Post-treatments of injected polyamides parts using supercritical fluids

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1. Introduction

High-tech fields require materials exhibiting a large range of physical properties (flame retardancy, long-term stability versus hydrolysis, color stability versus light, antistatic surface properties...). Unfortunately, it is very often difficult to provide all these properties by only adding chemicals during the extrusion/injection process. This is due to chemical incompatibility versus the various wished chemicals and the temperature required during the extrusion or injection. An elegant way to solve this issue is to post-process the plastic parts. This allows the injection/extrusion process to be simplified bringing a cost advantage and in second way to provide flexibility to the production chains (plastic parts will be tailored for a specific use). Therefore, it is also another way to bring higher value to low cost plastic parts. In this work, we looked for a way to improve the stability versus water moisture of injected commercial plastic parts [1] of PA 6-6 composites by using supercritical fluids impregnation of various chemicals. Polyamide (PA) materials are commonly used in many engineering components and structures. Among them, PA 6 and PA 6-6 showing strong hydrogen bond interaction in their chemical structures, which ensure high mechanical performances, are of particular interest. However according to the moisture –related works on these materials both PA 6 and PA 6-6 are sensitive to moisture. They absorb more than 8 wt % in 100% relative humidity at room temperature. Frequently their mechanical behaviors are improved by adding glass fibers; this also reduces the water uptakes at 5 wt%. This moisture absorption affects mechanical properties (Young modulus and tensile stress) damages and induces dimensions and shapes of the parts. We decided to modify by an impregnation post-treatment using supercritical fluids (CO2 and Butane) PA 6-6 composites plastic parts. In terms of chemistry, we modified the polyamide matrix by using isocyanate compounds and in situ radical polymerization with cocktails of monomers and oligomers [1]. The impact of both the supercritical fluids used and chemicals treatment on the mechanical performances and the water uptakes of the treated parts will be discussed. We worked on two kinds of commercial materials, both containing a similar loading rate in silica fibers but having different silica fibers sizing.

2. Materials and Methods

Our experiments have been carried out on standard traction test dogbones (ISO3167) coming from suppliers. 10 or 12 dogbones have been processed in the same manner. Before experiments, dogbones were conditioned at 100 °C during 12 h. Chemicals have been used without any kind of purification. Supercritical fluids were CO2 or butane. Ethanol, acetone and methanol have been used as co-solvents.

After post-processing, the dogbones have been conditioned at 90 °C in water steam during 1000 h before to perform the water intakes measurements. Mechanicals tests have been performed just after the post-processing and after the water steam conditioning.

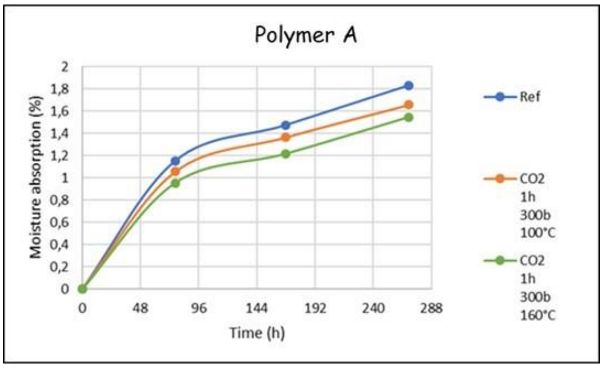

Figure 1. Impact of post-process treatments on the moisture absorption versus ageing in vapor water .

3. Results and discussion

The post-processes of composites PA 6-6 parts by chemicals grafting using supercritical fluids allowed the water uptakes to be reduced by 43% versus untreated parts after a 1000 h of ageing in hot water vapor.

However, we observed a loss of the initial mechanical performances due the supercritical fluids treatments even without any adding of chemicals. We compared the impact of two different supercritical fluids, CO2 and butane. It appeared that this sensitivity is probably due to the glass fiber sizing additives.

We evaluated the diffusion of chemicals inside the composite PA 6-6 by using fluorescent dyes both for supercritical CO2 and for supercritical butane. All the experiments in supercritical fluids have been carried out in the batch way. The impact of the process parameters will be discussed.

Finally, we will discuss of the impacts of used chemistry both on water uptake and on mechanical performances after 1000 h ageing in steam at 90 °C.

4. Conclusions

We clearly demonstrated that a post-process of composite PA 6-6 parts by covalent bonding chemistry using supercritical fluids as vector of impregnation could be advantageously used to modify physical properties. The flexibility, in terms of chemistry, of the supercritical fluid processing wherein the solvent properties can be tuned by acting on pressure, temperature opens a large field of investigation.

References

1. Olivier Poncelet, Daniel Getto, Olivier Renard EP3838570(A1)