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Abstract

Sol-Gel Complements Conventional Strategies for the Synthesis of Self-Extinguishing Hybrid Silica-Epoxy Nanocomposites †

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Introduction. Growing industrial demand, together with rising pollution and the depletion of phosphorus stores, is moving the scientific community towards the development of flame-retardant (FR) epoxy nanocomposites (ENCs) containing low levels of P and more sustainable additives [1].

Methods. High-resolution transmission electron microscopy (HRTEM) analysis was carried out to study the morphology of ENCs. Cone calorimetry (CC) and UL-94 vertical flame spread tests were performed to investigate the fire responses of all ENCs.

Results. The reaction of DGEBA (Bisphenol A diglycidyl ether)- or Novolac-based resins with APTES (3-aminopropyltriethoxysilane) allowed the production of organic-inorganic silanized epoxy moieties [2–4]. The hybrid moieties could condense with tetraethyl orthosilicate (TEOS), a silica precursor, to form an in situ silica phase through sol-gel reactions [3,5]. HRTEM analysis revealed that in the case of DGEBA, the silica phase was composed of well-ordered multi-lamellar nanoparticles (NPs). In contrast, the investigation of Novolac highlighted that fully amorphous silica NPs were embedded in the hybrid co-continuous polymer network [3]. The incorporation of DOPO-based FRs into silica-epoxy systems based on DGEBA/Novolac resin produced aliphatic nanocomposites with high transparency, no-dripping UL-94-V0 rating, and a strong decrease (up to 80%) in the peak of the heat release rate in CC tests, with a P loading of up to 3 wt.%. Regarding Novolac, the transparency remained even at silica NP loadings beyond 4 wt.% thanks to their amorphous nature [3]. More waste-to-wealth approaches involve the use of humic acids or biochar from spent coffee grounds, together with ammonium polyphosphate and urea, in APTES-modified DGEBA-based epoxy systems to obtain no-dripping, self-extinguishing systems, with a P content of only 1 wt.%.

Conclusions. The sol-gel in situ generation of inorganic phases has been explored in combination with DOPO-based FRs, bio-wastes, and other synergists to prepare no-dripping, self-extinguishing (V-0 rating in UL-94 flammability tests) aliphatic ENCs, even while keeping P at low loadings (1–3 wt.%).

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