Poly Flame

PolyFlame est une newsletter à destination des chercheurs et des industriels du domaine du « comportement au feu des matériaux organiques ». Cette newsletter périodique est publiée via la Société Chimique de France (SCF).

A travers cette newsletter, vous découvrirez les nouveautés et les dernières avancées dans le domaine du comportement au feu en matière de recherche et développement, la synthèse et la production de nouveaux systèmes de retardateurs de flamme, les besoins industriels. Pour faire avancer la connaissance et l'expertise, une partie de cette newsletter est consacrée à l'écoute des chercheurs et des industriels reconnus dans ce domaine.

Bonne Lecture,

Flame retardant strategies for the synthesis of hybrid epoxy nanocomposites

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Abstract

A combination of different hybrid strategies exploited in the flame retardation of bisphenol A diglycidyl ether (DGEBA) resin cured with a cycloapliphatic amine, namely isophorone diamine, is presented. First, epoxy nanocomposites were prepared through an in-situ sol–gel procedure for the modification of DGEBA resin with silanes. Hence, inorganic-organic hybrid structures were obtained, containing silica nanocrystals chemically bonded to the epoxy chains. Then, flame-retardant silica-epoxy materials were synthesized by incorporating two phosphorus-based flame-retardant additives (non-toxic DOPO derivatives) together with melamine as a nitrogen additive, followed by curing with isophorone diamine. The hybrid strategy allowed for achieving self-extinction (V0 rating in UL 94 test) for both systems with only 2 wt.% of P loading. Promising fire performances were also achieved by embedding phosphorus in the silica-based nanostructures using phosphoric acid through a hydrolytic sol-gel procedure. A similar synthetic approach was also effective in the formation of magnesium hydroxide nanocrystals in the epoxy matrix. The fire behavior of this system was experimentally studied and predicted by applying a machine learning method.

1. Introduction

Epoxy resins offer enhanced high-performances due to a combination of thermal stability and chemical resistance, which make these materials suitable for many industrial applications [1]. The use of aromatic amines as hardeners has adverse health effects, thus increasing the utilization of aliphatic or cycloaliphatic amines as green alternatives [2]. However, epoxy resins cured with cycloaliphatic curing agents are easily flammable and require the use of flame-retardant additives for fire-safe applications [3]. Halogenated flame-retardants have been commonly utilized in flame retardation of epoxy resins, although recently the usage of some of these additives has been regulated, also because of their potential toxicity [1, 2]. As an alternative, the development and use of silicon- and phosphorus-based flame-retardants is considered as a feasible option [4]. In this context, the application of an in-situ sol-gel method for the preparation of silica-epoxy nanocomposites, together with a strong phosphorus-nitrogen (P-N) synergism, represents a very interesting way to enhance the flame-retardant behavior of the epoxy matrix [5]. Such a strategy can give rise to self-extinguishing nanocomposites, even using a cycloaliphatic curing agent, keeping a relatively low P-loading (i.e., 2 wt.%). The in-situ growth mechanism of SiO_2 nanocrystals, based on controlled hydrolysis and

condensation reactions in an inverse micelle system, can be extended to other inorganic nanostructures, such as Si-P oxides [6] and magnesium hydroxide [7]. A new complementary approach to the experimental analysis of fire performances is offered by Artificial Intelligence, in particular exploiting artificial neural network models based on machine learning [8]. Applying algorithms to existing datasets of materials' properties, selected parameters can be predicted, addressing the assessment of the fire behavior of new materials and reducing the number of expensive destructive tests.

