

Inferring Particle Size in Reactive Polymer Processing via an In-Line Optical Detector

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The Residence Time Distribution RTD curve of a polymer inside an extruder is very important because it is related to its level of dispersion (in terms of variance) and the time the fluid is exposed to the shearing forces (in terms of average residence time). The standard procedure is to get the RTD curve injecting a predefined amount of tracer in a position of the extruder and measuring the tracer concentration at another point of the extruder (usually at its exit), as a function of time⁽¹⁾. In the past 15 years various in-line detection systems have being studied using a variety of tracers. In this paper we have used an in-line optical detector⁽²⁻³⁾ to study the dispersion of the second phase (PA6, PP-g-AA) in reactive polymeric blends during extrusion. The signal intensity was calibrated using known water/Al₂O₃ suspensions. The optical detector used operates delivering a voltage that ranges from a minimum base line value (V_0) to the maximum value (V_s). As the tracer passes through the light path the light intensity is reduced, producing a voltage (V) which can be normalised as V_N . Changes in the detector signal is proportional to the tracer concentration c as⁽⁴⁾:

$$\log\left(\frac{V_s - V_0}{V_s - V}\right) = \log\left(\frac{1}{1 - V_N}\right) = \frac{(\sigma + K)}{2.303} \cdot c \quad (1)$$

where σ is the scattering and K is the absorption coefficients.

Polypropylene (RP-347, OPP Petroq. S.A., Brazil, 10 g/10min) was used as flowing polymer in the extruder. The polymeric tracers were polyamide PA6 (Ultramid B3, BASF), and polypropylene grafted with acrylic acid PP-g-AA (Polybond 1001, Uniroyal Chemical). For calibration we used a water/alumina suspension with two average particle size 1 and 2 μ m, measuring at room temperature.. The optical detector fitted in a slit-die was assemble in a twin-screw extruder Werner Pfleiderer ZSK30⁽³⁾ with the following processing conditions were: 2 Kg/h, 60 rpm, 240°C In-line residence time distribution curves measurements were obtained following the standard procedure⁽¹⁾. Varied amounts of the second phase, in a form of pellets, was added in the extruder feeding port^(3, 4).

The reactive system studied is a PP/PA6 blend compatibilised with PP-g-AA. The reactive acrylic acid grafted group in the PP chain reacts with the amine end groups of the polyamide PA6 creating a diblock copolymer which tends to stay in the PP/PA6 interface. This reduces the interfacial tension, stabilising and reducing the particle size of the PA6 dispersed phase. The particle size reduction increases the backscattering reducing the total transmitted light intensity. Fig 1 shows the RTD curves (as normalised voltage) of the transient state of these reactive and non-reactive polymer blends. The transient state was obtained by adding enough pellets of PP-g-AA or PA6 or still PA6 + PP-g-AA in the feeding port of the ZSK-30, as a pulse in the flowing PP stream. The RTD curve of the pure PP-g-AA is barely visible, relying upon the yellowness of the grafted polymer. The addition of pure PA6 pellets in the PP flow yields a very clear RTD curve like trace. Upon adding as a pulse both, PA6 + PP-g-AA pellets, in the feeding port the trace is obtained with the same time characteristics of the pure PA6 pulse but the intensity scale is consistently enlarged. Fig. 2 shows the normalised detector voltage intensity for the water/alumina suspensions having two average particle size 1 and 2 μm . The light scattering is particle size dependent and so the detector, showing good sensitiveness to the particle size of the filler in the suspension.

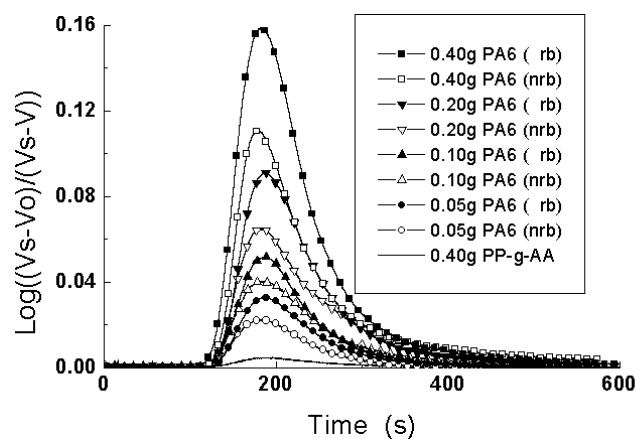


Fig. 1

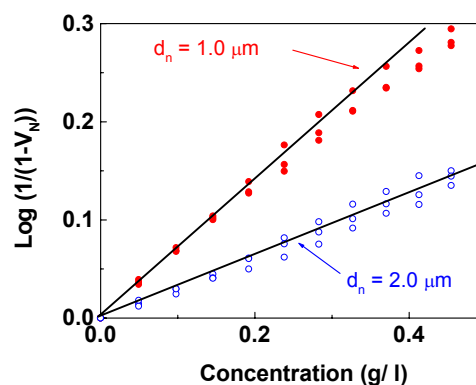


Fig. 2

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