



Review

Thermal Stability and Fire Resistance of Vinyl Ester Resin Composites: Effect of Various Fillers on Thermal Degradation and Flame Retardancy

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Abstract. This work systematizes published data on the thermal stability and fire resistance of vinyl ester resin (VER)-based composites filled with various inorganic and organic flame retardants, including aluminum trihydrate $\text{Al}(\text{OH})_3$, $\text{Mg}(\text{OH})_2$, dimethyl methylphosphonate immobilized on diatomaceous earth (DE@DMMP), ammonium polyphosphate, expandable graphite, montmorillonite, zinc borate, phosphate esters, and hybrid fillers. The study describes the synthesis and curing methods for ten representative VER composite systems and presents a comparative analysis of data from thermogravimetry, differential scanning calorimetry, heat release rate, and the oxygen index. It is shown that mineral hydroxide fillers increase the LOI to 30–31%, reduce the pHRR by 42–45%, and raise the decomposition onset temperature by 17–28 °C, whereas immobilized DMMP systems achieve an oxygen index of 50% with a UL-94 V-0 rating. For each type of filler, the trade-off between flame resistance and mechanical properties is discussed. The work is supplemented with data from publications over the past 3–5 years, covering bio-based VERs (based on magnolol, vanillin, protocatechuic aldehyde, and bisgwayacol), new-generation reactive phosphorus-containing modifiers (VIDOP, VIDHP/VIDPP, flexible-chain DOPO, DPPO, APP-MDO), and hybrid interphase solutions for fiber-reinforced composites, as well as a critical analysis of discrepancies in the literature and an assessment of the economic feasibility and scalability of the systems under consideration.

Keywords: vinyl ester resin; fire resistance; TGA; DSC; LOI; HRR; $\text{Al}(\text{OH})_3$; $\text{Mg}(\text{OH})_2$; ATH; DMMP; diatomite; ammonium polyphosphate; expandable graphite; nano-clay; bio-based flame retardants; DOPO; fire-resistant composites.

1. Introduction

This review identifies three of the most promising areas of research. First, the surface functionalization of mineral fillers—demonstrated in this work for ATH using silane-based treatments—should be systematically extended to $\text{Mg}(\text{OH})_2$ and hybrid filler mixtures, where the mechanical benefits may prove to be equally significant. Second, the immobilization strategy, first applied to DMMP in diatomite, warrants investigation with other phosphorus-containing compounds and carrier materials, including functionalized mesoporous silicon dioxide, with the aim of increasing the oxygen index (LOI) above the self-extinguishing threshold while preserving the advantages of low loading during processing. Third, combining reactive DOPO chemistry with mineral fillers at moderate loading levels may pave the way for the creation of composites that simultaneously meet stringent fire safety requirements and retain the mechanical properties necessary for structural applications. These areas form a clear research agenda for the development of a new generation of fire-safe VER composites [1], [2], [3], [4]

The relevance of developing such composites is determined by the combined influence of three groups of driving factors: fire safety requirements, structural (mechanical) requirements, and environmental and sustainable development requirements. It is precisely the area where these factors intersect that forms the subject of this review – new-generation fire-resistant VER composites (Figure 1).

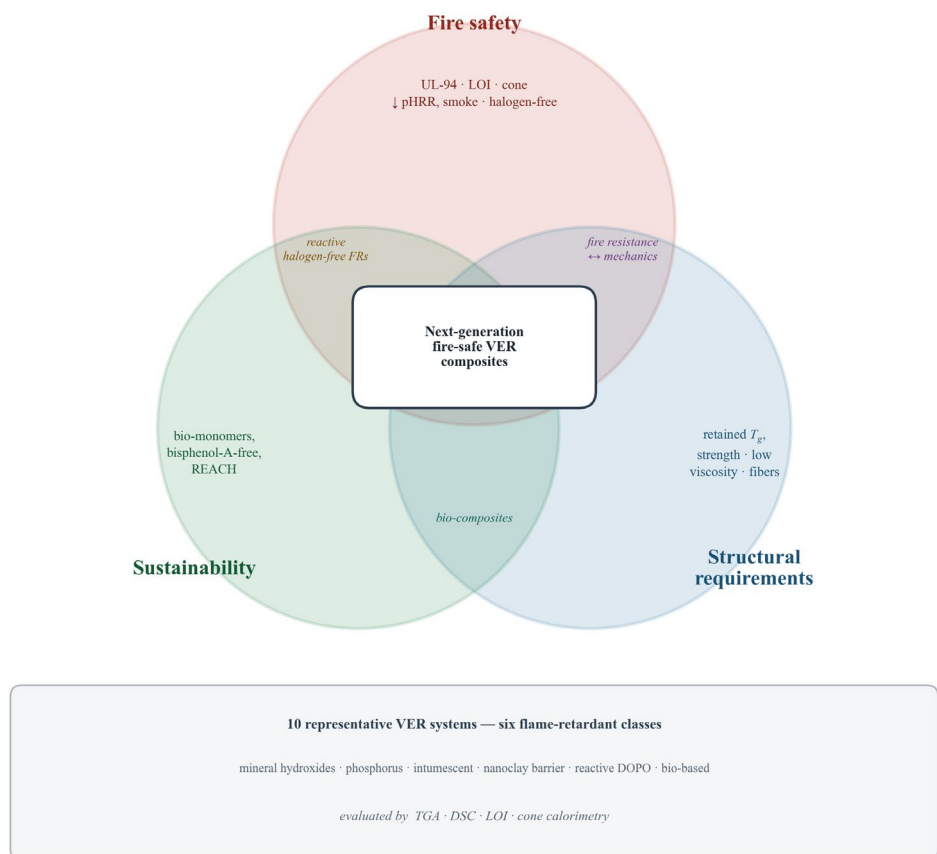


Figure 1 — The relevance of developing fire-resistant VER-based composites as an area where fire safety requirements, structural characteristics, and environmental sustainability intersect

One such potential filler is aluminum hydroxide— $\text{Al}(\text{OH})_3$, or ATH—whose fire-retardant effect is achieved through the release of 34.6 mass % of water at 180–220 °C, which helps cool the combustion zone and form a protective ceramic layer of Al_2O_3 . To effectively improve fire resistance, high concentrations of this material are required, which, in turn, leads to an increase in viscosity and a decrease in mechanical properties [5], [6].

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However, at high temperatures, aluminum hydroxide may decompose prematurely during processing; in such cases, magnesium hydroxide is preferable. Its dehydration range (300–360 °C) is significantly higher than that of aluminum hydroxide, and the byproduct—magnesium oxide (melting point 2852 °C)—provides a heat-resistant residue. For vinyl ester matrices, the fire-retardant effect of inorganic hydroxides, including magnesium-containing ones, has been confirmed by LOI, UL-94, and calorimetry methods at 50 kW/m² [10]. Studies have shown that the combined use of aluminum and magnesium hydroxides in a 2:1 ratio, even at high concentrations, yields an LOI of 31% and a 43% reduction in the peak heat release, with minimal deterioration in mechanical properties compared to using each filler separately [8], [9].

