

Polyoxomolybdate-based nanocoating on flexible polyurethane foams for efficient flame retardancy and smoke suppression

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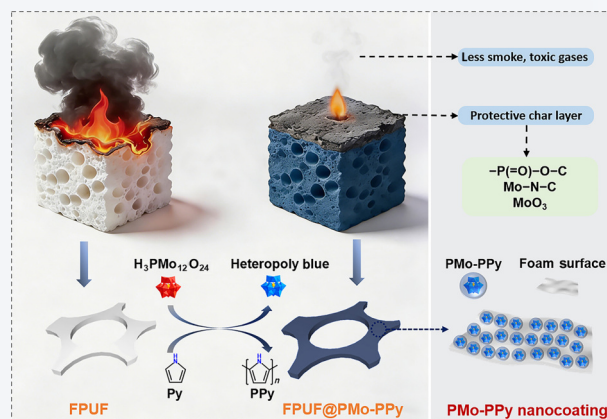
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Cite this article: Nano Research, 2026, 19, 94908619. <https://doi.org/10.26599/NR.2026.94908619>

ABSTRACT: Flexible polyurethane foams (FPUF) are commercially used cushioning materials, but severely suffer from fire risks due to their inherent flammability. Surface coating can localize flame-retardant functionality at the polymer-air interface without altering bulk properties. However, developing a facile nanocoating strategy beyond the layer-by-layer technique for FPUF remains challenging. This work reports novel flame-retardant FPUF@PMo-PPy composites, fabricated by uniformly depositing phosphomolybdic acid ($\text{H}_3\text{PMo}_{12}\text{O}_{40}$, PMo_{12}) clusters encapsulated within polypyrrole (PPy) via *in situ* polymerization onto the FPUF skeleton surface, based on electrostatic interactions. The PMo-PPy coating significantly mitigates the fire hazards of FPUF, exhibiting a maximum reduction of 66.7% in peak heat release rate (pHRR) and 73.5% in peak smoke production rate (pSPR). Notably, the maximum smoke density ($D_{\max}$) and smoke density at 10 min ($D_{10\text{ min}}$) were reduced by 55.7% and 60.8%, respectively, accompanied by lower pyrolysis gas toxicity (HCN and CO). This enhancement is attributed to decomposition products—phosphoric acid and MoO_3 —generated from the PMo-PPy coating, which improve the stability and compactness of the char layer. This PMo-PPy nanocoating strategy provides a security assurance for cushioning materials in industrial engineering, ensuring the security of life and property.



KEYWORDS: nanocoating, flame retardancy, smoke suppression, polyoxomolybdate, flexible polyurethane foam

1 Introduction

Flexible polyurethane foams (FPUF) are prevalent as cushioning materials in soft furniture, transportation, and power equipment applications [1, 2]. The polyether-type FPUF used in this study, typically synthesized from polyether polyols and diisocyanates, is characterized by an aliphatic ether-rich chemical structure. While this structure imparts excellent flexibility and comfort, it also renders the foam inherently flammable and thermally unstable. Consequently, FPUF ignites readily and propagates flames rapidly [3, 4], and its combustion releases dense smoke containing toxic

carbonyl compounds, carbon monoxide (CO), and isocyanate compounds, which are major contributors to fire-related fatalities [5, 6]. This significant lethality drives considerable research attention towards effectively reducing their fire hazards.

Surface engineering approaches offer an alternative to additive/reactive flame-retardant strategies, which face challenges like mechanical property degradation, environmental concerns, complex synthesis, and high costs [7]. Surface nanocoating is a representative surface engineering approach that localizes flame-retardant functionality at the polymer-air interface while preserving bulk properties [8]. Extensive research has demonstrated the viability of depositing flame-retardant coatings on foam substrates via techniques such as layer-by-layer assembly [9–15], flocculation assembly [16, 17] sol-gel processing [18, 19] and dip-coating [20, 21]. These approaches are further advantageous as they largely preserve the foam's intrinsic manufacturing process and overall physical performance. Unfortunately, the practical application is often hindered by their reliance on multistep treatments, complex

Received: January 9, 2026; Revised: March 3, 2026

Accepted: March 5, 2026

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processing, or organic solvents, thus rendering the process time-consuming and costly. To address these challenges, recent advances have focused on developing *in situ* deposition strategies that enable the direct, one-step formation of functional nanocoating during or immediately after foam fabrication. For example, Li et al. reported an *in situ* reactive self-assembly method to construct an ultrathin graphene oxide coating on silicone foam within the foaming process, achieving exceptional flame retardancy at an ultralow loading of 0.1 wt.% [22]. Similarly, Ma et al. designed a bioinspired copolymer coating applied via a simple rod-coating process, which combines strong adhesion with high fire-retardant efficiency [23]. These works highlight the potential of *in situ* or facile one-step coating approaches to simplify preparation, improve material utilization, and enhance interfacial integration without compromising the foam's porous structure.

Polyoxometalates (POMs) are a unique class of anionic metal oxide clusters, primarily composed of transition metals (e.g., V, Mo, W, Nb), which have attracted significant attention for catalytic applications [24–26]. Phosphomolybdic acid ($\text{H}_3\text{PMo}_{12}\text{O}_{40}$, PMo_{12}), a prominent Keggin-type heteropolyanion, is characterized by its redox activity, water solubility, tunable acidity, and nanosized structure [27]. These properties enable its uniform dispersion on FPUF substrates in aqueous media [28–30]. Notably, the high phosphorus [31–34] and molybdenum [35–37] content in $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ demonstrates promising potential for enhancing flame retardancy and smoke suppression, rendering it ideal for reducing fire hazards in FPUF. However, immobilizing POMs on flexible substrates remains challenging due to their weak interfacial adhesion. Few studies have addressed the role of $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ in improving flame retardancy and smoke suppression for FPUF.

In this study, we propose a facile approach for applying a surface nanocoating consisting of PMo_{12} clusters encapsulated within polypyrrole (designated as PMo-PPy) via *in situ* polymerization onto FPUF. The PMo-PPy nanocoating significantly improved the thermal stability, flame retardancy, and smoke suppression of the coated FPUF. The release of toxic volatile matter from the foams was markedly reduced. The flame-retardant mechanism was investigated in detail through analysis of the condensed and gaseous phases, revealing that the synergistic interaction of P and Mo elements enhanced char formation. Decomposition products—phosphoric acid and MoO_3 —promoted the formation of a stable, compact, and reinforced char layer incorporating $-\text{P}(=\text{O})-\text{O}-\text{C}$ and $\text{N}-\text{Mo}$ chemical bonds, thereby boosting the flame retardancy and smoke suppression performance. This single-

step and aqueous-phase coating strategy gives a viable solution to mitigate the fire risk associated with widely used FPUF, thereby contributing to enhanced life and property safety.

2 Experimental section

The synthesis route for FPUF@ PMo-PPy composites is illustrated in Fig. 1. The composites were obtained via a one-pot strategy by mixing FPUF, $\text{H}_3\text{PMo}_{12}\text{O}_{40}$, and pyrrole (Py). First, FPUF was presoaked in the $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ solution, imparting a positively charged surface. Upon introducing the Py monomer into this solution, polypyrrole (PPy) formed owing to the strong oxidative properties of $\text{H}_3\text{PMo}_{12}\text{O}_{40}$. Simultaneously, PPy encapsulated $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ during *in situ* polymerization, while $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ was reduced to heteropoly blue. Ultimately, the negatively charged spherical PMo-PPy nanoparticles (zeta potential: -33.9 mV) were uniformly deposited onto the FPUF framework surface via electrostatic interactions. Additional important information required for the text is supplied in the Electronic Supplementary Material (ESM).

