

# Copper-centered Anderson-type polyoxomolybdates as high-efficiency flame retardants for epoxy resins

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Received: 25 July 2026; Revised: 17 August 2026; Accepted: 21 August 2026

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Cite This: *Polyoxometalates*, 2027, 6, 9140155



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**ABSTRACT:** The increasing severity of fire-related hazards necessitates the development of advanced polymeric materials with integrated flame retardancy and smoke suppression. In this work, metal-centered Anderson-type polyoxomolybdates (POMs) are explored as multifunctional additives for epoxy resins (EP) to address both flammability and toxic fume emissions. A comparative study of a main-group aluminum-centered Anderson-type POMo (AlMo<sub>6</sub>) and its transition-metal copper-centered analogue (CuMo<sub>6</sub>) reveals the superior performance of the copper-containing system. Specifically, EP composites incorporating CuMo<sub>6</sub> exhibit markedly improved limiting oxygen index (LOI), significantly reduced peak heat release rate (p-HRR), and substantially suppressed carbon monoxide emissions during combustion. Raman spectroscopy of the residual chars indicates that CuMo<sub>6</sub> promotes a higher degree of graphitization, supporting copper-catalyzed carbonization. Furthermore, encapsulation of the inorganic POM clusters with hexadecyltrimethylammonium bromide (CTAB) markedly enhances their dispersion within the hydrophobic epoxy matrix, as confirmed by scanning electron microscopy (SEM). Thermogravimetric analysis (TGA) coupled with FTIR spectroscopy demonstrates that CTAB-modified CuMo<sub>6</sub> not only improves the thermal stability of the EP composites but also effectively reduces the release of flammable volatile decomposition products. This enhanced performance is primarily attributed to the catalytic action of CuMo<sub>6</sub>, which facilitates the formation of a dense, continuous, and thermally stable char barrier that limits heat and mass transfer during combustion.

**KEYWORDS:** Anderson-type polyoxomolybdates, CuMo<sub>6</sub>, epoxy resin, flame retardancy

## 1 Instruction

Epoxy resins (EP) are pivotal materials in modern industry owing to their excellent dielectric properties, high adhesion, and outstanding chemical resistance. Over the past few decades, EP has been extensively used in magnetic materials, high-performance adhesives, electronic encapsulation, and protective coatings [1]-[4]. However, its intrinsic flammability and substantial smoke emission during combustion severely limit its use in applications with stringent fire-safety requirements [5][6]. Dense smoke often poses a greater threat to life and property than the flame itself because of its toxicity and ability to obscure visibility. Consequently, enhancing both the flame retardancy and smoke suppression of EP has become an urgent objective [7][8].

Polyoxometalates (POMs) have recently attracted considerable attention across catalysis, magnetic materials, energy storage, and flame retardancy [9]. Most reported POM-based synergistic flame-retardant systems focus on Keggin-type POMs [10][14], whereas other structural variants have

remained comparatively underexplored [15][16]. Among the six classical POM structures, the Anderson-type ( $[\text{H}_2(\text{XO}_6)_6\text{M}_6\text{O}_{18}]^{n-}$ , X = central heteroatom, M = Mo<sup>VI</sup> or W<sup>VI</sup>) feature a distinctive structure with a central {XO<sub>6</sub>} octahedron surrounded by six edge-sharing {MO<sub>6</sub>} octahedra [17][18]. Prior studies have shown that Anderson-type POMs can act as effective precursors to metal oxides that catalyze polymer carbonization during thermal decomposition, thereby promoting the formation of a dense, thermally stable char layer [19]. However, the inorganic nature of POMs limits their compatibility with polymer matrices, substantially compromising their flame-retardant efficiency [20]-[23].

To improve dispersion and interfacial compatibility, strategies involving the covalent linkage of organic components have been employed. For example, Xu et al. developed an Anderson-type POM-based hybrid flame retardant by covalently linking organic molecules (bisDOPA) to Na-AlMo<sub>6</sub>, thereby improving its dispersion in EP [24]. Peng et al. grafted polyphosphazene onto NH<sub>4</sub>-AlMo<sub>6</sub>, simultaneously enhancing dispersion, mechanical properties, and flame retardancy in EP [25]. These approaches, however,

often require complex monomer syntheses and multi-step procedures. Therefore, there is an urgent need for simpler, more scalable routes to enhance the compatibility of Anderson-type POMs with the epoxy matrix.

Recent studies show that quaternary ammonium salts, widely used as cationic surfactants, possess chemical and physical attributes that enable substantial tuning of particle surface properties, underpinning their broad use in modifying inorganic fillers [26][33]. Their cationic moieties promote strong interfacial interactions with particle surfaces beyond simple physical adsorption [34]. Specifically, these modifiers regulate surface charge and tailor surface chemistry [35], bridging the polarity gap between inorganic particles and organic polymer matrices to enhance interfacial compatibility. Through facile ion exchange with quaternary ammonium cations, the interparticle interactions among highly surface-active inorganic POMs can be mitigated, facilitating uniform dispersion throughout the epoxy matrix.

In this study, two Anderson-type polyoxomolybdates (POMs) ( $\text{NH}_4\text{-AlMo}_6$  and  $\text{NH}_4\text{-CuMo}_6$ ) were investigated as flame retardants for EP. Limiting oxygen index (LOI) and cone calorimetry results show that the copper-centered Anderson-type POM ( $\text{NH}_4\text{-CuMo}_6$ ) exhibits better performance compared with its main-group aluminum-centered Anderson-type POMo ( $\text{AlMo}_6$ ). Raman spectra indicate that the  $\text{NH}_4\text{-CuMo}_6$  promotes a higher degree of graphitization in the residual char owing to the Cu-catalyzed carbonization. The dispersion of  $\text{NH}_4\text{-CuMo}_6$  in the epoxy matrix was further improved by modification with hexadecyltrimethylammonium bromide (CTAB). Thermogravimetric analyses (TGA) and TGA coupled with Fourier transform infrared spectroscopy (FTIR) demonstrate enhanced thermal stability and reduced release of flammable volatiles in EP after incorporation of CTAB-encapsulated  $\text{CuMo}_6$  (CTAB/ $\text{CuMo}_6$ ).

