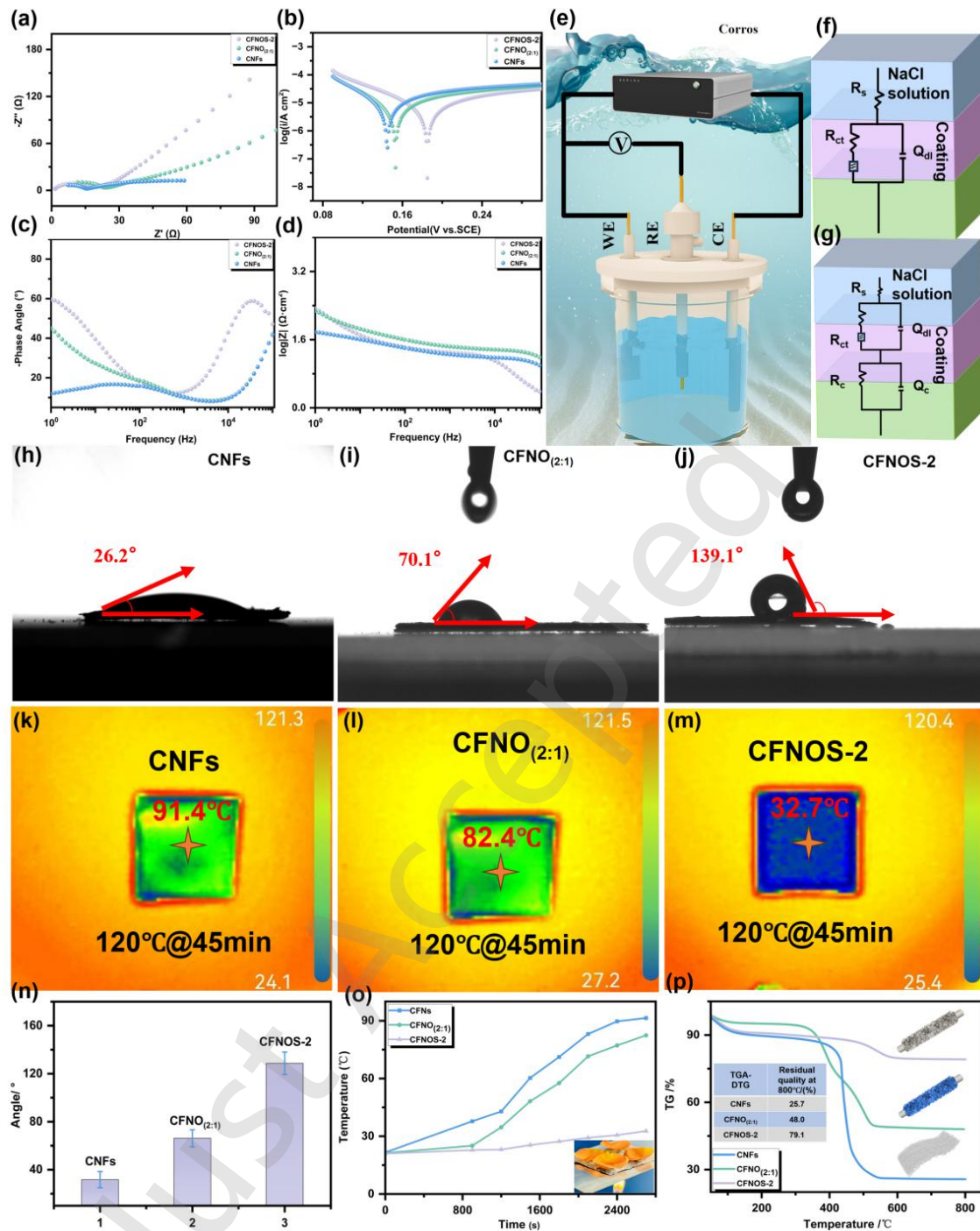


Fig. 6. (a-d) Electrochemical characterization of CNFs, CFNO_(2:1), and CFNOS-2 under neutral conditions: (a) Nyquist plot, (b) Tafel plot, (c) phase-frequency Bode plot, (d) impedance-frequency Bode plot. (e) Schematic diagram and (f-g) equivalent circuit models of coatings at different stages. (h-j) Water contact angles of CNFs, CFNO_(2:1), and CFNOS-2. (k-m) Infrared thermal images and (n) water contact angle error bars. (o) Curve showing the temperature changes on the sample surface during the heating process. (p) TG curves of CNFs, CFNO_(2:1), and CFNOS-2

4. Conclusions

In conclusion, in this paper, using polyacrylonitrile (PAN) and dimethylformamide (DMF) as raw materials, through process conditions such as electrospinning, hydrothermal treatment, and carbonization, a composite material consisting of nano-carbon fibers, nickel-iron dioxide (NiFe_2O_4), and silica nano-carbon fibers with high microwave absorption efficiency was designed and synthesized. The results showed that when the thickness was 1.68 mm, the minimum reflection loss reached -59.10 dB; simultaneously, when the matching thickness was 1.60 mm, the best EAB (Electromagnetic Absorption Bandwidth) was 5.04 GHz. Through reasonable structural design and chemical composition adjustment, a multi-component magnetic medium cooperative system was established, thereby achieving excellent electromagnetic wave absorption performance. During the synthesis process, NiFe_2O_4 thin sheets would regularly distribute on the surface of the carbon nanofibers, and they had a dense layered structure. Both surfaces were uniformly coated with silica, thus obtaining a relatively smooth surface. Due to the cooperative effect between the multi-component composite materials, these materials have rich heterogeneous interface structures, excellent impedance matching characteristics, high conductive loss and polarization loss. In addition, this composite material has excellent heat insulation, infrared stealth, hydrophobicity and corrosion resistance, which indicates that it has a broad application prospect as a good absorber and provides unique ideas and insights for the structural design of multi-component composite materials.

Electronic Supplementary Material: Supplementary material is available in the online version of this article.

Data availability

All data needed to support the conclusions in the paper are presented in the manuscript and/or the Electronic Supplementary Material. Additional data related to this paper may be requested from the corresponding author upon request.

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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.

Author contribution statement

MingboLiu: Investigation, methodology, writing-original draft, formal analysis. Jinyuan Liu: Conceptualization, validation, data curation, writing-review & editing. Qianqian Jia: Writing-original draft, formal analysis, software. Xueli Qi: Funding acquisition, methodology, validation. Weichen Ding: validation. Yujiao Wang: Formal analysis. Haoyu Zhao: Software. Xiaolong Chi: Data Curation. Qiang Luan: Funding acquisition. Yanrui Jiang: Visualization. Zhongning Tian: writing-original draft, visualization. Zhenjiang Li: Funding acquisition, writing-review & editing. Meng Zhang: Conceptualization, resources, data curation, project administration, funding acquisition, writing-review & editing. All the authors have approved the final manuscript.

Use of AI statement

During the preparation of this work, the authors used DEEPSEEK in order to polish the English grammar. The author used the Doubao software to draw the image of the Calabash Brothers, making the diagram more vivid and attractive. After using this tool/service, the authors reviewed, edited, and verified all AI-assisted outputs and take full responsibility for the content of this publication.

