

NEW DEVELOPMENTS IN MICRO-COMPOUNDING OF NANOCOMPOSITES

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Abstract

The fast pace in the research and development of new nanocomposites requires fast screening of potential candidates both in sample preparation and testing. Traditional methods of compounding can no longer accommodate an increasing demand for a large number of samples.

In this paper we present the most recent development in micro-scale compounding technology in material science. The equipment consists of a 0.12 meter conical twin-screw extruder co- and counter-rotating and housed in a vertical clamp shell barrel, which can be operated in batch and continuous modes ensured by a recirculation channel and a control valve built into the barrel. The design and versatility of this equipment allows the evaluation of a myriad of materials ranging from polymer blends to filled systems and nanocomposites with 2 to 15 grams of sample.

It is also possible to monitor changes in the rheological properties of the materials during processing allowing a better assessment of mechanisms such as polymer degradation and stability, reactive extrusion and crosslinking. Comparative analysis with other processing equipment such as mixing bowl and twin screw extruders on model systems and in terms of extent of dispersion and mixing is achieved.

Introduction

It is well known that in multi-phases systems, the resulting properties are morphology dependent, which in turn is dependent on the shear history during mixing [1]. Therefore a blend system can exhibit different end-use properties depending on how it was prepared. This can be challenging in product development if the degree of mixing and extent of dispersion are not scalable from laboratory to industrial scale.

Attempts in the literature to extract quantitative information from mixing devices with regard to the extent of shear and the determination of rheological properties of polymers and extent of dispersion for polymer blends have been somewhat successful [2-3] and provide an indication of the time scale under which each of the processing equipment operates. Others have shown the degree of dispersion obtained with mixing bowls as compared to a single sigma-blade type mixer [4-5]. While generally the degree of shearing in these mixers is rpm-dependent, it is

well known that the optimum particle size is governed by the break-up of stretched filament into droplet when a critical capillary number $Ca=R\mu\dot{\gamma}/\Gamma$ and a viscosity ratio are reached [6].

Experimental

Extruder design

Additional to the introduction, feeding is possible through a horizontal hopper mounted on the top of the barrel and can be used to feed polymer pellets, powder as well as solids and liquid additives. The design of the hopper also allows a precise feeding which can be useful when accurate compositions are required. In addition, an inert gas feed is allowed from the top of the extruder to ensure inert atmosphere during operation.

Instrumentation

Both barrel temperature and true melt temperature can be controlled (the melt thermocouple is located in the stream of processing material). Shear rate can be varied by two variables; one is the screw speed that can be varied between 1 and 250 rpm and the adjustable gap between the screws and the barrel walls. This clearance can be varied by adjusting the position of the barrel in the z-axis. Additionally, the barrel is positioned on a lever, which swivels around a stationary axis and counter balanced by a force transducer at the other end. The force transducer is typically 10 KN in range and measures the axial force exerted by the barrel opposing the shearing forces imposed by the screws towards the bottom. Both rpm and axial force can be controlled.

A schematic overview of the micro-scale compounder is shown in Figure 1:

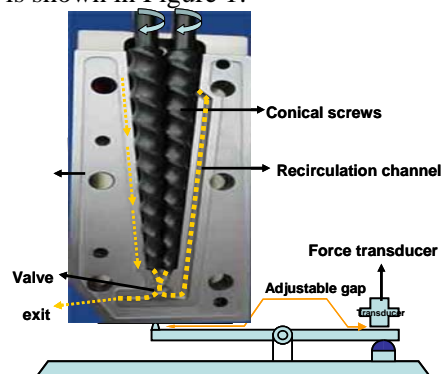


Figure 1. Schematic overview of the micro-scale compounder.

Case studies

Figures 2 and 3 are illustrative of the predictive capacity of the micro-compounder. Figure 2 shows the complex viscosity versus frequency of two commercial polyethylenes one is linear and the other containing long chain branching. In this example the data has been fit with the Carreau-Yasuda model to predict the rheological properties at high shear rates. It is well accepted in the literature that branched polyolefins possess a steeper shear thinning behavior compared to linear polyethylenes [7]. This is clearly shown in Figure 2, which indicates a crossover in the viscosity profile of the two samples at 180°C. Furthermore, the two samples are expected to behave differently during processing where the branched polyethylene can be processed at a higher throughput for the same head pressure. The validation of this difference can be done using the micro-compounder and is shown in Figure 3 indicating the axial force versus rpm at the same temperature. These results show similar behavior of the two samples under large deformations where at high rpm the axial force of the branched polyethylene is lower than that of the linear one. This confirms that the extruder can be used as a predictive tool for polymers with different molecular architecture under normal extrusion conditions.

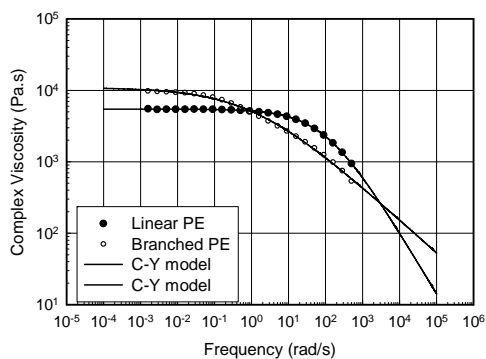


Figure 2. Viscosity-frequency data of linear PE versus branched PE at 180°C.

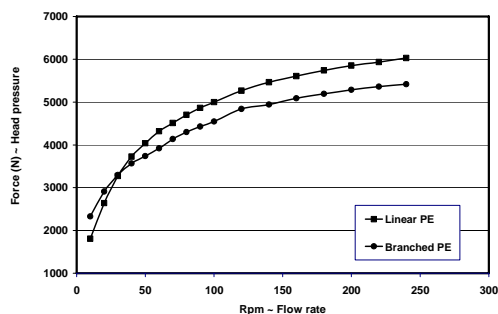


Figure 3. Axial Force versus Screw of linear versus branched PE at 180 °C.

The last example shown here is a commercial system of polyester/Epoxy blend containing a latent catalyst. This system is used in automotive coatings, where the catalyst is dispersed in the polyester and then is melt blended at temperatures below that of the catalyst activation. Figure 4 shows the curing curves of a polyester/epoxy system at different temperatures. The resulting curves are a result of an isothermal curing induced by heat and mixing and mimics better the process conditions under which the blend is prepared commercially. The kinetics of this reaction can then be easily determined as function of time and temperature. The ability of the extruder to perform such measurements allows more flexibility in R&D for stoichiometry studies and catalyst screening.

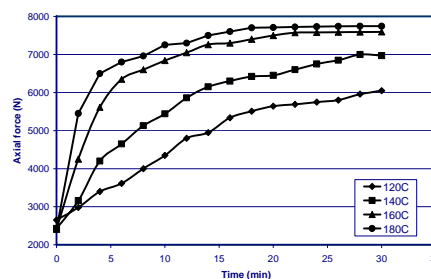


Figure 4. Kinetics study of a polyester/epoxy blend at different temperatures.

Conclusions

In this work, a new micro-compounder was introduced to the field of nanocomposites with supporting data to demonstrate the versatility of this instrument. The results presented in this work show a direct correlation between structure, properties and applications but in the same time demonstrate the utility of such equipment especially for fast product development and commercialization.

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