## 2 Experimental section

### 2.1 Materials

Ammonium molybdate tetrahydrate ( $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ ) was supplied by Beijing Inokai Science and Technology Co.,

Ltd. Anhydrous copper sulfate ( $\text{CuSO}_4$ ) and aluminum sulfate ( $\text{Al}_2(\text{SO}_4)_3$ ) were obtained from Shantou Guangxilong Chemical Factory. Cetyltrimethylammonium bromide (CTAB) was purchased from Saen Chemical Technology (Shanghai) Co., Ltd. The Anderson-type POMs ( $(\text{NH}_4)_4[\text{CuMo}_6\text{O}_{24}\text{H}_6]\cdot 5\text{H}_2\text{O}$  ( $\text{NH}_4\text{-CuMo}_6$ ) and  $(\text{NH}_4)_3[\text{AlMo}_6\text{O}_{24}\text{H}_6]\cdot 7\text{H}_2\text{O}$  ( $\text{NH}_4\text{-AlMo}_6$ ) were prepared according to the literature method [16]. Diethyltoluenediamine (DETDA) was purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. Bisphenol A diglycidyl ether (D.E.R. 332, BADGE) and 4,4'-diaminodiphenylmethane (DDM, > 97.0%) were obtained from Sigma Aldrich.

### 2.2 Preparation of CTAB/ $\text{CuMo}_6$

CTAB (0.90 g) was dissolved in anhydrous ethanol under stirring to give a clear solution.  $\text{NH}_4\text{-CuMo}_6$  (0.39 g), dispersed in anhydrous ethanol, was then added to the CTAB solution, and the mixture was stirred for 20 min, during which a fine precipitate formed. The resulting mixture was ultrasonically dispersed at 50 °C for 20 min and subsequently vacuum-filtered. The collected solid was dried under vacuum at 100 °C for 12 h, and is denoted as CTAB/ $\text{CuMo}_6$ .

### 2.3 Preparation of EP and its composites

Neat epoxy and its composites (EP/ $\text{AlMo}_6$ , EP/ $\text{CuMo}_6$ , and EP/CTAB/ $\text{CuMo}_6$ -x, where x is the mass fraction of CTAB/ $\text{CuMo}_6$ ) were prepared by thermal curing. Using EP/ $\text{CuMo}_6$  as an example, BADGE (30 g) and  $\text{NH}_4\text{-CuMo}_6$  (1.186 g, 3 wt%) were dispersed in excess ethanol at 50 °C under vigorous stirring. After a homogeneous dispersion was obtained, the ethanol was removed by rotary evaporation at 40 °C. The curing agent DDM (or DETDA for EP/CTAB/ $\text{CuMo}_6$ -x) was then added with continuous stirring. Owing to the high reactivity of DDM, premature crosslinking occurs during long-time stirring, resulting in excessive viscosity. Thus, DETDA was selected for EP/CTAB/ $\text{CuMo}_6$ -x samples to achieve sufficient filler dispersion and homogeneous curing. The mixture was degassed under vacuum and poured into a preheated PTFE mold for curing. The curing schedule was 100 °C for 2 h followed by 150 °C for 2 h. Formulations for EP and its composites are summarized in Table 1.

**Table 1** Formulation of EP and its composites.

| Samples                       | BADGE<br>(wt.%) | $\text{CuMo}_6$<br>(wt.%) | $\text{AlMo}_6$<br>(wt.%) | CTAB/ $\text{CuMo}_6$<br>(wt.%) | Curing agent<br>(wt.%) <sup>a</sup> |
|-------------------------------|-----------------|---------------------------|---------------------------|---------------------------------|-------------------------------------|
| EP                            | 78.2            | 0                         | 0                         | 0                               | 21.8                                |
| EP/ $\text{CuMo}_6$           | 75.9            | 3                         | 0                         | 0                               | 21.0                                |
| EP/ $\text{AlMo}_6$           | 75.9            | 0                         | 3                         | 0                               | 21.0                                |
| EP/CTAB/ $\text{CuMo}_6$ -0.5 | 77.8            | 0                         | 0                         | 0.5                             | 21.7                                |
| EP/CTAB/ $\text{CuMo}_6$ -1   | 77.4            | 0                         | 0                         | 1                               | 21.6                                |
| EP/CTAB/ $\text{CuMo}_6$ -2   | 76.6            | 0                         | 0                         | 2                               | 21.4                                |

<sup>a</sup> DDM for EP/ $\text{XMo}_6$  (X=Cu, Al) and their corresponding neat EP, whereas DETDA for EP/CTAB/ $\text{CuMo}_6$ -x and their neat EP.

### 2.4 Characterizations

The chemical structures of the specimens were characterized using a VERTEX70 Fourier transform infrared (FTIR)

spectrometer. The morphological features of the samples and their residues after combustion were examined using a Zeiss Supra 55 scanning electron microscope (SEM) operating at an

acceleration voltage of 20 kV.

The fire safety performance was systematically assessed. The limiting oxygen index (LOI) was determined according to ASTM D2863 using an oxygen index analyzer (Fire Testing Technology), with bar dimensions of 100 mm × 6.5 mm × 3 mm (n=5). Vertical burning behavior was evaluated via the UL-94 test (Motis Fire Technology) according to ASTM D3801, using bars of 125 mm × 13 mm × 3 mm (n=5). Combustion parameters, including heat release rate and smoke production, were obtained from cone calorimetry tests (Fire Testing Technology) performed in accordance with ISO 5660. Square samples (100 mm × 100 mm × 3 mm) were exposed to a radiant flux of 35 kW·m<sup>-2</sup> until flameout.

The thermal stability of the specimens was evaluated by thermogravimetric analysis (TGA). Primary TGA and derivative thermogravimetry (DTG) were conducted on a TA Q600 instrument, while complementary TGA data were acquired using a Netzsch STA 449 F3 Jupiter thermobalance to verify reproducibility. Both measurements were performed from room temperature to 800 °C at a heating rate of 10 °C/min under a nitrogen atmosphere. Additionally, the evolution of pyrolysis products was monitored in real-time via thermogravimetry-Fourier transform infrared (TG-FTIR) spectroscopy using a Mettler Toledo TGA/DSC 3+ instrument under identical heating conditions. Thermal transitions were analyzed by differential scanning calorimetry (DSC) on a TA Q600 instrument. The samples were heated from ambient temperature to 300 °C at a rate of 10 °C/min under nitrogen, and the data from the second heating cycle were used to determine the glass transition temperature.