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Electronic Supplementary Material

Multifunctional SiO₂-wrapped CNFs@NiFe₂O₄ nanocomposites for excellent electromagnetic wave absorption, hydrophobicity, and thermal insulation performance

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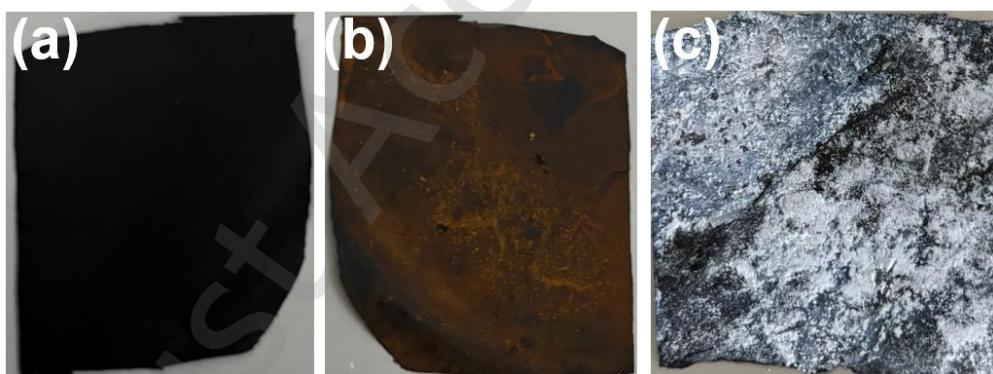


Fig. S1. Macroscopic photos of a) CNFs, b) CNFs@NiFe₂O₄, c) CNFs@NiFe₂O₄@SiO₂

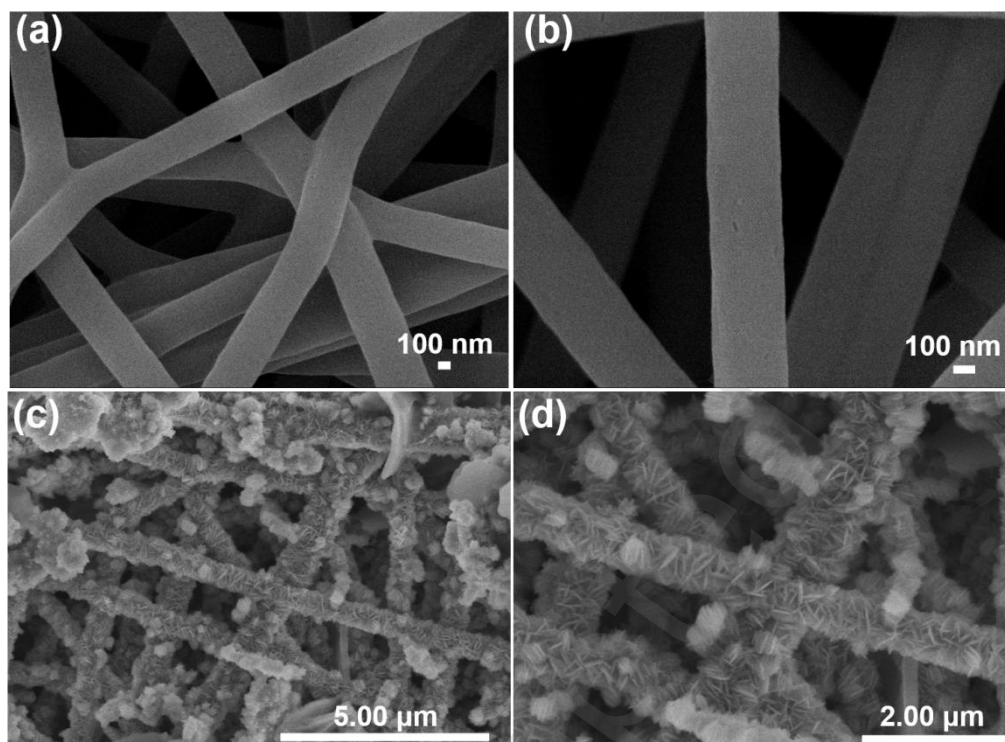


Fig. S2. a-b) SEM of CNFs, c-d) SEM of CFNO_(2:1)

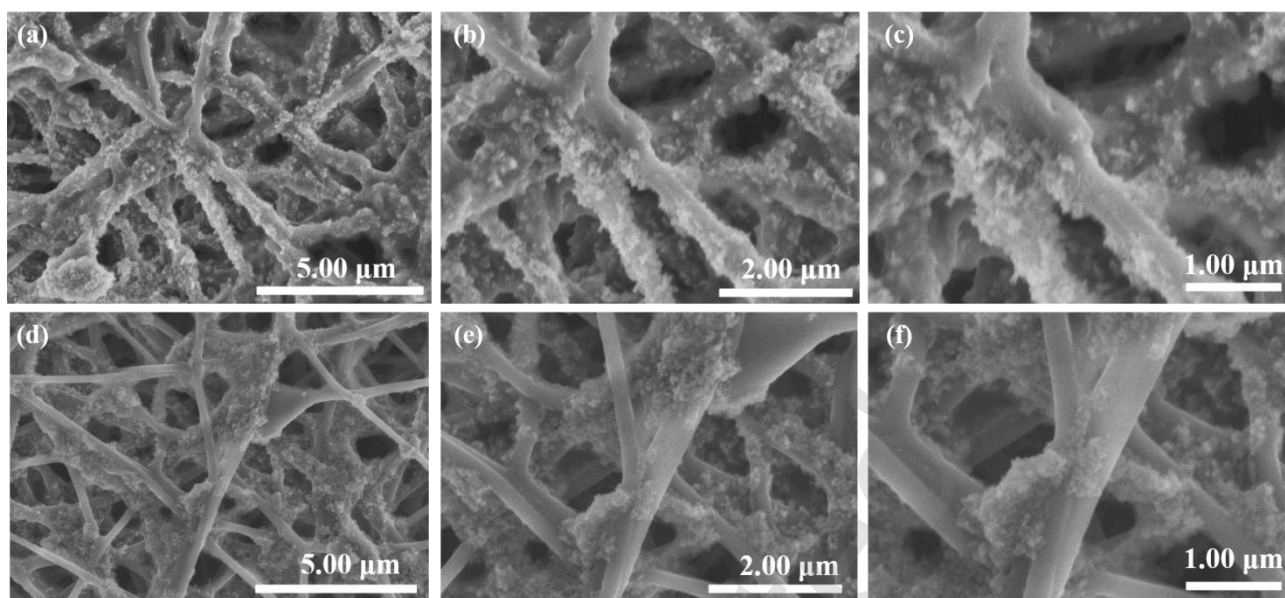


Fig. S3. a-c) SEM of CFNO_(1:1), d-f) SEM of CFNO_(3:1)

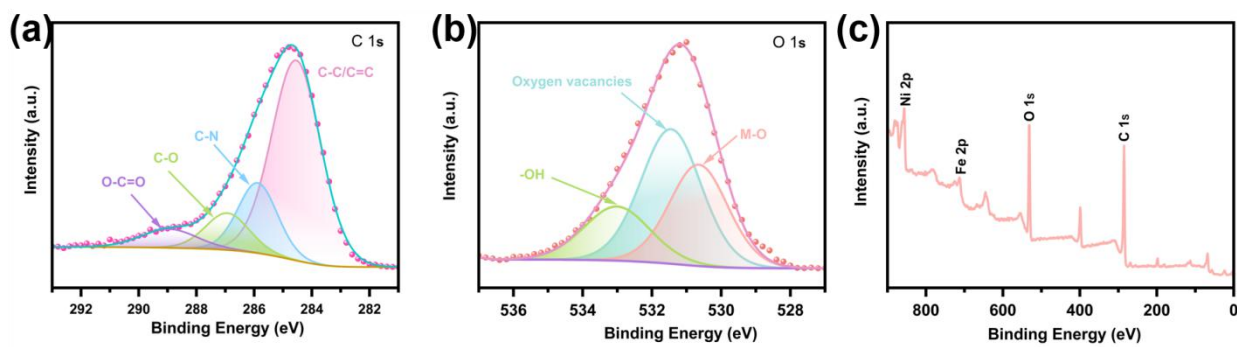


Fig. S4. XPS CFNO_(2:1).

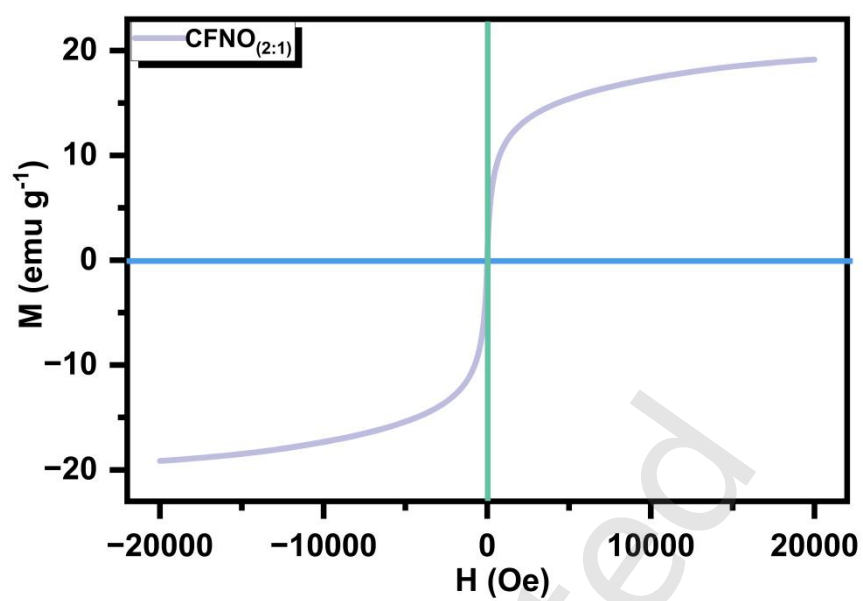


Fig. S5. The hysteresis loop of $\text{CFNO}_{(2:1)}$ nanocomposite material at room temperature

