Semana de Engenharia 2010
Guimarães, 11 a 15 de Outubro

IN-LINE RHEOLOGICAL CHARACTERIZATION: APPLICATION TO A MINI-EXTRUSION LINE

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KEYWORDS
In-line rheology, Rheo-optics, SALS.

ABSTRACT

Extruders are a fundamental part of any extrusion and compounding line. Co-rotating and counter-rotating twin-screw extruders are commonly used in plastic industry for applications ranging from melting and pumping of polymer for profile extrusion to compounding, mixing and chemical reaction. However, due to either the small quantities of materials available or their high cost, it is often necessary to have the capability to perform this work at small scale, laboratorial scales. In addition, in-line rheometers located in the die can help in detecting changes in structure, morphology, or composition of a material system, thus assisting materials research and processing optimization. The aim of this work is to meet the above goal with a novel small scale modular single / twin-screw extrusion system, with well-controlled outputs in the range 30-300 g/h and the capability to perform material characterization in-line: shear viscosity, normal stresses and extensional viscosity. Finally the implementation of optical techniques (small angle light scattering, microscopy,) in the in-line rheometer is reported, thus permitting direct structural characterization.

INTRODUCTION

In a slit die rheometry the material is forced through a thin rectangular channel or slit. The pressure drop and flow rate through the slit is used to determine the viscosity (η). In 1968 Brodbent et al. [1] first reported systematic errors in the measurement of pressures in flowing viscoelastic fluids using pressure transducers mounted at the base of small holes versus flush-mounted pressure transducers. The difference between the pressure measured by means of the flush mounted transducer (P1) and that measured by a transducer mounted at the based of a hole (P2) is referred to as the hole pressure (Ph). The source of the pressure hole is due to a bending of the streamlines into the hole. Where the streamlines are curved the normal stress tends to lift the fluid out of the hole. From the pressure difference (Ph) and depending on the pattern hole that we have, one will calculate normal stresses differences. A schematic of the slit die rheometer used in this research is shown in Figure1.

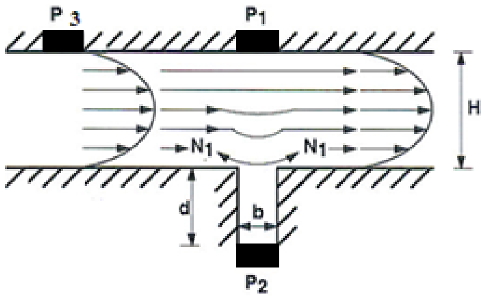

Figure 1 – Slit die rheometer.

The wall shear stress (σ_w) was obtained from the pressure gradient and the equation of motion as

$$\sigma_w = \frac{H}{2(1+H/W)}\frac{dP}{dx} \qquad (1.1)$$

where the dimension H e W is the height and width of the slit respectively, dx is the distance between P3 and P1and dP = P3-P1.

The apparent shear rate is obtained from

$$\dot{\gamma}_a = \frac{6Q}{WH^2} \qquad (1.2)$$

Where Q is the volumetric flow rate. The true wall shear rate was obtained by correcting for the non-parabolic velocity profile and is given by

$$\dot{\gamma}_w = \dot{\gamma}_a\left[\frac{2}{3}+\frac{d\ln\dot{\gamma}_a}{3d\ln\sigma_w}\right] \qquad (1.3)$$

The viscosity was obtained from $\sigma_w / \dot{\gamma}_w$ using equations (1.1) and (1.3).
The normal stress difference is obtained from

$$Ph = P1-P2 \quad (1.4)$$

$$N1= 2mPh \quad (1.5)$$

$$\text{Where } m = \frac{d\ln(Ph)}{d\ln(\sigma)} \quad (1.6)$$

The measured values of viscosity (η) from steady shear tests in a parallel plate TA ARG2 rotational rheometer and in a Bohlin capillary rheometer and values of first normal stress difference (N1) from steady shear tests in cone-plate are compared with the corresponding values from the slit die rheometer for a LDPE, at 150ºC in Figure 2. This shows a good agreement between all the data.