2. Experimental

2.1 Materials

Tetraethyl orthosilicate (TEOS), (3-aminopropyl)-triethoxysilane (AP), anhydrous ethanol, 2,4,6-triamino-1,3,5-triazine (melamine) were purchased (ACS grade) from Sigma-Aldrich (Switzerland) and used as received. A two-component epoxy resin system (Hexion GmbH, Germany), consisting of bisphenol A resin (Epikote™ Resin 827) and isophorone diamine (Epikure™ curing Agent 943, abbreviated as IDA) was employed for preparing the nanocomposites. 6H-dibenz[c,e][1,2]oxaphosphorin,6-[(1-oxido-2,6,7-trioxa-1-phosphabicyclo[2.2.2]oct-4-yl)methoxy]-6-oxide (DP) and (6-oxidodibenzo[c,e][1,2]oxaphosphinin-6-yl)propenamide (DA) (Figure 1) were synthesized following the procedures reported in the literature [9].
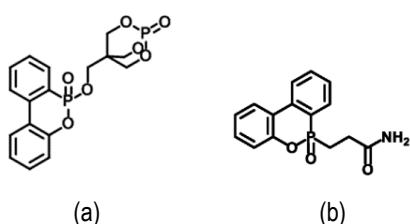

$$\text{(a)} \qquad \text{(b)}$$

Figure 1: Chemical structures of DP (a) and DA (b).

2.2 Preparation of in-situ silica-epoxy composites

To promote an in-situ sol-gel synthesis, such silica precursors as AP and TEOS, were added to the commercial epoxy resin system (DGEBA), giving a final silica content of 2.0 wt.%, followed by the addition of DA or DP (Figure 1), melamine (M), and mixed properly prior to the mixing with the hardener (IDA). The quantity of DA and DP added was calculated to achieve 2.0 wt.% of P in the final composites. The phosphorous content was also measured experimentally. The resulting mixtures were degassed and poured into a steel mould and taken for curing

(40 °C/3 h) and post-curing (150 °C/2 h) [10, 11]. Following a similar procedure, after functionalization of DGEBA with AP, epoxy-Si-P hybrid nanocomposites (EPOSP) were prepared using TEOS and phosphoric acid [6] and epoxy-Mg(OH)$_2$ nanocomposites (EPOMg) were obtained using magnesium ethoxide [7]. In the case of EPOSP and EPOMg, DEGBA and IDA were purchased as a two-component epoxy resin system (SX10) from MATES S.r.l. (Milan, Italy) and used as received. Both systems were cured at 60 °C for 24 h and then post-cured at 80 °C for 4 h.

Sample	TEOS/AP (mol/mol)	DA (g)	DP (g)	M (g)	H$_3$PO$_4$ (g)	P (%)
EPO	/	/	/	/	/	/
EDP	/	/	27	/	/	2.7
EDA	/	41	/	/	/	2.6
EPOS	2	/	/	/	/	/
ESDAM	2	41	/	14	/	2.2
ESDP	2	/	27	/	/	2.2
ESDPM	2	/	27	9	/	2.4
EPOSP	2	/	/	/	4.3	1.0

Table 1: Additives used to prepare epoxy composites. The amounts of epoxy and hardener were set at 107 and 27 g, respectively.

2.3 Analytical techniques

The thermal and fire behaviors of the obtained silica-epoxy nanocomposites were investigated in detail by means of differential scanning calorimetry (DSC), cone calorimetry (CC), pyrolysis combustion flow calorimetry (PCFC), UL 94 vertical flame spread tests. The gas evolved analysis for the study of flame-retardant mechanisms was performed by DIP-MS (direct insertion probe–mass spectrometry) and PY-GC-MS (pyrolysis–gas chromatography–mass spectrometry). The morphology of the obtained nanocomposites and residual chars was assessed by means of Scanning Electron Microscopy (SEM) and Energy-dispersive X-ray analysis (EDX).

3. Results and discussion

3.1 Epoxy-silica nanocomposites with P-based flame-retardant additives

Thermal and fire behavior. DA acts as plasticizer in the epoxy resin, leading to a lower glass transition temperature (Tg) compared to the neat polymer (Table 2). Conversely, DSC tests for DP indicate a significant increase (up to 5%) of Tg with respect to the pristine epoxy network, possibly due to hydrogen bonds and polar interactions of DP (Figure 1) with the matrix [10-12].

Sample	Tg (°C)
EPO	127
EDP	133
EDA	85
EPOS	112
ESDAM	91
ESDPM	131

Table 2: Glass transition temperature (Tg) of the prepared epoxy composites.