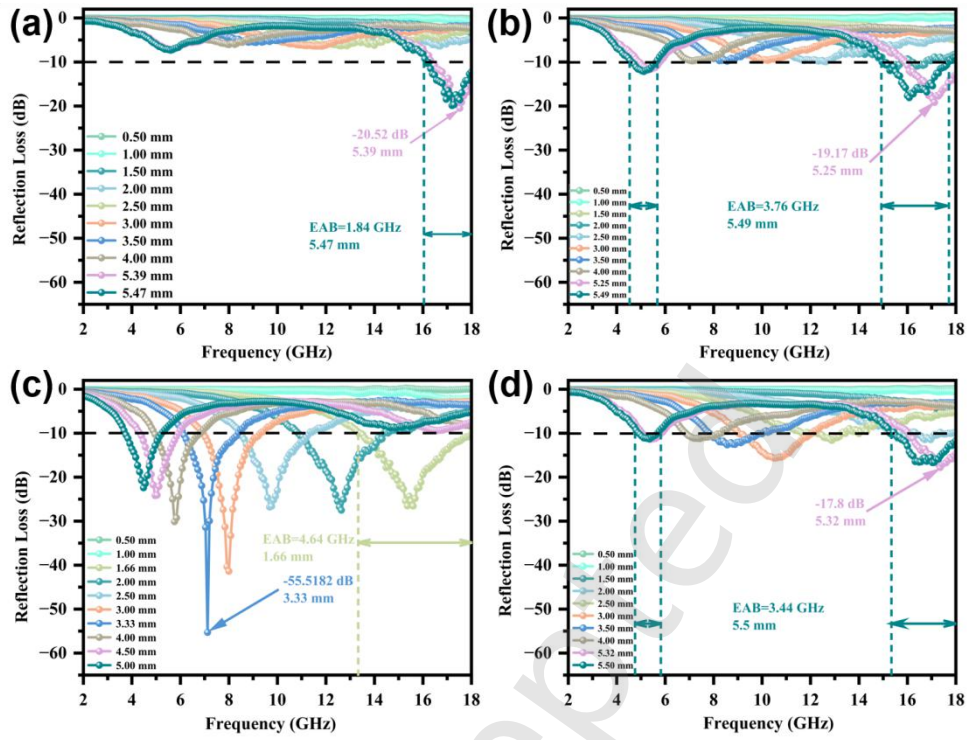


Fig. S6. The 2D RL value curves versus frequency of a) CNFs, b) CFNO_(3:1) sample, c) CFNO_(2:1) sample, d) CFNO_(1:1) sample.

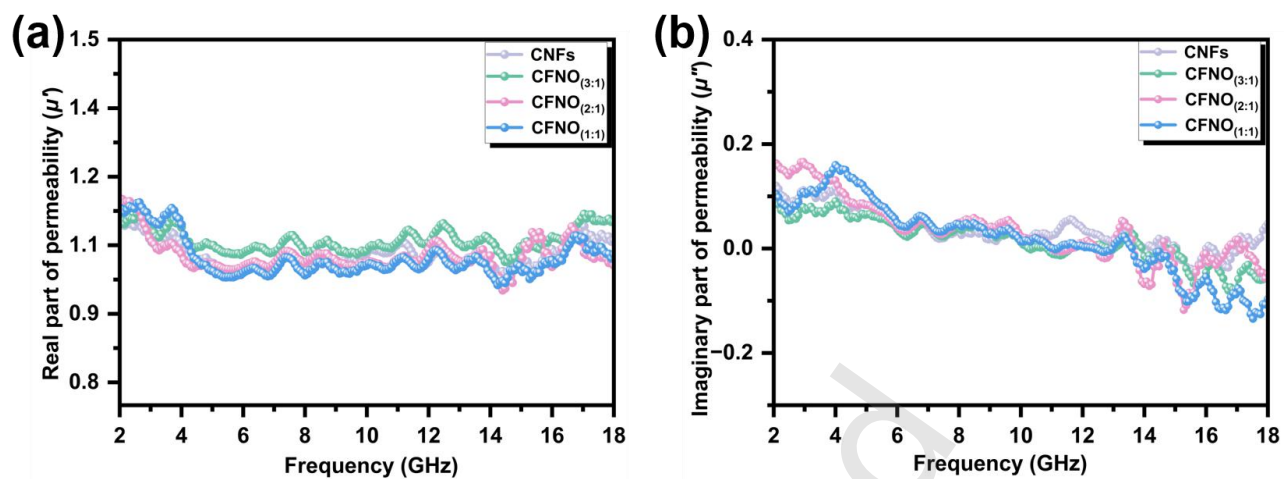


Fig. S7. a) real part of magnetic permeability, b) imaginary part of magnetic permeability.

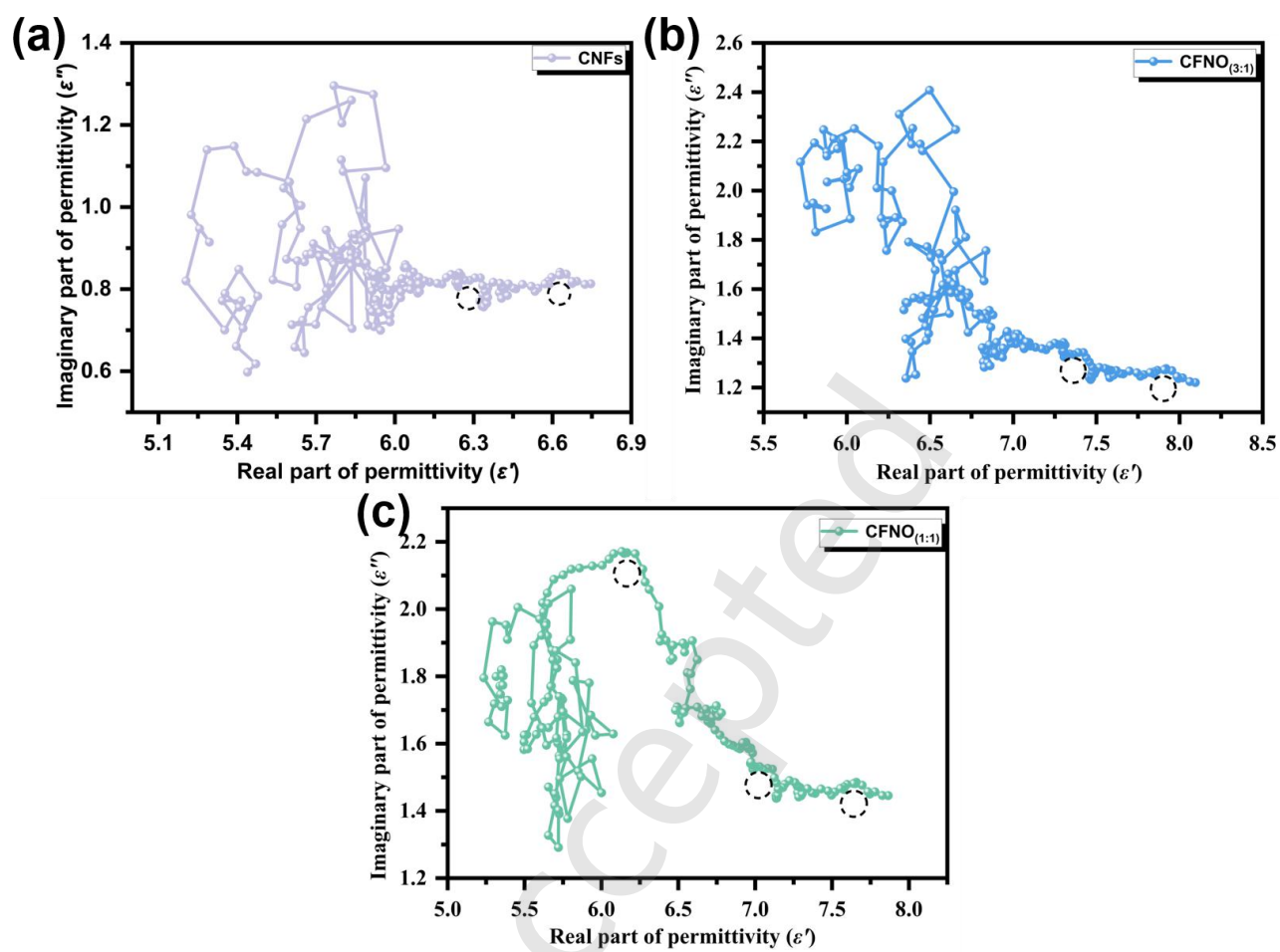


Fig. S8. Cole-Cole curve of a) CNFs, b) CFNO_(3:1), c) CFNO_(1:1).

