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NEW TRENDS IN ON-LINE RHEOMETRY TO STUDY THE RHEOLOGICAL PROPERTIES OF NANOCOMPOSITES

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ABSTRACT

On-line rheometers are usually employed for quality control purposes, but they can also be used to detect changes in structure, or composition of a given material system, thus assisting materials research and processing optimization. A new on-line rotational rheometer was developed with the intention of studying the morphological changes of complex materials during melt compounding. The scope of this work is to address on-line rheometry as an emergent characterization technique to study the evolution of exfoliation/intercalation of layered silicates during compounding and to validate its scale-up from the lab to the industrial scene.

INTRODUCTION

Nowadays it is widely known that polymer nanocomposites with interesting physical (e.g, barrier to gases, flame retardancy, electrical conductivity) and mechanical properties can be obtained by dispersing adequately specific particles, such as organoclays or carbon nanotubes, in a polymeric matrix (LeBaron, Wang et al. 1999; Koo 2010). The melt intercalation is accepted as the most promising nanocomposite production technique, as it can be scaled-up to industrial practice and is environmentally sounder, since it is undertaken via extrusion compounding. When preparing such materials by this technique, the engineer/scientist is confronted to a plethora of variables concerning both, material properties and processing conditions (Lertwimolnun and Vergnes 2006), that must be well understood in order to obtain a final product with tailored features for a given field of application. As it was already been stated by many authors (Galgali, Ramesh et al. 2001; Solomon, Almusallam et al. 2001; Bousmina 2006; Eslami, Grmela et al. 2010), rheology is a powerful tool for understanding the behaviour and morphological changes in polymer nanocomposites under flow. Thus, the use of on-line rheometers to monitor the evolution of dispersion and exfoliation of nanoparticle clusters during compounding seems to be striking. Such devices have been extensively used by the authors of this work (Covas, Carneiro et al. 2002; Covas, Maia et al. 2008; Mould, Barbas et al. 2009) as a means of measuring the flow properties of polymeric melts during/along the extrusion process.

EQUIPMENT

The new generation of on-line parallel plate rheometer with automatic capabilities developed by our group is illustrated on Figure 1. This equipment can be assembled to any type of extruder machine by means of a special adapter designed for the purpose:

Figure 1. On-line rotational rheometer attached to an extruder

EXPERIMENTAL VALIDATION

The equipment was assessed taking into account its mechanical and thermal validity. The results were compared with the results obtained a commercial benchtop rheometer (Paar Physika MCR300). The confrontation of the results, plotted on Figures 1 and 2, shows that there is a perfect match between the on-line device and the commercial equipment:

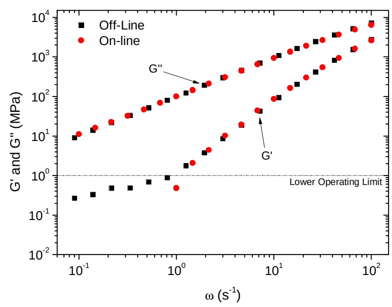

Figure 2. Mechanical validation results obtained from small amplitude oscillatory shear test using silicon oil (Wacker 100000, 100Pa.s)

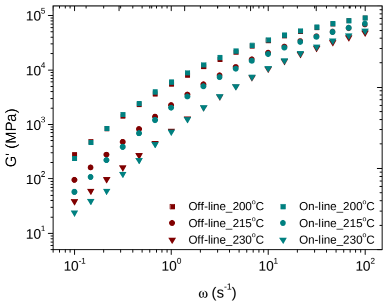

Figure 3. Thermal validation results obtained from small amplitude oscillatory shear test using PS (Edistir N 1840) at three different temperatures

POLYMER NANOCOMPOSITES: CASE OF STUDY

Several experiments with different polymer nanocomposites formulations and processing conditions were performed on-line in order to measure the viscoelastic properties to predict the nanoparticle dispersion of the systems tested. Figure 4 shows an example of how nanoclay loading can influence the flow behavior at the low frequency region, giving the material a solid-like behavior, possibly due to the formation of networks at the dispersed phase:

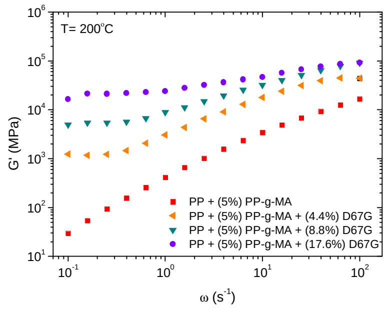

Figure 4. Storage modulus of a PP layered-silicate nanocomposite with different clay loadings

CONCLUSIONS

An improved version of an already existent on-line rotational rheometer was developed showing outstanding capabilities for on-line extrusion monitoring. The results obtained from validation are in good agreement with results obtained from commercial benchtop rheometers at the same test conditions. Also, as a concluding remark, we can consider that the on-line rheometer developed is sensitive to detect morphological changes in polymer nanocomposites. The main outcome of this work is a better understanding of the parameters concerning both, materials and processing conditions, which will help the scientist/engineer to develop new and better nanostructured materials with tailored properties.

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