Phosphorus-containing compounds function by trapping $\text{H}\cdot$ and $\text{OH}\cdot$ radicals, which sustain combustion in the gas phase. Dimethyl methylphosphonate (DMMP) is generally effective at low concentrations, but it has the ability to migrate within the polymer matrix and subsequently evaporate. In their study, Xie et al. mobilized DMMP into the pores of diatomaceous earth via vacuum impregnation, which also possesses fire-retardant properties, achieving LOI = 50% and UL-94

reached Class V-0. Differential scanning calorimetry analysis confirmed the elimination of the plasticizing effect of DMMP [11]. This approach was further developed in a study by the authors of this review, which presents an optimized protocol for the preparation of DE@DMMP with improved retention of the phosphonate component [12].

There are also flame retardants such as ammonium polyphosphate combined with pentaerythritol (APP/PER), which promote the accelerated formation of intumescent carbonaceous char at temperatures of 250–300 °C, thereby achieving LOI = 28.5%. At the same time, the reduction in mechanical properties is minimal [13]. This line of research led to the development of reactive APP derivatives: In the study by Chu et al. on APP-MDO, it was shown that modified APP containing reactive C=C bonds is capable of copolymerizing with the VER matrix, which reduces flame retardant migration and improves moisture resistance compared to physically incorporated APP systems [14], [15].

The use of graphite as a flame retardant causes it to expand and explode during heating, with almost no effect on the system's viscosity, but tensile strength decreases by up to 22% [16].

One type of flame-retardant filler is organically modified nano-clay, which also acts as a diffusion barrier for volatile decomposition products and reduces the pHRR by 30–40% even at low concentrations. However, this material must be used in combination with other flame retardants [13].

DOPO (9, 10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide) derivatives undergo copolymerization with VER during curing, which eliminates the migration problem. In this case, the LOI increases to 28–32% without an increase in viscosity, but a multi-step synthesis procedure is required here [14]. In recent years, the concept of reactive DOPO derivatives has expanded significantly. A liquid flame retardant called VIDOP—a combination of DOPO and 1-vinylimidazole salts—has been proposed, which provides both gas-phase and condensed-phase effects in VER and glass-fiber-reinforced plastics based on it [16]. The ionic liquid VIDHP in carbon fiber/VER composites made it possible to improve fire resistance while maintaining interlaminar strength [17]. A systematic comparison of VIDHP and VIDPP derivatives with different degrees of phosphorus oxidation showed that the oxidation state of P directly determines the balance between gas-phase and condensed-phase mechanisms and the resulting LOI and pHRR values [18]. Grafting DOPO via a flexible aliphatic chain eliminates the embrittlement typical of rigid DOPO-crosslinked networks while preserving the flame-retardant effect [19]. Concurrently, the synthesis of DPPO-containing modifiers via diisocyanate bridges is being developed, which improves the compatibility of the phosphorus fragment with the matrix [20].

Bio-based vinyl ester resins with intrinsic flame resistance have emerged as a distinct and rapidly developing field in recent years. A low-smoke bio-based VER based on magnolol (DBDBMA) with reduced smoke emission and intrinsic flame resistance has been [21]. Bio-based OPBMAR resins based on vanillin with Schiff base moieties, processed via the VARTM method into fiberglass composites [22], and OBBBMA resins based on protocatechuic aldehyde for glass fiber/VER systems [23]; both systems demonstrate enhanced flame resistance without the addition of external flame retardants. Bisphenol A-free VERs based on bisgwayacol have been developed, which simultaneously solves the problem of replacing the toxic monomer [24]. A bio-based P/N-containing flame retardant, HDBMA, based on vanillin, has recently been proposed [17].

For fiber-reinforced VER composites, interfacial and membrane solutions are also being developed: PES/PEPA nanofiber membranes intercalated between layers in CF/VER carbon-plastic composites [25]; the reactive flame retardant ADOTP in combination with carbon fiber [26]; an ionic design of the VER/glass fiber interfacial boundary [27]; a biomimetic “soil–root–mineral” architecture that replicates natural fire-retardant structures [28]. These studies demonstrate that fire protection in composites is increasingly being engineered at the interfacial level, rather than solely within the matrix volume.

Functionalized mesoporous silicon dioxide combines a ceramic fire barrier with encapsulated gas-phase agents, achieving a 35–45% reduction in pHRR at low concentrations while maintaining mechanical properties. The problem is the high cost, which consequently leads to low scalability.

Despite the wide variety of existing fillers with flame-retardant properties, an LOI higher than 30% has not yet been achieved in the VER matrix. A UL-94 V-0 rating was achieved with a filler consisting of DMMP immobilized in diatomite pores. However, the issue of preserving mechanical properties arises in all cases. Understanding the specific mechanisms underlying each factor contributing to the problem is a prerequisite for the rational design of composites. This review presents a comparison of the thermal properties of 10 VER composites made from different fillers, including thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), LOI, and cone calorimetry to identify directions for future research [6], [7].

1.1 Selection criteria for ten representative composite systems

The ten systems under consideration were selected from an extensive body of published literature based on the principle of comprehensively covering the main VER fire protection mechanisms, such that each system represents a distinct class of solutions:

- endothermic cooling and ceramic barrier—Al (OH)₃ (ATH) and Mg (OH)₂ as standalone mineral hydroxides (the latter with higher-temperature dehydration at 300–360 °C), as well as their synergistic mixture ATH + Mg (OH)₂ (2:1), which expands the operating temperature range;
- gas-phase radical inhibition with migration suppression—DE@DMMP, the only example described in the literature of low-molecular-weight phosphonate immobilization that achieved UL-94 V-0 rating;
- intumescent (expanding) mechanism—APP/PER as a reference intumescent system;
- physical expanding barrier—expanding graphite (EG);
- nanobarrier diffusion mechanism—organo-modified montmorillonite (MMT);
- condensed-phase synergy—ZB/ATH, where a glassy borate layer seals the ceramic residue;
- reactive (covalent) phosphorus incorporation without a filler phase—DOPO-MAA;
- hybrid reactive-ceramic approach—mesoporous SiO₂ functionalized with DOPO-silane.