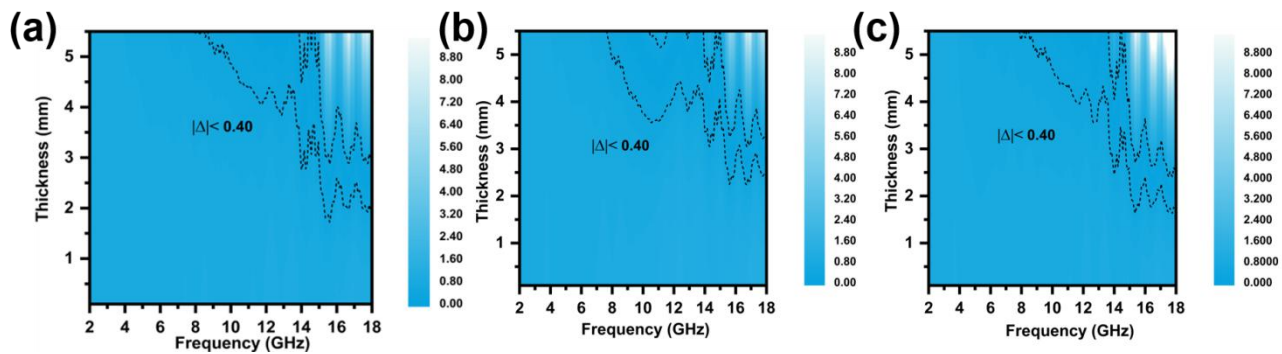


Fig. S9. $|\Delta|$ of a) CNFs, b) CFNO_(3:1), and c) CFNO_(1:1).

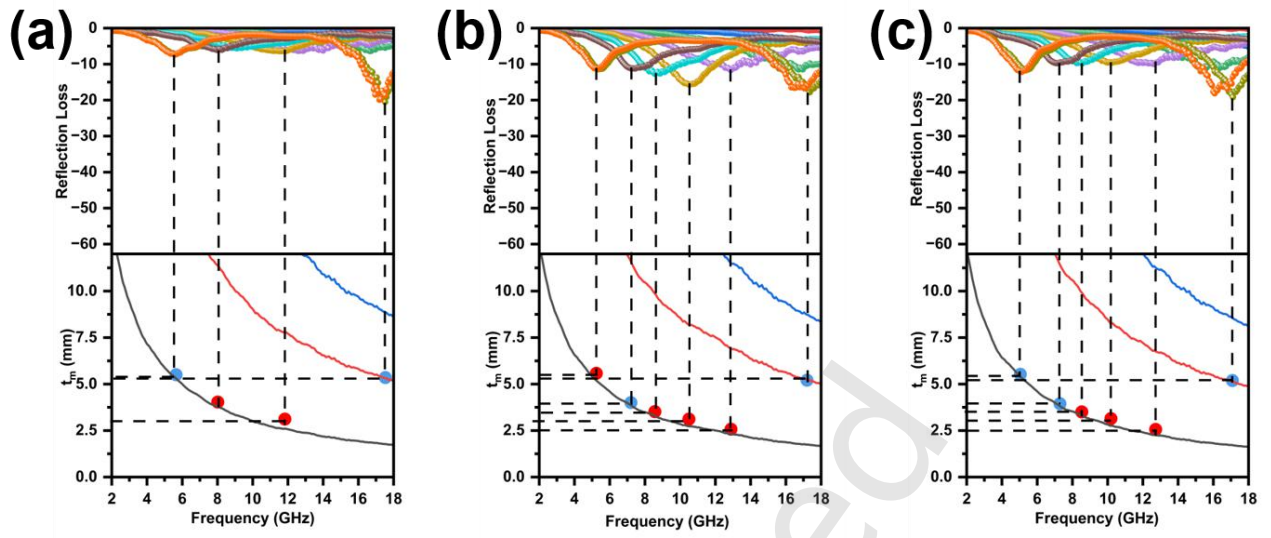


Fig. S10. One-quarter wavelength model for a) CNFs, b) CFNO_(3:1), CFNO_(1:1).

