

INTERNATIONAL SOL-GEL CONFERENCE BERLIN 1 – 6 SEPTEMBER 2024

BOOK OF ABSTRACTS

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

Tuesday, 3rd September - 15:15: Nano- and micro-structured materials - Oral

Dr. Aurelio Bifulco¹, Dr. Claudio Imparato¹, Prof. Giulio Malucelli², Dr. Sabyasachi Gaan³, Prof. Antonio Aronne¹

 Department of Chemical, Materials and Production Engineering (DICMaPI), University of Naples Federico II, P.le Tecchio 80, 80125 Naples, Italy, 2. Department of Applied Science and Technology, Politecnico di Torino, Viale Teresa Michel 5, 15121 Alessandria, Italy, 3. Laboratory for Advanced Fibers, Empa Swiss Federal Laboratories for Materials Science and Technology, Lerchenfeldstrasse 5, 9014 St. Gallen, Switzerland

Introduction

Epoxy resins are suitable for the manufacturing of different materials for many industrial applications. However, they are easily flammable, especially when cured with aliphatic amines, and thus the adoption of flame retardants (FRs) is commonly required. Among the available FRs, silicon(Si)- and phosphorus(P)-based FRs are spreading on the market. The in-situ sol-gel strategy allows for the preparation of no dripping self-extinguishing silica-epoxy nanocomposites. The in-situ growth mechanism of SiO₂ nanocrystals occurs through the formation of inverse micelle systems by hydrolysis and condensation reactions. This methodology can also lead to other inorganic nanostructures, for example Si-P oxides and Mg(OH)₂ [1].

Methods

Epoxy-based composites were prepared using 3-aminopropyltriethoxysilane (APTS) for the modification of DGEBA resin, tetraethoxysilane (TEOS), Mg(OC₂H₅)₂, and phosphoric acid respectively as Si, magnesium (Mg), and P precursors for the in-situ formation of inorganic phases, and a cycloaliphatic amine as hardener (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

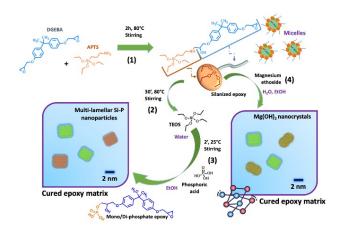
The chemical modification of DGEBA resin with APTS produces hybrid organic-inorganic epoxy moieties (see Figure), which can condensate with TEOS to form in-situ SiO₂ multi-lamellar nanoparticles. The concurrent addition of phosphoric acid and TEOS alters the multi-sheet morphology. Besides, replacing TEOS with $Mg(OC_2H_5)_2$ leads to the generation of in-situ $Mg(OH)_2$ nanoparticles. P-based FRs can act in synergy with the inorganic phases, promoting an effective flame retardancy during the combustion of epoxy nanocomposites.

Discussion

All the in-situ modified epoxy nanocomposites exhibit high transparency, and a strong decrease (up ~50%) in the peak of the heat release rate (pkHRR). 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., 1-2 wt.%). Finally, artificial neural networks can predict the heat release capacity of hybrid epoxy nanocomposites containing $Mg(OH)_2$ nanoparticles, limiting the number of experimental measurements [2].

References

[1] ACS Applied Nano Materials 6(9) (2023): 7422-7435.



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