This set of systems covers the entire practically relevant range of loadings (from 5 phr for MMT to 60 phr for ATH and Mg(OH)₂), types of interaction with the matrix (physical dispersion—surface-modified filler—immobilized component—covalently incorporated comonomer), and achievable performance metrics (LOI 22.1–31.0%; pHRR 230–420 kW/m²). All systems are described in peer-reviewed sources with reproducible preparation protocols using the same matrix (bisphenol-A vinyl ester resin), which ensures the validity of the comparative analysis and the generalizability of the conclusions to a broader class of fire-resistant VER composites.

2. Methods

All composites are prepared using a commercial bisphenol A-based vinyl ester resin (Derakane 411-350, Ashland Inc., USA): a BADGE/methacrylic acid prepolymer in styrene (~35 wt.%), viscosity 300–500 mPa·s (25 °C), equivalent epoxy weight 190–210 g/eq. Curing system: methyl ethyl ketone peroxide (MEKP, 2 wt.%) as the initiator and cobalt naphthenate (0.2 wt.%) as the accelerator. Curing: 23 °C, 24 h; post-curing: 80 °C, 2 h. The degree of cure was confirmed by DSC (≥95%) for all formulations.

The preparation of composites with hydroxide-based fillers—both Al (OH)₃ (ATH) and Mg (OH)₂ systems—is carried out according to a standard protocol. The fillers are dried at 120 °C for four hours to remove any adsorbed moisture, then added to VER and mixed in a planetary mill at a speed of 300 rpm for 15 minutes, followed by vacuum degassing at 10–15 mbar. They are then poured into silicone molds and cured at room temperature.

When modifying the surface of aluminum hydroxide, an additional step is required: the aluminum hydroxide particles are treated with a 1% solution of γ -methacryloxypropyltrimethoxysilane in ethanol at 60 °C for one hour, then filtered and dried at 110 °C. This results in more than just physical mixing and covalent bonding. The latter effect is confirmed

by FTIR—the appearance of a Si–O–C band at 1090 cm^{-1} . The modified aluminum hydroxide is then incorporated into the main matrix [9].

Of all ten composites, the preparation of the system with DMMP immobilized in the pores of diatomaceous earth is the most complex, as it involves calcining the diatomaceous earth at $200\text{ }^{\circ}\text{C}$ for four hours to completely remove air from its pore network, followed by the introduction of DMMP into the pores via vacuum impregnation at a pressure below 1 mbar for twelve hours at a DMMP-to-DE mass ratio of 1:2, ensuring complete pore filling without residual bulk DMMP on the surface. The prepared powder was incorporated into the VER matrix by mechanical mixing. FTIR analysis results showed a decrease in the intensity of the P=O and P–O–C bands, and SEM images revealed cross-sections of the particles, which together demonstrate that DMMP penetrated the pores of the diatomite rather than being adsorbed onto the surface [11], [12].

The APP/PER flame-retardant system was prepared by mechanical mixing in a mass ratio of 3:1—optimal for the kinetics of char formation—followed by incorporation into VER via standard mechanical mixing. To prevent premature hydrolysis of the polyphosphate chains, this system was encapsulated in a melamine shell [28].

The preparation of expandable graphite (EG) as a filler requires careful mixing, since rapid mixing can cause destruction even before curing. EG particles are very brittle layered structures; upon destruction, they lose their ability to expand, which is a key factor for fire resistance. Therefore, mixing was performed using a low-shear paddle mixer at 200 rpm for ten minutes. Additionally, vacuum degassing—the next step—is also carried out very carefully to prevent premature expansion of the graphite under reduced pressure [28].

An organic solvent is required to incorporate organoclay or montmorillonite (MMT) filler into VER, as the silicate structure of montmorillonite does not disperse through mechanical mixing. Acetone at a concentration of about 5% is used as the solvent; the swollen clay is then introduced into the resin matrix, and the solvent is removed by vacuum evaporation. A method using a solvent at $60\text{ }^{\circ}\text{C}$ was required to incorporate the organoclay. The intercalation of organoclay was confirmed by X-ray diffraction [13].

Zinc borate (Firebrake ZB, Rio Tinto) was introduced not as a standalone filler, but in combination with aluminum hydroxide (ZB/ATH) to achieve a synergistic mechanism: the former provides endothermic cooling, while zinc borate promotes the formation of a glassy borate layer in the condensed phase, which strengthens and stabilizes the carbon residue. Both powders were dry-mixed prior to addition to the VER and then processed according to the standard mixing protocol [14].

To use DOPO as a flame retardant, DOPO-methacrylamide (DOPO-MAA) is first synthesized by reacting DOPO with acrylamide in toluene at $85\text{ }^{\circ}\text{C}$ for six hours. The compound is then dissolved directly into VER, followed by copolymerization during the curing process. In this process, phosphorus atoms are incorporated as covalent nodes. When introduced into the resin, no filler phase is formed; consequently, the viscosity of the system does not increase. The incorporation as covalent nodes has been confirmed by ^{31}P -NMR spectroscopy [16].

A mesoporous silicon filler system is used in combination with DOPO-silane by functionalizing the latter within the silicon pores. Prior to this, DOPO-silane is grafted in toluene at $110\text{ }^{\circ}\text{C}$ for 24 hours. This approach ensures a more durable fixation of the flame retardant than impregnation, since the formed P–O–Si bonds are hydrolytically stable. The phosphorus content in the functionalized silicon dioxide was confirmed by ICP-OES. The material was incorporated into VER via high-shear mixing, and its large specific surface area ($800\text{ m}^2/\text{g}$) facilitated good distribution throughout the matrix [28].

Another flame-retardant system, consisting of a synergistic mixture of aluminum hydroxide and magnesium hydroxide, was prepared by dry mixing in a ratio of 2:1, then added to VER. This ratio was selected based on published optimization data, as it ensures both fire resistance and the preservation of mechanical properties, including a minimal increase in viscosity [8], [9].

The prepared composites were subjected to thermogravimetric analysis (TGA) (ISO 11358, N_2 , $10\text{ }^{\circ}\text{C}/\text{min}$, $25\text{--}700\text{ }^{\circ}\text{C}$) and DSC (ISO 11357, N_2 , $10\text{ K}/\text{min}$, 2nd heating cycle, $0\text{--}200\text{ }^{\circ}\text{C}$), LOI

evaluation (ASTM D2863/ISO 4589-2), and cone calorimetry (ISO 5660-1, heat flux 50 W/m², horizontal orientation). All formulations were tested for vertical burning according to UL-94 (IEC 60695-11-10), and their mechanical properties were determined (ISO 527, tensile strength; ISO 178, flexural strength).

3. Discussion

3.1 Thermogravimetric analysis (TGA)

Figure 1 shows the results of TGA curve measurements for pure VER and VER-based composites with various fillers in a nitrogen atmosphere. Pure VER exhibits a two-stage decomposition: an initial stage at ~ 298 °C ($T_{5\%} \approx 290$ °C), with the main peak occurring at approximately 355 °C, corresponding to the breakage of styrene chains, during which volatile substances are released, followed by secondary decomposition at 380–500 °C. The carbon residue at 700 °C is 2%, indicating the extremely low fire resistance of the pure sample [3], [7], [20].