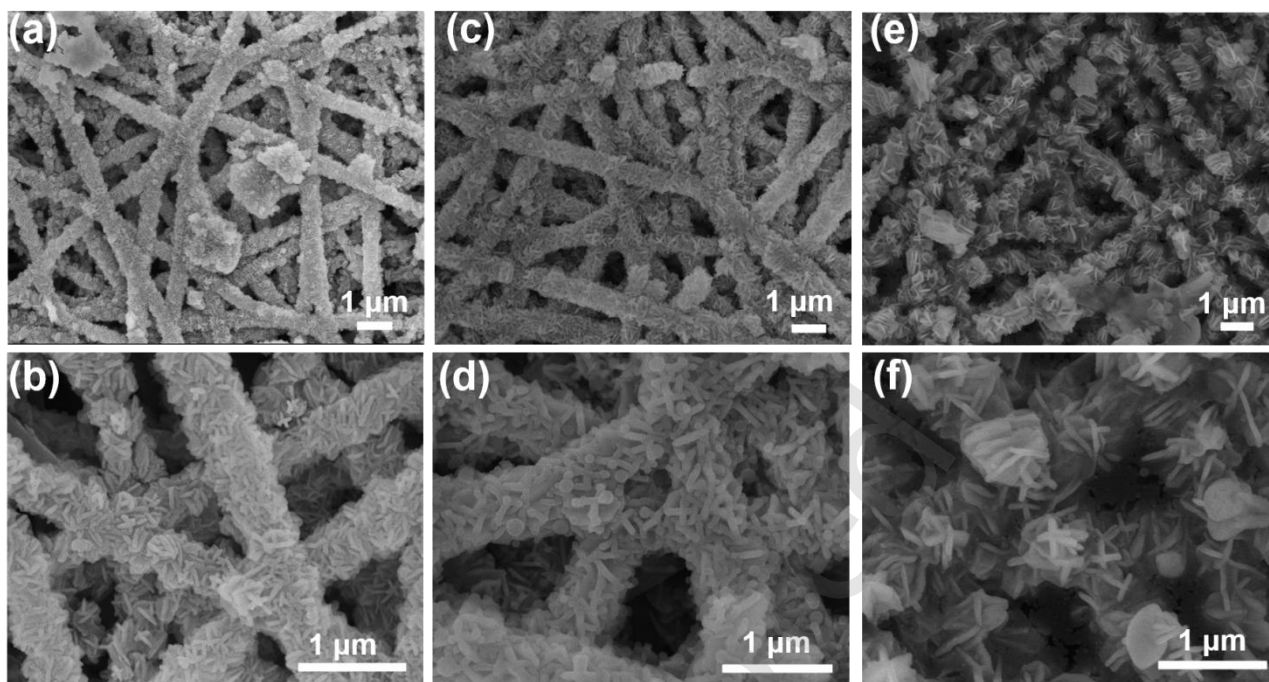


Fig. S11. a-b) SEM of CFNOS-1 , c-d) SEM of CFNOS-2 , e-f) SEM of CFNOS-3

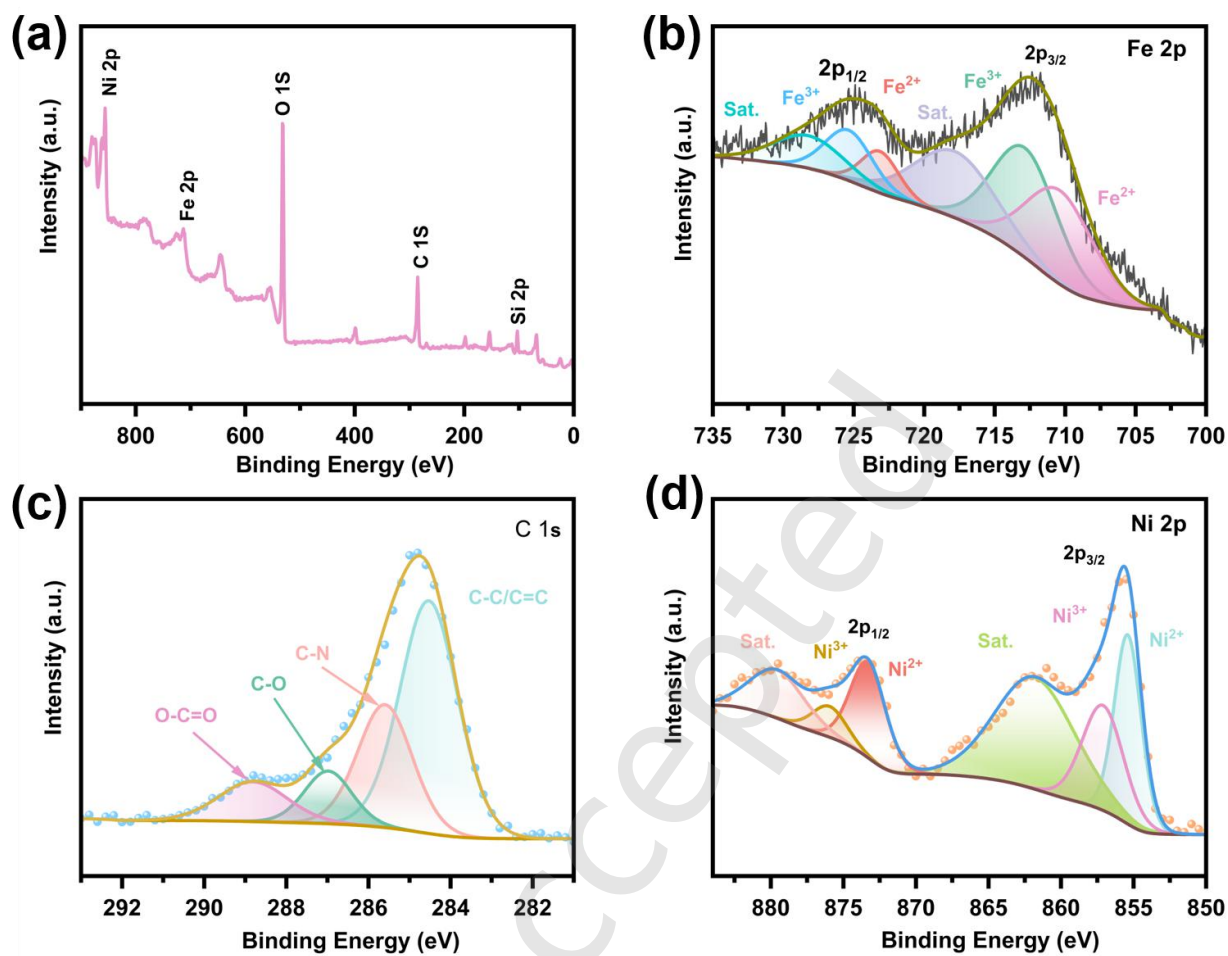


Fig. S12. XPS CFNOS-2.

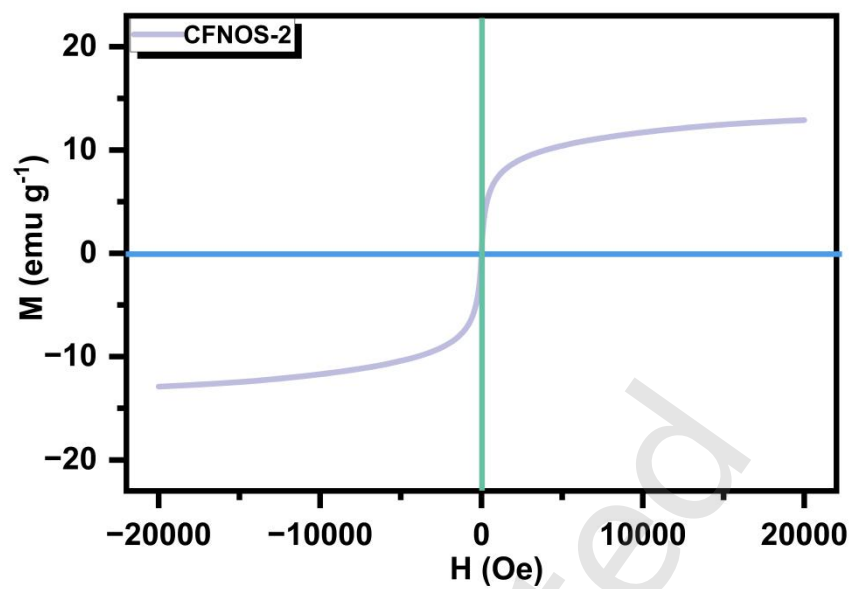


Fig. S13. The hysteresis loop of CFNOS-2 nanocomposite material at room temperature.

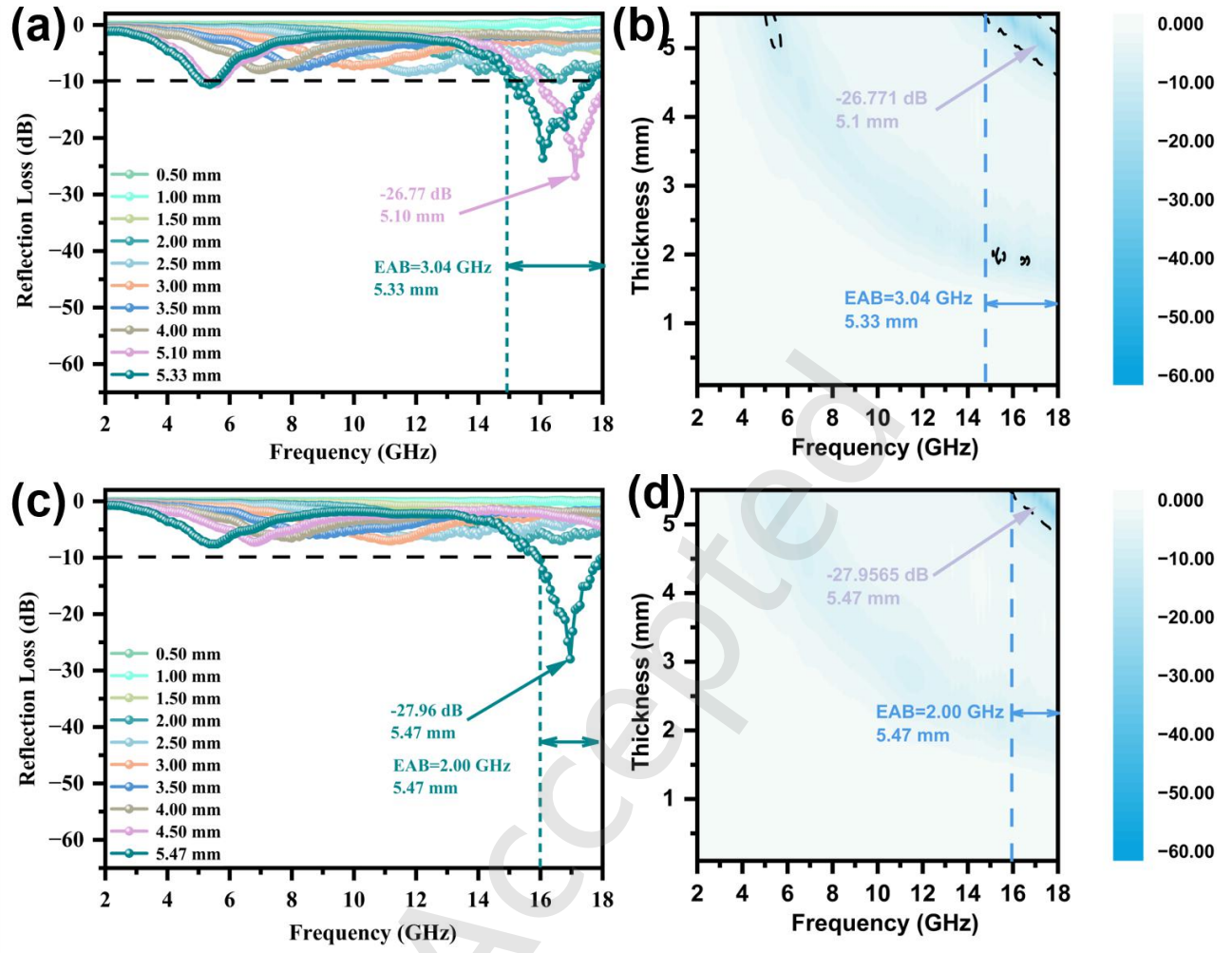


Fig. S14. The 2D RL value curves versus frequency and Contour plot of the relationship between RL values and frequency of a-b) CFNOS-1, c-d) CFNOS-2 sample.

