

Short communication: test method

Prediction of the flow curves of thermoplastic polymer/clay systems from torque data



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ABSTRACT

The aim of this work was to determine some relevant rheological parameters of polymer/clay systems using mixing torque and mixing speed data obtained during processing in an internal mixer. The method used was originally proposed by Marquez et al. for monophasic polymers, and is here applied to polymer/clay systems for the first time. Several clay-containing composites based on different polymer matrices (i.e., LDPE, HDPE, PA6, EVA) were used to verify the effectiveness of the method for measuring the flow curves of these polymer/clay systems. The results indicated that, for all the systems at low clay level, the rheological curves calculated with the Marquez method fit quite well those measured in both rotational and capillary rheometers. On the contrary, on increasing the amount of clay, the flow curves estimated by the melt mixing torque data still fit those measured by rotational rheometer while diverging from those measured by the capillary viscometer. Indeed, the polymer-clay systems do not obey the Cox-Merz rule at high filler content.

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1. Introduction

The usual way to measure the rheological properties of polymer based materials in the melt is to use offline rheometers. They offer a thorough, precise and reliable analysis of the rheological behavior. However, especially for industrial purposes, it would be very useful to have an *online* estimation, i.e., during processing, of the rheological behavior of the material. This could be extremely useful to correct promptly the formulation and the composition or to modify the processing parameters to achieve quick optimization of the whole process. Moreover, this opportunity is also useful for scientific studies, especially for multiphase systems.

Online measurement would provide the rheological properties under the effective flow conditions occurring inside the processing equipment, thus giving more reliable and relevant results compared with offline measurements in which, for instance, the morphology is perhaps modified [1,2]. In fact, it is well known that in multiphase polymer systems the melt morphology and, consequently, the rheological properties of these systems are strongly dependent on the processing conditions [3–7]. Thus, the flow field in rotational rheometers or in a capillary viscometer during the measurement can lead to some morphological changes of the material with respect to the morphology achieved during previous mixing [1,2].

Of course, knowledge of the flow curve in the shear rate range of the processing operation would be more than welcome in order to have higher control of the whole process. There have been several attempts to demonstrate the validity of using mixing torque and mixing speed data to produce a reliable flow curve of a polymeric system [8–

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Table 1

Code, commercial name, manufacturer, MFI of the polymers used in this work and mixing temperature adopted for the preparation of the composites.

Code	Commercial name	Manufacturer	MFI (g/10 min)	Mixing T (°C)
LDPE	Riblene FC30	Polimeri Europa	0.27	180
HDPE	MP94	Polimeri Europa	7	190
EVA 28	Greenflex HN70	Polimeri Europa	6	160
PA6	Radilon S35 NAT	Radicinova spa	–	240

17], including polymer blends and filled materials. This type of torque rheometer, is particularly interesting for filled polymers and other heterogeneous systems. Indeed, for these systems, the rheological properties depend on the dispersion of the second phase that depends, in turn, on the flow. Therefore, the measurements performed during mixing may ensure good and reliable rheological characterization.

In this respect, an important shortcoming when using the torque rheometer is the calibration of the measuring head because torque and rotational speed must be correlated to shear stress and shear rate, respectively, and this is not easy due to the complex geometry of the blades and of the flow during mixing. The studies of Goodrich and Porter [10], Blyler and Daane [11], Lee and Purdon [12], Marquez et al. [8] and Mallette et al. [9] proposed a calibration technique to obtain the power-law parameters of polymer melts, and then the related flow curves, from torque-rotational speed data. They demonstrated that the polymer systems were successfully characterized from a rheological point of view during mixing.

In this work, the analysis of Marquez et al. [8] was used for the first time to characterize clay-polymer systems. The use of this method can be very interesting with multiphase polymer systems as they do not usually follow the Cox-Merz rule i.e. it is not possible to obtain rheological

information from measurement carried out by a rotational rheometer, especially at the highest frequencies. On the other hand, a capillary viscometer cannot achieve such high shear rates. This implies the presence of a dark zone in the rheological curve that is often the most interesting for the industrial processes. The torque rheometer proposed in the present work may overcome all these issues offering the possibility to shed light on that dark zone that cannot be otherwise explored.

