

# Hierarchical engineering of MOF-on-MOF derived aerogels toward synergistic microwave absorption and multifunctional protection

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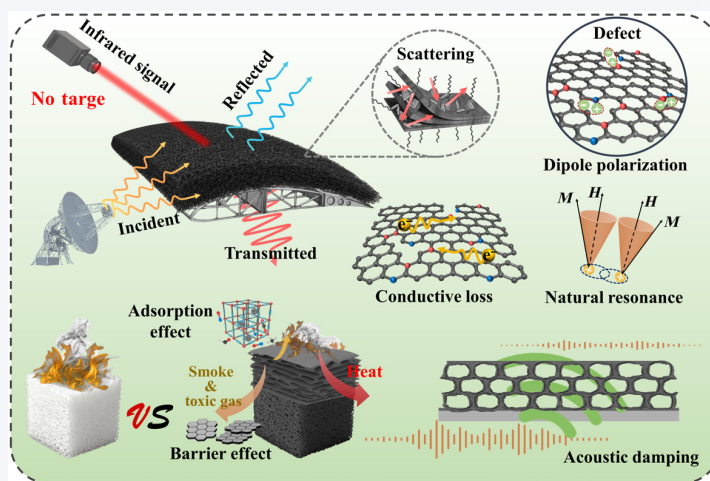
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**ABSTRACT:** Constructing multifunctional aerogels that simultaneously integrate electromagnetic microwave (EMW) absorption, flame retardancy, acoustic damping, and thermal protection remains a formidable challenge due to inherent trade-offs in structural design and compositional synergy. Herein, we propose a hierarchical assembly and controlled carbonization strategy to fabricate MXene-reinforced metal-organic framework (MOF)-on-MOF derived carbon aerogels ( $Z@F_xN_y-M/CA$ ), wherein the  $Fe^{3+}/Ni^{2+}$  molar ratio is precisely tuned to tailor the microstructure, defect chemistry, and interfacial characteristics. This design enables a unique synergistic interplay between a conductive MXene network, defect-rich carbon frameworks, and Fe/Co/Ni-derived magnetic components, collectively realizing efficient EMW attenuation via coupled conduction loss, polarization relaxation, and magnetic resonance. Remarkably, the optimized aerogel achieves an outstanding minimum reflection loss ( $RL_{min}$ ) of  $-60.36$  dB and a broad effective absorption bandwidth (EAB) of 5.06 GHz, outperforming most state-of-the-art absorbers. Beyond EMW absorption, the aerogel exhibits exceptional fire safety, with over 50% reduction in peak heat release rate (pHRR) and total heat release (THR), along with suppressed smoke emission and the formation of a dense graphitized char layer. Furthermore, it delivers superior acoustic damping with a noise reduction coefficient (NRC) of 0.66 and efficient thermal management capability. Such integrated multifunctionality is intrinsically linked to the finely engineered pore architecture, abundant heterogeneous interfaces, and compositionally modulated Fe/Co/Ni-derived phases. This work presents a paradigm-shifting MOF-on-MOF strategy for designing next-generation lightweight aerogels that harmoniously integrate EMW absorption, flame retardancy, thermal insulation, and acoustic protection, offering new insights into structure–property relationships in multimetal-derived multifunctional materials.

**KEYWORDS:** metal-organic framework (MOF)-on-MOF, aerogel, microwave absorption, flame retardant, multifunctional integration



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## 1 Introduction

With the rapid development of modern electronic systems and highly integrated electromagnetic devices, electromagnetic radiation pollution and interference have become increasingly severe, threatening information security, the reliable operation of precision instruments, and human health [1–4]. Meanwhile, in aerospace, national defense, and building engineering, materials are also required to simultaneously address fire hazards, noise pollution, and stringent thermal protection demands [5–9]. These service environments are typically characterized by multiphysical-field coupling and extreme working conditions, rendering conventional single-function protective materials inadequate. Consequently, the development of lightweight multifunctional materials integrating efficient electromagnetic microwave (EMW) absorption, flame retardancy, acoustic damping, and thermal protection has emerged as a critical challenge in materials science.

Aerogels, featuring ultralow density, high specific surface area, and three-dimensional (3D) porous architectures, offer intrinsic advantages for EMW absorption, thermal management, and sound attenuation [10–13]. In particular, carbon aerogels have been extensively explored as EMW absorbers due to their good electrical conductivity and tunable dielectric properties. Li et al. [14] realized reversible tuning of EMW absorption frequency and switching between absorption and transmission modes based on the controllable compression of chitosan-derived elastic carbon aerogels, achieving a minimum reflection loss ( $RL_{\min}$ ) of approximately  $-40$  dB. Xie et al. [15] reported a melamine-templated multidimensional carbon foam that simultaneously exhibited excellent EMW absorption performance (with an  $RL_{\min}$  of  $-56.02$  dB) and infrared stealth capability. On the other hand, to overcome the insufficient mechanical performance of carbon aerogels, Li et al. [16] constructed lightweight yet robust carbon aerogels by introducing biomass-derived magnetic Ni/C nanoparticles and aramid nanofibers, which maintained ultralow density and excellent elasticity while achieving strong EMW absorption of  $-49.5$  dB and favorable infrared thermal camouflage. Despite these advances in tunable electromagnetic responses, multidimensional stealth, and mechanical reinforcement, existing carbon aerogel systems still largely rely on single or limited loss mechanisms. Their impedance matching regulation, flame-retardant safety, and multiphysical-field synergistic protection capabilities remain inadequate, necessitating further breakthroughs through multicomponent and multiscale structural design.

In recent years, introducing heterogeneous functional components, such as magnetic nanoparticles, two-dimensional (2D) conductive materials, and porous coordination polymers, has been recognized as an effective route to enhance aerogel performance [17–20]. Among these, metal-organic frameworks (MOFs) are particularly attractive precursors owing to their highly designable metal nodes and organic ligands, which can be transformed into metal/metal oxide-carbon composites after carbonization [21]. Wang et al. [22] constructed highly porous Ni/C@rGO composite aerogels through graphene oxide (GO) nanosheet-mediated structural evolution of Ni-MOF, achieving broadband EMW absorption (5.2 GHz) and excellent thermal management performance while maintaining ultralow density and high porosity. Zhou et al. [23] combined *in situ* growth of zeolitic imidazolate framework-67 (ZIF-67) with a carbon aerogel skeleton to fabricate Co/N-doped carbon aerogels with hierarchical pore structures, which achieved  $-54.0$  dB EMW absorption and good

thermal insulation performance. Notably, our group has recently deposited MOFs onto the surface of black phosphorus nanosheets (BPNs) via an electrostatic self-assembly strategy and integrated them with chitosan aerogels, successfully fabricating a lightweight carbon aerogel. The resulting material exhibits outstanding EMW absorption performance together with thermal insulation and fire-resistant properties [24]. However, the performance of these systems is still constrained by the intrinsic limitations of single-MOF precursors. Single-metal or single-topology MOFs often yield relatively simple composite phases after pyrolysis, making it difficult to simultaneously construct continuous conductive networks, abundant heterogeneous interfaces, and efficient catalytic sites, thereby limiting dielectric-magnetic loss matching and synergistic multifunctional enhancement.

To overcome these limitations, the MOF-on-MOF strategy has been proposed to construct composite precursors with core-shell or multilayer heterogeneous interfaces by *in situ* growing a secondary MOFs on a parent MOFs [25, 26]. This approach enables precise regulation of composition distribution, interface structure, and defect characteristics at the microscopic scale, and has recently demonstrated remarkable advantages in multifunctional materials design [27, 28]. The  $NH_2$ -UiO-66@ $NH_2$ -MIL-125 structure constructed by Tang et al. [29], simultaneously enhanced mechanical properties and flame-retardant/smoke-suppression performance in polymer matrices. Liu et al. [30] realized synergistic regulation of lattice strain, defect states, and electrical conductivity by constructing so-called “Matryoshka”-like multilayer MOF-on-MOF heterostructures, significantly improving impedance matching and enhancing EMW absorption performance. Furthermore, Wu et al. [31] assembled and derived magnetic porous carbon materials using multi-level MOF-on-MOF assembly, achieving extremely low reflection loss while reducing the matching thickness, which further verified the unique advantages of MOF-on-MOF structures in the design of high-performance EMW absorbing materials. In addition, MXene is introduced as a highly conductive two-dimensional component to regulate dielectric properties, construct heterogeneous interfaces, and improve impedance matching, thereby contributing to enhanced electromagnetic wave absorption.