Upon the introduction of aluminum hydroxide, an additional endothermic step appears at 200–250 °C, corresponding to the dehydration of aluminum hydroxide, or at 300–360 °C—magnesium hydroxide, which is clearly visible on the shoulder. This requires 1050–1300 J/g, which contributes to effective cooling of the material during ignition. Fillers contribute to a shift in the onset of decomposition by 17–24 °C. This occurs because fillers create barriers to the diffusion of volatile components, thereby limiting the mobility of polymer chains. The carbon residue at 700 °C increases to 33–38%, which closely corresponds to the theoretical content of oxides (Al₂O₃ or MgO) [7], [8].

Pure magnesium hydroxide (60 phr) exhibits the highest decomposition onset temperature among hydroxide systems: since its dehydration occurs at 300–360 °C and does not precede the decomposition of the matrix, $T_{5\%}$ increases to 318 °C (+28 °C compared to pure VER), and the carbon residue (MgO) at 700 °C is $\approx 34\%$. In terms of cooling efficiency, Mg(OH)₂ is similar to ATH; however, its higher-temperature dehydration is better aligned with the main decomposition stage of VER (≈ 355 °C), which accounts for the composite's slightly higher thermal stability [8], [9], [10].

The DE@DMMP system (25% by weight) shows a more modest improvement: the ignition temperature shifts by only ~ 7 °C, and the carbon residue content increases to $\sim 12\%$. However, the porous DE support hinders DMMP migration, ensuring sustained suppression of gas-phase combustion throughout the material's service life—which is a significant advantage over DMMP systems obtained by physical mixing [29], [12].

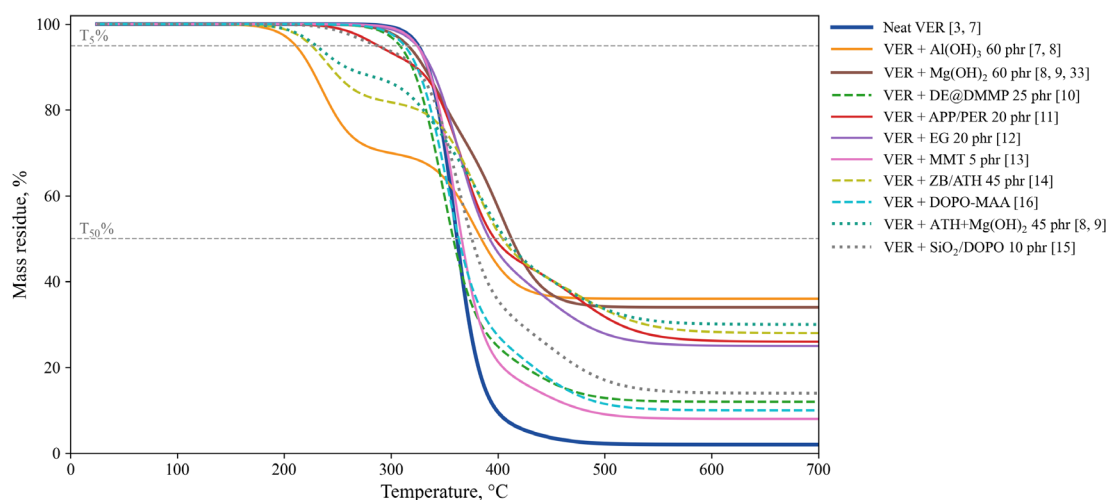


Figure 2 – TGA curves of neat VER and VER composites with different filler types (N₂, 10°C/min)[2], [3], [7], [8], [9], [10], [13], [14], [16], [29], [30], [31]

The TGA profile of the APP/PER filler showed that decomposition occurs in four stages (Figure 2): mass loss at 250–300 °C (about 8%), which corresponds to the precipitation of

polyphosphoric acid; at 350–480 °C, this is the combustion of the graphitized carbon residue; and at 500–600 °C, this reflects the combustion of the partially graphitized layer of the carbon residue. Compared to the pure sample, the decomposition onset temperature is shifted by 12 °C, which is an effective indicator and indicates the formation of a carbon residue [28].

In expanding graphite, no significant decomposition is observed below 300 °C due to the chemical inertness of the material. Already upon heating above 300 °C, graphite begins to expand, forming layers that slow heat transfer to the polymer matrix. The 5% decomposition point shifts by +10 °C to 308 °C, and the carbon residue at 700 °C reaches 25%. This is the highest value among carbon fillers [20].

Nanoclay has a negligible effect on mass loss and the decomposition onset temperature. (T5%) increases by only +4 °C, but the mass loss rate decreases by 18% compared to pure VER. The silicate plates hinder the volatilization of volatile products. The carbon residue is 8%, which is also a negligible value; the increase in LOI in this system is also negligible [13].

The ATH+Mg(OH)₂ system exhibits the following TGA profile: an ATH shoulder at 200–250 °C and a broad Mg(OH)₂ peak at 300–360 °C. The amount of heat absorbed is slightly lower than in the composite with a single filler (by 950–1100 J/g; T5% = 316 °C (+18 °C compared to pure VER), carbon residue 30%), which confirms the high degree of filler retention [8], [9].

The DOPO-MAA reactive system does not form a discrete filler phase; accordingly, its TGA curve lacks an additional mass loss step at temperatures below 300 °C. Covalently bound phosphorus atoms act in the gas phase, releasing PO• radicals, which capture H• and OH• chain carriers during thermal decomposition. This leads to a moderate increase in T5% to 306 °C (+8 °C) and the formation of a carbon residue of approximately 10%, which is higher than that of pure VER but lower than that of systems with a mineral filler. The main advantage of DOPO-MAA, according to TGA data, is the complete absence of a premature dehydration stage, which hinders the high-temperature processing of hydroxide-filled systems [2], [16]. Similar behavior is also characteristic of new-generation DOPO derivatives with flexible aliphatic side chains, for which a smoother curve is additionally observed in the 250–320 °C range [19].

Functionalized mesoporous SiO₂/DOPO-silane (10 phr) exhibits a TGA profile intermediate between those of the DOPO reactive system and composites with inorganic fillers. A slight mass loss in the 250–280 °C range is due to the decomposition of residual organic surface groups on the silica. The ceramic SiO₂ matrix persists up to 700 °C, accounting for approximately 9% of the carbon residue (total carbon = 14%). T5% = 309 °C (+11 °C). The high specific surface area of the carrier (800 m²/g) ensures uniform distribution of phosphorus centers throughout the matrix, resulting in a more homogeneous TGA curve without the sharp dehydration shoulder characteristic of hydroxide fillers [15].