3 Results and discussion

3.1 Structure and morphology of FPUF@ PMo-PPy composites

As shown in Fig. 2(a), the X-ray diffraction (XRD) pattern of pure FPUF sample exhibits a broad diffraction peak centered at $\sim 20.5^\circ$. In contrast, the FPUF@ PMo-PPy composite shows a new sharp peak below 10° and a much broader peak around 18° – 32° , attributable to the presence of PMo-PPy nanoparticles. Fourier transform infrared spectroscopy (FTIR) spectra of PMo-PPy and FPUF@ PMo-PPy composite (Fig. 2(b)) reveal four characteristic peaks at 1057, 953, 872, and 793 cm^{-1} , assigned to $\text{P}-\text{O}$, $\text{Mo}=\text{O}$, $\text{Mo}-\text{O}_b-\text{Mo}$, and $\text{Mo}-\text{O}_c-\text{Mo}$ stretching vibrations, respectively. FPUF exhibits no apparent absorption in this region, confirming the successful deposition of Keggin-structured PMo-PPy onto the FPUF matrix. X-ray photoelectron spectroscopy (XPS) analysis (Fig. 2(c)) further confirms the presence of C, N, O, P, and Mo in the FPUF@ PMo-PPy composite, indicating deposition of PMo-PPy nanoparticles. The Mo 3d spectrum of $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ (Fig. 2(d)) shows peaks at 232.7 and 235.9 eV corresponding to Mo^{6+} $3d_{5/2}$ and $3d_{3/2}$, respectively. For PMo-PPy and FPUF@ PMo-PPy (Figs. 2(e) and 2(f)), peaks at 231.2 and 234.1 eV correspond to partially

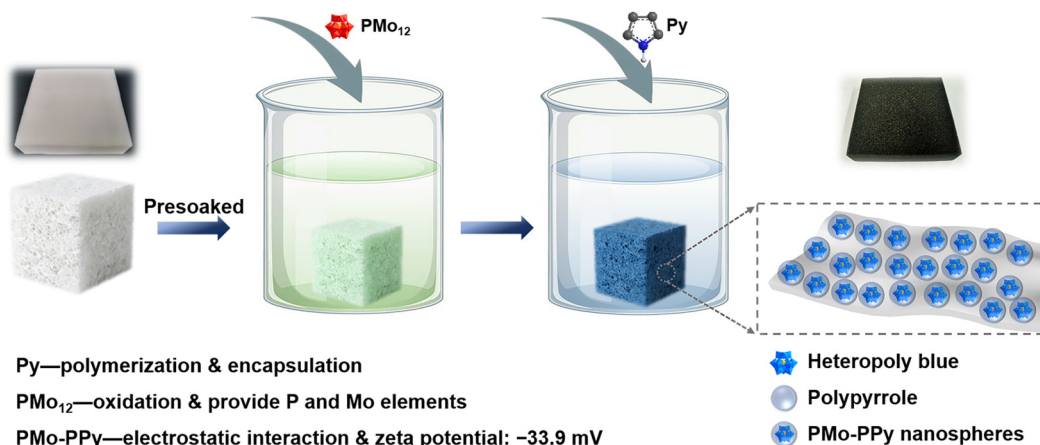


Figure 1 Schematic illustration of the fabrication procedure for FPUF@ PMo-PPy composites.

reduced Mo^{6+} , confirming Py polymerization under $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ oxidation and partial conversion to heteropoly blue [28]. Collectively, these results verify the successful synthesis of the FPUF@PMo-PPy composite.

Surface morphology of FPUF, PMo-PPy, and FPUF@PMo-PPy composites was further characterized by scanning electron microscopy (SEM) at different magnifications. PMo-PPy (Fig. S1(e) in the ESM) shows an agglomerated globular structure with an average particle size of ~ 90 nm (Fig. S1(d) in the ESM). FPUF (Fig. 3(a)) displays a porous three-dimensional open-cell framework. Upon PMo-PPy deposition, the FPUF@PMo-PPy composites retained their cellular structure (Figs. 3(b)–3(e)). At medium and

high magnification (Fig. 3(f)), pure FPUF exhibits a smooth surface. As the $\text{H}_3\text{PMo}_{12}\text{O}_{40}$ concentration increases, coated samples show progressively rougher surfaces (Figs. 3(g)–3(j)) due to a marked increase in the number of PMo-PPy nanospheres coating the FPUF skeleton (Figs. 3(k)–3(o)). With prolonged impregnation time, a similar pattern was observed, as depicted in Fig. S2 in the ESM. The weight gain exhibits a nonlinear increase that gradually saturates with increasing polyoxometalate dosage and impregnation time, indicating that the loading of PMo-PPy nanospheres is ultimately limited by the available adsorption capacity of the FPUF substrate surface (Figs. S1(a) and S1(b) in the ESM). Interestingly, when a small amount of Py is added, the coating mass gain rapidly reaches

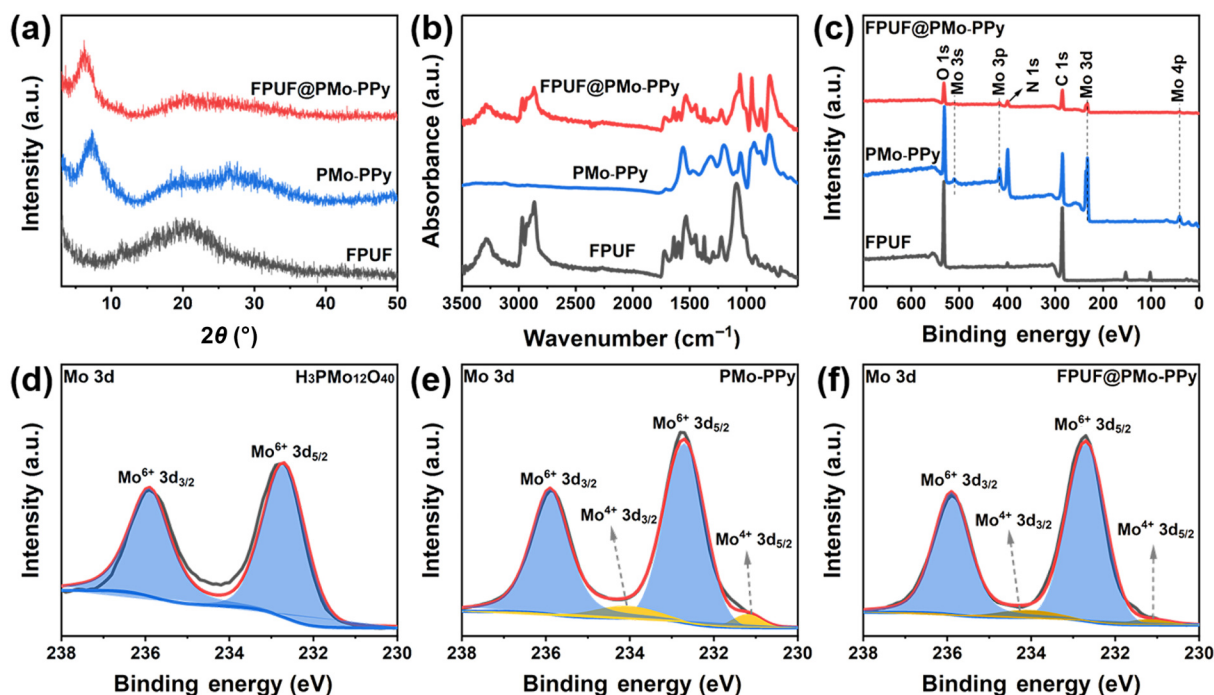


Figure 2 (a) XRD patterns, (b) FTIR spectra, and (c) full-XPS spectra of FPUF, PMo-PPy, and FPUF@PMo-PPy composite. Mo 3d XPS spectra for (d) $\text{H}_3\text{PMo}_{12}\text{O}_{40}$, (e) PMo-PPy, and (f) FPUF@PMo-PPy composite.