Based on these considerations, this work proposes a materials design concept of “hierarchical assembly and controlled pyrolysis”. Dopamine-modified melamine foam (MF) was employed as a three-dimensional elastic template, onto which MXene sheets and ZIF-67@ $NH_2$ -MIL-88B( $Fe_xNi_y$ ) MOF-on-MOF heterostructures with tunable  $Fe^{3+}/Ni^{2+}$  ratios were sequentially assembled, followed by controlled carbonization to construct a series of multifunctional aerogels ( $Z@F_xN_y$ -M/CA). As a result, the aerogels exhibit ultralightweight characteristics together with strong EMW absorption ( $RL_{\min}$  of  $-60.36$  dB), significantly enhanced flame-retardant performance (over 50% reduction in peak heat release rate), excellent broadband sound absorption (noise reduction coefficient up to 0.66), and effective thermal management capability. This work provides a viable strategy for constructing high-performance multifunctional protective materials and deepens the understanding of structure–composition–property relationships in multimetal MOF-on-MOF-derived systems.

## 2 Experimental

Detailed information was described in the Electronic Supplementary Material (ESM).

### 3 Results and discussion

#### 3.1 Synthesis and characterization

As shown in Fig. 1,  $Z@F_xN_y$ -M/MF composite aerogels were fabricated via a hierarchical assembly and interfacial regulation strategy (details in the ESM). MXene was first obtained by LiF/HCl etching of  $Ti_3AlC_2$  and subsequently functionalized with cetyltrimethylammonium bromide (CTAB), which reduced intersheet electrostatic repulsion and improved dispersion stability in alcoholic media, enabling uniform incorporation into the MF skeleton. Polydopamine (PDA) modification introduced catechol and amine groups onto the MF surface, enhancing MXene adhesion and providing active sites for subsequent *in situ* MOFs growth. Thereafter,  $NH_2$ -MIL-88B( $Fe_xNi_y$ ) and ZIF-derived phases were synergistically grown on the foam framework and MXene surfaces. Mild carbonization transformed MF into a carbon aerogel (CA), yielding a tightly coupled hierarchical structure consisting of conductive 2D nanosheets, multimetal MOF-derived phases, and a 3D porous carbon skeleton.

Scanning electron microscopy (SEM) images in Figs. 2(a)–2(c) show  $NH_2$ -MIL-88B( $Fe_xNi_y$ ) synthesized with different  $Fe^{3+}/Ni^{2+}$  ratios. All samples exhibit typical rod-like or spindle-shaped morphologies, indicating that bimetallic incorporation does not alter the MIL-88B crystal framework. Energy-dispersive X-ray spectroscopy (EDX) mapping of  $NH_2$ -MIL-88B( $Fe_xNi_y$ ) (Fig. S2 in the ESM) reveals homogeneous distributions of C, N, Fe, and Ni, confirming uniform bimetal coordination. Varying the  $Fe^{3+}/Ni^{2+}$  ratio markedly influences crystal size and surface morphology: the Ni-rich sample ( $Fe_1Ni_3$ , Fig. 2(b)) shows blunted edges and rough surfaces, whereas the Fe-rich sample ( $Fe_3Ni_1$ , Fig. 2(c)) displays more regular and elongated crystals. These results demonstrate that the  $Fe^{3+}/Ni^{2+}$  ratio effectively regulates crystal growth kinetics, providing a structural basis for subsequent derivation and performance optimization.

SEM comparison of pristine MXene and CTAB@MXene (Fig. S3 in the ESM) shows that CTAB modification converts MXene from a densely stacked structure to a more loosely packed morphology with enlarged interlayer spacing, facilitating uniform anchoring and

interfacial coupling of MOF-derived phases and enabling multi-interface synergistic architectures. SEM images of  $Z@F_xN_y$ -M ( $MXene$  loaded with  $ZIF-67@NH_2$ -MIL-88B( $Fe_xNi_y$ )) powders (Figs. 2(d)–2(f)) reveal that rod-like  $NH_2$ -MIL-88B crystals are uniformly decorated with abundant nanoscale particles or shell-like structures, confirming the successful loading and tight coupling of ZIF-derived phases. The incorporation of MXene not only provides a conductive platform for the growth of  $Z@F_xN_y$  particles but also facilitates the formation of a continuous electron transport network. The intimate contact between MXene sheets and MOF-derived components generates abundant heterogeneous interfaces, which are beneficial for interfacial polarization. The  $Fe^{3+}/Ni^{2+}$  ratio markedly influences particle distribution and surface morphology:  $Z@F_1N_3$ -M exhibits finer and more homogeneous particles, whereas Fe-rich  $Z@F_3N_1$ -M shows denser agglomeration, likely related to metal ion migration and rearrangement during coordination and thermal evolution.

To further verify the MOF-on-MOF heterostructure design, transmission electron microscopy (TEM) and EDX elemental mapping were performed on the representative  $Z@F_1N_1$ -M intermediate. As shown in Fig. S5 in the ESM, the coexistence of spindle-like  $NH_2$ -MIL-88B and polyhedral ZIF-67 crystals can be clearly distinguished. Notably, the ZIF-67 nanoparticles are tightly anchored on the surface of  $NH_2$ -MIL-88B, and a clear interfacial contact can be observed, indicating *in situ* growth rather than simple physical mixing. In addition, the EDX elemental mapping (Fig. S6 in the ESM) shows that Co is mainly distributed in the polyhedral regions, while Fe and Ni are concentrated in the spindle-like framework, confirming the spatial separation and interaction of the two MOF components. These results unequivocally confirm the formation of a well-defined ZIF-67@ $NH_2$ -MIL-88B MOF-on-MOF heterojunction, providing a robust structural foundation for the subsequent controlled carbonization and the resulting multifunctional properties.

As shown in Figs. 2(g)–2(l), carbonized  $Z@F_xN_y$ -M/CA aerogels exhibit a hierarchical multiscale porous structure. Low-magnification SEM images (Figs. 2(g), 2(i), and 2(k)) demonstrate that all samples retain the 3D interconnected MF framework, forming open macroporous networks (hundreds of micrometers)