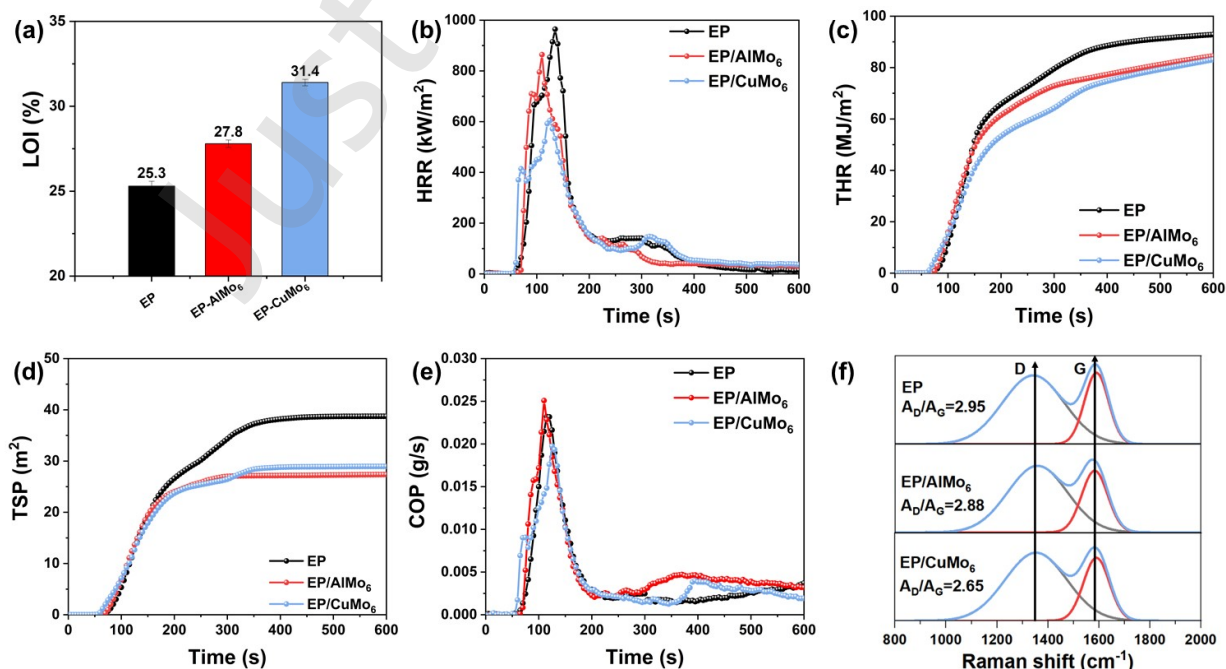
### 3 Results and discussion

#### 3.1 Flame retardant properties of EP and its composites

Fig. 1(a) presents the limiting oxygen index (LOI) values for neat EP and its composites, EP/AlMo<sub>6</sub> and EP/CuMo<sub>6</sub>. Neat EP

exhibited an LOI value of 25.3%, while the incorporation of 3 wt.% of NH<sub>4</sub>-AlMo<sub>6</sub> increased this value to 27.8%. A more substantial increase to 31.4% was achieved with the EP/CuMo<sub>6</sub> composite. These improvements are corroborated by cone calorimetry results, where the addition of the Anderson-type polyoxometalates significantly decreased both the peak heat release rate (*p*-HRR) and total heat release (THR) relative to pure epoxy (Figs. 1b and 1c). EP/CuMo<sub>6</sub> displayed the most pronounced suppression of combustion intensity, reducing the *p*-HRR from 964.41 kW·m<sup>-2</sup> for pristine EP to 604.4 kW·m<sup>-2</sup>. Furthermore, the polyoxometalates demonstrated high efficiency in mitigating smoke hazards, evidenced by reductions in time-dependent total smoke production (TSP) and carbon monoxide emissions (Figs. 1d and 1e). Notably, EP/CuMo<sub>6</sub> achieved the greatest reduction in the peak carbon monoxide production rate (COP), lowering it from 0.023 g/s for neat epoxy to 0.018 g/s. These assessments demonstrate that incorporating the Anderson-type polyoxometalates significantly enhances the flame retardancy of the epoxy matrix, with the EP/CuMo<sub>6</sub> composite exhibiting superior performance across all metrics.

To elucidate the mechanism underlying the enhanced flame retardancy, Raman spectroscopy was employed to evaluate the graphitization degree of the char residues from the cone calorimetry tests (Fig. 1f). A decreased area ratio of the D band (disordered carbon) to the G band (graphitic carbon), indicating a higher degree of graphitization, was observed in the chars of both EP/AlMo<sub>6</sub> and EP/CuMo<sub>6</sub>. The highest reduction in EP/CuMo<sub>6</sub>, where the ratio dropped from 2.95 for EP to 2.65, signifies the formation of a more ordered and graphitized char structure. This robust char layer acts as an effective physical barrier against heat and mass transfer, thereby inhibiting combustion and emission of toxic smoke.



**Figure 1** (a) Limiting oxygen index (LOI), (b) heat release rate (HRR), (c) total heat release (THR), (d) total smoke production (TSP), and (e) carbon monoxide production rate (COP) of EP and its composites, EP/AlMo<sub>6</sub> and EP/CuMo<sub>6</sub>. (f) Raman spectra of the residual chars from the cone calorimetry

tests.

The collective results (Fig. 1a–f) demonstrate that both  $\text{AlMo}_6$  and  $\text{CuMo}_6$  enhance the flame retardancy and fire safety of epoxy resin (EP), as evidenced by key performance indicators such as limiting oxygen index, heat release parameters, smoke and CO production, and char residue

quality. Notably,  $\text{CuMo}_6$  outperforms  $\text{AlMo}_6$  in most aspects, which can be attributed to its superior ability to catalyze the formation of a high-quality, graphitized char barrier. These findings confirm the greater potential of  $\text{CuMo}_6$  as an efficient flame-retardant filler for epoxy composites.