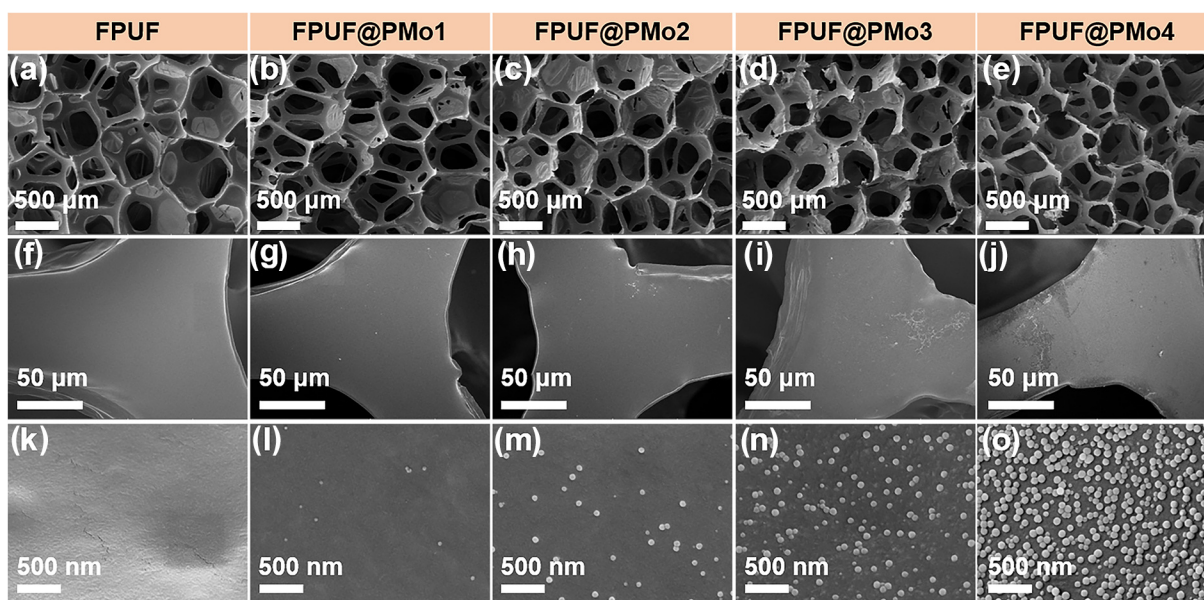


Figure 3 SEM images of FPUF@PMo-PPy at different magnifications with varying amounts of phosphomolybdic acid: ((a), (f), and (k)) FPUF, ((b), (g), and (l)) FPUF@PMo1, ((c), (h), and (m)) FPUF@PMo2, ((d), (i), and (n)) FPUF@PMo3, and ((e), (j), and (o)) FPUF@PMo4.

saturation. As the Py dosage continues to increase, the coating loading shows almost no further change (Fig. S1(c) in the ESM). As shown in Fig. S3 in the ESM, with increasing Py dosage, the agglomerated phosphomolybdic acid particles on the FPUF surface gradually transform into uniformly dispersed small spheres, indicating that the *in situ* polymerization of Py facilitates the uniform dispersion of phosphomolybdic acid on the FPUF surface. Elemental mapping (Figs. S1(f)–S1(i) in the ESM) reveals homogeneous distribution of PMo-PPy on the FPUF surface, clearly demonstrating successful coating.

3.2 Flame retardancy and smoke suppression properties

Cone calorimetry is used to assess the fire behavior of polymers under well-defined, radiant heat fluxes that simulate the early stages of a developing fire [38]. Figures 4(a) and 4(b) show the heat release rate (HRR) and total heat release (THR) curves for pure FPUF and FPUF@PMo-PPy composites, respectively; key parameters are listed in Table S1 in the ESM. Pure FPUF burned out within 100 s, exhibiting the highest pHRR (509.05 kW/m²) and THR at flame out (22.17 MJ/m²), confirming its high flammability. Increasing the H₃PMo₁₂O₄₀ loading significantly reduced the pHRR and THR at flame out. Compared to pure FPUF, FPUF@PMo3 exhibited a pHRR of 169.37 kW/m² and THR at flame out of 13.73 MJ/m², representing reductions of 66.7% and 38.1%, respectively, demonstrating the substantial flame retardancy enhancement imparted by the PMo-PPy coating. This trend was further corroborated by the influence of immersion time; as the immersion duration increased, the pHRR progressively decreased, with the sample immersed for 3 h achieving a reduction of approximately 68.5% (Figs. S5(a) and S5(b) in the ESM). Concurrently, the average effective heat of combustion (EHC) showed a gradual decreasing trend from 26.3 MJ/kg for pure FPUF to 22.5 MJ/kg for FPUF@PMo4 (Table S1 in the ESM), indicating a reduction in the actual heat released per unit mass of fuel vaporized. The fire growth potential, evaluated by the maximum average rate of heat emission (MARHE), was substantially lowered by approximately 65.8%

(Table S1 in the ESM), while the fire performance index (FPI) increased significantly (Fig. 4(c)). Notably, the FPI for the 3-hour immersion sample rose by over 217% (Fig. S5(c) in the ESM), reflecting enhanced flame retardancy. These quantitative improvements, combined with a marked increase in char yield (e.g., 29.71% for FPUF@PMo3, Fig. S4(b) in the ESM) and a reduced mass loss rate (Figs. S4(a) and S5(d) in the ESM), demonstrate that the PMo-PPy coating effectively protects the FPUF substrate through promoting char formation in the condensed phase.

In real fires, smoke obstructs visibility, hindering escape and rescue. Peak smoke production rate (pSPR) and total smoke release (TSR) are critical parameters for predicting fire hazards. The pSPR and total smoke production (TSP) of FPUF@PMo-PPy composites decreased significantly compared to pure FPUF (Fig. 4(d) and Table S2 in the ESM). The pSPR decreased progressively with increasing PMo-PPy loading, with FPUF@PMo4 exhibiting the lowest value, representing a 73.5% reduction. More notably, the TSR was dramatically suppressed from 227.93 m³/m² for pure FPUF to 12.69 m³/m² for FPUF@PMo4, indicating a profound reduction in the total amount of smoke generated. This smoke suppression trend was consistent with the effects of immersion time, where both SPR (Fig. S5(e) in the ESM) and TSP (Fig. S5(f) in the ESM) were markedly reduced. The smoke suppression performance was further investigated using smoke density testing. Figures 4(e) and 4(f) show that both the maximum smoke density ($D_{\max}$) and smoke density at 10 min ($D_{10\min}$) of the composites decreased progressively. FPUF@PMo4 achieved the lowest values, with its $D_{\max}$ (163) and $D_{10\min}$ (144) decreased by 55.7% and 60.8%, respectively, compared to pure FPUF. Furthermore, the smoke density per unit thickness ($D_{\min}$) also shows a consistent declining trend, dropping from 14.73 mm⁻¹ for pure FPUF to 6.52 mm⁻¹ for FPUF@PMo4 (Table S2 in the ESM). These collective results demonstrate the effective smoke suppression capability of the PMo-PPy coating across multiple evaluation metrics.