Figure 2 shows that the presence of silica guarantees the formation of a continuous and stable char after the combustion [4], although unfilled epoxy resin and silica-epoxy nanocomposites without any flame-retardants could not be classified in UL 94 tests. Epoxy resin containing DA alone could achieve a V0 rating (self-extinction) in UL 94 test at 2 wt.% P, though the presence of silica nanoparticles was crucial to achieve a V0 non-dripping rating [10]. Conversely, the incorporation of DP (2 wt.% P) alone in the epoxy could already prevent dripping phenomena. However, when DP was used as flame-retardant, the combined effects of melamine together with silica nanoparticles were necessary to achieve a V0 rating (Figure 2) [11].
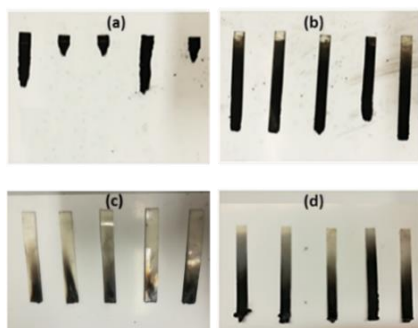

Figure 2: Residues after UL 94 vertical flame spread tests of EPO (a), EPOS (b), ESDAM (c), and ESDPM (d).

CC tests showed a remarkable reduction (up to 75%) of the heat release rate (HRR) when DA and DP were used as flame-retardants in the nanocomposites (Figure 3 and Table 3). The adoption of both additives in the manufacturing of the epoxy nanocomposites accounted for an increase in CO/CO_2 ratio, indicating their gas phase flame inhibition behavior (Table 3) [9]. In addition, a higher char formation and intumescence were observed for the samples containing DA and DP with melamine, especially in the case of DP, because of a predominant activity in the condensed phase (see below) [10,11].
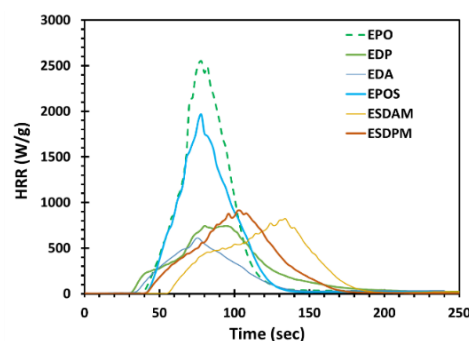

Figure 3: Heat release rate (HRR) observed in EPO, EDP, EDA, EPOS, ESDAM, ESDPM.

Sample	HRR (kW/m²)	ΔHRR (%)	Residue (wt.%)	CO/CO_2
EPO	532	/	1	0.06
EDP	135	-75	16	0.08
EDA	160	-70	3	0.15
EPOS	412	-23	4	0.04
ESDAM	249	-53	4	0.16
ESDPM	247	-54	10	0.08

Table 3: Results from cone calorimetry tests for the investigated samples (irradiative heat flux: 35 kW/m²).

Residue analysis. The abundant char residues after cone calorimetry test are shown in Figure 4. Table 4 collects the composition of the char residue after UL 94 vertical flame spread tests, evidencing a significantly higher %P retention in the residue of DP-containing samples compared to their initial P content (Table 1). The increased P content confirms a condensed phase activity of DP. In addition, the presence of melamine promotes a higher %P content in the residue of ESDPM with respect to EDP and ESDP (Table 4), which clearly indicates their condensed phase action [11, 13].
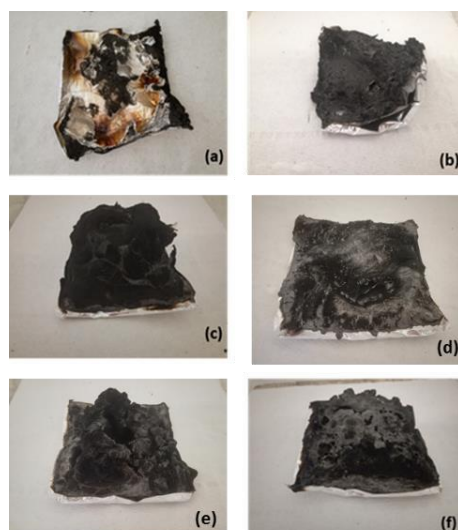

Figure 4: Char residues after CC tests for EPO (a), EPOS (b), EDP (c), EDA (d), ESDAM (e), ESDPM (f).

