The control of hydroxyl density on glass particulates surface: Application to anionic polymerization of polyamide 6

Achraf Belkhiri, Nick Virgilio, France Chabert, Olivier de Almeida, Hélène Welemane, Valérie Nassiet

The control of hydroxyl density on glass particulates surface: Application to anionic polymerization of polyamide 6

BELKHIRI Achraf[1], VIRGILIO Nick[3], CHABERT France[1], DE ALMEIDA Olivier[2], WELEMANE Hélène[1] and NASSIET Valérie[1]

1 : Laboratoire Génie de Production (LGP), Université de Toulouse, INP-ENIT, Tarbes, France
E-mail : achraf.belkhiri@enit.fr, france.chabert@enit.fr, helene.welemane@enit.fr , valerie.nassiet@enit.fr
2 : Institut Clément Ader, Université de Toulouse, CNRS UMR 5312, IMT Mines Albi, UPS, INSA, ISAE-SUPAERO, Campus Jarlard, 81013 Albi, France
E-mail : olivier.dealmeida@mines-albi.fr
3: CREPEC, Department of Chemical Engineering, Polytechnique Montréal, Montréal, Canada
nick.virgilio@polymtl.ca

Monomer polymerization after the impregnation of reinforcements is a relevant option for the manufacturing of thermoplastic composites by liquid processes (infusion or RTM). In the case of polyamide 6 (PA6) synthesized by this process, the system is composed of the monomers, the catalyst, and the activator, which react within the fibrous environment. Therefore, controlling the chemistry of the fiber surface is crucial because it influences not only the polymerization and crystallization processes, but it also controls the fiber-matrix adhesion and the resulting mechanical properties. However, the hydroxyl groups typically present on the glass fiber surface inhibit the polymerization process.

The objective of this project is to adapt and control the surface chemistry of glass particulate reinforcements in order to promote the polymerization and crystallization processes of PA6. This work was carried out with glass microbeads comparable in size to the diameter of glass fibers.

First, the evolution of the hydroxyl groups surface density on the glass beads, as a function of calcination time, was monitored by thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR). The hydroxyl groups surface density was then tuned in order to allow the synthesis of anionic polyamide 6 (PA6), which was monitored by differential scanning calorimetry (DSC). Next, in order to further promote the polymerization and crystallization of PA6, and to improve interfacial adhesion between the PA6 and glass particulates, the grafting process of an amino-silane coupling agent at the particulates surface was investigated by TGA and FTIR. The results demonstrate that the competition between re-hydroxylation and condensation of the silane on the surface during the grafting process needs to be carefully balanced in order to maintain fast polymerization kinetics, as revealed by DSC analysis. Overall, the systematic methodology presented in this work can be adapted for various combinations of reactive resins and solid fillers, allowing for the preparation of high performance (nano)composite materials.