The protective effectiveness of the PMo-PPy coating was further evaluated under the higher heat flux intensities (Fig. S6 in the

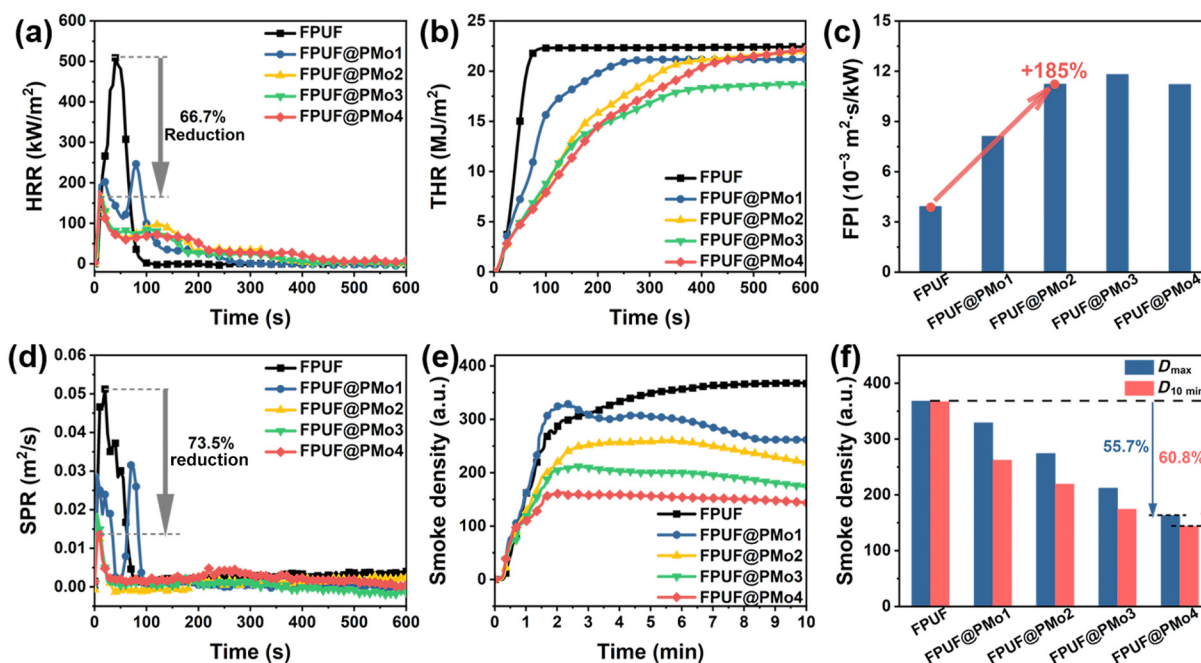


Figure 4 (a) HRR, (b) THR, (c) FPI, (d) SPR, (e) smoke density curves, and (f) characteristic data for pure FPUF and FPUF@PMo-PPy composites.

ESM). Across all conditions (35, 50, and 65 kW/m²), the coating consistently and significantly reduced peak heat release rate. Smoke production was substantially suppressed at heat fluxes up to 50 kW/m², and even under the highest heat flux (65 kW/m²), the coated sample maintained low smoke output during the early stage of combustion.

The critical role of the Py component in achieving this high performance was confirmed through comparative experiments. In the absence of Py, the loading of phosphomolybdic acid was low and its dispersion was poor (Fig. S3(f) in the ESM), resulting in inferior flame retardant and smoke suppression properties (Fig. S7 in the ESM). This finding underscores the essential function of Py in facilitating a high and uniform loading of the PMo-PPy hybrid coating, which is crucial for the formation of a robust protective barrier during combustion.

The flammability of the samples was evaluated by exposing the foams to a butane torch until flame extinction. As shown in Video S1 in the ESM, pure FPUF ignited immediately, burned vigorously with significant melt dripping—which ignited the underlying cotton—and was completely consumed (Fig. 5). In contrast, the PMo-PPy-coated foams exhibited restrained flaming and no melt dripping (Videos S2–S5 in the ESM). With increasing PMo-PPy loading, the resulting char residue maintained higher structural integrity. Notably, FPUF@PMo4 displayed self-extinguishing behavior: when ignited again from the opposite side at 24 s in Video S5 in the ESM, the flame self-extinguished before propagating across the entire surface. The results of limiting oxygen index (LOI) and horizontal flame tests are listed in Table S3 in the ESM. The LOI increased from 17.6% for pure FPUF to 22.3% for FPUF@PMo4, indicating a progressively enhanced resistance to ignition in an oxygenated atmosphere. In horizontal burning tests, while all coated samples exhibited burning rates between 2.1–2.5 mm/s, slightly higher than that of pure FPUF (1.6 mm/s).

The marked increase in LOI and the observed self-extinction, potentially attributed to the char-forming capability of the PMo-PPy coating during combustion, are indicative of the formation of a continuous protective char layer that enhances flame-retardant performance. This protective effect was further examined in the following condensed-phase analysis.

We compared its flame-retardant performance and fabrication efficiency with previously reported coating technologies for FPUF (Table S4 in the ESM). Conventional methods often involve multi-step procedures, precise parameter control, prolonged immersion times (up to days), or high mass gains. In contrast, this work requires only 3 h of immersion, achieves a mass gain of 20.6%, and delivers a 68.5% reduction in pHRR and an 84.0% reduction in TSP. These values surpass most literature data, demonstrating that the proposed method offers a promising strategy for high-performance flame-retardant foams.

3.3 Condensed phase analysis

The flame-retardant and smoke-suppression properties of polymer materials are closely linked to the morphology, structure, and chemical composition of the char residue. SEM images of the FPUF@PMo-PPy char residue at different magnifications, particularly in Figs. 6(a) and 6(b), reveal that the porous foam structure was largely preserved due to a protective char layer tightly surrounding the FPUF matrix. Figure 6(b) shows the protective carbon layer has an average thickness of 200–400 nm, with nanospheres of varying sizes uniformly embedded within the outermost layer. XRD analysis of the protective carbon layer (Fig. 6(c)) reveals reflections corresponding to the (020), (110), (040), (021), (111), and (060) planes of MoO₃ (PDF Card No. 35-0609). Raman spectrum of the protective carbon layer (Fig. 6(d)) shows peaks at 667, 822, and 996 cm⁻¹, assigned to MoO₃ stretching

