A STUDY ON THE FILTRATION OF NANO AND MICRO PARTICLES IN LIQUID COMPOSITE MOLDING PROCESSES

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Introduction

When liquid composite molding (LCM) techniques are employed using a filled resin, particles are transported by the resin and can be captured by the fibrous bed, which acts as a filter. The trapped particles can decrease preform porosity and therefore its permeability, increasing the resistance to flow [1]. Moreover, as the filtration takes place, a gradient in the suspension concentration can be developed along the flow path. This inhomogeneous particle distribution remains in the material microstructure after the resin cures, leading to uneven properties of the finished part [2]. Understanding filtration phenomena during LCM processing of composite materials using particle filled resins is important to manufacture defect free composite parts. In this work, composites were obtained by RTM using two types of fillers: glass micro spheres and nano clays. The filtration of the particles and its effect on processing conditions and composite final microstructure were studied.

Experimental procedure

Glass fiber random mat (300 g/m^2) was used as the reinforcement and an epoxy-amine resin system was used as the polymeric matrix. The resin was loaded with 5, 10 and 20% by weight of glass microspheres (diameter 80 μm) and 1% 2.5% and 5% by weight of organically modified bentonite (Cloisite 30B). In order to improve clay dispersion in the liquid resin, samples were immersed in an ultrasonic bath for 30 minutes at 30^{0}C before adding the cycloaliphatic amine, to prevent reaction to occur during that stage. Rheological tests were conducted under nitrogen atmosphere. The viscosity evolution with temperature was monitored from 14^{0}C to 24^{0}C (laboratory temperature range for the infusion experiments). Unidirectional injection experiments were performed in a rectangular steel mold (3.2mm thick) with a 30 mm acrylic lid. Different fiber volume fractions were obtained by changing the number of fabric layers. The volume of suspension filtrated by the fibrous bed was normalized to obtain comparable results among the different tests. The resin was cured at 105 ^{0}C for 1h. A discontinuous preform conformed by several strips of reinforcement was used, which allowed obtaining cured composite samples named as C1, C2…, C_i (to be used for the observation of particle deposition mechanisms by scanning electron microscopy); and cured suspension samples named as S1, S2,…S_i (to estimate the particle content as a function of the filtration length, by thermo gravimetric analysis).

Results

The effect of the fillers on the resin viscosity is shown in Figure 1. Results can be summarized as follows: adding 5% of glass spheres did not affect the neat resin viscosity; above that value, the higher the concentration of glass spheres, the higher the viscosity of the suspension; due to their very small size and low sphericity, very small amounts of nano clays significantly increased the viscosity of the suspension, which was higher with 1% of nano clays than with 20% of glass spheres; the highest viscosity was shown by the suspension with 2.5% of nano clays. In fact, increasing the particle concentration to 5% reduced the suspension viscosity. This was attributed to the high tendency of the nano particles to agglomerate, leading to the formation of clusters which are bigger and more spherical than individual clay platelets. For this reason, 2.5% was chosen as the clay content for the experiments. The permeability of the reinforcement was measured using standard procedure and it was found that both fillers decreased the measured value of this property (Figure 2). Glass micro spheres dramatically reduced this property, as a consequence of the cake formed at the beginning of the preform. Figure 3 shows pictures of the first 12 cm of the cured composite parts. It was impossible to detect clay filtration by visual observation of the samples, at any fiber volume fraction. On the other hand, glass microspheres were partially filtrated when 27% fiber volume content was set, leading to a deep filtration mechanism, in which the suspension flows within the preform but it progressively captures particles. When a fiber volume content of 47% was set, glass micro spheres could not enter into the microstructure of the fabrics, thus surface filtration occurred where the gradual accumulation of particles caused a filtration cake to grow as the injection proceeded. TGA experiments are currently being carried out in order to quantify the % of trapped particles as a function of the preform length. Furthermore, scanning electron microscopy will be performed to analyse filtration mechanisms. These results are expected to be ready and presented at the FPCM-12 conference.

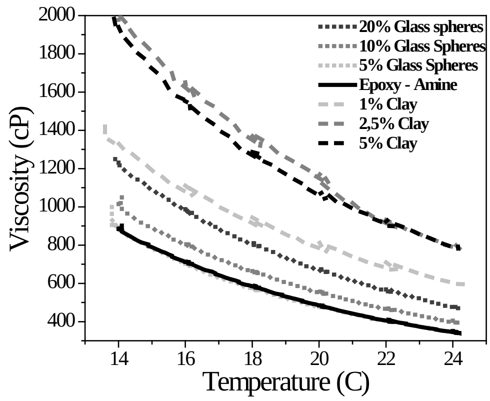


Figure1: *Rheology results.*

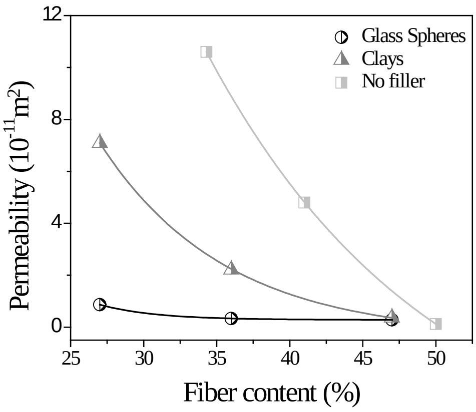
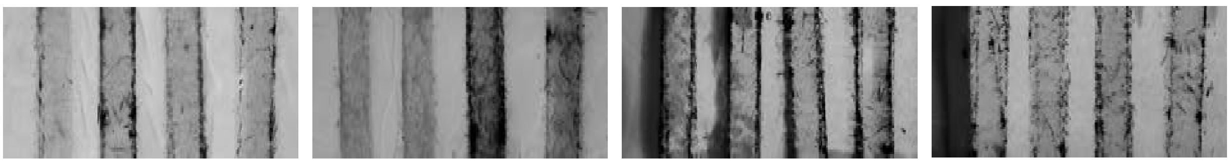


Figure 2: *Permeability results.*

Vf = 0.27 Vf=0.47 Vf = 0.27 Vf=0.47



2.5% Nano Clays 20% Glass Spheres

Figure 3: *Visual observation of the cured composites. The inlet is located at the left of each picture.*

References

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