ON-LINE OPTICAL CHARACTERIZATION OF THE MIXING LEVEL OF A PS/PA6 IN CO-ROTATING TWIN-SCREW EXTRUSION

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ABSTRACT

This work measured residence time distribution curves (RTD) obtained at various axial positions along three different kneading blocks and under a range of screw speeds to ascertain the mixing performance. For this purpose, one of the barrel segments of the extruder was modified in order to incorporate four sampling devices and, in addition to that, slit dies containing optical windows were attached to them. The experiments consisted in reaching steady extrusion and then add a small amount of tracer. Upon opening each sampling device, material was laterally detoured from the local screw channel, and its turbidity and birefringence were measured by the optical detector. It is hypothesized the area under each RTD curve, is a good indicator of dispersive mixing, whereas variance can be used to assess distributive mixing. The experimental data confirmed that these mixing indices are sensitive to changes in processing conditions, and that the expected behavior for each kneading block geometry was observed.

Keywords: *Twin-screw extruders; Mixing performance; On-line characterization; Optical properties;*

INTRODUCTION

Extrusion is one of the most popular industrial process to manufacture polymer products. It consists of obtaining shaped products by passing the molten material through a die, forming, after its solidification, a product with constant cross section(1). Mixing is an essential feature of any polymer processing, in which, it involves the spatial re-arrangement of the formulation components leading to uniformity by imposing a certain shear deformation history (distributive mixing), as well as the progressive decrease in size of initial agglomerates, or droplets of a suspended liquid phase, by the exertion of hydrodynamic stresses during a certain time (dispersive mixing)(2). The complete description of the state of mixing of a given system requires the identification of the size, shape, orientation, and spatial location of every particle or droplet of the minor component along the processing equipment(3,4). This must be obtained either through numerical modeling or experimentally, which is not straightforward, making practical mixing assessment during processing a complex topic.

This work used information extracted from RTD curves obtained by means of on-line turbidity and birefringence measurements to characterize the distributive and dispersive mixing abilities of kneading blocks with different geometries of a twin-screw extruder (TSE), operating under a range of screw speeds. For this purpose, a blend of PS/PA6 is processed, PS being the matrix and PA6 the tracer. Measurements are made at various axial locations, thus evidencing the kinetics of mixing along each kneading block.

MATERIALS AND METHODS

Materials

A commercial grade of a general-purpose polystyrene, PS (Styrolution 124N/L, manufactured by INEOS Styrolution), with MVR of 12 cm^3/10 min (5.0 kg, 200 °C) was extruded as matrix. A polyamide 6, PA6 (Domamid® 6NC01, manufactured by DOMO Chemicals), with MVR of 165 cm^3/10 min (5.0 kg, 275 °C) was used as tracer/pulse.

Experimental Set-up

The experiments were performed in a modular co-rotating intermeshing twin-screw extruder Collin ZK 25P with a screw diameter of 25 mm and L/D = 48. The modular barrel is composed of eight interchangeable segments, each with its own temperature control. As illustrated in Figure 1, one of these was replaced by a modified segment (1) containing two axial rows of sampling devices (2a and 2b). Each device consists of an on-off valve which, when rotated 90°, allows the material to flow out of the extruder, along a circular side-channel linking the inner and outer barrel walls. A multi-slit die (3) (containing 4 slits, each 30 mm long, 15 mm wide, and 1.5 mm thick, each aligned with the circular channel of one sampling device through a conical connection) was fixed to the top row of the sampling devices (2a). Each slit contains a pair of directly opposed transparent circular windows with a diameter of 10 mm, so that changes in light intensity transmitted through the polymer melt flow can be analyzed by an optical detection system (4-6). The latter contains an aligned pair of light emitters (6a) and light receiver (6b), which are kept in position by a C-shaped support (4). The entire contrivance can slide axially along the barrel segment, in order to make measurements at the various slit dies.