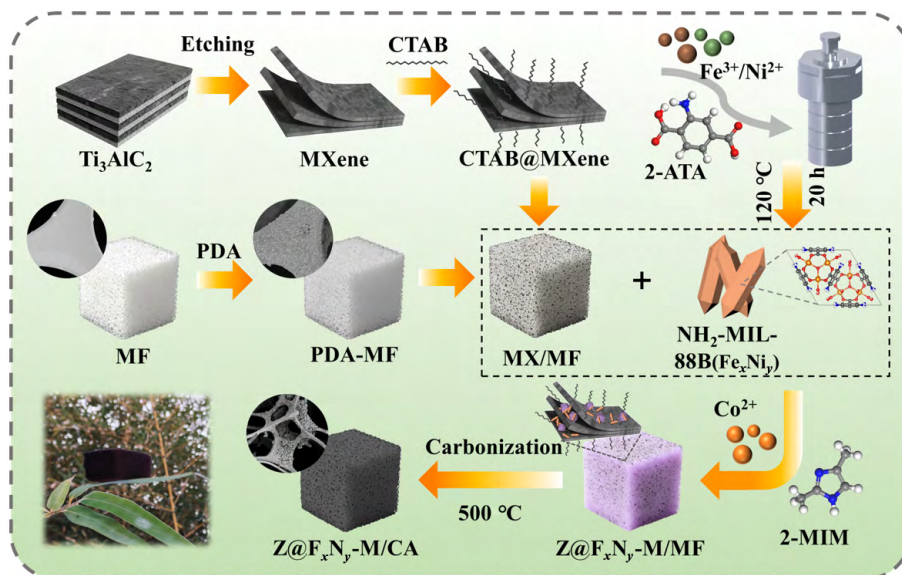


**Figure 1** Schematic diagram of the synthesis process of  $Z@F_xN_y$ -M/MF composite aerogel.

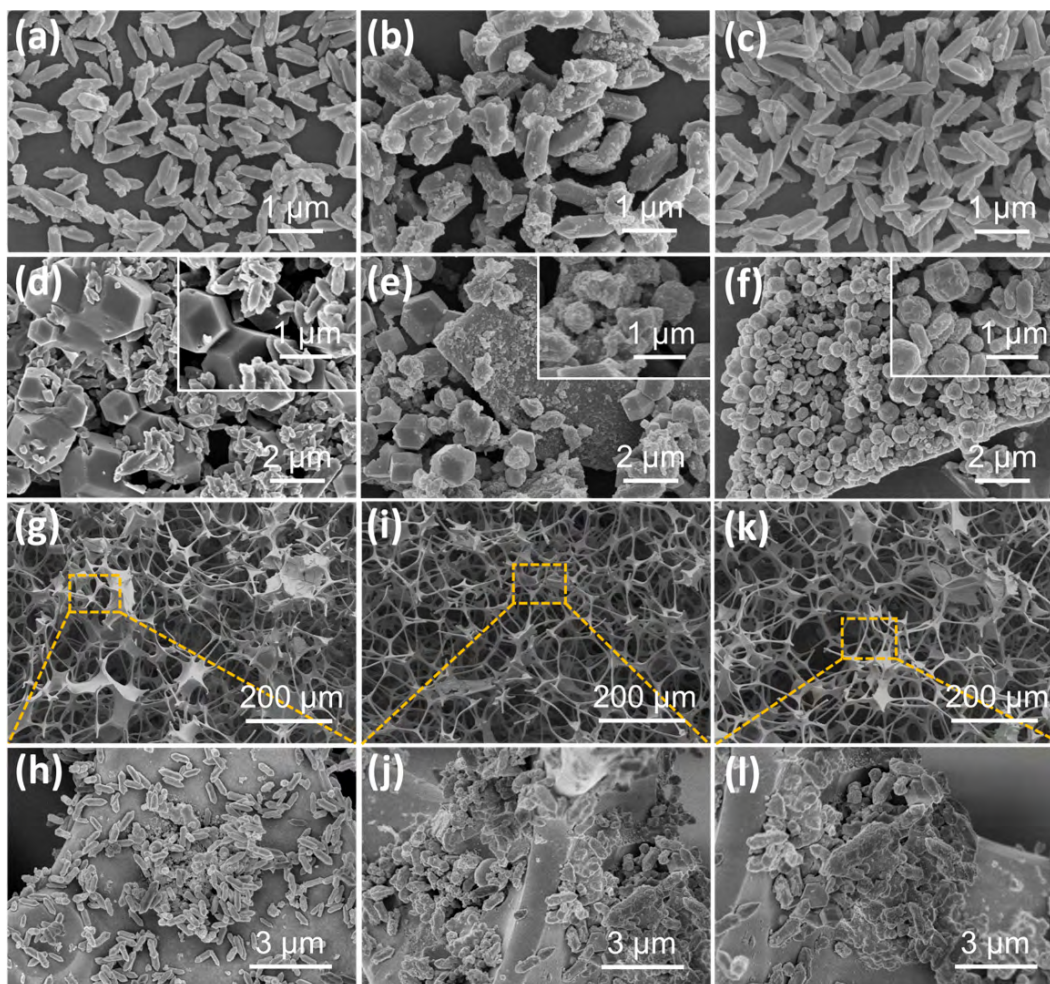


**Figure 2** SEM images of (a)  $\text{NH}_2\text{-MIL-88B(Fe}_3\text{Ni)}$ , (b)  $\text{NH}_2\text{-MIL-88B(Fe}_3\text{Ni)}$ , (c)  $\text{NH}_2\text{-MIL-88B(Fe}_3\text{Ni)}$ , (d)  $\text{Z@F}_1\text{N}_1\text{-M}$ , (e)  $\text{Z@F}_1\text{N}_1\text{-M}$ , (f)  $\text{Z@F}_1\text{N}_1\text{-M}$ , (g) and (h)  $\text{Z@F}_1\text{N}_1\text{-M/CA}$ , (i) and (j)  $\text{Z@F}_1\text{N}_1\text{-M/CA}$ , and (k) and (l)  $\text{Z@F}_1\text{N}_1\text{-M/CA}$ .

favorable for heat, gas, and EMW transport as well as multiple scattering. High-magnification images (Figs. 2(h), 2(j), and 2(l)) further reveal uniform decoration of the carbon skeleton with MOF-derived nanoparticles intimately coupled with MXene nanosheets. Consistent with Fig. S4 in the ESM, PDA modification roughens the MF surface and provides abundant anchoring sites, enabling uniform inorganic phase deposition in M/MF (MXene-modified melamine foam) and validating the hierarchical interfacial construction strategy. This “micrometer-scale skeleton-nanometer-scale active phase” architecture increases interfacial polarization sites and underpins synergistic electromagnetic loss, catalytic charring, and acoustic dissipation.

The X-ray diffraction (XRD) pattern is shown in Fig. 3(a), samples with different  $\text{Fe}^{3+}/\text{Ni}^{2+}$  ratios exhibit distinct structural evolution before and after carbonization.  $\text{NH}_2\text{-MIL-88B(Fe}_x\text{Ni}_y)$  displays typical MIL-88B diffraction peaks in the low-angle region, indicating that varying the  $\text{Fe}^{3+}/\text{Ni}^{2+}$  feed ratio does not alter the MOF crystalline phase, while the absence of impurity peaks confirms successful  $\text{Ni}^{2+}$  incorporation. In  $\text{Z@F}_x\text{N}_y\text{-M}$  composites, characteristic reflections of MXene, ZIF-67, and  $\text{NH}_2\text{-MIL-88B}$  are simultaneously observed, demonstrating effective integration of all components. After carbonization, the ordered MOFs peaks nearly vanish, accompanied by a broad diffraction band at  $2\theta \approx 24^\circ$  assigned to amorphous or poorly graphitized carbon [32]. Meanwhile, broadened peaks at  $\sim 44.3^\circ$ ,  $51.6^\circ$ , and  $75.8^\circ$  correspond

to the (111), (200), and (220) planes of a face-centered cubic (fcc) FeCoNi alloy, indicating partial reduction and reconstruction of multimetal species, which provides a solid-phase basis for magnetic loss and EMW absorption.

Thermogravimetric analysis (TGA, Fig. 3(b)) shows that  $\text{Z@F}_x\text{N}_y\text{-M/CA}$  exhibits higher residual mass and slower weight loss than pristine  $\text{NH}_2\text{-MIL-88B}$ , reflecting enhanced thermal stability arising from the synergistic barrier effect of MXene, MOF-derived phases, and the carbon framework. Notably, the sample with an  $\text{Fe}^{3+}/\text{Ni}^{2+}$  ratio close to 1:1 delivers the highest char yield above  $600^\circ\text{C}$ , suggesting a stronger tendency to form a stable inorganic-carbon protective layer and thus improved flame-retardant performance.

Figures 3(c)–3(g) show the X-ray photoelectron spectroscopy (XPS) results of  $\text{Z@F}_x\text{N}_y\text{-M/CA}$  aerogels. The survey spectrum (Fig. 3(c)) confirms the presence of C, N, O, Fe, Co, and Ni. The C 1s spectrum (Fig. 3(d)) displays peaks for C–C/C=C, C–N/C–O, C=O, and O–C=O, with dominant C–C/C=C indicating graphitized  $\text{sp}^2$  carbon for conductive loss [33]. Fe 2p shows  $\text{Fe}^{3+}$  peaks (712.1 and 720.2 eV) and minor  $\text{Fe}^0$  signals (707.2 and 717.7 eV) [34]. Co 2p confirms metallic Co<sup>0</sup> (778.3 and 793.4 eV) and a small fraction of Co<sup>2+</sup> (780.2 and 796.4 eV), while Ni 2p indicates Ni<sup>0</sup> (854.4 and 870.0 eV) and Ni<sup>2+</sup> species (856.0 and 873.8 eV) [35]. These results indicate that Fe, Co, and Ni mainly exist in metallic states, jointly forming FeCoNi alloys, while partial surface oxidation generates thin metal oxide shells. This

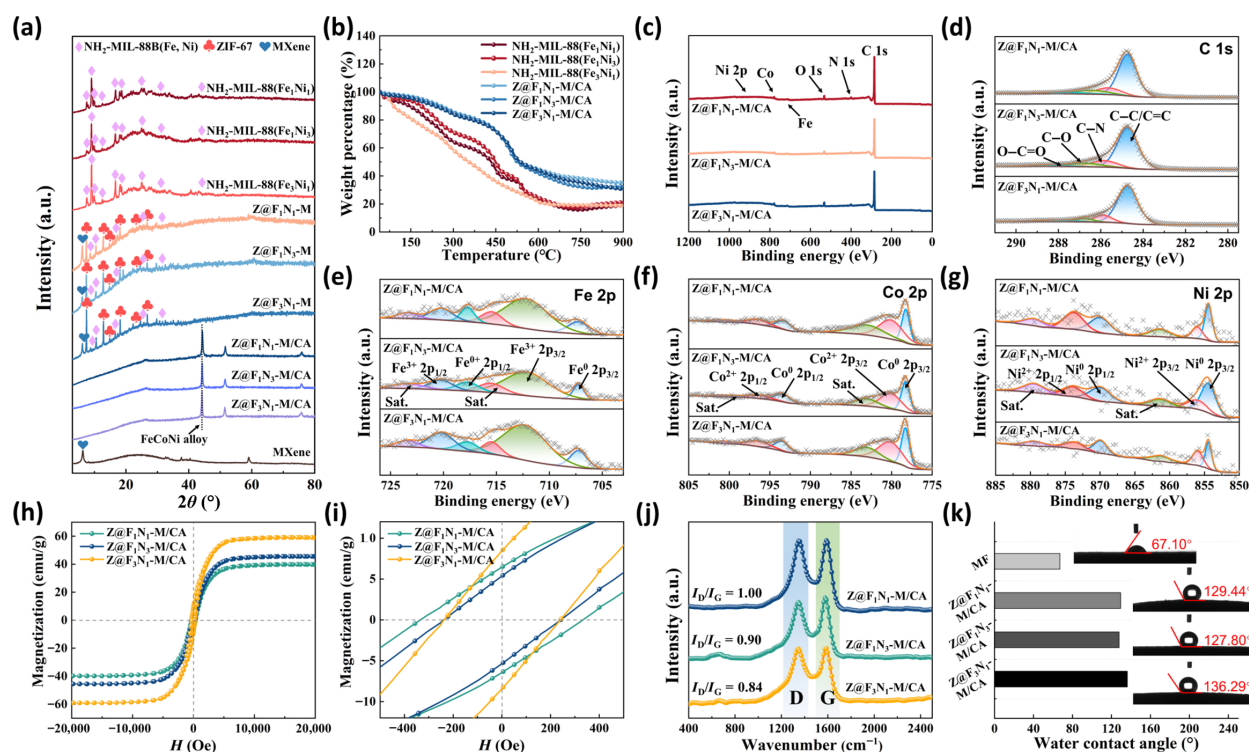


**Figure 3** (a) XRD spectra; (b) TGA; XPS spectra of Z@F<sub>x</sub>N<sub>y</sub>-M/CA: (c) survey scan, (d) C 1s, (e) Fe 2p, (f) Co 2p, and (g) Ni 2p; (h) and (i) vibrating sample magnetometer (VSM), (j) Raman spectra, and (k) contact angle of Z@F<sub>x</sub>N<sub>y</sub>-M/CA.

