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Mechanism of Action of New Sustainable Flame Retardant Additives in Polyamide 6,6

Authors: I. Belyamani, M. K. Hassan, J. U. Otaigbe, W. R. Fielding, K. A. Mauritz, J. S. Wiggins, W. L. Jarrett

Abstract: We have investigated the flame-retardant efficiency of special new phosphate glass (P-glass) compositions having different glass transition temperatures (Tg) on the processing conditions of polyamide 6,6 (PA6,6) and the final hybrid flame retardancy (FR). We have showed that the low Tq P glass composition (i.e., ILT 1) is a promising flame retardant for PA6,6 at a concentration of up to 15 wt. % compared to intermediate (IIT 3) and high (IHT 1) Tg P glasses. Cone calorimetry data showed that the ILT 1 decreased both the peak heat release rate and the total heat amount released from the PA6,6/ILT 1 hybrids, resulting in an efficient formation of a glassy char layer. These intriguing findings prompted to address several questions concerning the mechanism of action of the different P glasses studied. The general mechanism of action of phosphorous based FR additives occurs during the combustion stage by enhancing the morphology of the char and the thermal shielding effect. However, the present work shows that P glass based FR additives act during melt processing of PA6,6/P glass hybrids. Dynamic mechanical analysis (DMA) revealed that the Tg of PA6,6/ILT 1 was significantly shifted to a lower Tg (~65 oC) and another transition appeared at high temperature (~ 166 oC), thus indicating a strong interaction between PA6,6 and ILT 1. This was supported by a drop in the melting point and crystallinity of the PA6,6/ILT 1 hybrid material as detected by differential scanning calorimetry (DSC). The dielectric spectroscopic investigation of the networks' molecular level structural variations (i.e. hybrids chain motion, Tg and sub-Tg relaxations) agreed very well with the DMA and DSC findings; it was found that the three different P glass compositions did not show any effect on the PA6,6 sub-Tg relaxations (related to the NH2 and OH chain end groups motions). Nevertheless, contrary to IIT 3 and IHT 1 based hybrids, the PA6,6/ILT 1 hybrid material showed an evidence of splitting the PA6,6 Tg relaxations into two peaks. Finally, the CPMAS 31P-NMR data confirmed the miscibility between ILT 1 and PA6,6 at the molecular level, as a much larger enhancement in cross-polarization for the PA6,6/15%ILT 1 hybrids was observed. It can be concluded that compounding low Tg P-glass (i.e. ILT 1) with PA6,6 facilitates hydrolytic chain scission of the PA6,6 macromolecules through a potential chemical interaction between phosphate and the alpha-Carbon of the amide bonds of the PA6,6, leading to better flame retardant properties.

Keywords: broadband dielectric spectroscopy, composites, flame retardant, polyamide, phosphate glass, sustainable

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