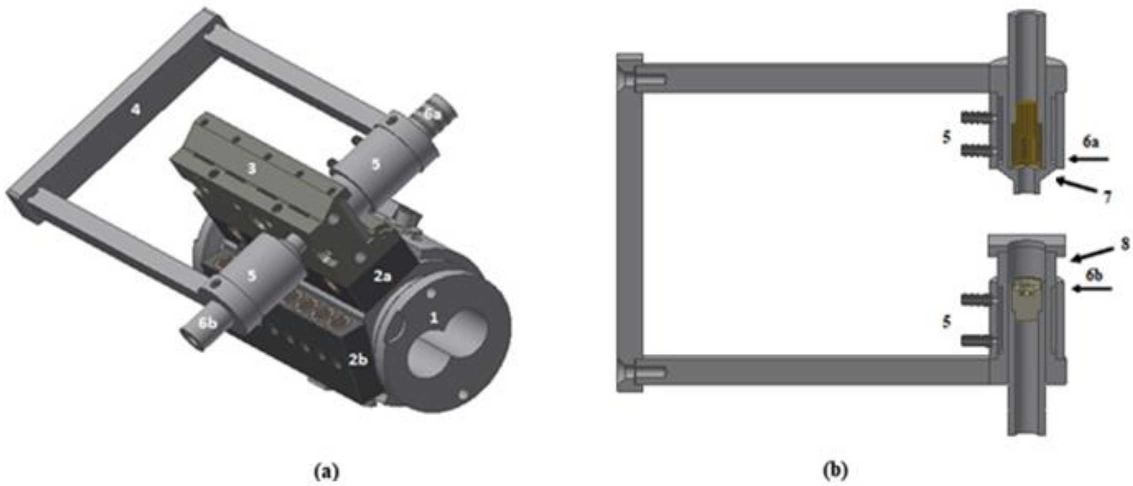

Figure 1: (**a**) Experimental set-up: modified barrel segment (1) with two axial rows of sampling devices (2a, 2b); multi-slit die (3); sliding optical detector with a C-shaped support (4); water-cooling system (5); light source (6a) and receptor (6b) for optical measurements and (**b**) Optical detector system: highlighted the LED (6a) with a polarizer (7) and two LDRs (6b) with a polarizing filter (8).

A white LED (Light Emitting Diode) with a polarizer (7) was used as light source and two LDRs (Light Dependent Resistor) as photodetectors, the first one to quantify changes in the light beam intensity, i.e., in melt flow turbidity, and the second one, positioned behind a polarizing filter (8) and aligned 45° with respect to the flow and 90° to the LED's polarizer, to quantify birefringence. The signals from the two LDRs measuring synchronously turbidity and birefringence are: i) collected at a frequency of 0.1 MHz (with an accuracy of 5%), ii) converted into digital signals by means of an analogic-digital interface (USB data acquisition NI-DAQ 6812), iii) transmitted to a personal computer running the software developed in the LabVIEW 8.6 NI platform (National Instruments) which averages (compresses) the data to present it at 10 Hz, iv) makes the real-time calculations, v) screen presentation, and vi) data saving. Data collection should not be affected by the inherent extruder vibrations caused by the drive elements, as the corresponding frequency ranges are quite distinct (0.1 MHz for data collection, 0-100 Hz for the mechanical vibrations[5]). A detailed description of the set-up can also be found elsewhere[6].

Figure 2 presents the upstream part of the extruder, including the modified barrel segment, together with the three screw profiles studied. The geometry downstream was kept constant in all experiments. The feed rate is controlled by a K-Tron gravimetric feeder. The collecting ports are located at L/D = 13, 14, 15 and 16. Since the aim here is to investigate the influence of screw design on the mixing efficiency: i) the three screws have the same configuration up to L/D = 13, consisting of conveying elements with decreasing pitch downstream. ii) the three screws have the same configuration from L/D = 16 onwards, starting with two left-handed (LH) elements (each 15 mm long), in order to ensure that they worked fully filled upstream, at least at L/D = 16, so that a material sample could be collected for an optical measurement. iii) between L/D = 13 and L/D = 16, three distinct mixing zones were assembled: 1) four kneading blocks with positive 45° stagger, each containing five 3 mm thick disks (KB45-3); 2) two kneading blocks with 45° positive stagger, each containing five 6 mm thick disks (KB45-6); 3) two kneading blocks with neutral 90° stagger, each containing five 6 mm thick disks (KB90-6).