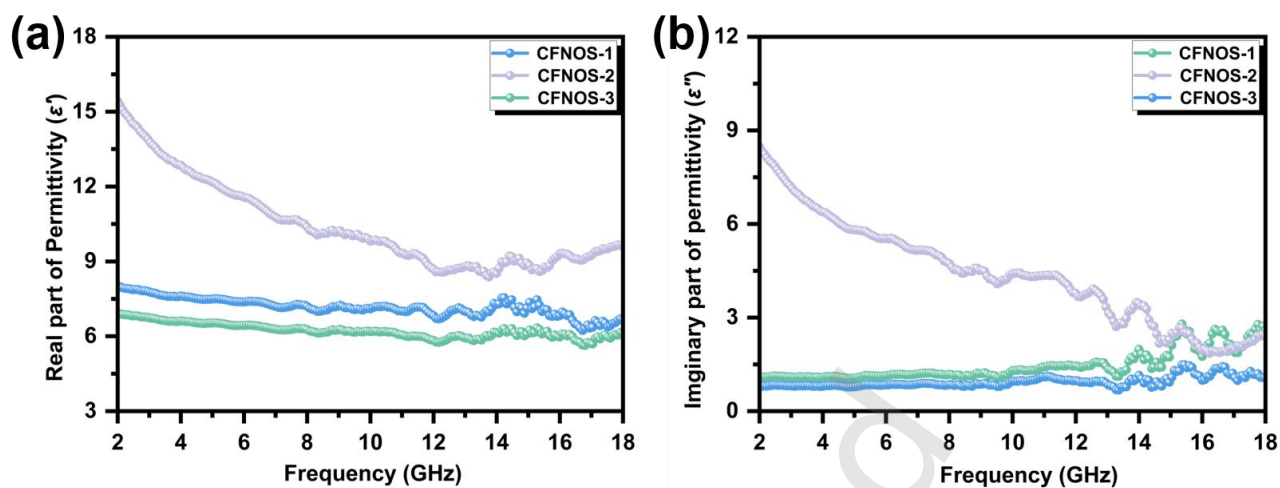


Fig. S15. a) Dielectric real part, b) Dielectric imaginary part.

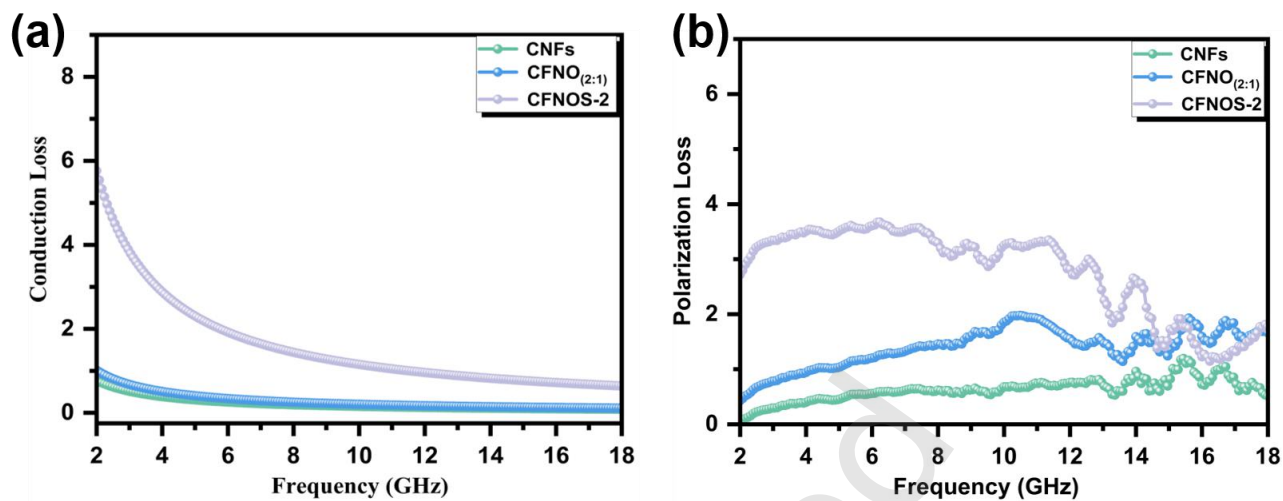


Fig. S16. a) Conduction Loss, b) Polarization Loss.

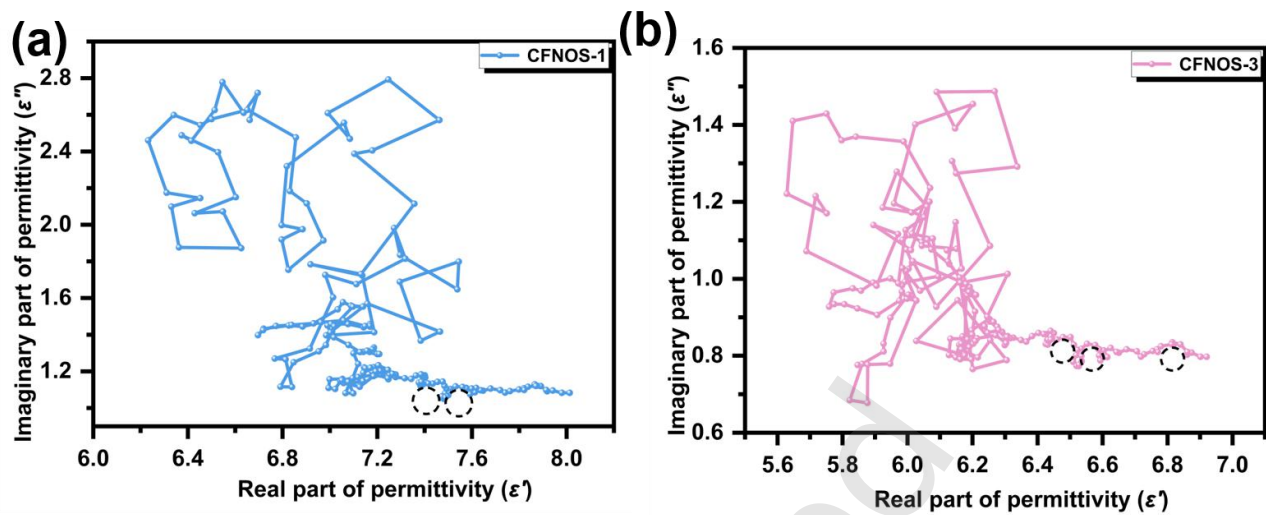


Fig. S17. Cole-Cole curve of a) CFNOS-1, b) CFNOS-3.

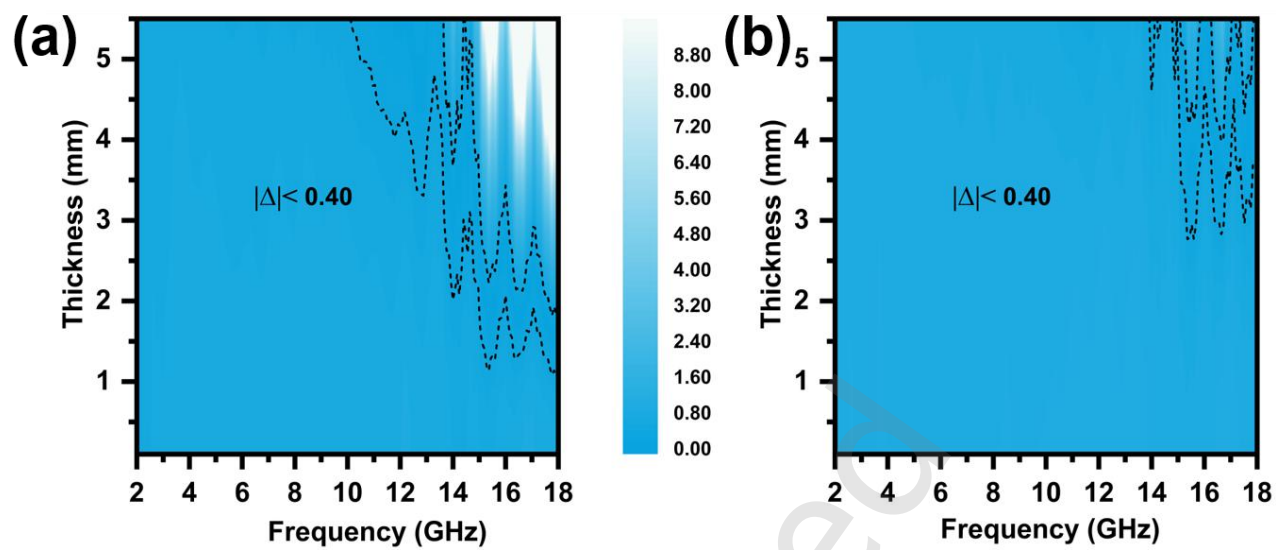


Fig. S18. $|\Delta|$ of a) CFNOS-1, b) CFNOS-3.