heterogeneous “alloy core@oxide shell” structure, together with defect-rich carbon matrices, provides multiple interfacial and polarization loss pathways, accounting for the enhanced EMW absorption performance.

As shown in Figs. 3(h) and 3(i), all Z@F<sub>x</sub>N<sub>y</sub>-M/CA aerogel samples exhibit typical ferromagnetic hysteresis behavior at room temperature, characterized by well-defined hysteresis loops and saturation magnetization ( $M_s$ ), indicating the successful *in situ* generation of magnetic components during the carbonization process. From the overall magnetization curves (Fig. 3(h)), it can be observed that the saturation magnetization gradually increases with increasing Fe content. Among the samples, Z@F<sub>3</sub>N<sub>1</sub>-M/CA shows the highest  $M_s$  value (59.0 emu/g). In the enlarged low-field region (Fig. 3(i)), all three samples display small coercivity ( $H_c$ ) and remanent magnetization ( $M_r$ ), indicative of typical soft magnetic characteristics. Such soft magnetic behavior is beneficial for rapid magnetization–demagnetization processes under alternating electromagnetic fields, thereby enhancing magnetic loss mechanisms such as natural resonance and exchange resonance.

Figure 3(j) presents the Raman spectra of the Z@F<sub>x</sub>N<sub>y</sub>-M/CA aerogels. All samples exhibit the characteristic D band and G band located at approximately 1350 and 1580 cm<sup>-1</sup>, corresponding to disordered carbon or defect structures and sp<sup>2</sup>-hybridized graphitic carbon structures, respectively [36]. This indicates that the carbonized materials are composed of both defective carbon and graphitized carbon, providing a structural basis for electromagnetic loss and interfacial polarization. Notably, the  $I_D/I_G$  ratios vary significantly among samples with different Fe<sup>3+</sup>/Ni<sup>2+</sup> ratios: Z@F<sub>1</sub>N<sub>1</sub>-M/CA shows an  $I_D/I_G$  of 1.00, Z@F<sub>1</sub>N<sub>3</sub>-M/CA exhibits an  $I_D/I_G$  of 0.90, while Z@F<sub>3</sub>N<sub>1</sub>-M/CA presents the lowest value of 0.84. A higher  $I_D/I_G$  ratio implies a higher defect density and more edge sites, which are favorable for introducing dipolar polarization centers and enhancing interfacial polarization loss. In contrast, a

lower  $I_D/I_G$  ratio suggests a higher degree of structural order and graphitization, which contributes to improved electrical conductivity and enhanced conductive loss capability.

To quantitatively evaluate the structural robustness and elastic recovery of the aerogels, cyclic compression tests were performed at a maximum strain of 70% for 20 cycles (Fig. S6 in the ESM). As shown in Fig. S7 and summarized in the inset table in the ESM, all Z@F<sub>x</sub>N<sub>y</sub>-M/CA aerogels exhibit significantly enhanced compressive strength compared to pristine MF. At 70% strain, the maximum compressive stress reaches 0.0095, 0.0068, and 0.0067 MPa for Z@F<sub>1</sub>N<sub>1</sub>-M/CA, Z@F<sub>1</sub>N<sub>3</sub>-M/CA, and Z@F<sub>3</sub>N<sub>1</sub>-M/CA, respectively, representing a 2 to 3-fold enhancement over pure MF (0.0028 MPa). This reinforcement is attributed to the stiff MXene nanosheets and the rigid MOF-derived carbon coating that strengthen the cell walls against deformation. Notably, all samples display excellent elastic recovery with minimal plastic deformation after 20 cycles, retaining over 85% of the initial maximum stress. The closed hysteresis loops observed are typical of viscoelastic energy dissipation within the carbon skeleton. These results confirm that the hierarchical coating strategy not only preserves the intrinsic elasticity of the MF-derived carbon framework but also imparts enhanced mechanical robustness, ensuring the structural integrity required for practical multifunctional applications.

Contact angle measurements (Fig. 3(k)) demonstrate pronounced hydrophobicity of the carbonized aerogels, with static water contact angle of 129.44°, 127.80°, and 136.29° for Z@F<sub>1</sub>N<sub>1</sub>-M/CA, Z@F<sub>1</sub>N<sub>3</sub>-M/CA, and Z@F<sub>3</sub>N<sub>1</sub>-M/CA, respectively, compared with 67.10° for pristine MF. This transition arises from the formation of low-surface-energy carbon and hierarchical micro/nanoscale roughness generated by MOF-derived porous carbon and MXene nanosheets, which stabilizes the Cassie–Baxter state by trapping air and reducing solid–liquid contact.

### 3.2 Electromagnetic performance and radar cross-section (RCS) simulation results

As shown in Figs. 4(a1)–4(a3), pristine MF exhibits negligible EMW absorption over 2–18 GHz, with RL values remaining above –10 dB at all thicknesses due to its low conductivity, lack of magnetic response, and single dielectric loss mechanism [37]. In contrast,  $Z@F_xN_y$ -M/CA aerogels show markedly enhanced EMW absorption across the entire frequency range (Figs. 4(b1)–4(d3)). Notably,  $Z@F_1N_1$ -M/CA delivers continuous strong absorption over a wide thickness range, achieving an outstanding  $RL_{\min}$  of –60.36 dB and a broad effective absorption bandwidth (EAB, 5.06 GHz at 2.1 mm) even at small thicknesses, demonstrating a typical “thin-wide-strong” absorption behavior [38].  $Z@F_1N_3$ -M/CA ( $RL_{\min}$  = –48.19 dB at 3.46 GHz) and  $Z@F_3N_1$ -M/CA ( $RL_{\min}$  = –44.61 dB at 15.46 GHz) exhibit deep absorption valleys in the low- and high-frequency regions, respectively, indicating that absorption characteristics can be effectively tuned by adjusting the  $Fe^{3+}/Ni^{2+}$  ratio. These results confirm that bimetallic MOF synergistic

regulation enables precise matching of intrinsic loss capability with target frequency bands.

Furthermore, the EMW performance of  $Z@F_xN_y$ -M/CA was compared with that of similar aerogel systems reported in recent years (Table 1). Compared with MF-based aerogels modified by single carbon materials or conventional fillers, the MOF-on-MOF-derived system constructed in this work demonstrates distinct advantages in both absorption intensity and thickness adaptability.  $Z@F_1N_1$ -M/CA achieves an  $RL_{\min}$  of –60.36 dB at a relatively low filler loading of only 20 wt.%, outperforming most comparable materials. In comparison with our previous study based on the ZIF@MIL/MXene/MF system [39], the present work further optimizes the  $RL_{\min}$  from –55.82 to –60.36 dB through precise regulation of the  $Fe^{3+}/Ni^{2+}$  ratio, resulting in a significant enhancement in absorption strength. This improvement further confirms the critical role of bimetallic synergistic effects in regulating impedance matching and loss mechanisms.