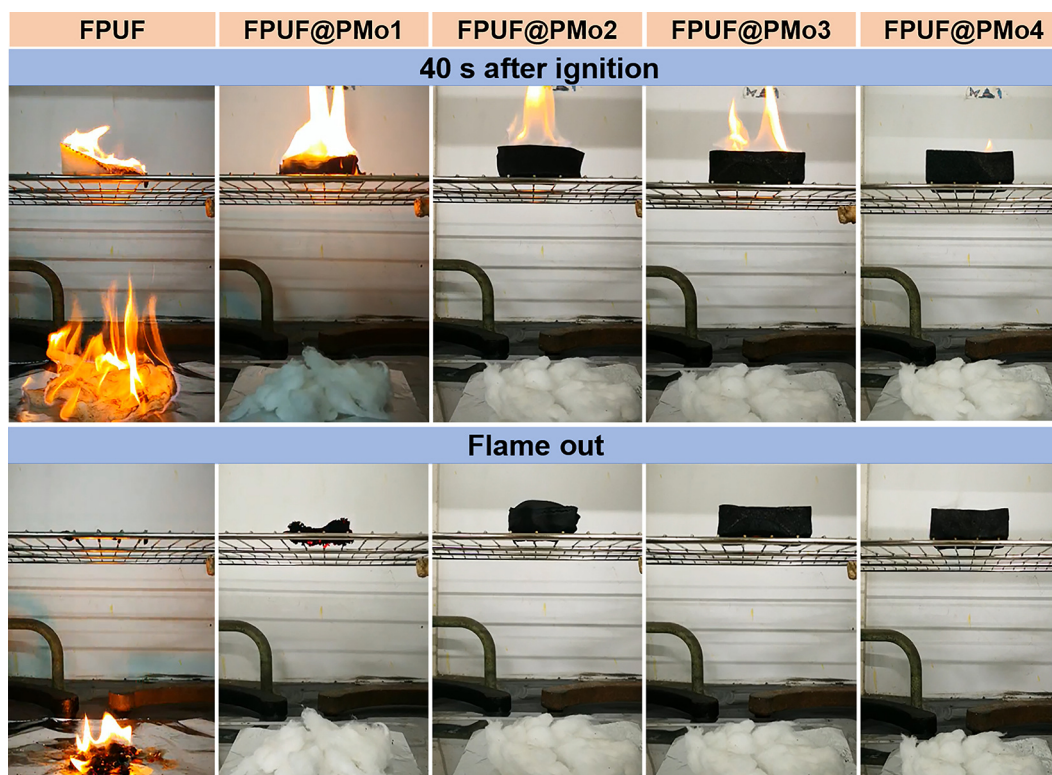


Figure 5 Photographs of pure FPUF and FPUF@PMo-PPy after ignition 40 s and flaming out.

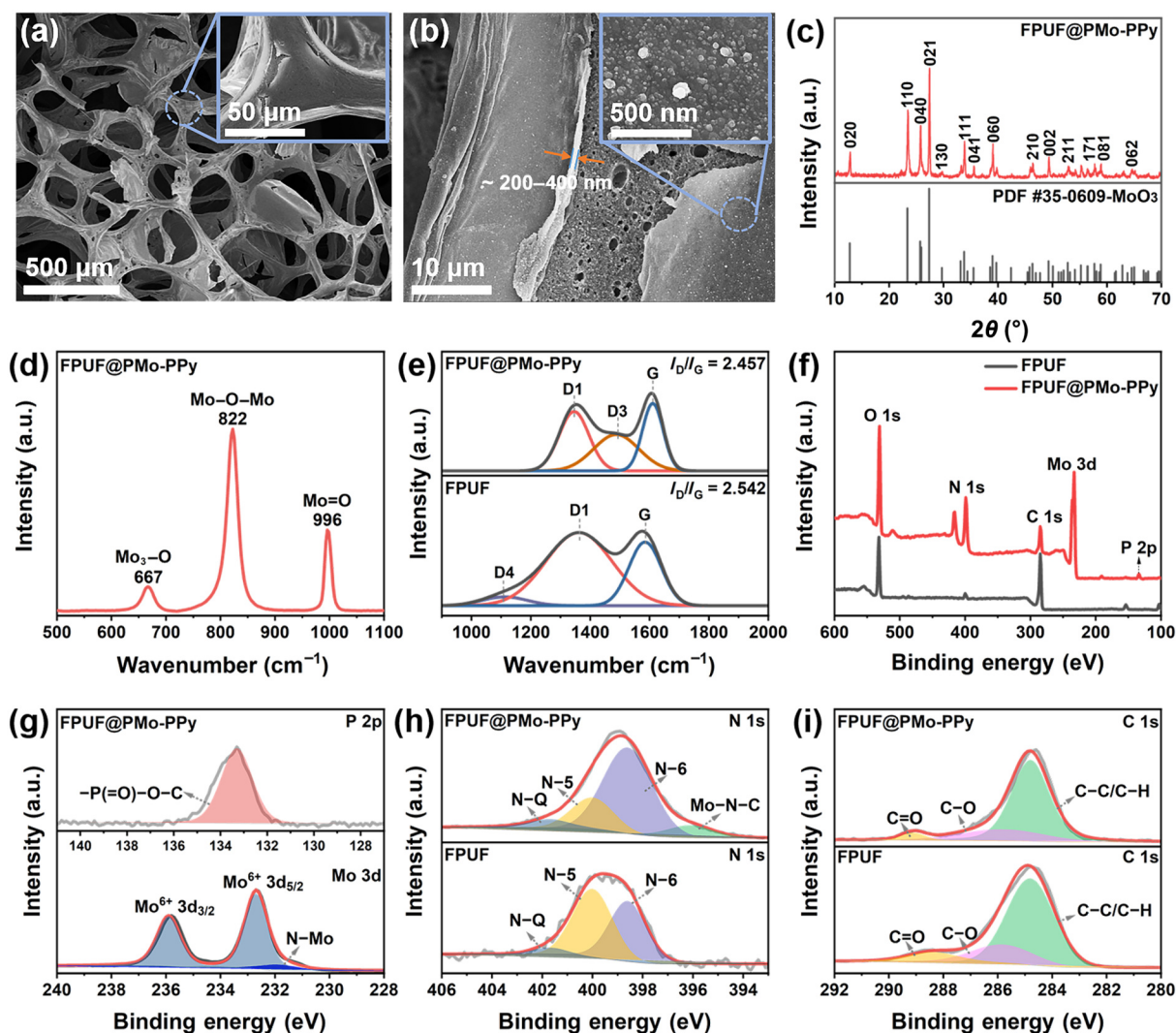


Figure 6 SEM images of the char residue of the FPUF@PMo-PPy composite after combustion at (a) low and (b) high magnification. (c) XRD patterns and ((d) and (e)) Raman spectra of the char residue for composites after cone calorimetry tests. (f) Full-XPS survey spectra and high-resolution XPS spectra of the char residues showing (g) P 2p and Mo 3d, (h) N 1s, and (i) C 1s regions for FPUF and FPUF@PMo-PPy composite.

modes of Mo₃-O (triply connected bridge-oxygen), Mo-O-Mo (doubly connected bridge-oxygen), and Mo=O (terminal oxygen), respectively. These results confirm MoO₃ formation during combustion.

To further elucidate the thermal decomposition behavior and evolution of MoO₃, temperature-dependent XRD analysis was conducted on PMo-PPy and FPUF@PMo-PPy composites (Figs. S8(a)–S8(c) in the ESM). The results indicate that MoO₃ forms when the temperature reaches 200–300 $^\circ\text{C}$, demonstrating that the PMo-PPy coating readily decomposes to generate MoO₃ upon heating. Furthermore, the CO oxidation capability of MoO₃ was also evaluated. As shown in Fig. S8(d) in the ESM, the conversion of CO initiates when the temperature exceeds 200 $^\circ\text{C}$, with the highest conversion efficiency observed between 300 and 400 $^\circ\text{C}$. These results are consistent with the TG-FTIR analysis presented in Fig. 7(e). This confirms that MoO₃ derived from the PMo-PPy coating possesses intrinsic CO oxidation capability, which contributes to reducing the toxicity of the smoke during combustion. In addition, MoO₃ acts as a Lewis acid, promoting char formation via Friedel-Crafts alkylation of alkene linkages, thereby enhancing flame retardancy and smoke suppression [39,

40].

