Influence Study of the Molar Ratio between Solvent and Initiator on the Reaction Rate of Polyether Polyols Synthesis

Authors : María José Carrero, Ana M. Borreguero, Juan F. Rodríguez, María M. Velencoso, Ángel Serrano, María Jesús Ramos Abstract : Flame-retardants are incorporated in different materials in order to reduce the risk of fire, either by providing increased resistance to ignition, or by acting to slow down combustion and thereby delay the spread of flames. In this work, polyether polyols with fire retardant properties were synthesized due to their wide application in the polyurethanes formulation. The combustion of polyurethanes is primarily dependent on the thermal properties of the polymer, the presence of impurities and formulation residue in the polymer as well as the supply of oxygen. There are many types of flame retardants, most of them are phosphorous compounds of different nature and functionality. The addition of these compounds is the most common method for the incorporation of flame retardant properties. The employment of glycerol phosphate sodium salt as initiator for the polyol synthesis allows obtaining polyols with phosphate groups in their structure. However, some of the critical points of the use of glycerol phosphate salt are: the lower reactivity of the salt and the necessity of a solvent (dimethyl sulfoxide, DMSO). Thus, the main aim in the present work was to determine the amount of the solvent needed to get a good solubility of the initiator salt. Although the anionic polymerization mechanism of polyether formation is well known, it seems convenient to clarify the role that DMSO plays at the starting point of the polymerization process. Regarding the fact that the catalyst deprotonizes the hydroxyl groups of the initiator and as a result of this, two water molecules and glycerol phosphate alkoxide are formed. This alkoxide, together with DMSO, has to form a homogeneous mixture where the initiator (solid) and the propylene oxide (PO) are soluble enough to mutually interact. The addition rate of PO increased when the solvent/initiator ratios studied were increased, observing that it also made the initiation step shorter. Furthermore, the molecular weight of the polyol decreased when higher solvent/initiator ratios were used, what revealed that more amount of salt was activated, initiating more chains of lower length but allowing to react more phosphate molecules and to increase the percentage of phosphorous in the final polyol. However, the final phosphorous content was lower than the theoretical one because only a percentage of salt was activated. On the other hand, glycerol phosphate disodium salt was still partially insoluble in DMSO studied proportions, thus, the recovery and reuse of this part of the salt for the synthesis of new flame retardant polyols was evaluated. In the recovered salt case, the rate of addition of PO remained the same than in the commercial salt but a shorter induction period was observed, this is because the recovered salt presents a higher amount of deprotonated hydroxyl groups. Besides, according to molecular weight, polydispersity index, FT-IR spectrum and thermal stability, there were no differences between both synthesized polyols. Thus, it is possible to use the recovered glycerol phosphate disodium salt in the same way that the commercial one.

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Keywords : DMSO, fire retardants, glycerol phosphate disodium salt, recovered initiator, solvent

Conference Title : ICP 2017 : International Conference on Polymer

Conference Location : Paris, France **Conference Dates :** June 25-26, 2017