RCS simulations (Fig. 4(g)) reveal significant RCS reduction for

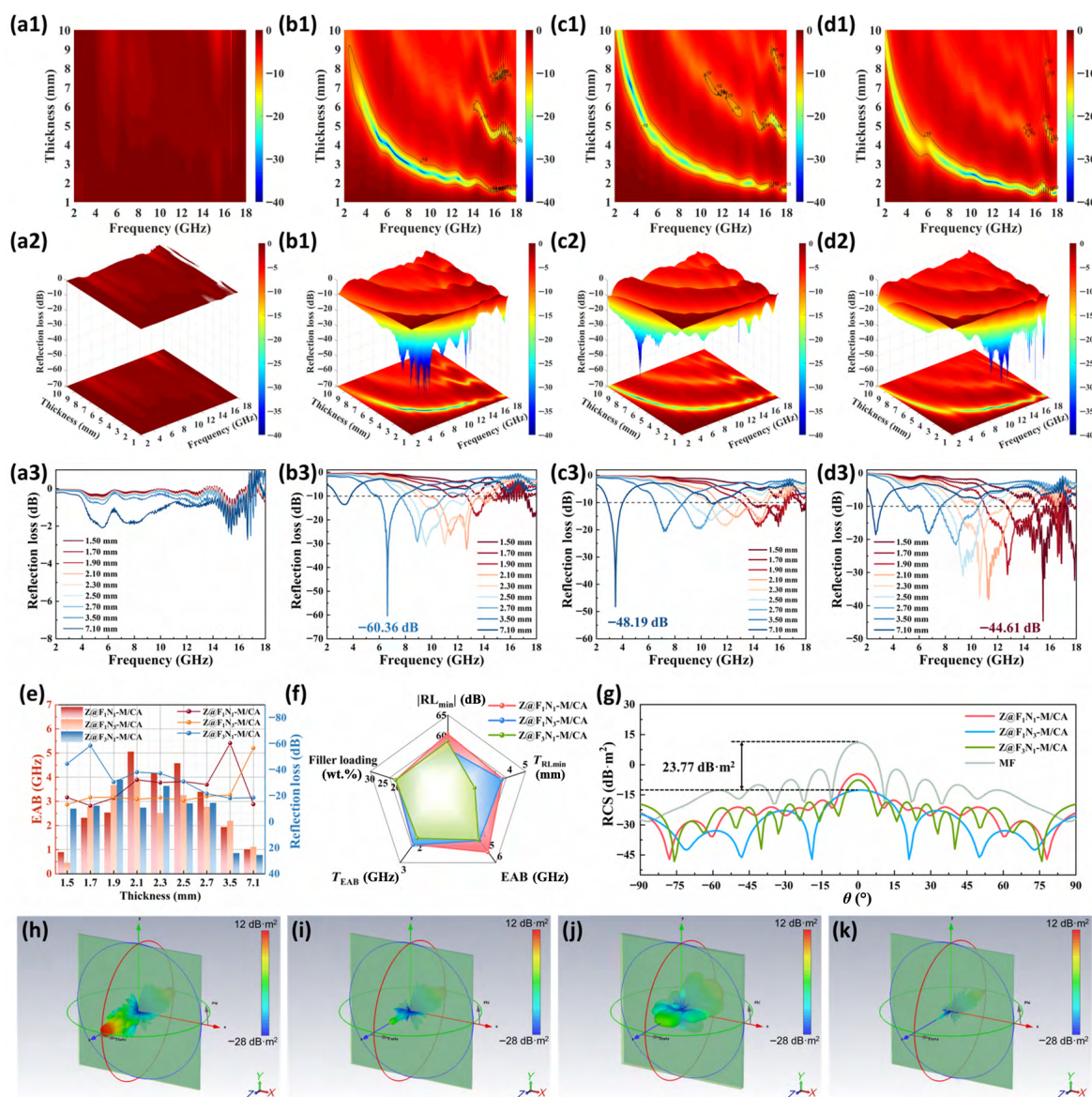


**Figure 4** RL plots of ((a1)–(a3)) MF, ((b1)–(b3))  $Z@F_1N_1$ -M/CA, ((c1)–(c3))  $Z@F_1N_3$ -M/CA, and ((d1)–(d3))  $Z@F_3N_1$ -M/CA. (e) Comparison maps of  $RL_{\min}$  and EAB at different thicknesses. (f) EMW performance radar chart; (g) RCS curve; RCS simulation of (h) MF, (i)  $Z@F_1N_1$ -M/CA, (j)  $Z@F_1N_3$ -M/CA, and (k)  $Z@F_3N_1$ -M/CA.

**Table 1** Similar aerogels in recent years and their EMW absorption performance

| Sample                                                      | Filler loading (wt.%) | RL <sub>min</sub> (dB) | $f_{RLmin}$ (GHz) | $T_{RLmin}$ (mm) | EAB (GHz) | $T_{EAB}$ (mm) | Ref.               |
|-------------------------------------------------------------|-----------------------|------------------------|-------------------|------------------|-----------|----------------|--------------------|
| Co <sub>3</sub> O <sub>4</sub> @N-C                         | 40                    | −46.58                 | 10.72             | 3.3              | 5.4       | 3.3            | [40]               |
| RGO/MWCNT-MF                                                | —                     | −50.43                 | 9.50              | 4.6              | 10.8      | 4.6            | [41]               |
| Co-QDs/CF                                                   | 20                    | −24.12                 | 18.00             | 1.9              | 8.7       | 2.5            | [42]               |
| MF-MXene/Fe <sub>3</sub> Si/g-C <sub>3</sub> N <sub>4</sub> | —                     | −44.30                 | 9.41              | 1.5              | 1.9       | 1.5            | [43]               |
| Graphite/MF                                                 | 50                    | −41.70                 | —                 | 2.5              | 9.8       | 2.6            | [44]               |
| MF/MXene/FeNi-PBA                                           | 20                    | −24.83                 | 18.00             | 1.3              | 4.7       | 1.5            | [45]               |
| CNTs/PyC/SiBCN                                              | —                     | −47.50                 | 9.20              | 4.6              | —         | —              | [46]               |
| MDCF-EP                                                     | —                     | −47.63                 | 9.83              | 4.6              | 8.4       | 4.6            | [47]               |
| N-CF                                                        | 20                    | −21.51                 | 13.20             | 3.0              | 7.04      | 3.0            | [48]               |
| MF@MXene/CoNi@C                                             | 30                    | −24.10                 | 3.06              | 1.68             | 6.88      | 1.56           | [49]               |
| ZIF@MIL/MXene/MF                                            | 20                    | −55.82                 | 15.58             | 1.96             | 5.20      | 4.0            | Previous work [39] |
| Z@F <sub>1</sub> N <sub>1</sub> -M/CA                       | 20                    | −60.36                 | 6.62              | 3.5              | 5.06      | 2.1            | This work          |
| Z@F <sub>1</sub> N <sub>3</sub> -M/CA                       | 20                    | −48.19                 | 3.46              | 7.1              | 3.94      | 2.1            |                    |
| Z@F <sub>3</sub> N <sub>1</sub> -M/CA                       | 20                    | −44.61                 | 15.46             | 1.7              | 3.90      | 1.9            |                    |

all Z@F<sub>x</sub>N<sub>y</sub>-M/CA samples compared with MF over incident angles from  $-90^\circ$  to  $90^\circ$ , with a maximum attenuation of 23.77 dB·m<sup>2</sup>. Z@F<sub>1</sub>N<sub>3</sub>-M/CA exhibits the lowest and most stable RCS responses, indicating enhanced EMW dissipation and scattering suppression. 3D RCS maps (Figs. 4(h)–4(k)) further show contracted and more uniform scattering lobes relative to MF, confirming that the hierarchical porous structure and multiple loss mechanisms effectively weaken radar echoes. It is important to note that this simulation employs an idealized flat-plate perfect electric conductor (PEC) backing model. While this approach does not fully account for the complex scattering contributions from surface roughness and the macroscopic 3D porous architecture of the actual aerogel, it serves as a standardized and widely adopted methodology for evaluating the intrinsic radar signature reduction capability derived from the material's fundamental electromagnetic parameters. The synergistic integration of MXene-based conductive networks, Fe/Co/Ni-induced magnetic and polarization losses, and 3D porous carbon frameworks enables simultaneous strong absorption, broadband response, and low RCS, highlighting the potential of Z@F<sub>x</sub>N<sub>y</sub>-M/CA aerogels for electromagnetic stealth and multifunctional protection.

Figure 5 present the electromagnetic parameters of Z@F<sub>x</sub>N<sub>y</sub>-M/CA aerogels and pristine MF over 2–18 GHz. As shown in Figs. 5(a)–5(c), all aerogels exhibit significantly higher permittivity than MF, confirming that composite construction and carbonization markedly enhance dielectric response. The real permittivity ( $\epsilon'$ ) of the aerogels decreases with frequency and remains in the range of 5–12, reflecting typical polarization relaxation behavior [50]. The imaginary permittivity ( $\epsilon''$ ), representing dielectric loss, ranges from 1.5 to 6.5 and shows multiple fluctuations, indicating the coexistence of several polarization relaxation processes. Accordingly, the dielectric loss tangent ( $\tan\delta_\epsilon$ ) of the aerogels (0.15–0.65) far exceeds that of MF, evidencing strong dielectric loss. Such enhanced dielectric loss originates from abundant heterogeneous interfaces (e.g., FeCoNi alloy/carbon and MXene/carbon) that induce interfacial polarization, together with dipolar polarization associated with nitrogen doping, defects, and residual functional groups.