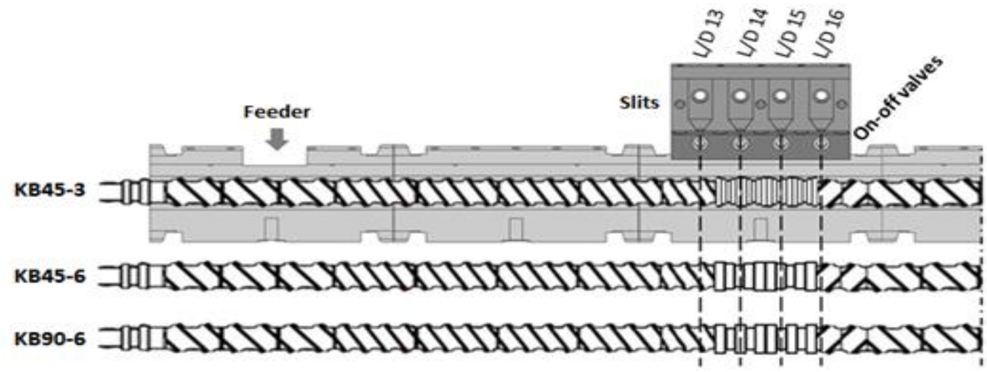

Figure 2: Screw profiles containing a 60 mm long mixing zone with different geometries: KB45-3: four kneading blocks with positive 45° stagger, each containing five 3 mm thick disks; KB45-6: two kneading blocks with 45° positive stagger, each containing five 6 mm thick disks; KB90-6: two kneading blocks with neutral 90° stagger, each containing five 6 mm thick disks.

In order to keep the experimental effort within reasonable limits, all the experiments were performed with a feed rate of 2 kg/h and a uniform barrel temperature set to 230 °C, while the screw speed was varied between 100 rpm and 500 rpm. For each processing run, upon reaching steady state extrusion of PS, a pulse of PA6 is added (0.105 g, corresponding to a concentration lower than 0.1 % w/w relative to the matrix), the valve of a specific sampling device is opened and the optical detector starts synchronously recording the transmitted light intensity as turbidity and the cross-polarized transmitted light intensity as birefringence. The presence of the dispersed phase in the flow through the slit-die produces light scattering and retardation, which are recorded in real-time. In both cases, the data comes out as a typical residence time distribution (RTD) curve.

RESULTS AND DISCUSSION

The RTD curves obtained by following melt flow turbidity were measured between L/D = 14 and 16, for the three screw profiles and screw speeds ranging from 100 to 500 rpm, and barrel set to 230 °C, and are shown in Figure 3. All the curves exhibit the typical pulse shape, becoming broader and being shifted to longer times as they are obtained more downstream. Differently, increasing screw speeds shift the curves to shorter times while widening them. These results were to be expected, as they reflect the typical progression of the material along the screws of a co-rotating twin screw extruder with a corresponding enhancement of mixing[7,8].