The zinc borate filler in aluminum hydroxide retains the endothermic dehydration feature of ATH at 200–250 °C, while a glassy layer forms on the surface of the carbon residue starting at 410 °C. T5% = 312 °C (+14 °C) and a carbon residue of 28% at 700 °C. The glassy residue with a high ZnO/B₂O₃ content seals cracks in the Al₂O₃ layer, thereby limiting oxygen access—a result not achieved when using the fillers separately [14].

3.2 Differential scanning calorimetry (DSC)

Analysis of the DSC curves for all samples revealed significant differences in glass transition temperatures (T_g) and changes in heat capacity (ΔC_p).

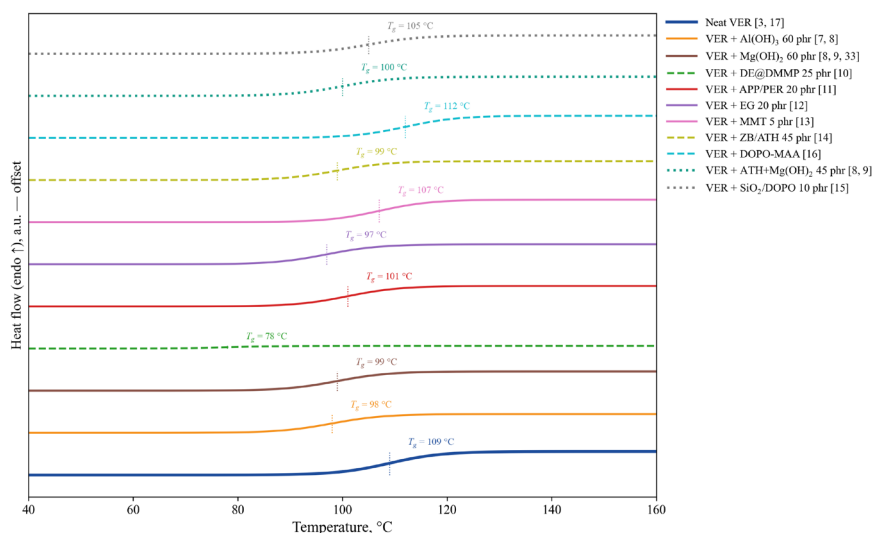


Figure 3 – DSC curves of neat VER and VER composites (2nd heating cycle, N₂, 10 K/min) [3], [7], [8], [9], [10], [13], [14], [16], [32], [10]

The pure VER sample exhibited the highest values of $T_g(\text{max}) = 109\text{ }^{\circ}\text{C}$ and $\Delta C_p = 0.135\text{ J/g}\cdot\text{K}$. Following the pure sample, the MMT-based composite exhibited $T_g = 107\text{ }^{\circ}\text{C}$ and $\Delta C_p = 0.130\text{ J/g}\cdot\text{K}$, attributed to its low concentration and a purely physical fire-retardant mechanism.

The remaining composites demonstrated the following values: Al (OH)₃ – $98\text{ }^{\circ}\text{C}$, EG - $97\text{ }^{\circ}\text{C}$, ZB/ATH - $99\text{ }^{\circ}\text{C}$, ATH+Mg(OH)₂ - $100\text{ }^{\circ}\text{C}$, APP/PER - $101\text{ }^{\circ}\text{C}$, and SiO₂/DOPO - $105\text{ }^{\circ}\text{C}$, with ΔC_p values in the range of $0.105\text{--}0.118\text{ J/g}\cdot\text{K}$. The smallest decrease in T_g is observed for DE@DMMP ($-78\text{ }^{\circ}\text{C}$, $\Delta C_p = 0.016\text{ J/g}\cdot\text{K}$), associated with the effective immobilization of DMMP in the diatomite pores, which limits the mobility of the polymer matrix chains.

In contrast, DOPO-MAA significantly increases $T_g(\text{max})$ ($112\text{ }^{\circ}\text{C}$, $\Delta C_p = 0.128\text{ J/g}\cdot\text{K}$). DSC data confirm that reactively incorporated fillers prevent a decrease in T_g , whereas all particle-based systems lower T_g to some extent depending on the loading and interfacial compatibility. The following Table 1 shows the key DSC parameters for all systems.

Table 1 – DSC parameters of neat VER and VER composites (2nd heating cycle, N₂, 10 K/min) [3], [7], [8], [9], [10], [11], [13], [14], [15], [16], [20], [28], [29], [30], [31], [32]

System	$T_g(\text{max})$, $^{\circ}\text{C}$	ΔC_p , $\text{J/g}\cdot\text{K}$
Neat VER	109	0.135
VER + Al (OH) ₃ 60 phr	98	0.108
VER + Mg (OH) ₂ 60 phr	99	0.111
VER + DE@DMMP 25 phr	78	0.016
VER + APP/PER 20 phr	101	0.118
VER + EG 20 phr	97	0.115
VER + MMT 5 phr	107	0.130
VER + ZB/ATH 45 phr	99	0.109
VER + DOPO-MAA	112	0.128
VER + ATH+Mg(OH) ₂ 45 phr	100	0.110
VER + SiO ₂ /DOPO 10 phr	105	0.105