Raman spectroscopy was also used to analyze the graphitization degree of char residue after cone calorimetry tests (Fig. 6(e)). To accurately deconvolute the complex spectrum of the disordered carbonaceous char, a multi-peak fitting model incorporating D1 ($\sim 1350\text{ cm}^{-1}$), D3 ($\sim 1500\text{ cm}^{-1}$), D4 ($\sim 1200\text{ cm}^{-1}$), and G ($\sim 1580\text{ cm}^{-1}$) bands was employed. The spectrum for pure FPUF char was fitted with D1, D4, and G bands, while that for FPUF@PMo-PPy char was fitted with D1, D3, and G bands. The appearance of the D3 band (associated with amorphous carbon) and the disappearance of the D4 band in the coated sample indicate a change in the chemical environment of the char. The I_D/I_G ratio (integrated intensity ratio of D and G bands) inversely correlates with graphitization degree. The significantly lower I_D/I_G ratio for FPUF@PMo-PPy composite (2.457) compared to FPUF (2.542) indicates that the PMo-PPy coating enhances char graphitization and compactness. This dense, continuous char layer effectively shields the polymer matrix, hindering heat transfer and the release of combustible volatiles.

XPS analysis elucidated the elemental composition and valence states of the char residues after cone calorimetry. Figure 6(f) shows

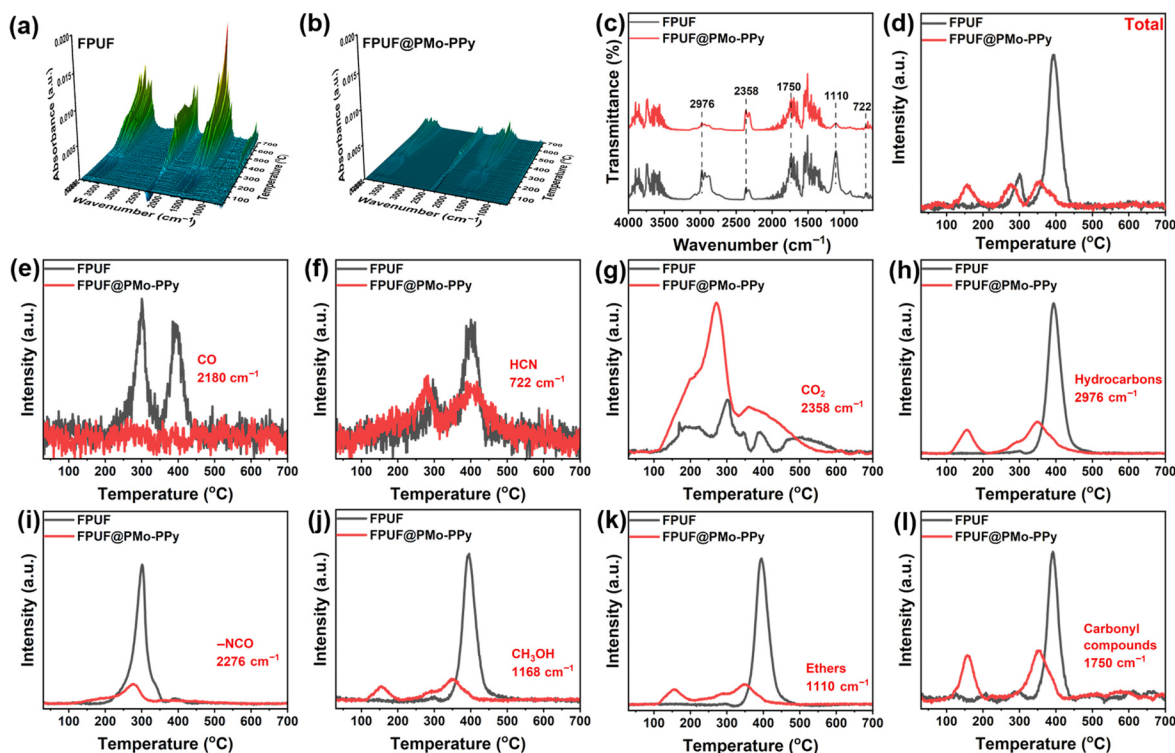


Figure 7 Three-dimensional TG-IR spectra of (a) pure FPUF and (b) FPUF@PMo-PPy composite. (c) FTIR spectra of their volatilized pyrolysis products at the maximum evolution rate. Absorption intensity of gaseous pyrolysis products: (d) Gram-Schmidt curves, (e) CO, (f) HCN, (g) CO₂, (h) hydrocarbons, (i) -NCO-containing compounds, (j) CH₃OH, (k) ethers, and (l) carbonyl compounds.

XPS full spectra indicating C, N, and O presence in both char residues. New P and Mo peaks in the FPUF@PMo-PPy char confirm PMo-PPy coating involvement in charring, with decomposition products retained. The P 2p spectrum (Fig. 6(g)) shows a peak at 133.3 eV attributable to P(=O)-O-C linkages, stabilizing the carbon residue. The high-resolution Mo 3d spectrum (Fig. 6(g)) reveals Mo⁶⁺ in MoO₃ (232.7 and 235.9 eV) and, notably, an N-Mo bond signal (231.9 eV, Mo 3d) in FPUF@PMo-PPy, indicating strong electron interaction between MoO₃ and N-containing compounds (N-carbon). Deconvolution of the N 1s spectra (Fig. 6(h)) shows peaks at 398.6 eV (pyridinic-N, N-6), 400 eV (pyrrolic-N, N-5), and 401.7 eV (graphitic-N, N-Q). The appearance of an N-Mo bond signal (396.1 eV, N 1s) further confirms strong interaction between MoO₃ and the N-carbon matrix. To further investigate the evolution of this interaction during thermal decomposition, temperature-dependent XPS analysis was conducted (Figs. S8(e) and S8(f) in the ESM). Upon heating to above 300 °C, the FPUF@PMo-PPy composite clearly exhibits the formation of Mo-N-C bonds. This result suggests that MoO₃, derived from the decomposition of phosphomolybdic acid, is highly dispersed within the matrix. Concurrently, pyridinic nitrogen generated during pyrolysis possesses lone-pair electrons and exhibits strong coordination capability, actively bonding with molybdenum atoms to form N-Mo coordination bonds. The condensation degree of the polyaromatic network can be assessed by the N-Q/(N-6 + N-5) ratio (Table S5 in the ESM). The higher N-Q/(N-6 + N-5) value (0.087) for FPUF@PMo-PPy implies a more stable char layer, consistent with Raman analysis. C 1s spectra (Fig. 6(i)) show peaks at 284.8 eV (C-C, C-H), 285.8 eV (C-O), and 289 eV (C=O). The C_{ox}/C_a ratio (C_{ox}: oxidized carbons; C_a: aliphatic/aromatic carbons) reflects the thermal oxidative resistance

of the char residue. The lower C_{ox}/C_a value for FPUF@PMo-PPy (Table S6 in the ESM) indicates improved thermal oxidative resistance imparted by PMo-PPy. These analyses confirm that N, P, and Mo elements from PMo-PPy enhance the compactness, stability, and thermal oxidative resistance of the char layer. This robust char acts as a protective shield in the condensed phase, slowing down the combustion process and thereby enhancing flame retardancy and smoke suppression.