In contrast to non-magnetic MF ( $\mu' \approx 1$ ,  $\mu'' \approx 0$ ), the aerogels exhibit pronounced magnetic responses (Figs. 5(d)–5(f)), with  $\mu'$

values of 0.7–1.4 and  $\mu''$  values of 0.1–0.8. Distinct resonance peaks, particularly near  $\sim 4$  and  $\sim 13$  GHz, indicate effective magnetic loss introduced by *in situ* formed FeCoNi alloy nanoparticles, consistent with XRD and XPS results. These resonances are attributed to natural resonance at low frequencies and exchange resonance at higher frequencies, while the  $\tan\delta_\mu$  curves confirm significant magnetic loss mainly in the 2–10 GHz range.

Cole–Cole plots (Fig. 5(g)) show multiple semicircles for all Z@F<sub>x</sub>N<sub>y</sub>-M/CA samples, indicating the coexistence of multiple Debye relaxation processes [51]. This arises from abundant heterogeneous interfaces that induce strong interfacial polarization via space-charge accumulation, as well as dipolar polarization from carbon defects, N dopants, and residual functional groups [52]. In this context, MXene plays a crucial role in modulating dielectric loss behavior. Its high electrical conductivity contributes to conduction loss, while its surface functional groups and defects further enhance dipolar polarization. Moreover, the heterogeneous interfaces among MXene, MOF-derived carbon, and magnetic nanoparticles introduce additional interfacial polarization, thereby promoting dielectric relaxation. In contrast, MF exhibits a nearly negligible Cole–Cole curve, confirming its weak dielectric loss. These results demonstrate that the aerogels contain abundant polarization loss centers responsible for dielectric dissipation.

Cole–Cole plots (Fig. 5(g)) show multiple semicircles for all Z@F<sub>x</sub>N<sub>y</sub>-M/CA samples, indicating the coexistence of multiple Debye relaxation processes. This arises from abundant heterogeneous interfaces that induce strong interfacial polarization via space-charge accumulation, as well as dipolar polarization from carbon defects, N dopants, and residual functional groups. In contrast, MF exhibits a nearly negligible Cole–Cole curve, confirming its negligible dielectric loss. These results demonstrate that the aerogels contain abundant polarization loss centers responsible for dielectric dissipation.

The  $C_0$  curves (Fig. 5(h)) of the aerogels vary markedly with frequency, particularly in the 2–10 GHz range, indicating that magnetic loss is not governed solely by eddy current effects. The pronounced frequency-dependent fluctuations of  $C_0$  indicate that magnetic loss cannot be solely attributed to eddy current effects but is dominated by multiple resonance mechanisms. In general, natural resonance, arising from magnetic anisotropy-induced spin

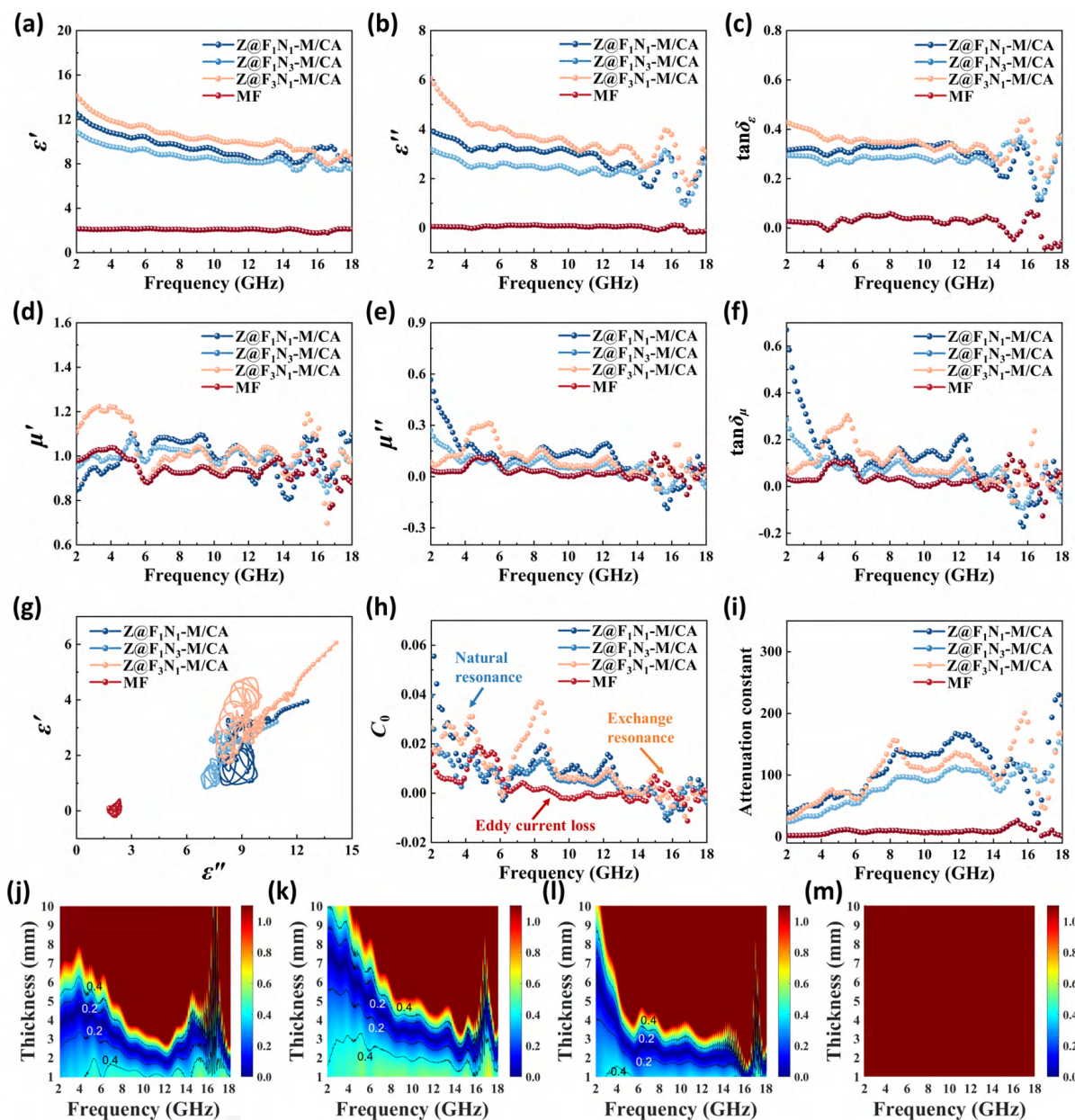


**Figure 5** Z@F<sub>1</sub>N<sub>1</sub>-M/CA, Z@F<sub>1</sub>N<sub>3</sub>-M/CA, Z@F<sub>3</sub>N<sub>1</sub>-M/CA and MF: (a)  $\epsilon'$ , (b)  $\epsilon''$ , (c)  $\tan \delta_\epsilon$ , (d)  $\mu'$ , (e)  $\mu''$ , (f)  $\tan \delta_\mu$ , (g) Cole-Cole curve, (h)  $C_0$ , (i) attenuation constants, and (j)–(m)  $|\Delta|$  diagrams.

precession, typically occurs in the lower GHz region, whereas exchange resonance, associated with short-range exchange interactions among magnetic moments, appears at higher frequencies. The observed variation of  $C_0$ , together with the corresponding resonance peaks in  $\mu''$  (Fig. 5(e)), is consistent with the coexistence of these mechanisms in FeCoNi alloy nanoparticles. The pronounced fluctuations are associated with natural (low-frequency) and exchange (high-frequency) resonances originating from FeCoNi alloy nanoparticles, suggesting that magnetic loss results from the synergy of eddy current loss and multiple resonance mechanisms.

The attenuation constant ( $\alpha$ ) quantitatively describes the overall capability of a material to attenuate EMW [53]. In Fig. 5(i),  $\alpha$  of all aerogels is substantially higher than that of MF over 2–18 GHz and generally increases with frequency, reflecting enhanced polarization relaxation and resonance effects. Z@F<sub>1</sub>N<sub>1</sub>-M/CA exhibits the

highest  $\alpha$  values across most frequencies, indicating the strongest intrinsic attenuation. The  $|\Delta|$  function maps (Figs. 5(j)–5(m)) visually illustrate the impedance matching degree of the materials at different thicknesses and frequencies in the form of 2D contour plots, where deep blue regions ( $|\Delta| < 0.4$ ) represent good impedance matching [54]. By tuning the Fe<sup>3+</sup>/Ni<sup>2+</sup> ratio, balanced dielectric and magnetic losses are achieved, allowing more EMW to enter and dissipate within the material. Notably, Z@F<sub>1</sub>N<sub>1</sub>-M/CA shows excellent impedance matching at thin thicknesses (~2.0–3.0 mm) over a wide frequency range, highlighting its promise for lightweight, broadband EMW absorption.