3.3 Limiting oxygen index (LOI) and cone calorimetry

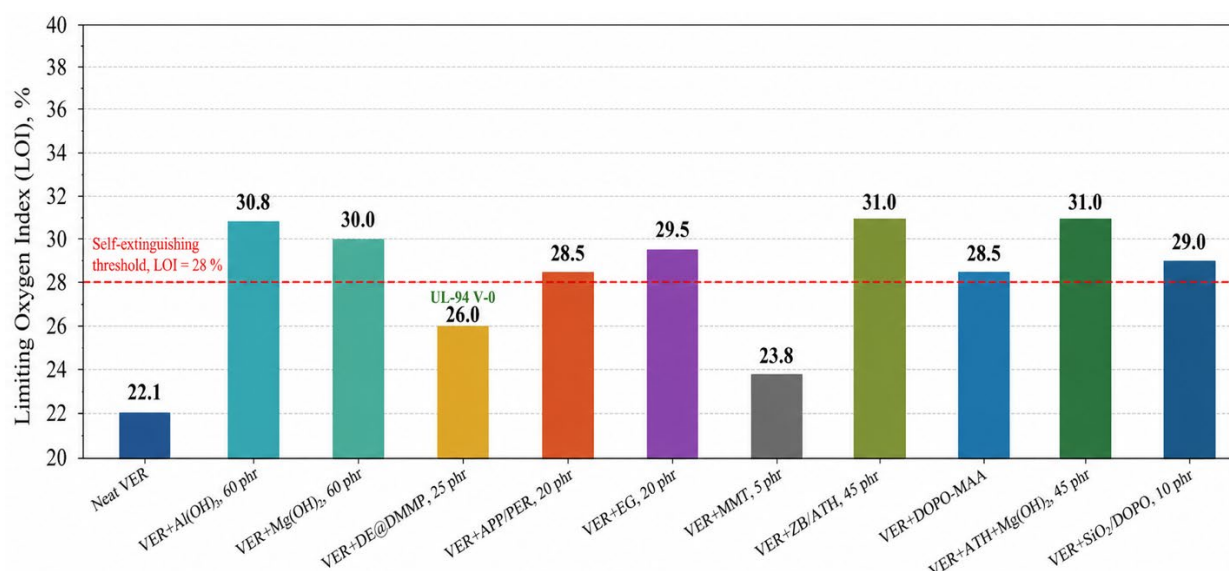


Figure 4 – LOI values of VER composite systems [7], [8], [9], [10], [12], [13], [29], [30]

Among all composite systems, the highest LOI is exhibited by VER composites containing aluminum hydroxide and magnesium hydroxide (LOI = 31%), as well as those containing zinc borate (LOI = 31%). The LOI of the composite filled solely with aluminum hydroxide is close to this value. Next, in descending order of LOI, are the following composites: VER+EG, VER+SiO₂/DOPO, VER+APP/PER, and VER+DOPO-MAA.

VER+MMT and DE@DMMP slightly increase the LOI compared to the pure sample, reaching 23.8 and 26, respectively [20]. Calorimetry is a powerful tool for assessing a material's fire resistance and fire safety. The following Table 2 shows the measured characteristics.

Table 2 – Cone calorimetry data for neat VER and VER composites (50 kW/m² heat flux)

System	TTL, s	pHRR, kW/m ²	ΔpHRR	THR, MJ/m ²	Ref.
Neat VER	28	420	—	95	[3], [7]
VER + Al(OH) ₃ 60 phr	48	230	−45%	60	[7], [8]
VER + Mg(OH) ₂ 60 phr	45	245	−42 %	61	[8], [9], [10]
VER + DE@DMMP 25 phr	36	310	−26%	78	[12]
VER + APP/PER 20 phr	40	270	−36%	72	[29]
VER + EG 20 phr	42	260	−38%	68	[30]
VER + MMT 5 phr	33	295	−30%	82	[13]
VER + ZB/ATH 45 phr	44	250	−40%	63	[14]
VER + DOPO-MAA	31	330	−21%	79	[2], [16], [31]
VER + ATH+Mg(OH) ₂ 45 phr	46	240	−43%	62	[8], [9]
VER + SiO ₂ /DOPO 10 phr	38	275	−35%	74	[16]

The heat release rate (pHRR) of pure VER is reported to be 420 W/m²; the total heat release (THR) is ~95 MJ/m² [3], [7]. The ignition time is approximately 28 seconds.

The best performance was recorded for the composite with aluminum hydroxide: pHRR decreases to 230 kW/m² and the ignition time increases to 48 s. Aluminum oxide serves as the diffusion barrier in the condensed phase [7], [8]. Similar results were also observed for the system combining aluminum hydroxide and magnesium hydroxide. Similar results were obtained for Mg (OH)₂ alone (pHRR = 245 kW/m²) and for a system combining aluminum and magnesium hydroxides. The VER + DOPO-MAA system exhibited a pHRR of 330 kW/m² and an ignition time of 31 s, which is the lowest value among all 10 proposed components. The values correspond to the data in Tables 1 and 2; for the carbon residue of the APP/PER system, an estimate based on the TGA curve (Figure 2) is provided.

3.4 Comprehensive multi-parameter comparison of systems

To simultaneously evaluate the improvement in fire resistance and its “cost” in terms of changes in thermomechanical properties, Table 3 and Figure 5 summarize the key performance indicators for all ten systems: LOI, pHRR, time to ignition, THR, Tg, carbon residue at 700 °C, and the decomposition onset temperature T_{5%}.

Table 3 — Summary of a multi-parameter comparison of the fire-resistant and thermal characteristics of ten VER systems [3], [7], [8], [9], [10], [12], [13], [14], [16], [29], [30], [31], [32]

System	LOI, %	pHRR, kW/m ² (Δ pHRR)	TTI, c	Tg, °C	Residue at 700 °C, %	T _{5%} , °C
Neat VER	22.1	420 (—)	28	109	2	290
VER + Al(OH) ₃ 60 phr	30.8	230 (−45 %)	48	98	33–38	307–314
VER + Mg(OH) ₂ 60 phr	30.0	245 (−42 %)	45	99	34	318
VER + DE@DMMP 25 phr	50.0	310 (−26 %)	36	78	~12	297
VER + APP/PER 20 phr	28.5	270 (−36 %)	40	101	~26	302
VER + EG 20 phr	29.5	260 (−38 %)	42	97	25	308
VER + MMT 5 phr	23.8	295 (−30 %)	33	107	8	294
VER + ZB/ATH 45 phr	31.0	250 (−40 %)	44	99	28	312
VER + DOPO-MAA	28.5	330 (−21 %)	31	112	~10	306
VER + ATH+Mg(OH) ₂ 45 phr	31.0	240 (−43 %)	46	100	30	316
VER + SiO ₂ /DOPO 10 phr	29.0	275 (−35 %)	38	105	14	309