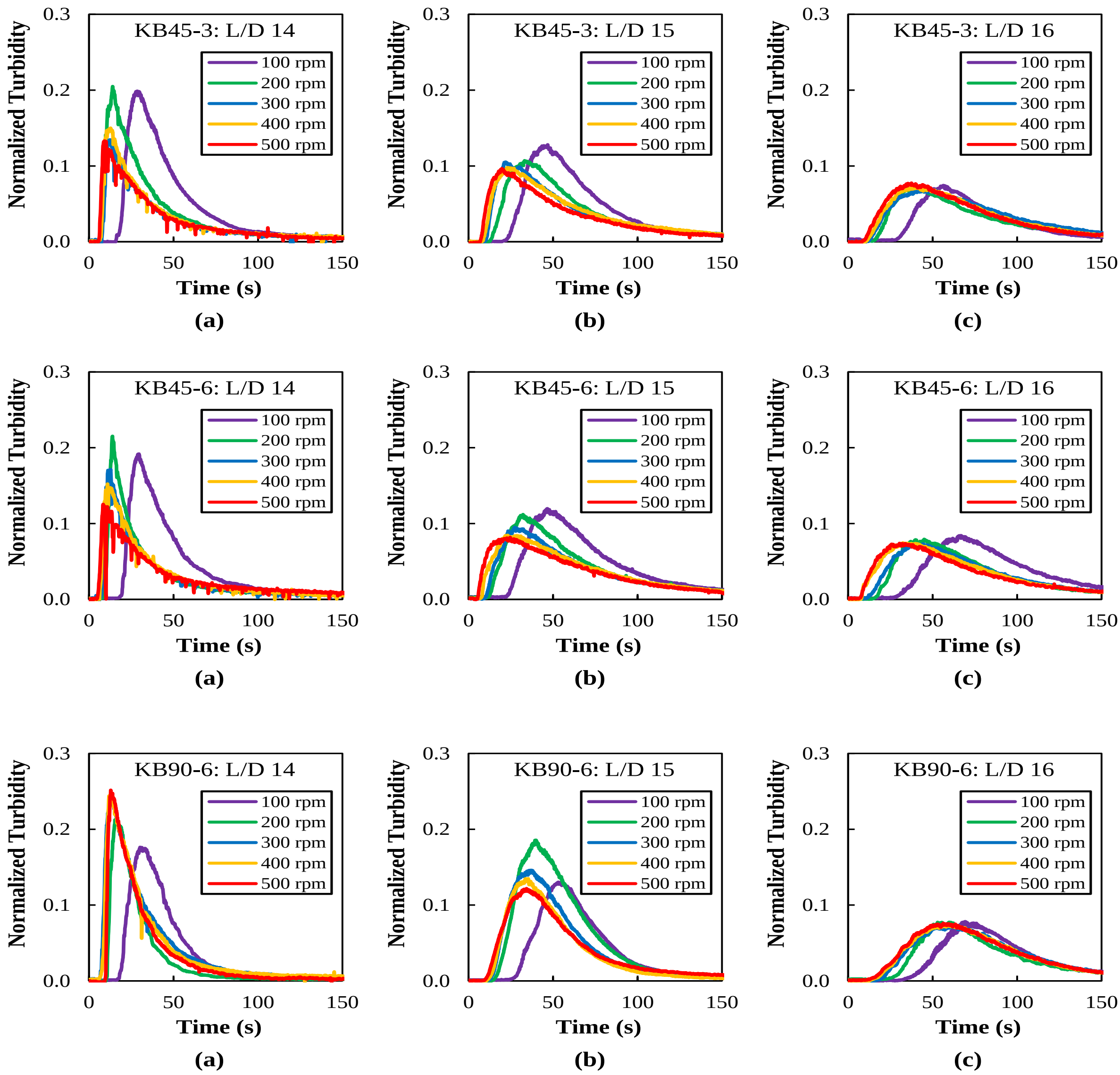

Figure 3: Residence time distribution (RTD) curves measured with the barrel set to 230 °C along the mixing zone at ports: (**a**) L/D = 14, (**b**) L/D = 15, and (**c**) L/D = 16 for different kneading blocks (KB45-3, KB45-6, and KB90-6) as a function of screw speed (100 to 500 rpm). The time scale was shortened to 150 s to better show the major peak area.

Figure 4 maps the dispersive and distributive mixing performance of the three kneading blocks investigated in this study, considering the distinct axial locations (at L/D = 15 and 16) and the range of screw speeds tested. The data shift to higher levels of distributive and dispersive mixing as the melt progresses downstream. Generally, higher distributive and dispersive mixing levels are attained when using KB90-6, while the performance of KB45-3 and KB45-6 is virtually undistinguishable, i.e., the thickness of the KB45 disks (either 3 or 6 mm) does not seem to impart a significant effect. The highest dispersive mixing levels are attained with lower speeds due to the associated higher residence times, whereas distributive mixing is promoted by higher speeds.