Sample	N (wt.%)	Si (wt.%)	P (wt.%)
EPO	1.8	/	/
EDP	/	/	4.5
EPOS	0.7	8.3	/
ESDP	3.4	4.3	2.9
ESDPM	4.1	3.8	8.6

Table 4: Energy-dispersive X-ray analysis (EDX) elemental composition of the char residue of the prepared epoxy composites.

Evolved gas analysis and flame-retardant mechanism. Evolved gas analysis with DIP-MS and PY-GC-MS of the epoxy samples showed the formation of a noticeable amount of flame inhibitors (i.e., active phosphorus species), responsible for gas flame inhibition especially in the case of DA (Figure 5), which confirmed a main gas phase activity in its flame-retardant mechanism [10]. Based on the fire studies and residue analysis, DP acted predominantly in the condensed phase, where its decomposition products reacted with the ammonia produced by melamine degradation, leading to the formation of P-N-O polymeric substructures and polyphosphoric acids in the char [11, 13]. This latter showed a higher retention of P (Table 4) and, especially in the presence of silica, acted as a thermal shield and oxygen barrier during the combustion [4].
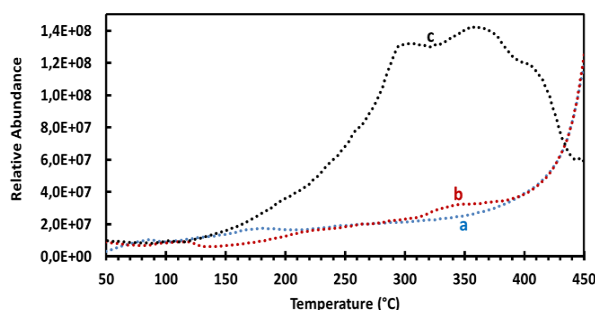

Figure 5: DIP-MS total ion thermograms of EPO (a), ESDPM (b), and ESDAM (c).

3.2 Epoxy-Si-P hybrid nanocomposites

Structure and morphology. The adopted synthetic strategy allowed for the incorporation of P moieties during the in-situ growth of silica lamellar nanostructures. The generation of these nanocrystals, resulting from TEOS hydrolysis within polar nanodomains embedded into an inverse micelle system, was accompanied by the formation of stable Si–O–P linkages inside the silica nanodomains, as suggested by solid-state NMR spectra [6]. A uniform dispersion of the inorganic component at the nanometric size was achieved in the polymeric matrix. EPOSP samples is visible in the TEM micrographs and P

(Figure 6). When DGEBA resin was modified by phosphoric acid alone, worm-like chain structures were observed, while in the presence of silane precursors, two families of nanocrystals with different interlayer distances were formed [6].
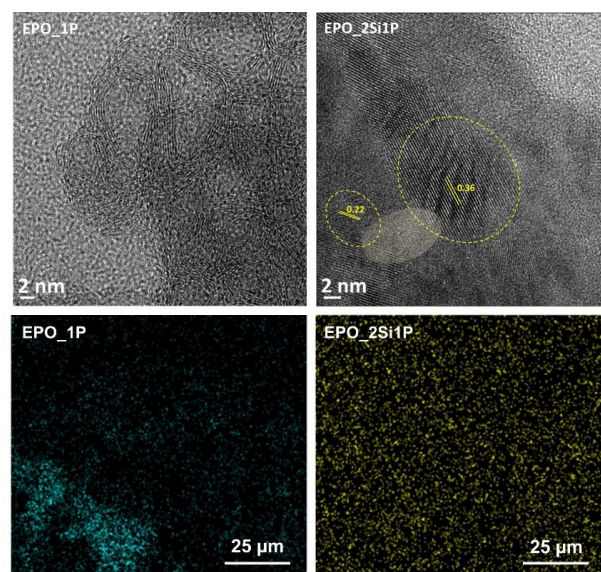

Figure 6: HRTEM images (top) and SEM-EDX maps of P distribution (bottom) of an epoxy-P (EPO_1P) and an epoxy-Si-P (EPO_2Si1P) sample [6]. Adapted from [6].