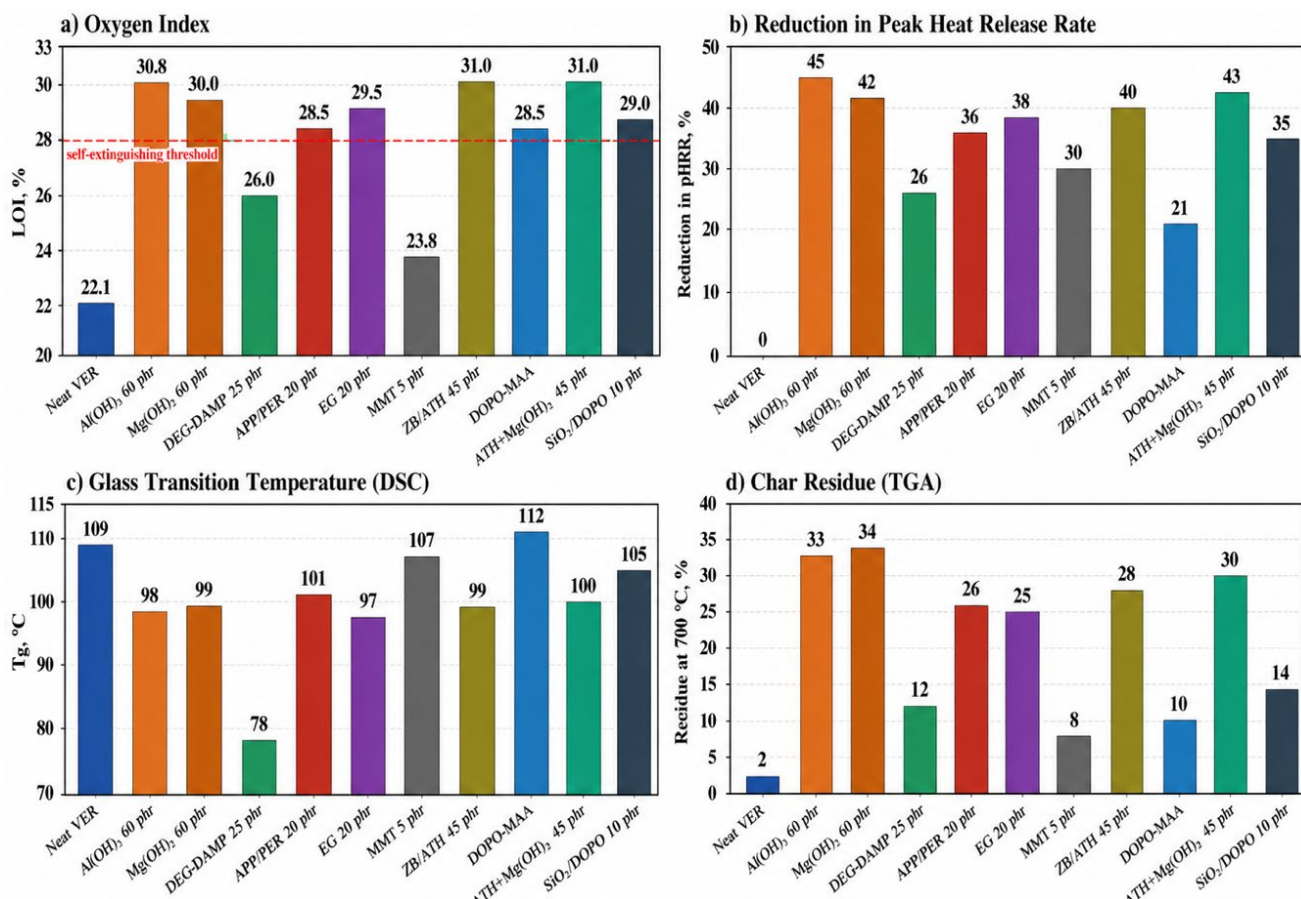


Figure 5 – Multiparameter comparison of ten VER systems: a) LOI; b) pHRR reduction; c) glass transition temperature; d) carbon residue at 700 °C (based on data from Tables 1–3; ([3], [7], [8], [9], [10], [12], [13], [14], [16], [10], [32] [17], [33])

Tables 3 and Figure 5 show that no single system achieves maximum values for all parameters simultaneously. Hydroxide systems (ATH, Mg (OH)₂, ATH + Mg (OH)₂, ZB/ATH) lead in terms of LOI, reduction in pHRR, and time to ignition, but “pay” for this with a 9–11 °C decrease in T_g and a high loading (45–60 phr). The reactive DOPO-MAA system is the only one that increases T_g (up to 112 °C), but it results in the smallest reduction in pHRR (–21%). DE@DMMP—the only system with a UL-94 V-0 rating—is characterized by the most pronounced decrease in T_g (down to 78 °C), due to the residual plasticizing effect of the phosphonate. These trade-offs quantitatively illustrate the thesis formulated in the introduction regarding the need for a rational choice of filler tailored to specific operational requirements.

3.5 Critical analysis of discrepancies in published data

The values of thermal and combustion parameters reported by different authors for nominally identical systems often differ. An analysis of the primary sources reveals six main reasons for these discrepancies.

First, for systems containing ATH, published LOI values range from ~28 to 35%, depending on (a) the shape and size of the particles (micro- and nanoscale ATH form Al₂O₃ barriers with varying degrees of continuity), (b) the presence of silane surface treatment, which increases the LOI by 1–2% due to improved adhesion and uniformity of distribution [9], and (c) the total loading: at 60 phr, the LOI is 2–3% higher than at 40 phr [7], [8]. Therefore, a direct comparison of LOI values without considering particle shape and loading is incorrect.

Second, for VER + MMT nanocomposites, the reported values for pHRR reduction fall within a wide range of 25–40%, which is determined primarily by the degree of clay exfoliation: effective exfoliation (confirmed by the shift of the d(001) peak in the X-ray diffraction pattern) determines the upper limit of the range, whereas aggregated tactoid structures determine the lower limit [13]. The shear mixing method and the choice of solvent are critical.

Third, for DOPO derivatives, LOI values in the literature range from 26 to 32% and depend significantly on the structure of the modifier: the degree of DOPO substitution, the presence of a nitrogen-containing moiety (imidazolium salts VIDOP, VIDHP), and the form of introduction (monomer for copolymerization or prepolymer) [2], [14], [16], [18], [19]. A systematic comparison of derivatives with different degrees of phosphorus oxidation [18] showed that P(+V) forms shift the reaction to the condensed phase (higher coke yield), whereas P(–I)/P(+III) forms are more effective in the gas phase; this, in particular, explains the discrepancies in pHRR values for systems with similar compositions.

Fourth, the differences in T_g values are explained by the different nature of the “phosphorus–matrix” bond: physically incorporated DMMP acts as a plasticizer and lowers T_g by 25–30 °C, whereas covalently bound DOPO-MAA does not lower T_g (or slightly increases it due to an increase in crosslinking density), while immobilized DE@DMMP occupies an intermediate position, since immobilization only partially eliminates plasticization [12], [16].

Fifth, for APP/PER intumescent systems, char yield and LOI are sensitive to the APP:PER ratio (optimum 3:1) and to moisture protection: without melamine encapsulation, exposure to high humidity leads to partial hydrolysis of the polyphosphate and a noticeable loss of effectiveness [29], [15]. Differences in the conditioning history of the samples are a common cause of discrepancies in data from different laboratories.

Sixth, the time to ignition and pHRR systematically depend on the calorimetry conditions (heat flux of 35/50/75 kW/m², sample orientation, plate thickness); therefore, absolute values from different studies are comparable only after being normalized to identical test conditions. In this review, all calorimetry data are reported for 50 kW/m² and horizontal orientation (ISO 5660-1).

Thus, in most cases, the observed discrepancies do not reflect inconsistencies in the data but rather differences in filler morphology, method of incorporation, loading, conditioning, and test conditions. This is precisely why Table 3 lists the sources of each value, and the comparison of systems is based on data obtained under comparable conditions.