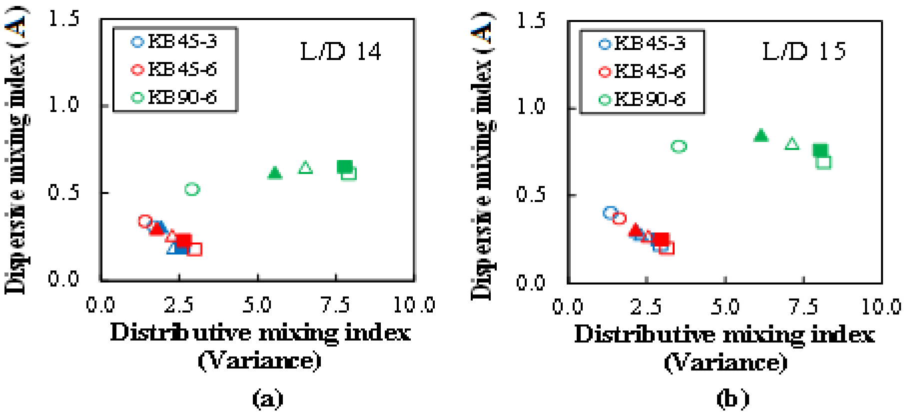

Figure 4: Mapping the mixing performance: Dispersive mixing index (A) versus distributive mixing index (σ^2) for different kneading blocks (KB45-3, KB45-6, and KB90-6) and screw speeds (○ - 100 rpm, ▲ - 200 rpm, Δ - 300 rpm, ■ - 400 rpm, and □ - 500 rpm) at ports: (**a**) L/D = 14 and (**b**) L/D = 15.

CONCLUSIONS

In this work, the melt mixing behavior of a diluted PS/PA6 system along the mixing section was evaluated in real-time by an on-line optical detection system operating the extruder in transient mode, i.e., with the insertion of the second phase component as a pulse. The area under an RTD curve (A) and the variance of the RTD curves (σ^2) were used as dispersive and distributive mixing indices, respectively. It was observed that higher dispersive mixing levels were attained for the mixing zone consisting of disks staggered at 90°, as it is associated to higher residence times than those having lower angles (45°). Distributive mixing increases with increasing screw speed, although it was shown that the indices may be influenced by the size (specifically, the scattering cross-section) of the particles generated during flow along the screw. Maps of distributive and dispersive mixing were built to readily characterize the performance of each type of mixing zone. Therefore, the methodology used here can contribute to quickly assess the mixing performance of screw zones, which is useful for assembling a suitable screw configuration, setting the processing conditions, or designing new screw elements.

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REFERENCES

1. CHARRIER, J.M. Polymeric Materials and Processing: Plastics, Elastomers and Composites. New York: Hanser Publisher, 1990.
2. MANAS-ZLOCZOWER, I. Mixing and Compounding of Polymers: Theory and Practice. Munich: Carl Hanser Verlag GmbH & Co, 2009.
3. TADMOR, Z.; GOGOS, C.G. Principles of Polymer Processing. New Jersey: John Wiley & Sons, 2006.
4. DOMINGUES, N.; GASPAR-CUNHA, A.; COVAS, J.A. A quantitative approach to assess the mixing ability of single-screw extruders for polymer extrusion. J. Polym. Eng., v. 32, p. 81-93, 2012.
5. FOZILOV, M.M.; CHISTYAKOVA, T.B.; POLOSIN, A.N. Computer system of vibration analysis for evaluating extrusion equipment operability at productions of multi-assortment polymeric films. J. Phys. Conf. Ser., v. 1902, p. 12-44, 2021.
6. BICALHO, L.A.; COVAS, J.A.; CANEVAROLO, S.V. Online optical monitoring of polymer melting in a twin-screw extruder. Polym. Eng. Sci., v. 60, p. 2163-2175, 2020.
7. KOHLGRÜBER, K. Co-rotating Twin-screw extruders: Fundamentals, Technology and Applications. Munich: Carl Hanser Verlag GmbH & Co, 2007.
8. CARNEIRO, O.S.; COVAS, J.A.; FERREIRA, J.A.; CERQUEIRA, M.F. On-line monitoring of the residence time distribution along a kneading block of a twin-screw extruder. Polym. Test., v. 23, p. 925-937, 2004.