Thermal and fire behavior. EPOSP nanocomposites showed Tg values around 79 °C, about 16% lower than the neat resin network, because the presence of crystalline domains negatively affects the mobility of polymer chains by disturbing the inter-chain interactions and curing in the polymer matrix [6]. Their peculiar morphology translated into interesting flame-retardant features, even with Si and P contents around 1 wt.%: no dripping phenomena during UL 94 tests, the formation of a large amount of coherent and continuous char in nitrogen and air atmospheres, and the low CO/CO_2 ratio in the smokes obtained by CC, indicating a prevalent condensed phase mechanism.

3.3 Epoxy-Mg(OH)$_2$ nanocomposites

Morphology and fire behavior. Mg(OH)$_2$-based nanocrystals with pseudo-hexagonal symmetry were obtained in the presence of silanes by the in-situ sol-gel approach [7]. The EPOMg nanocomposite, despite the low loading (5 wt.%) of the nanofiller, revealed remarkably improved fire parameters: a decrease in heat release capacity (HRC, ~-34%) measured by PCFC, in HRR (~-29%) and peak of HRR (~-37%), as assessed by CC. Moreover, total smoke release and smoke extinction area were reduced by about 22 and 5%, respectively, suggesting that the embedded Mg(OH)$_2$ nanostructures may be effective as smoke suppressants [14].

Machine learning approach. An artificial neural network-based system was developed and tested to predict the HRC value of the prepared EPOMg nanocomposite, starting from the physico-chemical properties of a range of polymers and their PCFC parameters [14]. A machine learning approach was used, splitting the data into a training set and a test set, and a sensitivity analysis was performed to select the most relevant parameters and prove the accuracy of the implemented algorithm. Finally, the cross-validation method was employed, and relatively low error values were found. This example demonstrates that machine learning is a promising way to predict important properties of novel polymer composites.

Conclusion

Self-extinguishing in-situ silica-epoxy composites cured with cycloaliphatic curing agents can be prepared by the incorporation of melamine and phosphorus-based flame-retardants (i.e., DA and DP), even at relatively low P-loading. DA and DP show flame-retardant activity in both condensed and gas phases, through flame inhibition and thermal shielding mechanisms. These chemical modification strategies based on sol-gel chemistry represent sustainable processes to incorporate inorganic nanostructures into thermoset matrices employing inexpensive and safe reagents and solvents, and mild temperatures. The fine dispersion of the so-obtained nanoparticles in the polymer network accounts for considerable flame-retardant effects to be observed even with limited loadings (e.g., 2-5 wt.%). Furthermore, the application of machine learning methods opens advantageous perspectives for the study of thermal and fire performances of novel materials.

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Engineered Glass Powders as Fire Barrier Additives

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Glass frits, milled to a powder, are enjoying a resurgence of interest as environmentally friendly fire barrier and structural additives. Long used in a variety of polymers that include plastics, adhesives, sealants, fibre and wood products, a steady number of papers continue to be published showing interest across a broad spectrum of polymers (see Figure I).

Polymer	2023	2022	2021	2020	2019	2018	2017	2016	2015	2014	Previous	Total
Coatings		1									2	3
EDPM				1		1		1			1	4
Epoxy		1	1	1						1	4	8
Ethylene-vinyl Acetate (EVA)	1					1	1		1	1	1	6
Polydimethylsiloxane (PDMS)	1											1
Polyethylene	1										2	3
Polymethyl methacrylate (PMMA)		1										1
Silicon Carbide (SiC)			1									1
Silicone Rubber	2	1		1			1		2	1	8	16
Styrene-butadiene Rubber (SBR)					1			1			1	3
Other											14	14
	5	4	2	3	1	2	2	2	3	3	33	60

Figure 1

At least half of the papers call out the ceramification properties of specifically engineered frits as a primary source of functionality, acting both as a barrier and as a structural component (maintaining char). Additionally, many of the papers indicate fluxing and synergistic behaviours. Fluxing is most often discussed in terms of the melting glasses creating an environment where other associated elements can suspend and blend. Synergistic behaviour is often discussed in terms of char structure being enhanced and sometimes in terms of eutectic mixtures impacting the temperature characteristics of the combined elements.