**Table 2** LOI CCT and Raman data of EP and its composites ( $35 \text{ kW}\cdot\text{m}^{-2}$ ).

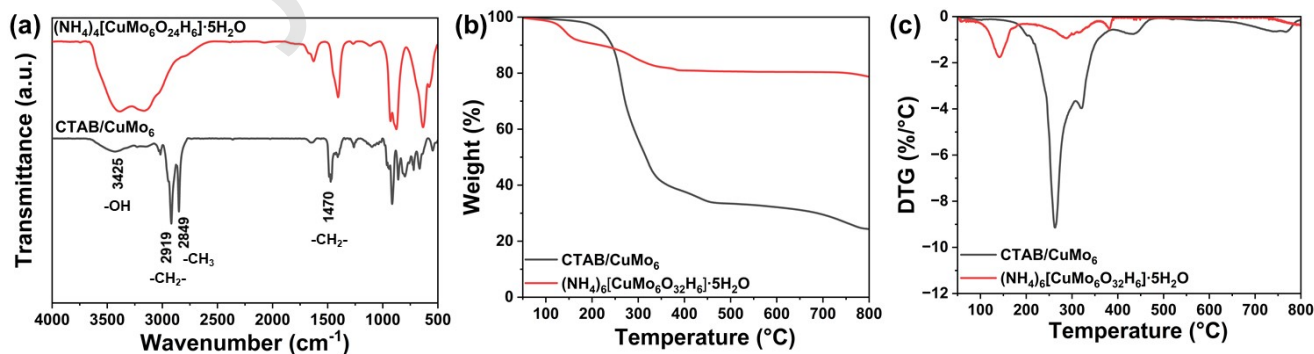
| Samples             | LOI  | $p\text{-HRR}$<br>( $\text{kW}\cdot\text{m}^{-2}$ ) | THR<br>( $\text{MJ}\cdot\text{m}^{-2}$ ) | TSP<br>( $\text{m}^2$ ) | $I_D/I_G$ |
|---------------------|------|-----------------------------------------------------|------------------------------------------|-------------------------|-----------|
| EP                  | 25.3 | 964.41                                              | 92.84                                    | 38.74                   | 2.95      |
| EP/ $\text{CuMo}_6$ | 31.4 | 604.40                                              | 84.37                                    | 28.95                   | 2.65      |
| EP/ $\text{AlMo}_6$ | 27.8 | 864.28                                              | 83.08                                    | 27.36                   | 2.88      |

### 3.2 Characterization of CTAB/ $\text{CuMo}_6$

Despite the remarkable flame-retardant efficiency of the Anderson-type polyoxometalates, the inherent incompatibility between these inorganic particles and organic matrices remains a critical challenge. To address this, surface modification using surfactants is a widely adopted strategy to enhance interfacial adhesion. Herein, hexadecyl trimethyl ammonium bromide (CTAB) was adopted to modify the surface of  $(\text{NH}_4)_4[\text{CuMo}_6\text{O}_{24}\text{H}_6]\cdot 5\text{H}_2\text{O}$ , which had demonstrated superior flame-retardant performance. CTAB modifies  $\text{CuMo}_6$  via electrostatic attraction between its cationic groups and negatively charged POM surfaces. The exposed alkyl chains reduce filler-matrix polarity mismatch, alleviate particle agglomeration, and strengthen interfacial compatibility with epoxy, enabling uniform dispersion of flame-retardant fillers. The structure of the resulting hybrid, CTAB/ $\text{CuMo}_6$ , was analyzed by Fourier transform infrared spectroscopy (FTIR). Four characteristic peaks at  $930 \text{ cm}^{-1}$ ,  $878 \text{ cm}^{-1}$ ,  $637 \text{ cm}^{-1}$ , and  $576 \text{ cm}^{-1}$ , observed in both CTAB/ $\text{CuMo}_6$  and  $(\text{NH}_4)_4[\text{CuMo}_6\text{O}_{24}\text{H}_6]\cdot 5\text{H}_2\text{O}$ , are attributed to the stretching vibrations of terminal  $\text{Mo}=\text{O}$ , edge-bridging  $\text{Mo}-\text{O}-\text{Mo}$ , corner-sharing  $\text{Mo}-\text{O}-\text{Mo}$ , and inner  $\text{Mo}-\text{O}-\text{Cu}$  linkages, respectively (Fig. 2a). Sharp absorption bands in the CTAB/ $\text{CuMo}_6$  spectrum at  $2919 \text{ cm}^{-1}$ ,  $2849 \text{ cm}^{-1}$ , and  $1470 \text{ cm}^{-1}$  correspond to the stretching and bending vibrations of the methylene ( $-\text{CH}_2-$ ) groups within the CTAB alkyl chains. These results confirm the successful functionalization of

$(\text{NH}_4)_4[\text{CuMo}_6\text{O}_{24}\text{H}_6]\cdot 5\text{H}_2\text{O}$  with CTAB while preserving the integrity of the core inorganic structure.

The thermal stability of CTAB/ $\text{CuMo}_6$  was evaluated by thermogravimetric analyses (TGA) under a nitrogen atmosphere. For  $(\text{NH}_4)_4[\text{CuMo}_6\text{O}_{24}\text{H}_6]\cdot 5\text{H}_2\text{O}$ , a mass loss occurs below  $200^\circ\text{C}$ , assigned to the release of adsorbed and lattice water. CTAB/ $\text{CuMo}_6$  shows no loss in this region, since the ammonium cations have been replaced by cetyltrimethylammonium and the material contains no lattice water (Fig. 2b). The absence of volatiles below  $200^\circ\text{C}$  is relevant to processing, as water released during curing would promote void formation at the filler-matrix interface. As shown in the DTG curves (Fig. 2c), the primary weight loss peak for CTAB/ $\text{CuMo}_6$  centers around  $260^\circ\text{C}$ , which corresponds to the thermal decomposition of the organic CTAB component[36][37]. Following this decomposition, the residual weight of CTAB/ $\text{CuMo}_6$  decreases to 24.3% at  $800^\circ\text{C}$ , compared with 78.7% for  $\text{NH}_4\text{-CuMo}_6$ . Taking the residue as the inorganic  $\text{CuMo}_6$  moiety, CTAB/ $\text{CuMo}_6$  contains ca. 30.9 wt.%  $\text{CuMo}_6$  and ca. 69.1 wt.% organic component, close to the mass ratio of the two starting materials used in the synthesis (69.8: 30.2). The corresponding CTA<sup>+</sup>:  $\text{CuMo}_6$  molar ratio (ca. 8.3: 1) exceeds the 6:1 required for charge balance, indicating that CTAB is not only ion-paired with the  $\text{CuMo}_6$  anion but also partially co-precipitated. CTAB/ $\text{CuMo}_6$  is therefore regarded as a surfactant-encapsulated composite rather than a compound of fixed stoichiometry.