2. Experimental

The polymeric matrices used in this work were: low density polyethylene (LDPE); high density polyethylene (HDPE); ethylene-vinyl acetate copolymer containing 28% of vinyl acetate (EVA 28); polyamide 6 (PA6). Some relevant information is reported in Table 1.

Cloisite 15A (15A, Southern Clay Products), a montmorillonite modified by 125 meq/100 g of dimethyl bis(hydrogenated tallow alkyl)ammonium cations was used as nanofiller. The organoclay is commercialized in the form of a fine white powder with an initial average dimension of about 8 μm .

The composites were prepared by melt mixing using a batch mixer Brabender Plasticorder PLE 330. The mixing temperatures depended on the polymer matrix used, as reported for each system in Table 1. Five mixing speeds were used: 32 rpm, 50 rpm, 60 rpm, 100 rpm, 200 rpm, while the mixing time was 5 min - enough to achieve a steady value of the mixing torque. The clay contents were 1% w/w and 10% w/w for all the polymer matrices with, in some cases, additionally a concentration of 5% w/w.

The morphology of the samples was analyzed by a scanning electron microscope (SEM) (Quanta 200F ESEM, FEI). All the samples were fractured in liquid nitrogen and then sputter-coated with a thin layer of gold to avoid electrostatic charging under the electron beam.

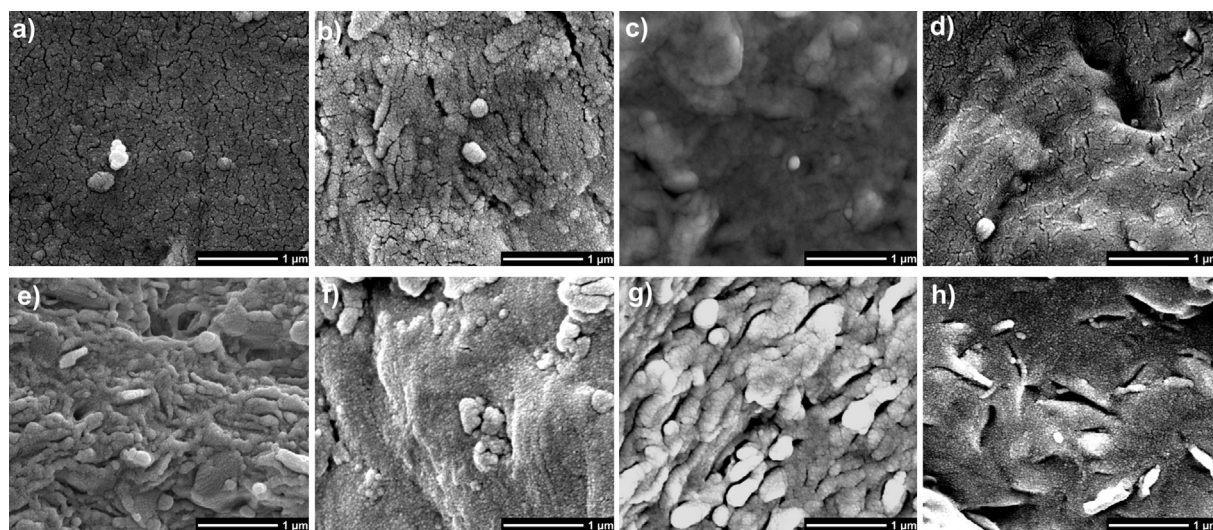


Fig. 1. SEM micrographs of all the systems with 1% and 10% of 15A mixed at 100 rpm: a) LDPE + 15A 1%, b) HDPE + 15A 1%, c) EVA 28 + 15A 1%, d) PA6 + 15A 1%, e) LDPE + 15A 10%, f) HDPE + 15A 10%, g) EVA 28 + 15A 10%, h) PA6 + 15A 10%.

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