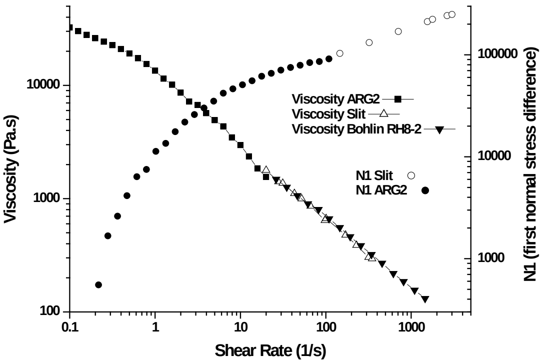

Figure 2 – Viscosity and first normal stress difference versus shear rate for a LDPE at 150 ºC.

Many sources of errors can interfere with the N1 measurements. Furthermore, we must emphasize that the hole pressure is less than 2% of the measured pressure and, hence, any source of error can significantly influence the accuracy.
In order to perform morphological analysis a new slit equipped with upper and lower sapphire optical windows was developed. In a SALS experiment light emerging from the laser (1) (see Figure 3) is sent through the sample that is passing in the modular rheo-optical slit die (2). Then the light is collected on a semi-transparent screen (3) that is mounted at the end. A high resolution CCD camera (4) connected to a frame grabber is used to capture and digitize the 2D light scattering patterns [2]. Figure 3 shows a general view of the modular rheo-optical slit die, coupled to a mini extruder (5) developed in the polymer engineering department, of University of Minho. It is a modular system that can work in single or twin screw modes. One can accurately control the throughput in the order of a few hundreds of grams per hour and perform in-line rheo-optical characterization. In addition a polarized optical microscope (6) can be used.

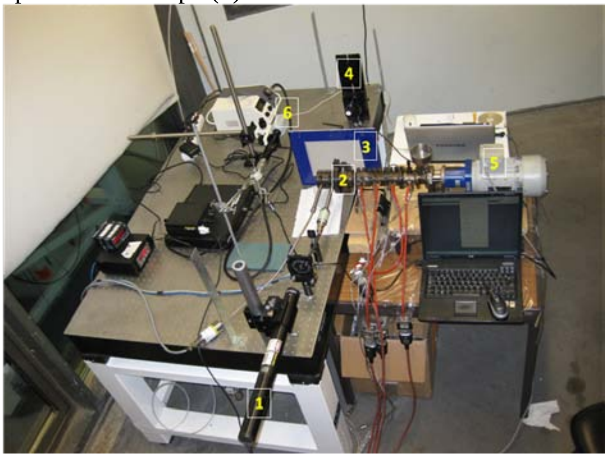

Figure 3 – Rheo-optical slit die coupled to SALS set-up and mini-extrusion line.

Figure 4 presents an example of material characterization: viscsosity, microscopy and SALS. As expected the Newtonian viscosity of PDMS is not affected by the low amount of fillers, and no structure develops under flow (isotropic SALS pattern).

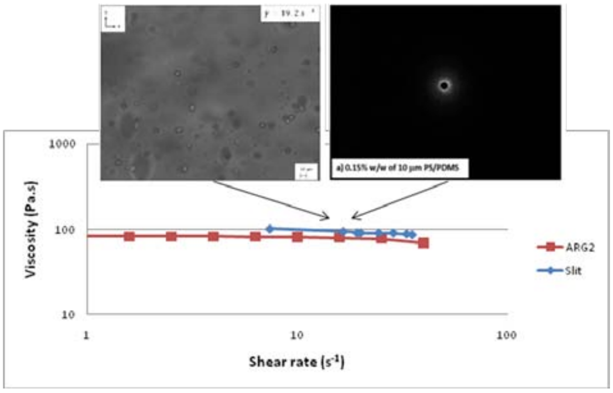

Figure 4 – 0.15% (W/W) of 10µm PS particles in PDMS

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Paulo Teixeira was born in Braga, Portugal and went to University of Minho where he studied Polymer Engineering and obtained his degree in 2005. He is a PhD student in Materials Science and Engineering – Polymer Department, financially supported by FCT.