**Figure 2** (a) FTIR spectra, (b) thermogravimetric analyses (TGA), and (c) corresponding derivative thermogravimetric curves (DTG) of  $(\text{NH}_4)_4[\text{CuMo}_6\text{O}_{24}\text{H}_6]\cdot 5\text{H}_2\text{O}$  and CTAB/ $\text{CuMo}_6$ .

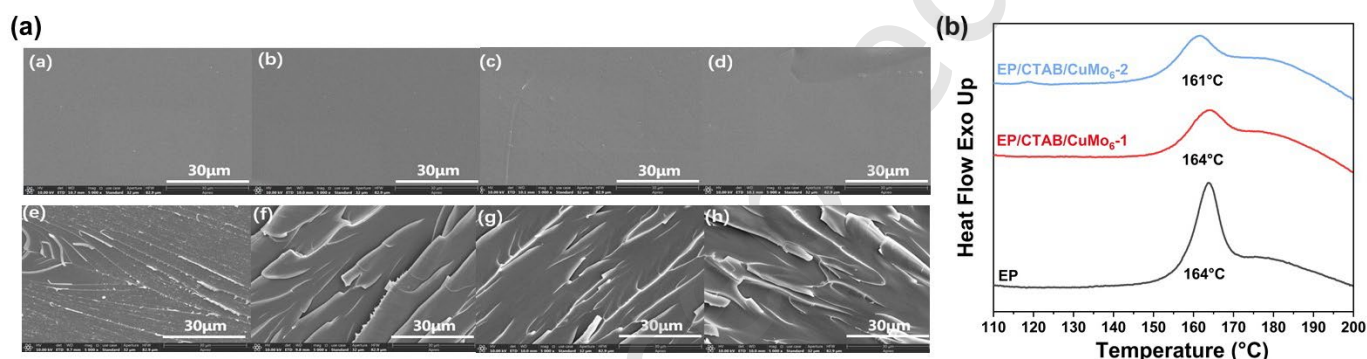
### 3.3 Dispersion and Curing of EP/CTAB/ $\text{CuMo}_6$

### composites

The dispersion state of fillers and their interfacial compatibility with the polymer matrix are critical factors governing both the flame-retardant efficiency and long-term service stability of epoxy-based composites [38][40]. SEM characterization (Figs. 3a(d)) demonstrates that CTAB-modified  $\text{CuMo}_6$  (CTAB/ $\text{CuMo}_6$ ) is uniformly dispersed within the epoxy matrix at filler loadings of 0.5-2 wt%, with no discernible agglomeration, thereby confirming the effectiveness of the surface modification. Cross-sectional images (Figs. 3a(e-h)) further reveal an oriented lamellar structure induced by processing-driven shear flow, which is consistent with the homogeneous distribution of the filler [41][43]. The fracture surface exhibits typical brittle fracture topography, characterized by elongated crack trajectories and well-defined layered morphologies, both of which attest to satisfactory interfacial compatibility between the CTAB/

$\text{CuMo}_6$  hybrid and the epoxy matrix.

DSC results (Fig. 3b) show that the glass transition temperature ( $T_g$ ) of the EP/CTAB/ $\text{CuMo}_6$  composite is 161 °C, only 3 °C lower than that of neat EP (164 °C). This slight decrease suggests a negligible change in  $T_g$ , indicating that the inorganic-organic hybrid filler imposes minimal constraint on polymer chain mobility. Both the neat and composite systems display a single exothermic curing peak, whose position, shape, and intensity correspond to the curing onset temperature, reaction rate, and degree of crosslinking, respectively. Overall, these observations confirm that the introduction of CTAB/ $\text{CuMo}_6$  does not compromise the curing kinetics or thermal transitions of the epoxy resin, thereby preserving its thermomechanical integrity.

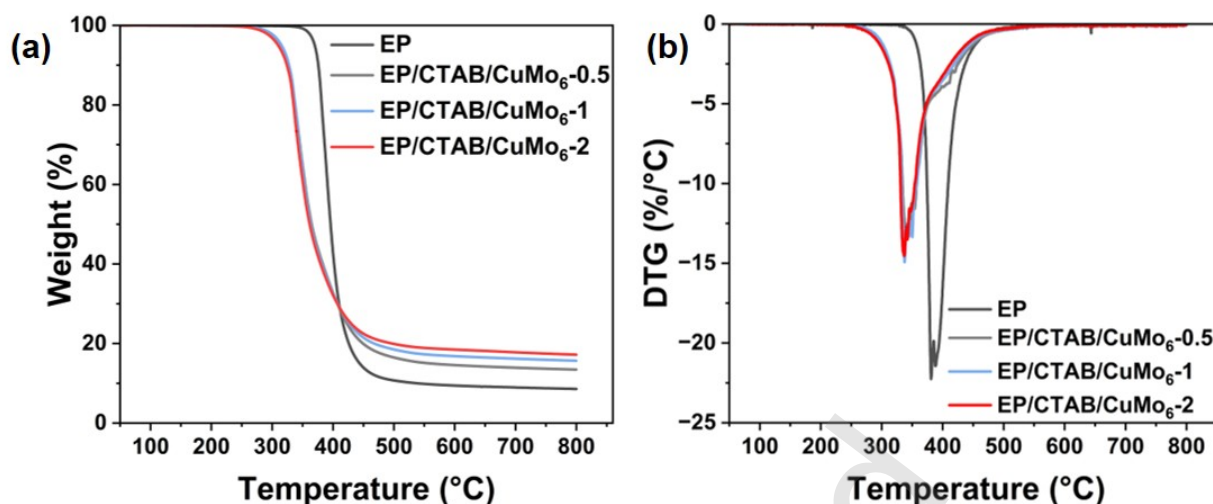


**Figure 3 a.** (a–d) Surface SEM images of epoxy composites with different filler loadings (0, 0.5, 1, and 2 wt%); (e–h) corresponding cross-sectional SEM images; **b.** DSC curves of EP and its composites.