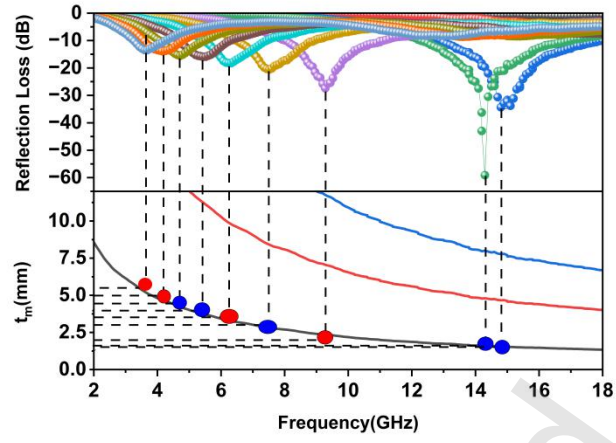


Fig. S19 One-quarter wavelength model for CFNOS-2 sample

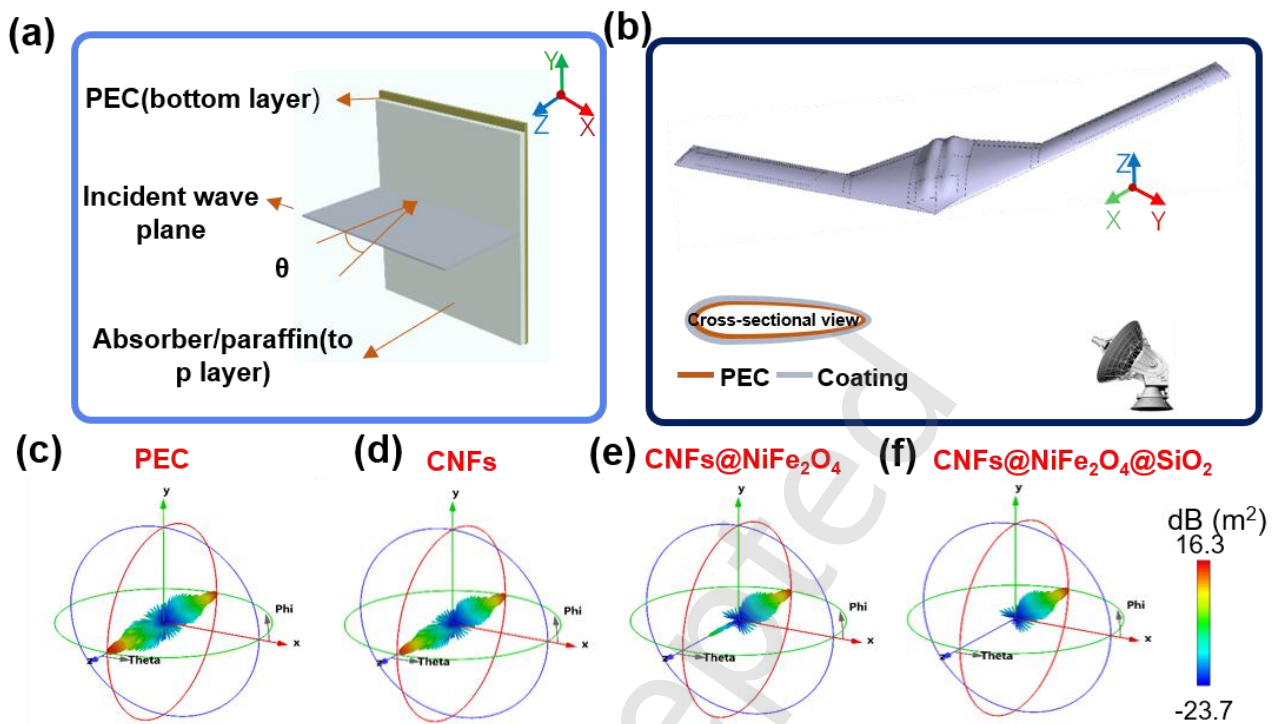


Fig. S20. (a) PEC covering material; (b) Stealth fighter aircraft with the PEC covering material and the ultra-wideband electromagnetic absorption element structure. (c) PEC; (d) CNFs; (e) CNFs@NiFe₂O₄; (f) The far-field simulation results of the CST of the PEC covered with CNFs@NiFe₂O₄@SiO₂.

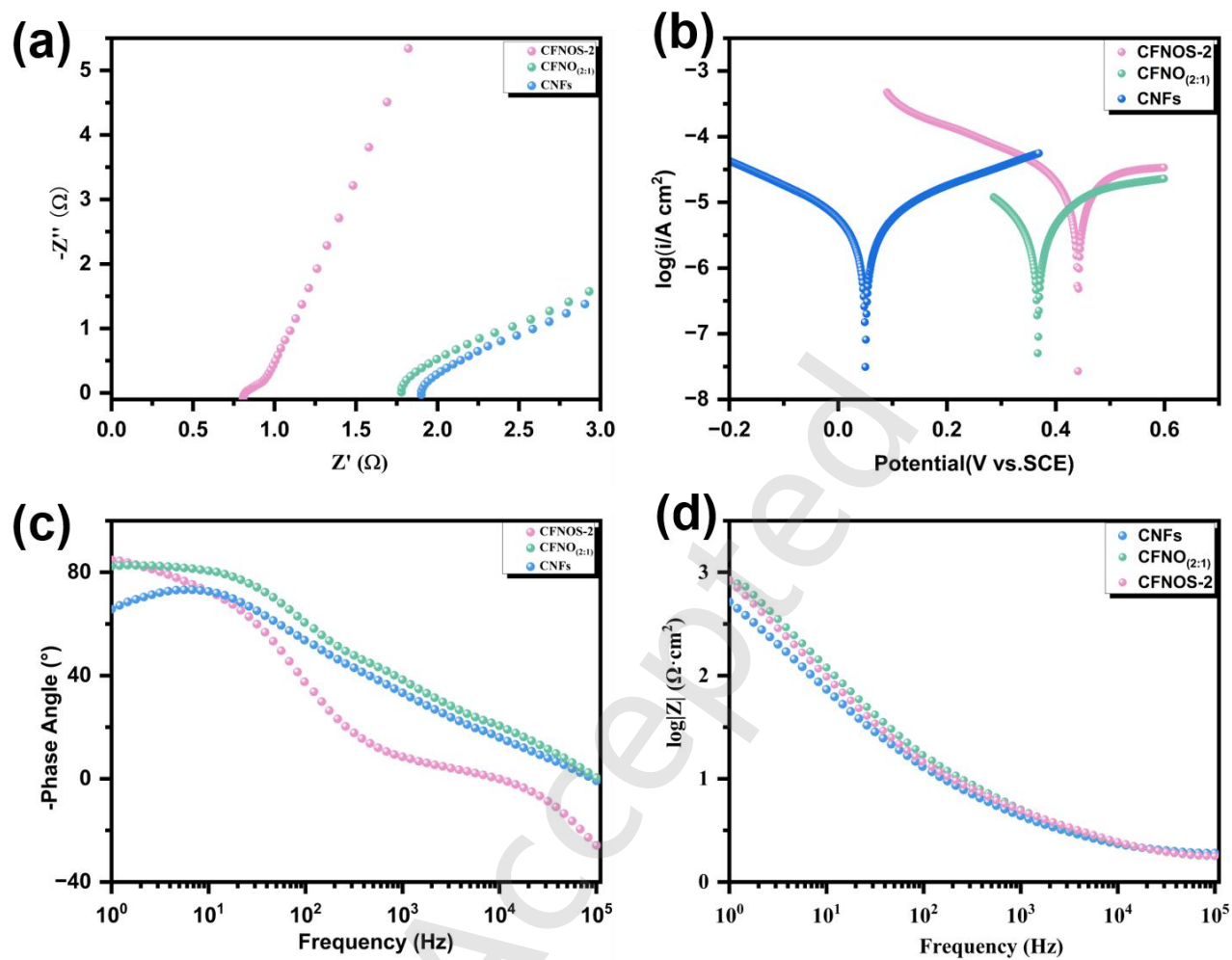


Fig. S21. Electrochemical characterization of CNFs, CFNO_(2:1) and CFNOS-2 under acidic conditions.