4. Practical aspects and prospects for further research

4.1 Economic feasibility

The cost of the fire-retardant systems discussed varies by an order of magnitude. Mineral hydroxides (ATH, $\text{Mg}(\text{OH})_2$) are the least expensive large-volume flame retardants, making them the preferred choice for mass-production structural applications (construction profiles, ship panels, containers), despite the high loading levels required. APP/PER systems and intumescent graphite occupy the mid-price segment at moderate loading levels (20 phr). The most expensive are reactive DOPO derivatives (multi-stage synthesis) and functionalized mesoporous SiO_2 (sol-gel synthesis of the carrier and grafting of DOPO silane in toluene), which currently limits their use to niche, mission-critical applications—aviation, electronics, and transportation [31]. DE@DMMP occupies an intermediate position: both diatomite and DMMP are inexpensive industrial products, and the main cost comes from the vacuum impregnation stage [12]. Simplifying the synthesis of reactive phosphorus-containing modifiers—for example, single-step schemes based on diisocyanate bridges [30]—is an important means of reducing costs.

4.2 Scalability of synthesis

Technologies for producing composites with mineral fillers (ATH, $\text{Mg}(\text{OH})_2$, ZB/ATH) are fully compatible with existing mixing and casting equipment and can be scaled up without restrictions; the only limiting factor remains the increase in viscosity at high loading levels. Bio-based VERs with intrinsic flame resistance (OPBMAR, OBBBMA, HDBMA) have already been processed using VARTM methods into fiberglass composites on a laboratory and pilot scale, demonstrating their compatibility with industrial infusion technologies [22], [23], [33]. The interlayer incorporation of PES/PEPA nanofiber membranes requires an additional step during layup but is compatible with automated processes for the production of layered composites [25]. The most critical processes for scaling up are the multi-stage processes—vacuum impregnation of DMMP into diatomite and the functionalization of mesoporous SiO_2 with DOPO-silane; optimizing these process parameters (reducing impregnation time, replacing toluene with safer solvents) remains a priority engineering task [12], [31].

4.3 Industrial applicability and environmental requirements

The choice of system is determined by the application. For construction and shipbuilding products, where cost and smoke emission are critical factors, hydroxide systems and their combinations with zinc borate are the most suitable. For electrical and transportation applications, where binder viscosity during fiber impregnation and the preservation of T_g are critical, reactive DOPO/DPPO modifiers and ionic interfacial solutions are preferred [17], [20], [27]. Tighter environmental regulations (REACH and similar regulations) restricting halogenated flame retardants and bisphenol A make bio-based VERs derived from magnolol, vanillin, protocatechuic aldehyde, and bisgwayacol strategically important [17], [21], [22], [23], [24]: they simultaneously eliminate toxic monomers and provide intrinsic flame resistance without additive migration. Biomimetic architectures [29] suggest the potential for further efficiency gains while maintaining environmental sustainability. Over the next 3–5 years, the following appear to be the most realistic candidates for industrial implementation: (i) surface-modified ATH/ $\text{Mg}(\text{OH})_2$ hybrids; (ii) bio-based VERs with incorporated phosphorus and nitrogen; (iii) reactive DOPO derivatives with flexible interconnections for fiber-reinforced structures.

4.4 Research priorities

From a scientific standpoint, the three areas outlined in the introduction remain key: the systematic extension of silane functionalization to $\text{Mg}(\text{OH})_2$ and hybrid mixtures; the development of new “phosphorus compound–porous support” pairs, analogous to DE@DMMP (including those based on mesoporous SiO_2) to overcome the self-extinguishing threshold at low loading levels; and the combination of reactive DOPO/VIDHP chemistry with moderate amounts of mineral fillers to simultaneously achieve $\text{LOI} > 31\%$, UL-94 V-0 classification, and maintain $T_g \geq 100^\circ\text{C}$. To these should be added a fourth direction identified in the analysis of the 2022–2026 literature: the design of fire protection at the fiber–

matrix interface (ionic interactions, nanofiber interlayers, biomimetic structures), which allows for combining fire resistance with an increase—rather than a decrease—in mechanical properties[17], [18], [25], [27], [28].

5. Conclusions

This review examines VER-based composite systems, the primary function of which is to enhance the fire resistance and other thermal properties of the host matrix while only slightly altering the mechanical properties inherent to VER. Physical testing methods such as TGA, DSC, LOI, and cone calorimetry allow for an assessment of the increase in the fire resistance of composites upon the introduction of a particular filler.

Among all the systems considered, an aluminum hydroxide-based filler stands out, having demonstrated both a high LOI and effective cone calorimetry characteristics.

In cases where high operating temperatures or technological constraints limit the ATH content, a synergistic blend of ATH and Mg (OH)₂ with a total content of 45 phr becomes an important practical alternative. Phosphorus-based fillers, such as the DE@DMMP system, achieve UL-94 V-0 rating—a result that hydroxide-based systems can only achieve at much higher concentrations—while maintaining moderate viscosity and mechanical loss values. A limitation is an LOI of 50%, so DE@DMMP is best used in combination.

APP with pentaerythritol achieves an LOI threshold of 28% or higher at a lower loading than hydroxide-based systems and represents the most mechanically favorable route to self-extinguishing properties when moisture resistance requirements are not stringent; new-generation reactive APP derivatives [15] also overcome this limitation. Montmorillonite's fire resistance mechanism is achieved through physical barrier methods, and its ability to reduce pHRR by 30–40% is due to its effective synergistic component in combination with primary flame retardants.

Although DOPO derivatives do not exhibit effective thermal properties, they offer a significant advantage: no loss of viscosity and the absence of a “particle-matrix” interface, which makes them attractive for use in fiber-reinforced materials. Recent studies on VIDOP, VIDHP/VIDPP, and DOPO with flexible crosslinks [16], [17], [18], [19], as well as bio-based VERs with inherent flame resistance [21], [22], [23], [33] confirm that reactive phosphorus chemistry can bridge the gap between the V-2 and V-0 categories without compromising processability.

A recurring pattern is observed in all ten systems: fire safety metrics – LOI, pHRR, and UL-94 rating – do not always improve simultaneously. A system can achieve a V-0 rating while remaining below the LOI self-extinguishing threshold, as demonstrated by the DE@DMMP example, since these two tests evaluate different aspects of combustion behavior: LOI measures flame spread under steady-state conditions, while UL-94 assesses the tendency toward self-extinguishing after the ignition source is removed. Therefore, presenting a single indicator as representative of fire safety is insufficient, and future work should regularly combine these complementary characteristics. The results of this review are useful for research and development of a new generation of fire-safe VER composites.

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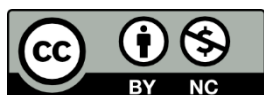
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