### 3.4 Thermal properties of EP/CTAB/ $\text{CuMo}_6$ composites

The thermal stability of EP/CTAB/ $\text{CuMo}_6$  composites was further evaluated via thermogravimetric analysis (TGA) under a nitrogen atmosphere (Fig. 4a, Table 3). Neat EP displayed a 5% weight loss temperature ( $T_5\%$ ) of 370 °C, whereas the EP/CTAB/ $\text{CuMo}_6$  composites exhibited significantly diminished initial decomposition temperatures, with  $T_5\%$  values ranging from 309 to 313 °C. This modest reduction suggests that CTAB/ $\text{CuMo}_6$  promotes premature degradation, plausibly by facilitating molecular chain scission through attenuated intermolecular interactions. Complementary DTG analysis (Fig. 4b) provides deeper insight into the degradation kinetics. Neat EP displays a single, sharp decomposition peak centered at approximately 386 °C, corresponding to the dominant chain-scission event of the epoxy backbone. In contrast, the CTAB/ $\text{CuMo}_6$ -containing composites exhibit the principal DTG peak displaced to lower

temperature (from 386 to ~340 °C) with reduced peak intensity, together with a shoulder near 380 °C corresponding to the original position of neat EP. These changes are attributed to catalytic dehydration and early charring of the matrix promoted by the  $\text{CuMo}_6$  moiety. This broadening, combined with the smooth and symmetrical peak shape, indirectly confirms the uniform dispersion of the modified filler within the matrix (as corroborated by SEM images, Fig. 3a), thereby preventing localized heterogeneous degradation. At temperatures exceeding 400 °C, the catalytic activity of CTAB/ $\text{CuMo}_6$  becomes dominant, leading to a marked enhancement in thermal stability through the formation of a protective carbonaceous char. The char yield at 800 °C increased substantially from near-zero levels for neat EP to 7.69-17.19 wt% for the composites, demonstrating the exceptional efficiency of the additive in promoting residue accumulation.



**Figure 4.** (a) TG and (b) DTG curves for EP and its composites in N<sub>2</sub> atmosphere.

**Table 3** TGA data of EP and its composites.

| Samples                        | T <sub>5%</sub> (°C) | T <sub>max1</sub> (°C) | T <sub>max2</sub> (°C) | Char yield (wt%) at 800°C |
|--------------------------------|----------------------|------------------------|------------------------|---------------------------|
| EP                             | 370                  | 386                    | 428                    | 7.69                      |
| EP/CTAB/CuMo <sub>6</sub> -0.5 | 313                  | 337                    | 415                    | 12.59                     |
| EP/CTAB/CuMo <sub>6</sub> -1   | 315                  | 339                    | 414                    | 15.66                     |
| EP/CTAB/CuMo <sub>6</sub> -2   | 309                  | 343                    | 414                    | 17.19                     |

T<sub>5%</sub> represents the temperature at which 5% mass loss occurs.

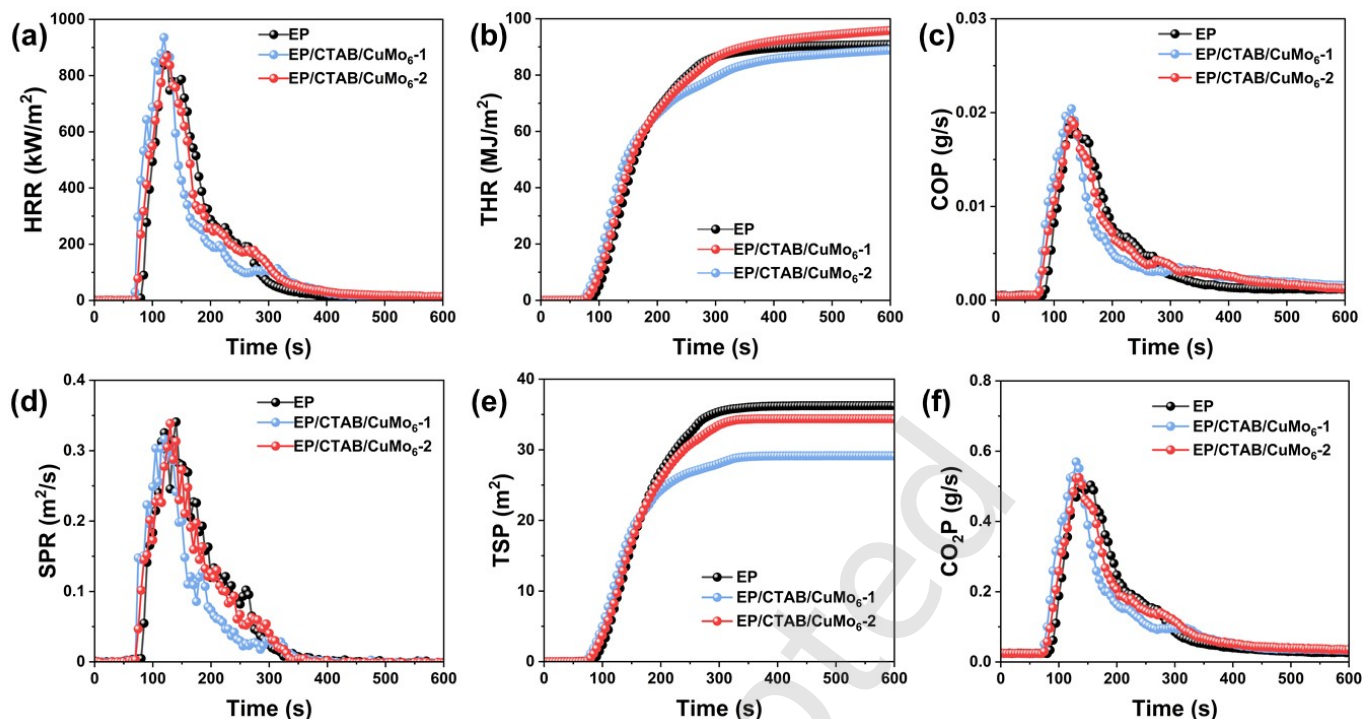
T<sub>max1</sub> and T<sub>max2</sub> denote the temperatures corresponding to the first and second maximum mass loss rates, respectively, during thermal degradation.