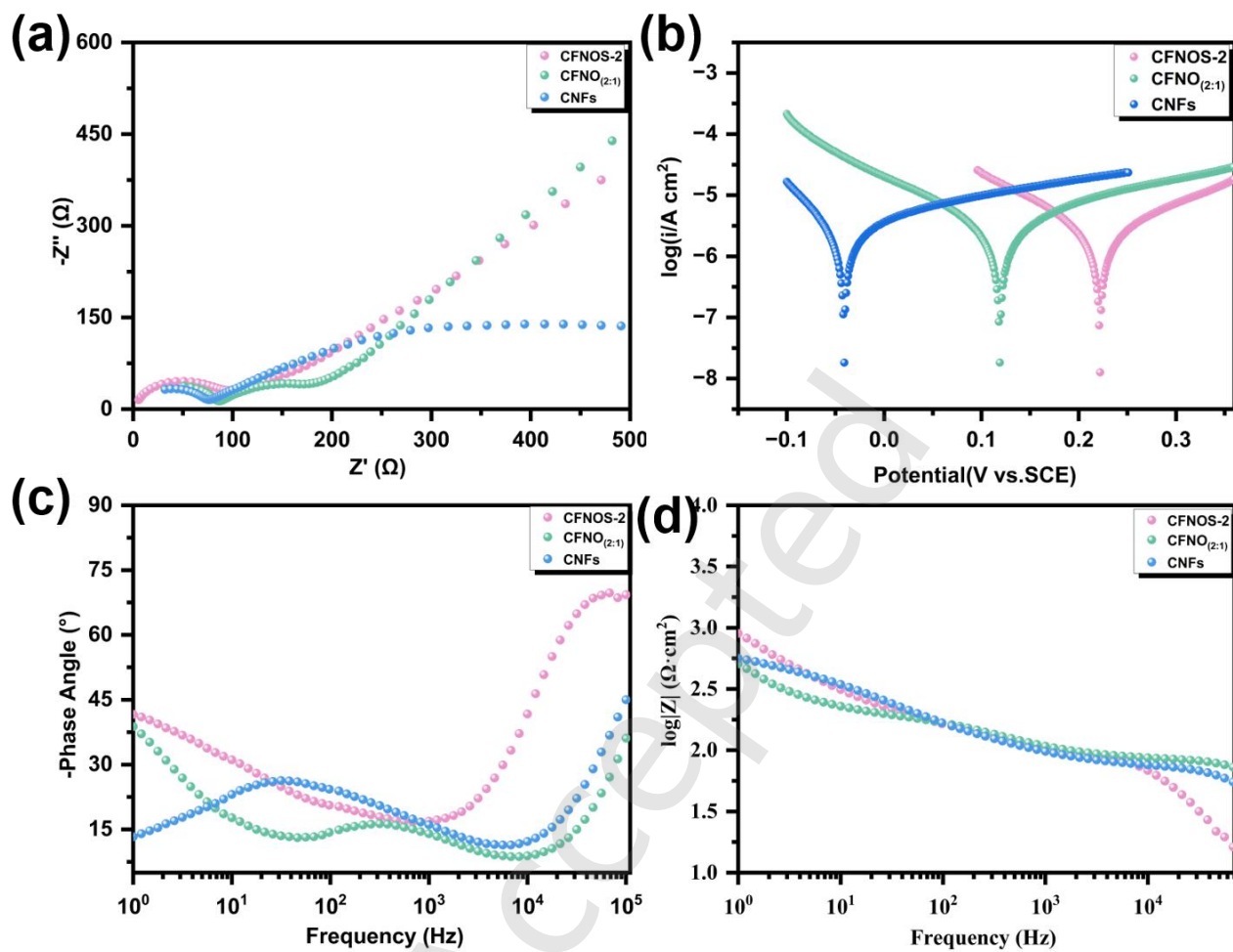


Fig. S22. Electrochemical characterization of CNFs, CFNO_(2:1) and CFNOS-2 under alkaline conditions.

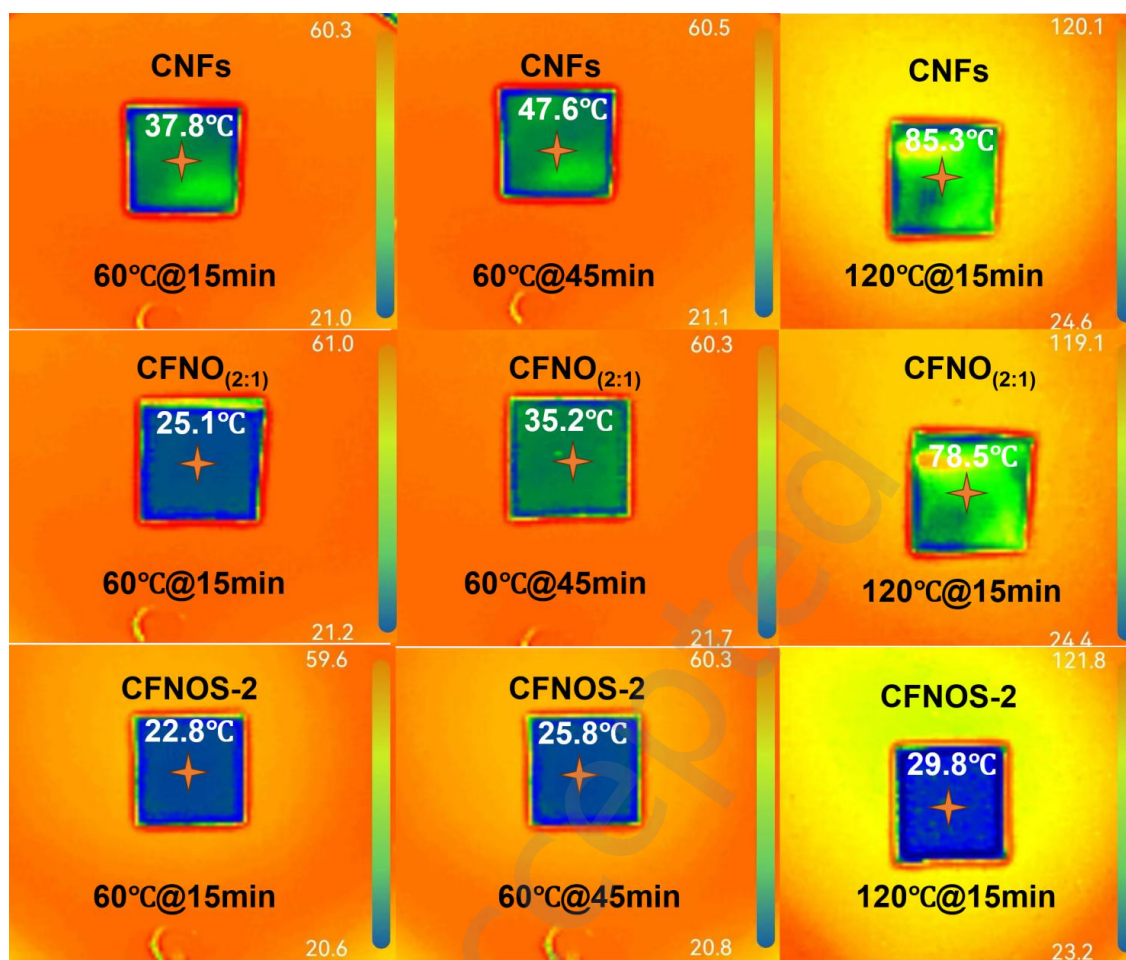


Fig. S23. infrared thermal images.