Engineered Frits and Blends

Clearly, both fluxing and ceramification are desirable properties in fire-retardant systems. The fluxing process is an endothermic reaction that absorbs heat while reducing smoke and toxic fumes. Whereas the ceramification transition is desirable as a support structure and to create an impenetrable fire barrier.

Figure 2 Frits (above) milled to Powder

This is precisely the characteristics that M&CT Ltd., a St. Helens UK firm, developed using their depth of glass formulation knowledge. Its original and flagship product, manufactured and sold under the '8008' series consists of engineered glass frits smelted to precise formulations, which are then blended and milled to a powder for use in a variety of polymers. The original formula for more than 25 years has been performing successfully and remains a leading seller for the firm. The blend is composed of a low-melting frit with a higher temperature 'devitrifying' frit that, as blended, addresses the desired low melting endothermic fluxing with the higher temperature devitrifying structure-forming component.

How '8008' Works

In short, the actions of '8008' within polymers are as follows:

- Below 350°C: '8008' is an inert component in the host material.

- 400 - 450°C: Endothermic low-melt process begins, ultimately flowing over and around the burning host material and beginning to form a char.

- 750 - 800°C: The devitrifying component begins to act – passing from a glassy to a crystalline state creating structural and barrier properties.

- 1200°C: '8008' continues to act, retaining its integrity and protecting the host material.

A great advantage is that the action of '8008' is a physical change in state rather than a chemical process - making it compatible with all known fire retardant systems.

Evolution of '8008' and Associated Products

As indicated above, while the standard '8008' remains a leading seller, customer demand has driven the introduction of several variations and extensions upon the original formulation. Most significant among these include:

- Granularity. The standard '8008' is produced to a d50 of 30μm. Demand is increasing for particle size in the range of d50 of 5μm (classified as 'microfine'). Within this microfine range we also offer the removal of the coarse tail as an option. The higher surface area of the microfine products also form a continuous surface more readily.

- pH buffering. This has mainly addressed needs in acrylic resin systems where the normal alkaline '8008' is buffered to resolve stability issues.

- Recycled fillers. In this product line, a large proportion of the active glasses are blended with recycled glass (>60%) to address circular economy concerns and achieve a desirable price point.

- Low melt frits. These have been designed specifically to address the desire for fluxing characteristics in specialized applications.

Conclusion

With the above advantages, one would be amiss not to point up some known watch issues for those who may be contemplating the use of engineered glass powders as fire barrier additives. These include:

- '8008' is a high temperature fire barrier, meaning that ignition suppressant materials will likely be required.

- Devitrification is a bonding process and by its nature exothermic. This means that there will be an increase in the heat release rate during the devitrification process.

- While beyond the scope of this paper, there are possible electrical disadvantages when the frit mix fuses in a fire situation, particularly in electrical cable covering material. The inclusion of materials with free positive ions can be of considerable benefit.

In conclusion, engineered glass powders as fire barrier additives can be an important component of any researcher or formulator seeking to improve the fire performance of at-risk polymers and products.

About the Author

Dean Baker acquired M&CT Ltd in St Helens, Merseyside UK in 2021. In part, it was his interest in environmentally inert fire-retardant additives that drove his investment in the firm. A US citizen, in 2016 he became a resident of the UK moving with his wife and family. He resides in the Cotswolds region with his family.

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AIMPLAS is glad to announce that will be hosting the fourth edition of EcoFRam, en event promoted by the French Chemical Society that will take place on 22 and 23 May. Over the two days, the conferences will be devoted to the state of the art and the latest developments of fundamental and applied research concerning eco-friendly flame retardant additives and materials. CALL FOR SPEAKERS OPEN! Send us your abstract to marketing@aimplas.es

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