3.4 Thermal decomposition behaviors and gaseous phase analysis of FPUF@PMo-PPy composites

Thermal decomposition behaviors of pure FPUF and FPUF@PMo-PPy composites were investigated under both inert (nitrogen) and oxidative (air) atmospheres using thermogravimetric analysis (TGA). Under the nitrogen atmosphere (Fig. S9 and Table S7 in the ESM), the incorporation of PMo-PPy notably reduced the onset decomposition temperature ($T_{5\%}$) from 256.9 °C for pure FPUF to 146.6 °C for FPUF@PMo4, and similarly lowered the temperature at maximum decomposition rate (T_{max}). Concurrently, the char residue at 700 °C increased substantially from 2.64 wt.% for FPUF to 36.94 wt.% for FPUF@PMo4, indicating a dramatically enhanced char-forming ability. This apparent decrease in the onset decomposition temperature is, in fact, a crucial aspect of the coating's flame-retardant strategy [41, 42]. The premature degradation of the PMo-PPy coating itself initiates the formation of a protective char layer at a lower temperature. This char layer effectively insulates the underlying FPUF matrix, thereby enhancing the material's safety and stability during exposure to high temperatures (> 400 °C) in a fire scenario by reducing heat and mass transfer. Under the air atmosphere (Fig. S9 and Table S8 in the ESM), the composites also exhibited higher residue yields and

shifted decomposition profiles, consistent with promoted thermo-oxidative char formation. The increased char yield is critical for fire retardancy, as it can reduce the release of flammable volatiles and act as a protective barrier during combustion.

Gaseous degradation products from FPUF and FPUF@PMo-PPy were analyzed by TG-IR under a nitrogen atmosphere. Three-dimensional FTIR spectra and FTIR spectra of volatilized pyrolysis products at maximum evolution rate are shown in Figs. 7(a)–7(c). The types of gaseous products were similar for both samples, identifiable by FTIR signals: water ($-\text{OH}$, $3450\text{--}3800\text{ cm}^{-1}$), hydrocarbons (C-H , $2870\text{--}2982\text{ cm}^{-1}$), CO_2 (2358 cm^{-1}), carbonyl compounds (C=O , 1750 cm^{-1}), and CH_3OH (1168 cm^{-1}). However, compared to FPUF, the absorption intensity and temperature profiles of characteristic peaks for FPUF@PMo-PPy changed significantly. The Gram–Schmidt curves (Fig. 7(d)) show FPUF has two major peaks between $250\text{--}320$ and $320\text{--}450\text{ }^\circ\text{C}$, corresponding to volatile release during decomposition. The thermal decomposition of FPUF@PMo-PPy occurred in three stages, which is consistent with the TGA (N_2) results shown in Fig. S9 in the ESM. In the first stage ($100\text{--}200\text{ }^\circ\text{C}$), the early decomposition of the PMo-PPy coating was observed, accompanied by the release of a small amount of gaseous products, as indicated by the lower FTIR peak intensities of evolved gases. The second and third stages correspond mainly to the decomposition of the FPUF substrate itself.

The effect of PMo-PPy on specific gaseous products was selectively studied. Figures 7(e)–7(l) show significant reductions in organic volatiles: hydrocarbons (2976 cm^{-1}), -NCO-containing compounds (2276 cm^{-1}), carbonyl compounds, CH_3OH , and ethers (C-O-C , 1110 cm^{-1}). The reduction in flammable gas generation, which curtails fuel supply to the flame, directly contributes to the observed decreases in pHRR and THR, while the simultaneous suppression of smoke precursors leads to lower smoke production. Notably, toxic gases harmful to human health, including HCN and

CO, were significantly suppressed, indicating PMo-PPy effectively reduces smoke toxicity. Conversely, CO₂ release from FPUF@PMo-PPy was much higher than from FPUF, due to the oxidation of CO by MoO₃. This increased CO₂ dilutes flammable gases in the gas phase, contributing to flame retardancy and smoke suppression.

3.5 Mechanism of flame retardancy and smoke suppression

Based on the preceding analyses, the proposed mechanism for the PMo-PPy nanocoating on FPUF is illustrated in Fig. 8. Initially, PMo-PPy nanospheres uniformly coat the FPUF framework surface. Upon heating, the PMo-PPy coating itself decomposes first, releasing decomposition products such as phosphorus-containing or molybdenum-containing compounds. These active species then promote the subsequent degradation of the FPUF substrate, facilitating the rapid cross-linking of decomposition intermediates. This ultimately leads to the formation of a stable, compact char layer incorporating -P(=O)-O-C and Mo-N-C chemical bonds. This char layer effectively impedes heat and mass exchange between the flame zone and the underlying polymer matrix, while also contributing to smoke particle retention. Simultaneously, the secondary free radicals ($\text{H}\cdot$, -OH) are gradually captured by the generated free radicals ($\text{PO}\cdot$ and $\text{PO}_2\cdot$), effectively slowing down the combustion rate and blocking the chain reaction [43]. Furthermore, the oxidation of CO by MoO_3 leads to a higher yield of non-flammable CO_2 , which dilutes combustible volatiles in the gas phase and further suppresses combustion. Consequently, FPUF@PMo-PPy composites achieve superior flame retardancy and smoke suppression.

3.6 Compression performance and water resistance

Compression tests of the coated foam were evaluated to assess the practical performance trade-offs. Specifically, the compressive stress of the FPUF@PMo-PPy composite demonstrates a notable increase

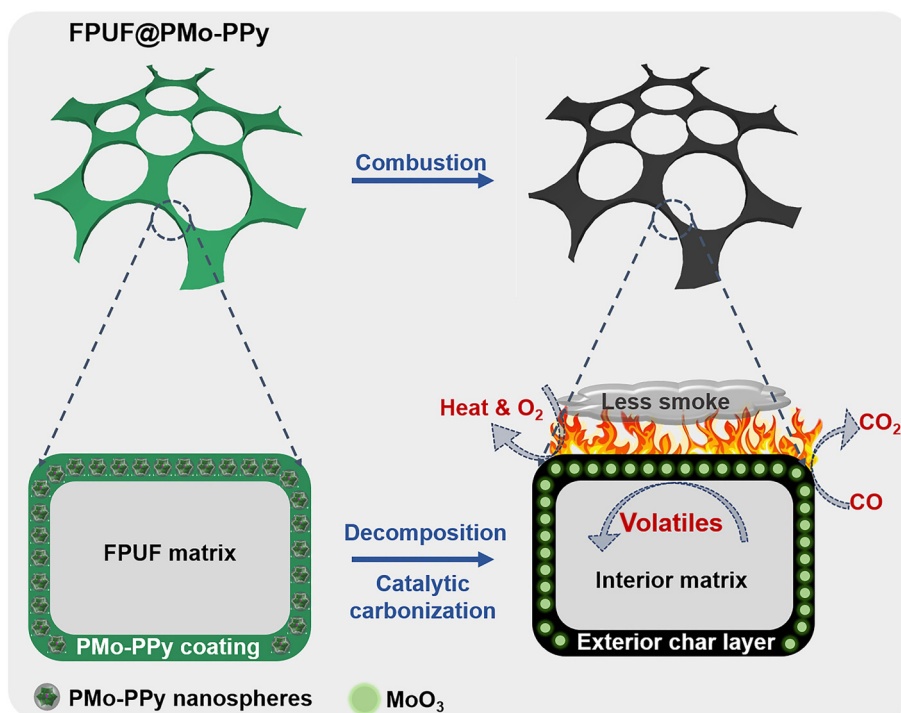


Figure 8 Proposed mechanism of PMo-PPy nanocoating for improving flame retardancy and smoke suppression of FPUF.

