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Tailoring glass fiber surface for the polymerization and crystallization of anionic polyamide 6

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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 synthesized by this process, the system composed of monomers, catalyst and activator reacts within the fibrous environment. Therefore, fiber surface chemistry is a crucial parameter because it does not only influence the polymerization and crystallization processes, but it also controls the fiber-matrix adhesion.

This project aims at adapting the surface chemistry of glass reinforcements so as to strengthen the interactions at the composite interfaces, but also in order to promote the polymerization and crystallization processes of anionic polyamide 6 (PA6). Indeed, the chemistry of surface reinforcement strongly affects polymerization kinetics and therefore final properties of glass fibers / PA6 composites. This work was carried out with glass microbeads comparable in size to the diameter of glass fibers.

First, the glass particulates must be adequately calcinated since the reaction catalyst is highly sensitive to moisture and surface-bound hydroxyl groups. As a result, the evolution of the hydroxyl groups surface was monitored as a function of calcination time by thermogravimetric analysis (TGA) and Fourier transform infrared spectroscopy (FTIR). The hydroxyl groups surface density was then carefully controlled in order to enhance the synthesis of anionic PA6. Then, a silane coupling agent was grafted on the glass particulates to improve interfacial adhesion and to promote even further the polymerization and crystallization of PA6. The grafting process was assessed by TGA and FTIR. Differential scanning calorimetry (DSC) analyses demonstrate that the competition between re-hydroxylation and condensation of the silane on the glass surface during the grafting process needs to be carefully balanced in order to maintain fast polymerization kinetics.

Once the silane grafting protocol was optimized, different silane-based coupling agents were compared. Three types of interactions at the particulate-matrix interface were considered. First, the glass surface was treated with a hydrophobic silane minimizing the interactions at the interface. Second, an amino-silane leading to the formation of weak bonds (hydrogen bonds) between the glass surface and the matrix was investigated. Finally, the glass beads were also treated with a silane end-functionalized with the reaction activator, allowing the formation of covalent bonds between the particulates surface and the matrix.

After treatment, the glass particulates surfaces were characterized using contact angle measurements, TGA, FTIR, X-ray photoelectron spectroscopy (XPS) and confocal microscopy. The results confirm successfully silane grafting onto the glass surface. Then, glass particulates/anionic PA6 composites synthesis was investigated by DSC to examine the influence of the chemical nature of the treatment on the polymerization time and the degree of crystallinity. The DSC results, combined with the surface characterization results, allowed to determine the effect of the surface treatment on the polymerization and crystallization, and thus to optimize the synthesis process.

Next, the fiber-matrix interfacial adhesion will be evaluated by means of tensile tests on composite specimens, allowing to quantify the influences of the grafting process and type of silane on the resulting macroscopic mechanical properties. Conclusions will help in getting an optimal interaction at the reinforcement / matrix interface, and in improving the design of composite fiber reinforced polymers.