### 3.3 Sound absorption and thermal protection performance

As shown in Figs. 6(a) and 6(b), Z@F<sub>x</sub>N<sub>y</sub>-M/CA aerogels exhibit markedly higher sound absorption than pristine MF in both the

low-frequency (100–450 Hz) and mid-to-high-frequency (450–6000 Hz) ranges. At low frequencies, the absorption coefficients of all samples increase with frequency, mainly due to hierarchical porous structures that prolong air vibration paths and enhance viscous and thermal dissipation. In contrast, MF maintains low absorption, reflecting the limited low-frequency dissipation ability of a single fibrous skeleton. The MOF-on-MOF-derived porous carbon/metal framework in  $Z@F_xN_y-M/CA$  improves pore connectivity and acoustic scattering, thereby enhancing low-frequency absorption.

In the mid-to-high-frequency range,  $Z@F_xN_y-M/CA$  shows a rapid increase in absorption, maintaining coefficients above 0.9 over a wide band and approaching near-total absorption, far outperforming MF. This arises from multiscale hierarchical pores that enable wavelength matching, multiple reflections, and efficient energy dissipation, while the MOF-derived skeleton increases local mass and interfacial complexity, strengthening acoustic-solid coupling. The noise reduction coefficient (NRC) values (Fig. 6(c)) further confirm this trend, with  $Z@F_3N_1-M/CA$  (0.66) >  $Z@F_1N_1-M/CA$  (0.58) >  $Z@F_1N_3-M/CA$  (0.52) > MF (0.50). These results indicate that a moderate Fe content optimizes the balance between pore structure and skeleton stiffness, maximizing acoustic energy dissipation while preserving high porosity and overall sound absorption performance.

As shown in Figs. 6(d)–(i), infrared thermography reveals the dynamic thermal responses of the samples on a 200 °C heating platform. Compared with MF,  $Z@F_xN_y-M/CA$  maintains consistently lower surface temperatures throughout the test, indicating superior thermal insulation. This is mainly due to the

highly porous aerogel structure, which suppresses solid- and gas-phase heat conduction, while the carbonized framework and MOF-derived interfaces enhance phonon scattering [55]. As shown in Fig. S8 in the ESM,  $Z@F_xN_y-M/CA$  exhibits a distinct temperature evolution compared with MF, characterized by an initial rise followed by gradual decrease and stabilization, reflecting effective thermal regulation rather than simple heat blocking. MF rapidly heats to 81.0 °C within 600 s, whereas all  $Z@F_xN_y-M/CA$  samples stabilize below 62.0 °C. Overall,  $Fe^{3+}/Ni^{2+}$  ratio tuning enables synergistic optimization of thermal management and acoustic performance in  $Z@F_xN_y-M/CA$ , offering an effective strategy for designing multifunctional acoustic-thermal protective materials.

To quantitatively evaluate the intrinsic thermal insulation capability of the aerogels, the thermal conductivity of all samples was measured at 25, 100, and 200 °C, as presented in Fig. S9 in the ESM. All  $Z@F_xN_y-M/CA$  aerogels exhibit lower thermal conductivities than pristine MF across the entire temperature range. At room temperature (25 °C),  $Z@F_1N_1-M/CA$  displays the lowest thermal conductivity of 32.95 mW/(m·K), compared to 39.95 mW/(m·K) for MF. Notably, at 200 °C (the same temperature used for the infrared thermography evaluation)  $Z@F_1N_1-M/CA$  maintains a thermal conductivity of 54.55 mW/(m·K), which is approximately 17.3% lower than that of MF (65.94 mW/(m·K)). This significant reduction in solid-phase heat conduction is primarily attributed to the introduction of abundant heterogeneous interfaces (such as MXene/carbon and FeCoNi alloy/carbon) by the MOF-on-MOF assembly, which enhance phonon scattering via interfacial thermal resistance (Kapitza resistance). And the refined

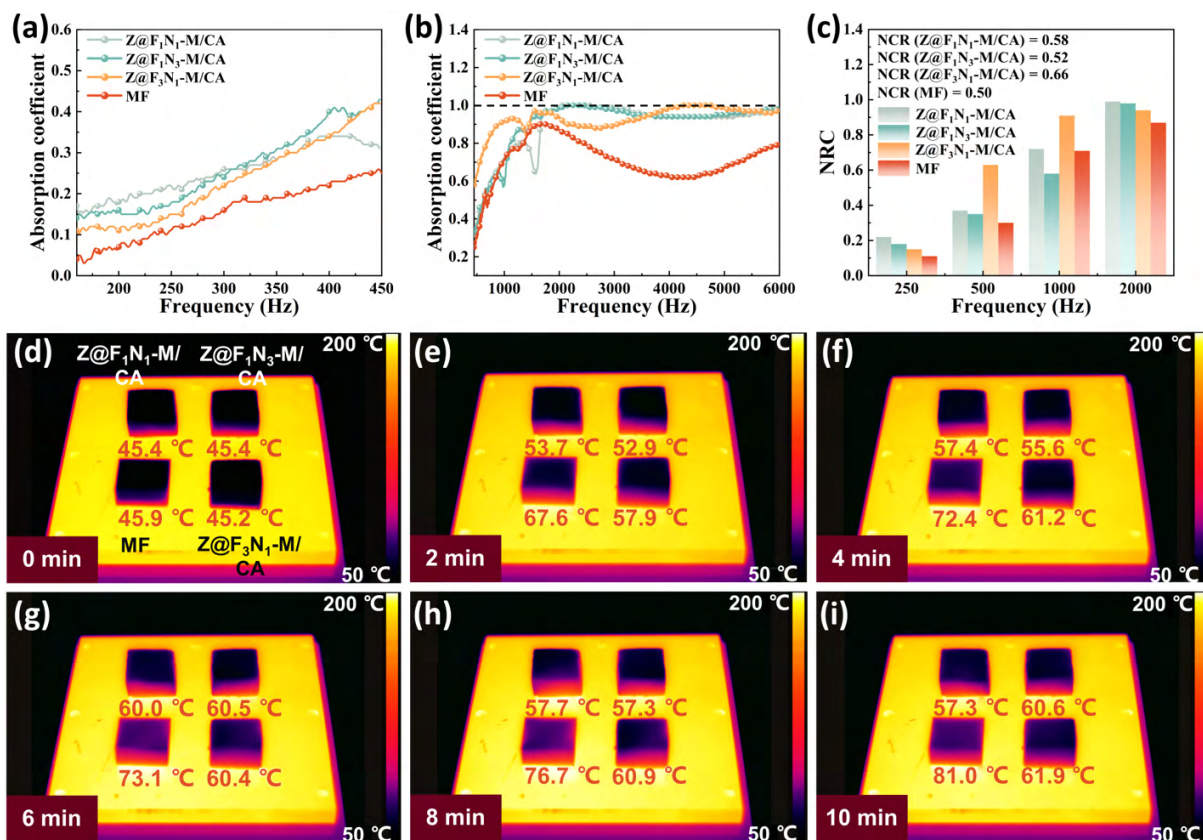


**Figure 6** (a) and (b) Sound absorption coefficient curves and (c) NRC of  $Z@F_1N_1-M/CA$ ,  $Z@F_1N_3-M/CA$ ,  $Z@F_3N_1-M/CA$ , and MF; (d)–(i) infrared thermography of samples on 200 °C heating platform.

hierarchical pore architecture, which restricts gas-phase thermal transport through the Knudsen effect. These quantitative thermal conductivity data, together with the dynamic infrared thermography results, unequivocally establish the superior thermal insulation and thermal management performance of the  $Z@F_xN_y$ -M/CA aerogels.

Table S1 in the ESM summarizes the key cone calorimetry test (CCT) parameters. As shown in Figs. 7(a)–7(d),  $Z@F_xN_y$ -M/CA aerogels exhibit markedly improved flame-retardant performance over MF in combustion intensity, heat release, and smoke suppression. In the HRR curves (Fig. 7(a)), MF shows a sharp peak heat release rate (pHRR) of 119.93 kW/m<sup>2</sup>, indicating intense early-stage combustion. In contrast, all  $Z@F_xN_y$ -M/CA samples display significantly reduced and broadened HRR peaks, evidencing effective combustion suppression. Notably,  $Z@F_1N_3$ -M/CA achieves the lowest pHRR (55.45 kW/m<sup>2</sup>), demonstrating the strongest flame inhibition and clear “peak-shaving” behavior. The total heat release (THR) curves (Fig. 7(b)) further indicate that MF undergoes rapid and continuous heat accumulation, whereas  $Z@F_xN_y$ -M/CA exhibits much lower THR values and slower growth rates, confirming effective limitation of combustible participation.

$Z@F_1N_3$ -M/CA shows a particularly reduced THR, highlighting the barrier effect of the MOF-derived inorganic/carbon hybrid skeleton in the condensed phase. Smoke suppression is evidenced by the smoke production rate (SPR) and total smoke production (TSP) curves (Figs. 7(c) and 7(d)). MF presents a high early peak smoke production rate (pSPR) and large TSP, while  $Z@F_xN_y$ -M/CA shows markedly reduced pSPR, smoother smoke release, and much lower TSP, demonstrating efficient inhibition of smoke and volatile products and mitigating secondary fire hazards.

