

CURING KINETICS OF CYANATE ESTER RESIN WITH 3-AMINOPROPYL OLIGOSILSESQUIOXANE

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In the present study, the kinetic peculiarities of dicyanate ester of bisphenol E (DCBE) polycyclotrimerization to high-performance polycyanurate (PCN) in the presence of 3-aminopropyl oligosilsesquioxane (AP-OSS) with contents of 0.1, 0.5 and 1.0 wt.% were investigated by DSC technique. AP-OSS was synthesized by the hydrolysis and polycondensation of trifunctional monomer γ -aminopropyltrimethoxysilane. Mass spectrometry with matrix-assisted laser desorption ionization (MALDI) technique has been used to characterize AP-OSS synthesized. The investigations were performed using DSC 25 differential scanning calorimeter in nitrogen atmosphere by heating from 25 to 350 °C at a heating rate of 5 °C/min.

The catalytic effect of AP-OSS was found for all the compositions studied. The values of the exotherm peak temperature (T_p) and the reaction enthalpy (ΔH) of the cured PCN-based nanocomposites were determined. It was established that for the individual DCBE sample, a T_p value was equal to ~272 °C (Fig. 1a). For the DCBE/AP-OSS compositions the corresponding T_p values shifted toward much lower temperatures, viz. to $T_p \sim 248$ –261 °C in dependence on the content of AP-OSS. Furthermore, the enthalpy of the cure reaction ΔH for the individual DCBE was calculated to be $\Delta H \sim 974$ J/g and it decreased significantly (to $\Delta H \sim 817$ –860 J/g) depending on the content of AP-OSS for all the compositions studied. The catalytic effect of the AP-OSS is also confirmed by the time dependencies of the conversion (α) of cyanate groups of DCBE since for the DCBE/AP-OSS compositions the reduction of induction period of the reaction was observed and the polymerization started early (Fig. 1b).

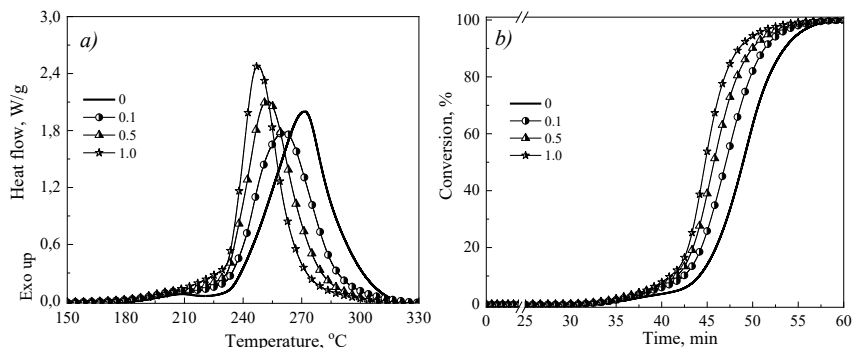


Fig. 1. DSC kinetic profiles (a) and time dependencies of conversion (b) for the individual DCBE and DCBE/AP-OSS compositions (AP-OSS content indicated in the plot)

We conclude that the thermal polycyclotrimerization of DCBE is catalyzed by the reactive primary amino groups of synthesized AP-OSS, that provides polymerization of DCBE at lower temperatures.