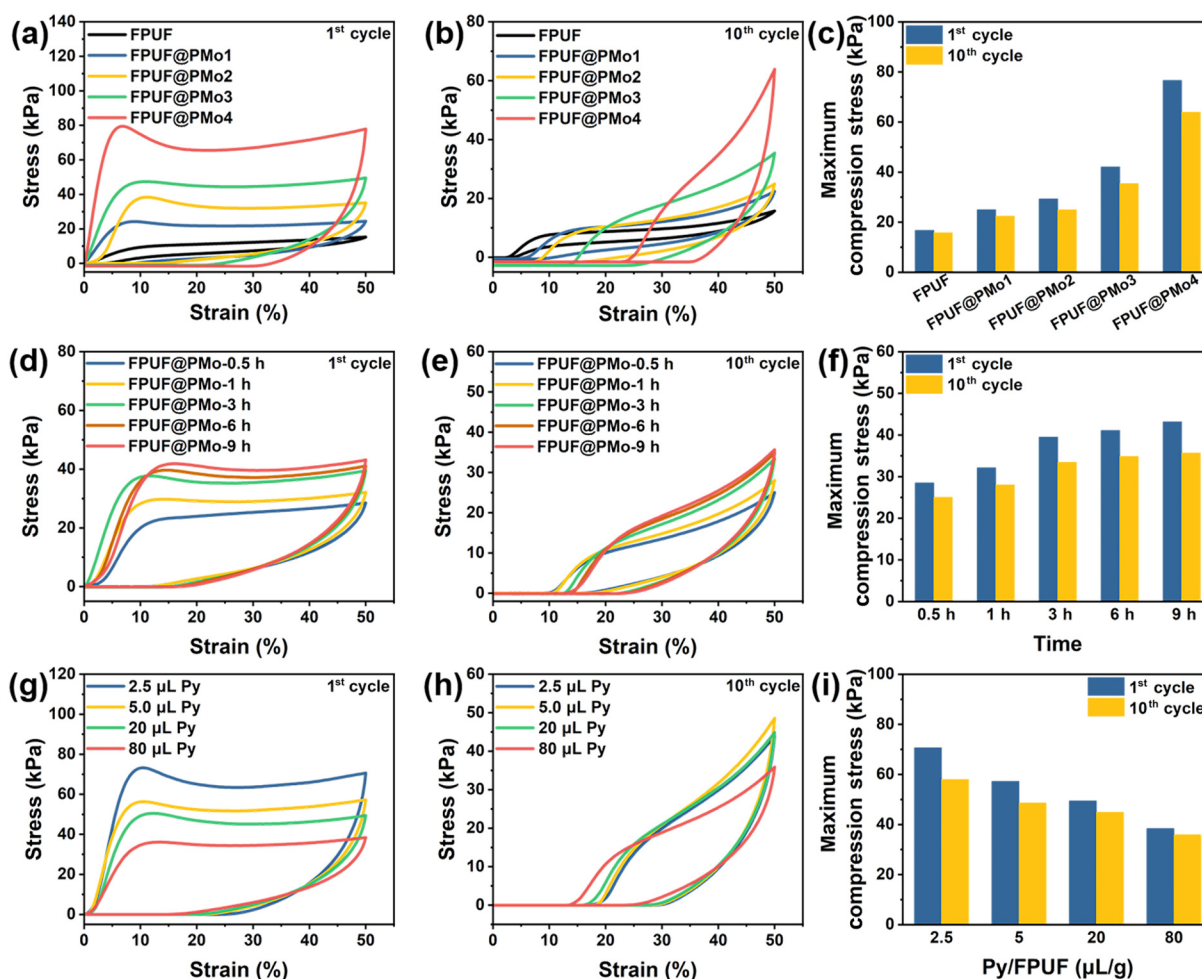


Figure 9 Compressive properties of FPUF and FPUF@PMo-PPy. Effect of PMo₁₂ content: (a) and (b) stress-strain curves (1st and 10th cycle, 50% strain) and (c) maximum stress. Effect of impregnation time: (d) and (e) Stress-strain curves (1st and 10th cycle, 50% strain) and (f) maximum stress. Effect of Py amount: (g) and (h) stress-strain curves (1st and 10th cycle, 50% strain) and (i) maximum stress.

with either a higher phosphomolybdic acid content or a longer impregnation time (Fig. 9), indicating the PMo₁₂ with a rigid structure can improve the compression resistance of FPUF [44]. In contrast, increasing the amount of Py monomer led to a gradual decrease in compressive stress (Fig. 9). As a flexible component, PPy improved the flexibility of the coated FPUF. In most cases, the maximum compressive strength decreased after multiple compression cycles, which is consistent with the trend reported for other coating methods in the literature (Table S9 in the ESM). For instance, the FPUF@PMo-3 h sample exhibited an increase in initial maximum compressive stress from 16.7 to 39.5 kPa, which then declined to 33.4 kPa after 10 cycles, retaining approximately 85% of its initial enhanced value. These trends are attributed to the competitive effects of coating mass, network reinforcement, and structural uniformity [45]. The coating also exhibited excellent resistance to water. No visible peeling was observed after repeated mechanical squeezing in water (Video S6 in the ESM), and the coating remained intact even after 2 h of ultrasonication (Fig. S10 in the ESM; FPUF@PMo-3 h refers to the sample prepared with a 3 h impregnation time). Furthermore, the flame-retardant and smoke-suppression properties of the ultrasonicated samples were largely preserved (Fig. S11 in the ESM). This robust interfacial stability is attributed to the strong electrostatic interactions between the PMo-PPy coating and the FPUF substrate.

4 Conclusions

In summary, we have developed a facile strategy for depositing PMo-PPy nanospheres onto FPUF. Multiple characterizations confirmed the successful and uniform nanocoating. The content of loaded PMo-PPy nanosphere is tunable by varying the concentration of H₃PMo₁₂O₄₀ aqueous solution. In terms of functional performance, the PMo-PPy coating modifies the decomposition behavior of FPUF, promoting the formation of a protective char layer which in turn improves its fire performance and prevents melt dripping during ignition. Cone calorimetry and smoke density tests demonstrated substantial fire hazard reduction: FPUF@PMo-PPy exhibited 66.7% and 38.1% reductions in pHRR and THR at flame out, respectively, while FPUF@PMo-PPy showed 73.5%, 55.7%, and 60.8% reductions in pSPR, $D_{\max}$, and $D_{10 \min}$, respectively, compared to pure foam. Furthermore, the release of CO and HCN toxic compounds was markedly suppressed, indicating the reduction of fire toxicity. The enhanced performance is attributed to the formation of a stable, compact char layer incorporating -P(=O)-O-C and N-Mo chemical bonds, which effectively blocks flammable gases, smoke particles, and heat transfer, thereby achieving superior flame retardancy and smoke suppression. This work demonstrates the potential of a functional coating to improve the flame retardancy and smoke suppression of

flexible polyurethane foam, providing insights for industrial practice.

Electronic Supplementary Material: Supplementary material (experimental materials, synthesis of composites, characterization and measurements, weight gain, SEM images and EDX mapping images of composites, test data related to cone calorimeter, TGA, XPS spectra and XRD patterns of composites, CO conversion curves of MoO₃, water resistance, table of LOI, burning rate, cone calorimeter and compression properties, comparison of flame retardancy performances with other coating technologies) is available in the online version of this article at <https://doi.org/10.26599/NR.2026.94908619>.

Data availability

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

Acknowledgements

This work is financially supported by the National Natural Science Foundation of China (No. 22288102), the Zhejiang Provincial Natural Science Foundation of China (No. LMS25B060002), and the Quzhou Municipal Science and Technology Bureau (No. 2024K015).

Declaration of competing interest

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

Author contribution statement

H. Y. R.: Methodology, investigation, experimental design, data curation, writing – original draft, writing – review & editing. X. Y. J. and W. Z.: Investigation, writing – review & editing, supervision. Y. Y.: Methodology, investigation, project administration, funding acquisition. Y. J. L.: Conceptualization, writing – review & editing, supervision, project administration, funding acquisition. All the authors have approved the final manuscript.

Use of AI statement

None.

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