### 3.5 Flame retardant properties of EP/CTAB/CuMo<sub>6</sub> composites

The flame-retardant properties of EP/CTAB/CuMo<sub>6</sub> composites were evaluated using LOI, UL-94 vertical burning, and cone calorimetry tests. Although none of the composite formulations attained a UL-94 rating due to filler agglomeration at higher loadings, all exhibited substantially enhanced LOI values (27.1 and 27.9, Table 4) compared to neat EP (22.4), confirming improved early-stage flame resistance.

Cone calorimetry data revealed that all composites

displayed progressively suppressed smoke production rates and total smoke release with increasing filler content (Fig 5). Concurrently, elevated peaks in both CO<sub>2</sub> and CO evolution indicated that incomplete combustion was promoted, a phenomenon that facilitates the formation of a protective char layer. This thermally insulating char barrier effectively retards heat and mass transfer, curtails volatile release, and dilutes flammable gases through the release of non-combustible byproducts [44][45]. Collectively, these results substantiate the role of CTAB/CuMo<sub>6</sub> in redirecting decomposition pathways and augmenting fire safety, despite its limited efficacy in vertical burning tests.



**Figure 5** (a) HRR, (b) THR, (c) COP, (d) SPR, (e) TSP and (f)  $\text{CO}_2\text{P}$  of EP and its composite from cone calorimeter test.

**Table 4** . LOI and CCT data of EP and EP composites ( $35 \text{ kW m}^{-2}$ ).

| Samples                      | LOI  | $p\text{-HRR}$<br>( $\text{kW}\cdot\text{m}^{-2}$ ) | THR<br>( $\text{MJ}\cdot\text{m}^{-2}$ ) | $p\text{-SPR}$<br>( $\text{m}^2\cdot\text{s}^{-1}$ ) | TSP<br>( $\text{m}^2$ ) | AEHC <sup>a</sup><br>( $\text{MJ}\cdot\text{kg}^{-1}$ ) | $t_p^b$<br>(s) |
|------------------------------|------|-----------------------------------------------------|------------------------------------------|------------------------------------------------------|-------------------------|---------------------------------------------------------|----------------|
| EP                           | 22.4 | 872.23                                              | 90.86                                    | 0.34                                                 | 36.25                   | 23.00                                                   | 125            |
| EP/CTAB/CuMo <sub>6</sub> -1 | 27.1 | 935.54                                              | 88.87                                    | 0.32                                                 | 29.14                   | 23.57                                                   | 120            |
| EP/CTAB/CuMo <sub>6</sub> -2 | 27.9 | 868.37                                              | 95.83                                    | 0.34                                                 | 34.39                   | 24.02                                                   | 125            |

**a** AEHC is the average of the effective heat of combustion from time of ignition to the end of the test (60 to 360 s)

**b**  $t_p$  is the time corresponding to  $p\text{-HRR}$

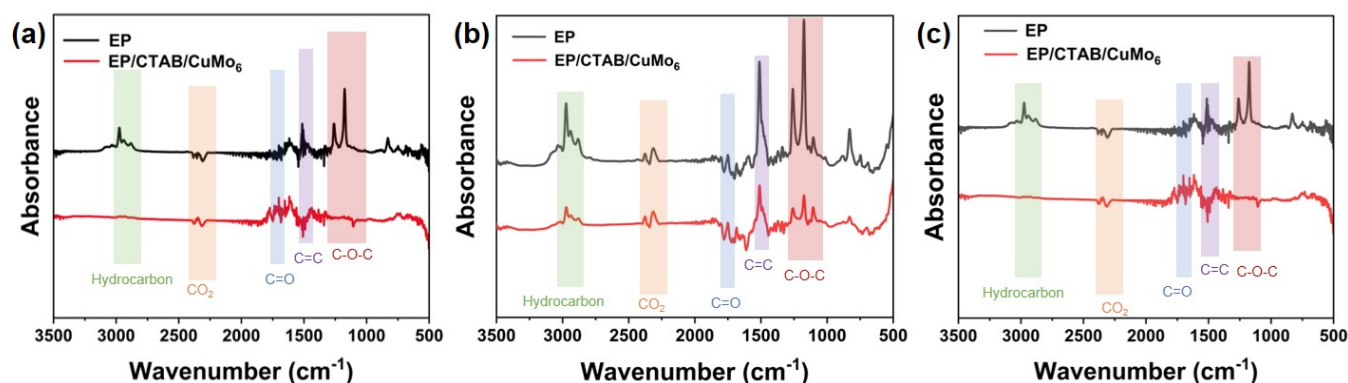
Table 4 summarizes the cone calorimetry data for all specimens. While the total heat release (THR) of the composites generally remained below that of neat EP, the 2 wt% loading exhibited a countertrend, likely arising from the intrinsic combustibility of CTAB and compromised filler dispersion at elevated concentrations, which promote discontinuous char formation. In terms of smoke suppression, EP/CTAB/CuMo<sub>6</sub>-1 composite achieved a 5.88% reduction in peak smoke production rate ( $p\text{-SPR}$ ) and a 19.61% decrease in total smoke production (TSP) relative to neat EP, confirming the pronounced smoke-suppressing capability of CTAB/CuMo<sub>6</sub>.

The average effective heat of combustion (AEHC) remained essentially constant across all formulations [46][47], indicating that gas-phase combustion behavior and volatile reactivity were largely unaffected by filler loading. This consistency implies that CTAB/CuMo<sub>6</sub> exerts its flame-retardant action primarily through condensed-phase mechanisms, with negligible perturbation of the combustion chemistry of volatiles in the flame zone.

### 3.6 Flame Retardant Mechanisms of Epoxy Cured Compounds

To elucidate the flame-retardant mechanism of the EP/CTAB/CuMo<sub>6</sub> composites, TG-FTIR analysis was conducted to characterize the gaseous products evolved during thermal decomposition (Fig. 6). The spectra reveal that the absorption features of EP/CTAB/CuMo<sub>6</sub>-1 are essentially indistinguishable from those of neat EP, indicating that the presence of CTAB/CuMo<sub>6</sub> does not fundamentally modify the intrinsic thermal degradation pathway of the epoxy resin. The characteristic absorption bands, assigned to specific volatiles, are summarized as follows:  $2190\text{--}2379 \text{ cm}^{-1}$  ( $\text{CO}_2$ ),  $1511 \text{ cm}^{-1}$  (aromatic C=C skeletal vibrations),  $1260 \text{ cm}^{-1}$  (aromatic C-O stretching),  $832 \text{ cm}^{-1}$  (para-substituted aromatic rings),  $2973 \text{ cm}^{-1}$  (aliphatic C-H stretching), and  $1106\text{--}1175 \text{ cm}^{-1}$  (aliphatic C-O stretching).