Residue analyses further verify the flame-retardant mechanisms. XRD patterns (Fig. 7(e)) reveal crystalline  $Fe_3O_4$  and  $NiCo_2O_4$  in  $Z@F_xN_y$ -M/CA chars, indicating *in situ* formation of stable metal oxides that act as structural supports and char-formation catalysts [56]. MF residues show no distinct crystalline features. Raman spectra (Fig. 7(f)) show lower  $I_D/I_G$  ratios for  $Z@F_xN_y$ -M/CA, reflecting higher graphitization and more ordered char layers.  $Z@F_1N_3$ -M/CA exhibits the lowest defect density, suggesting that higher Ni content promotes catalytic aromatization and carbon rearrangement, yielding a dense and stable protective char [57].

Figures 7(g)–7(n) show the macroscopic and microscopic morphologies of char residues after CCT. Pure MF (Figs. 7(m) and

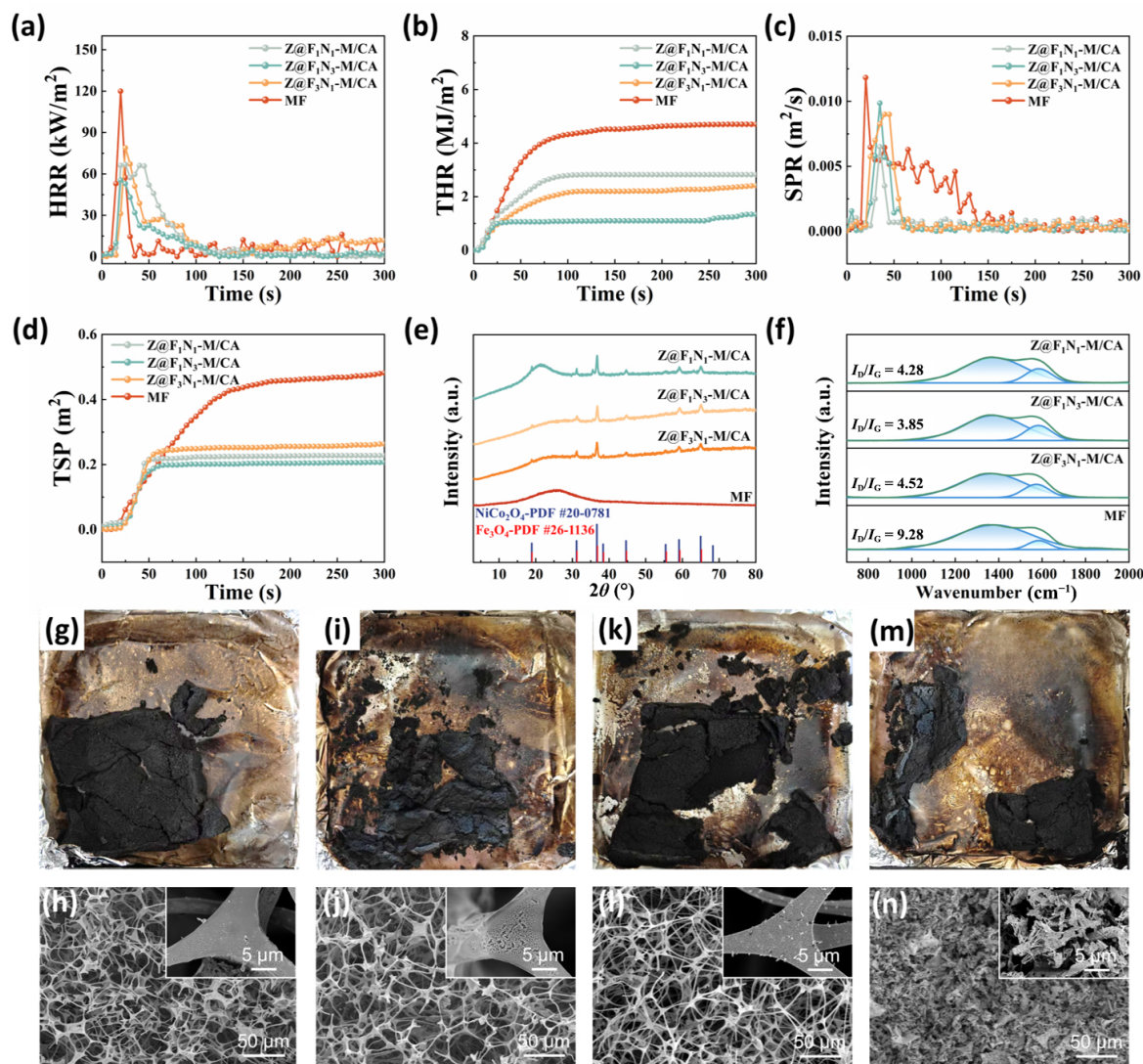


**Figure 7** (a) HRR, (b) THR, (c) SPR, and (d) TSP of  $Z@F_1N_3$ -M/CA,  $Z@F_1N_2$ -M/CA,  $Z@F_3N_1$ -M/CA, and MF; (e) XRD, (f) Raman spectra, digital photographs and SEM images of ((g) and (h))  $Z@F_1N_3$ -M/CA, (i) and (j)  $Z@F_1N_2$ -M/CA, (k) and (l)  $Z@F_3N_1$ -M/CA, and ((m) and (n)) MF of char residues after CCT.

7(n)) almost completely disappears after combustion and fails to form a continuous char layer, reflecting severe pyrolysis and extremely poor flame retardancy. In contrast, all  $Z@F_xN_y\text{-M/CA}$  aerogels retain intact char skeletons with shapes largely preserved, demonstrating excellent charring ability and structural stability. SEM images reveal that MF char consists of fragmented, sparse fibers with disordered porosity, offering little resistance to heat and gas diffusion. Conversely,  $Z@F_xN_y\text{-M/CA}$  chars exhibit robust hierarchical porous architectures. Notably,  $Z@F_1N_1\text{-M/CA}$  forms a continuous, dense micro/nanoscale “ceramified” protective layer derived from MOF- and MXene-originated metal oxide/carbon phases, which seals cracks, blocks oxygen ingress and gas escape, and is accompanied by abundant closed and honeycomb-like pores that effectively retard heat transfer.

Overall, MOF-on-MOF-derived multiphase synergy endows  $Z@F_xN_y\text{-M/CA}$  aerogels with catalytic charring, stable inorganic/carbon barrier formation, and effective suppression of heat and smoke release, markedly improving fire safety. Among them,  $Z@F_1N_1\text{-M/CA}$  shows the most pronounced heat suppression, char quality, and structural stability, highlighting the key role of  $\text{Fe}^{3+}/\text{Ni}^{2+}$  ratio regulation.

## 4 Conclusions

This work develops a multifunctional  $Z@F_xN_y\text{-M/CA}$  aerogel by integrating 2D MXene, bimetallic MOF-derived phases, and a 3D porous carbon framework via a MOF-on-MOF strategy and multilevel structural synergy. Regulation of the  $\text{Fe}^{3+}/\text{Ni}^{2+}$  molar ratio effectively tailors MOFs nucleation and growth, as well as the resulting metal/alloy phases, defect density, and conductive carbon networks after pyrolysis, enabling tunable EMW absorption performance. Notably, the optimized aerogel achieves a  $\text{RL}_{\min}$  of  $-60.36$  dB and a broad EAB of  $5.06$  GHz. Meanwhile, it exhibits significantly enhanced fire safety with over 50% reduction in pHRR and THR, along with suppressed smoke emission. In addition, the aerogel delivers a high NRC of up to  $0.66$  and maintains excellent thermal insulation performance, with surface temperatures stabilized below  $62^\circ\text{C}$  under a  $200^\circ\text{C}$  heating condition. Adjusting the  $\text{Fe}^{3+}/\text{Ni}^{2+}$  ratio modulates the balance and synergy between dielectric loss and magnetic loss, thereby optimizing impedance matching, energy dissipation, and absorption bandwidth, while the hierarchical porous architecture enhances multiple scattering and prolongs EMW propagation paths. Overall, this work provides a general strategy for designing lightweight multifunctional materials that integrate efficient EMW absorption with flame-retardant, thermal management, and acoustic protection.

**Electronic Supplementary Material:** Supplementary material (detailed information on the materials and experimental procedures, relevant calculation equations, and supplementary experimental results) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908751>.

## Data availability

All data needed to support the conclusions in the paper are presented in 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

All the contributing authors report no conflict of interests in this work.

## Author contribution statement

H. M. D.: Conceptualization, methodology, writing – original draft. J. H. H.: Date curation, investigation, methodology. X. Y. W.: Investigation. Y.-T. P.: Funding acquisition, writing – review & editing. M. L. M.: Methodology, software. G. W. X.: Project administration, writing – review & editing. Z. N. H.: Investigation. N. W.: Validation, writing – review & editing. All the authors have approved the final manuscript.

## Use of AI statement

None.

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