Sol-gel chemistry as a facile way for the synthesis of flame retardant hybrid epoxy nanocomposites

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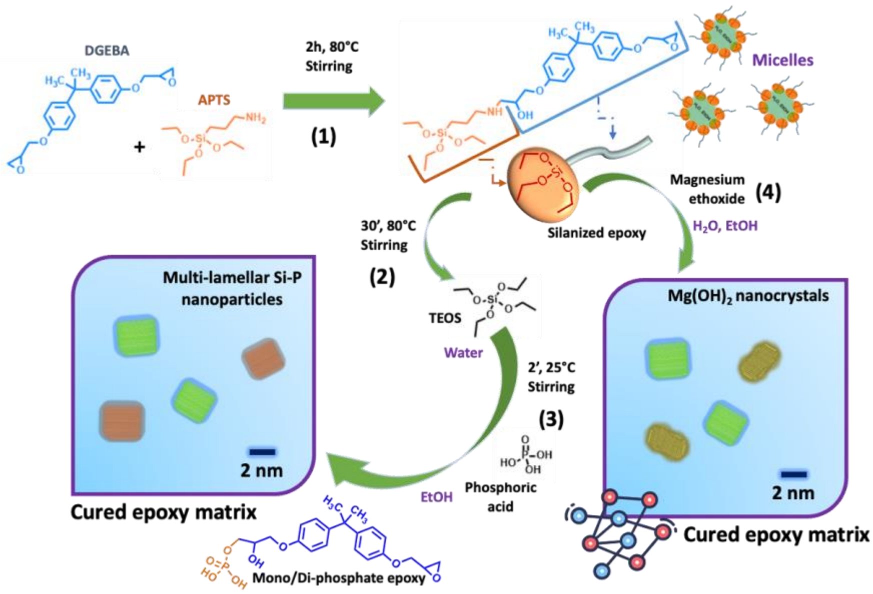

Figure. Possible routes for the sol-gel synthesis of flame retardant hybrid epoxy nanocomposites.

Introduction

Epoxy resins are suitable for the manufacturing of different materials for many industrial applications [1]. However, they are easily flammable, especially when cured with aliphatic amines, and thus the adoption of specific flame retardants (FRs) is commonly required. Among the available FRs, silicon- and phosphorus-based FRs are spreading on the market [1]. The in-situ sol-gel strategy allows for the preparation of no dripping self-extinguishing silica-epoxy nanocomposites. The in-situ growth mechanism of SiO_2 nanocrystals occurs through the formation of inverse micelle systems by hydrolysis and condensation reactions [1, 2]. This methodology can also lead to other inorganic nanostructures, for example Si-P oxides and $Mg(OH)_2$ [1-3].

Methods

Epoxy-based composites were prepared using APTS for the modification of DGEBA resin, TEOS, magnesium ethoxide, and phosphoric acid respectively as Si, Mg, and P precursors for the in-situ formation of inorganic phases, and a cycloaliphatic amine as the curing agent (see Figure). The morphological study of the obtained nanostructures was carried out by high-resolution transmission microscopy. Cone calorimetry and UL-94 vertical burning tests were performed to evaluate the composites' fire behavior and flammability, respectively.

Results

The chemical modification of DGEBA resin with APTS produces hybrid organic-inorganic epoxy moieties (see Figure), which can condensate with TEOS to form in-situ SiO_2 multi-lamellar nanoparticles [1, 2]. The concurrent addition of phosphoric acid and TEOS alters the multi-sheet morphology. Besides, replacing TEOS with magnesium ethoxide leads to the generation of in-situ $Mg(OH)_2$ nanoparticles [1, 3]. P-based FRs can act in synergy with the inorganic phases, promoting an effective flame retardancy during the combustion of epoxy nanocomposites [1, 3].

Discussion

All the in-situ modified epoxy nanocomposites exhibit high transparency, no dripping behavior, and a strong decrease (up ~50%) in the peak of the heat release rate (pkHRR) [1]. The use of DOPO-derivatives, or alternatively biowastes and ammonium polyphosphate, in silica-epoxy nanocomposites, accounts for UL-94 V-0 rating without any dripping and a reduction of pkHRR up to ~80%, even with a low P loading (i.e., 2 wt.%). Finally, fully connected feed-forward artificial neural networks can predict the heat release capacity of hybrid epoxy nanocomposites containing $Mg(OH)_2$ nanoparticles, limiting the number of required experimental measurements [1-3].

References

[1] Bifulco, Aurelio, et al. Journal of Sol-Gel Science and Technology (2022): 1-25.
[2] Bifulco, Aurelio et al. ACS Applied Nano Materials 6(9) (2023): 7422-7435.
[3] Bifulco, Aurelio, et al. Polymer Testing 127 (2023): 108175.