Notably, EP/CTAB/CuMo<sub>6</sub>-1 exhibited significantly lower absorption intensities across all identified gaseous species compared to neat EP. This suppression effect encompasses both toxic aromatic fragments, generated from the cleavage of benzene rings, and aliphatic hydrocarbons, which are recognized as key smoke precursors.



**Figure 6.** TG-FTIR analysis: FTIR spectra of gaseous products at (a)  $T_{5\%}$ ; (b)  $T_{\max 1}$  and (c)  $T_{\max 2}$  of EP/CTAB/CuMo<sub>6</sub>-1.

## 4 Conclusion

In summary, we report the successful modification of Anderson-type polyoxometalate  $(\text{NH}_4)_4[\text{CuMo}_6\text{O}_{24}\text{H}_6] \cdot 5\text{H}_2\text{O}(\text{CuMo}_6)$  using cetyltrimethylammonium bromide (CTAB) to develop an efficient flame-retardant filler for epoxy resin. Although pristine  $\text{CuMo}_6$  imparted appreciable flame retardancy to EP (LOI = 31.4), its practical application was constrained by inherent hygroscopicity (5.2% mass loss below 100 °C) and inadequate dispersion within the hydrophobic polymer matrix. CTAB modification effectively addressed these challenges: the resulting CTAB/ $\text{CuMo}_6$  exhibited markedly reduced moisture absorption (1.8% mass loss below 100 °C) and significantly improved dispersion characteristics. The EP/CTAB/ $\text{CuMo}_6$ -2 composite achieved an LOI of 27.9, representing a 24.55% enhancement over neat EP, while the EP/CTAB/ $\text{CuMo}_6$ -1 formulation yielded simultaneous reductions in total heat release ( $88.87 \text{ MJ} \cdot \text{m}^{-2}$ ), peak smoke production rate ( $0.32 \text{ m}^2 \cdot \text{s}^{-1}$ ), and total smoke production ( $29.14 \text{ m}^2$ ). Mechanistic studies confirmed that CTAB/ $\text{CuMo}_6$  hybrid preserves the catalytic char-forming capability of pristine  $\text{CuMo}_6$  cluster while enhancing interfacial compatibility with the epoxy matrix. The modified filler promotes the formation of a compact, thermally stable char layer that effectively impedes heat and mass transfer during combustion. Collectively, these findings establish CTAB surface modification as a viable and effective strategy for overcoming the practical limitations of polyoxometalate-based flame retardants, thereby providing a scalable approach to the development of fire-safe, high-performance epoxy composites for advanced applications.

## Data availability

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

## Acknowledgements

This work is financially supported by the National Natural Science Foundation of China (22471017, 22501265, and 21831001), the Beijing Natural Science Foundation (2242017), the Recruitment Program of Global Experts (Young Talents), and BIT Excellent Young Scholars Research Fund. The instrumental support from the National

Engineering Research Center of Flame Retardant Materials and the Analysis and Testing Center of BIT is also highly appreciated.

## Declaration of competing interest

The authors have no competing interests to declare that are relevant to the content of this article.

## Author contribution statement

M. S.: Data curation, formal analysis, visualization, writing – original draft. Y. L.: Visualization, validation. Y. Z.: Resources, funding acquisition, writing – review & editing. H. L.: Conceptualization, project administration, supervision, funding acquisition, writing-reviewing & editing. G.-Y. Y.: Resources, supervision, funding acquisition, project administration. All authors have approved the final version of the manuscript.

## Use of AI statement

None.

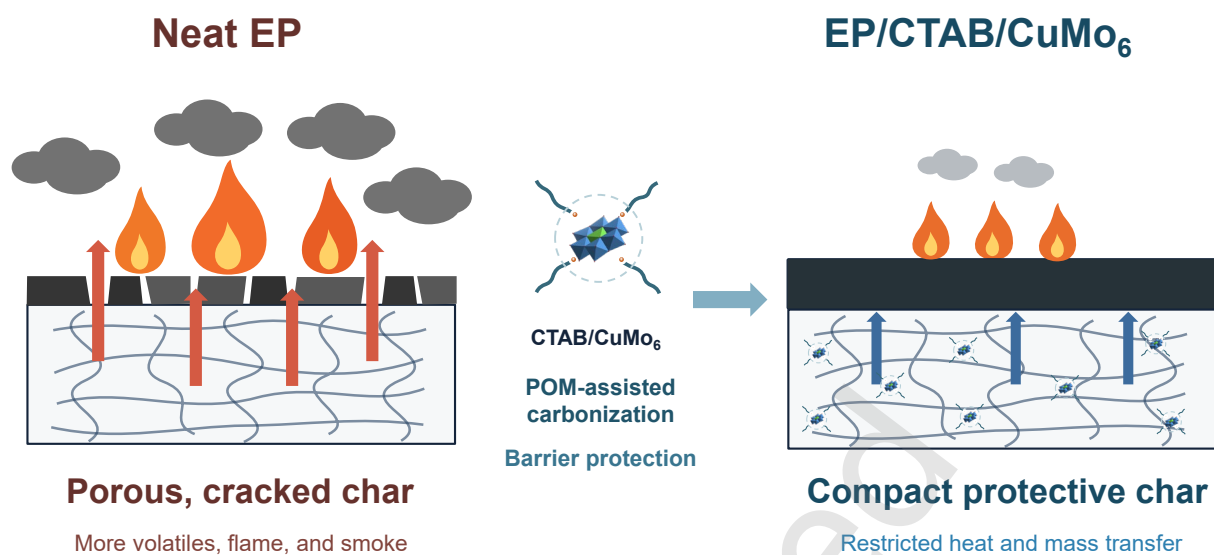
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CTAB-modified Anderson-type polyoxomolybdate (CTAB/CuMo<sub>6</sub>) as multifunctional flame retardants: catalytic charring for fire-safe and low-smoke epoxy composites