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SILSESQUIOXANE SYNTHESIS AND INCORPORATION INTO ORGANIC/INORGANIC HYBRIDS BASED ON EPOXY RESIN

Letícia Oliveira Alves[(1)], Elizabete Yoshie Kawachi[(1)], Silvana Navarro Cassu[(2)]

[(1)]Instituto Tecnológico de Aeronáutica, Brazil
leticia.olialves@gmail.com, bete@ita.br
[(2)]Instituto de Aeronáutica e Espaço, Brazil
silvanasnc@iae.cta.br

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Summary: Silsesquioxane structures have been incorporated into polymeric matrices in order to improve certain properties, such as mechanical and thermal resistance. Nonetheless, the rules which govern the behavior of such organic/inorganic hybrids are not always clear. In this study, a silsesquioxane containing epoxy functionality (SSQO-E) was incorporated into DGEBA (diglycidyl ether of bisphenol A) in different concentrations, up to 20 wt%. In this epoxy system, DETA (diethylenetriamine) was used as the curing agent. The SSQO-E was synthesized from GPTMS (3-glycidoxypropyl trimethoxysilane) through a hydrolytic condensation reaction, using tetramethylammonium hydroxide as a catalyst. The molar mass of the SSQO-E was estimated by SEC to have a value close to the cubic cage silsesquioxane structure (SSQO-T8). 29Si NMR and 1H NMR spectra of SSQO-E were also similar to the SSQO-T8, presenting predominately completely condensed silsesquioxane molecules. In addition, DGEBA/SSQO-E/DETA hybrids were prepared in a tetrahydrofuran/DGEBA medium to ensure the homogeneous dispersion of SSQO-E into the epoxy resin, which was verified by the single glass transition temperature (Tg) values (determined by differential scanning calorimetry, DSC) shown by the organic/inorganic hybrids. It was observed that Tg decreased with SSQO-E content, with a lowering of approximately 20 ºC for the 20 wt% SSQO-E content hybrid in relation to the Tg of epoxy resin, whilst the free volume (investigated by comparing the heat capacity change, Δcp, measured during the material glass transition) of the organic/inorganic hybrids showed no significant variation in relation to the epoxy resin. The cross-link density (evaluated by the swelling of the systems in THF solvent) was observed to increase with SSQO-E content despite the increase in bulkier SSQO-E molecules. The opposite effects of increasing in cross-link density and decreasing in Tg for higher SSQO-E content observed in the organic/inorganic hybrids may be attributed to: the presence of the more flexible 2-hydroxypropyl-n-propylether ((CH2)3-O-CH2-CH(OH)-CH2-) groups in DGEBA/SSQO-E/DETA hybrids in contrast to the less flexible hydroxypropyl ether (-O-CH2-CH(OH)-CH2-) groups present in epoxy resin; and to the higher number of flexible groups in hybrids (eight flexible groups per SSQO-E molecule) compared to the number in epoxy resin (two flexible groups per DGEBA molecule).