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Evaluation of Temperature Impact During Extrusion on the Mixing Levels of PS/PA6 by Optical Monitoring

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DOI: https://doi.org/10.51573/Andes.PPS39.GS.EX.2

December 2024

View Online

Export Citation

Evaluation of Temperature Impact During Extrusion on the Mixing Levels of PS/PA6 by Optical Monitoring

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Abstract: This work uses on-line monitoring of turbidity to ascertain the temperature impact on the mixing performance of kneading blocks with different geometries. For this purpose, one of the barrel segments of the extruder was modified to incorporate four sampling devices and a slit die containing optical windows. The experiments consisted in reaching steady extrusion and then adding a small amount of tracer. Upon opening each sampling device, material was laterally detoured from the local screw channel and its turbidity were measured by the optical detector. Residence time distribution (RTD) curves were obtained at various axial positions along three different kneading blocks and under different temperatures. It is hypothesized that K, a parameter related to 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 they translate the expected behavior of each kneading block geometry.

Keywords: Mixing, Optical Properties, Temperature, Twin-Screw Extrusion, RTD Curves

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Introduction

Mixing with co-rotating twin-screw extruders (TSE) has been the focus of numerous modeling and experimental studies, which usually aim to characterize either the distributive or the dispersive aspects of the process. In the case of distributive mixing, Fard et al. [1] proposed a mapping method based on the tracking of particles in the velocity fields, while Wang et al. [2] calculated the evolution of the Renyi relative entropies of the minor component along the extruder. However, most studies predicted and/or measured the residence time distribution (RTD) [3-6]. The width of the RTD curves is generally considered a measure of distributive mixing, which can be interpreted as the variance factor (Equation 1), but other parameters of the curves (e.g., minimum and mean residence times) can be used to gain an understanding of the effect of screw geometry and operating conditions on the machine's response. Another parameter, the total area under a RTD curve (A), is a direct and quantitative measure of tracer content and can be calculated accordingly Equation 2.

$$\sigma^2 = \int_0^{\infty}(t - t_n)^2 E(t)dt = \sum_0^{\infty}(t - t_n)^2 E(t)\Delta t \qquad 1)$$

$$A = \int_0^{\infty} cdt = \sum_{t_i}^{t_f} c_i * \Delta t = \sum_{t_i}^{t_f} V_{Ni} * (t_i - t_{i-1}) \qquad 2)$$

In these equations, $E(t)$ is the residence distribution function, t_i and t_n are the minimum and mean residence time, Δt is the time interval determined by the on-line data collection frequency, usually at 5 Hz or 0.2 s and, c is the tracer/pulse concentration at time t, which is taken here as V_N, the normalized transmitted light intensity response. To better analyze the experimental curves obtained from the measurements, they were fitted by theoretical pulse curves accordingly Equation 3 [7],

$$I = I_0 + K\left[1 - e^{-\left(\frac{t-t_i}{R_1}\right)}\right]^p . e^{-\left(\frac{t-t_i}{R_2}\right)} \qquad 3)$$

where I_0 is the initial intensity or base line value (here set to zero), K is an area constant, t_i is the minimum residence time, R_1 and R_2 are the rise and fall time rates, respectively, and p is a power exponent. R_1 relates to the first part of the RTD curve, before the maximum, hence representing its rise time rate. The higher the R_1, the lower this rate. R_2 is associated with the region of the RTD curve beyond the maximum, thus quantifying its fall time rate. Again, the greater its value, the lower the rate of the RTD curve returning to its base line. The p parameter shifts the peak of the RTD curve down and forward, thus spreading the curve and reducing its area. This means a reduction in the number of particles and an increase in the axial spreading of the dispersed phase. Changes

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in p are much more perceptive than changes in R_2. R_1 is even less sensitive, as it is obtained from fitting a short portion of curve. K is a constant related to the area under the curve and is very sensitive both to changes in intensity of the RTD and to changes in R_1, R_2, and p, i.e., with changes in the number and dispersion of particles.

Here, we propose using the parameter K as an indicator of dispersive mixing performance, whereas the variance is used to assess distributive mixing. Both were chosen because they are related to the number and shape of dispersed particles and they are simple and easy to quantify, especially when compared to other more widespread mixing indicators.

Materials and Methods

Materials

A commercial grade of polystyrene, Styrolution 124 N/L with MFR of 12 cm^3/10 min (5.0 kg, 200°C) and density of 1.04 g/cm^3 was extruded as matrix. A polyamide 6, Domamid® 6NC01 with MFR of 165 cm^3/10 min (5.0 kg, 275°C) and density of 1.00 g/cm^3 was used as tracer/pulse.

Experimental Set-up

The experiments were carried out on a Collin ZK 25P co-rotating modular twin-screw extruder with L/D = 48. Figure 1(a) shows the modified barrel segment (1) containing two axial sets of on-off valves (2a and 2b). Each assembly consists of 4 openings that allow material to flow out of the extruder. A multislit die (3) containing 4 slits (L/D = 13, 14, 15, and 16) was fixed to the upper assembly (2a). Figure 1(b) shows the optical detection system used to quantify any changes in the intensity of light transmitted through the polymeric flow. The latter contains an aligned pair of light emitters (6a) and light receiver (6b), which are held in position by a C-shaped support (4).

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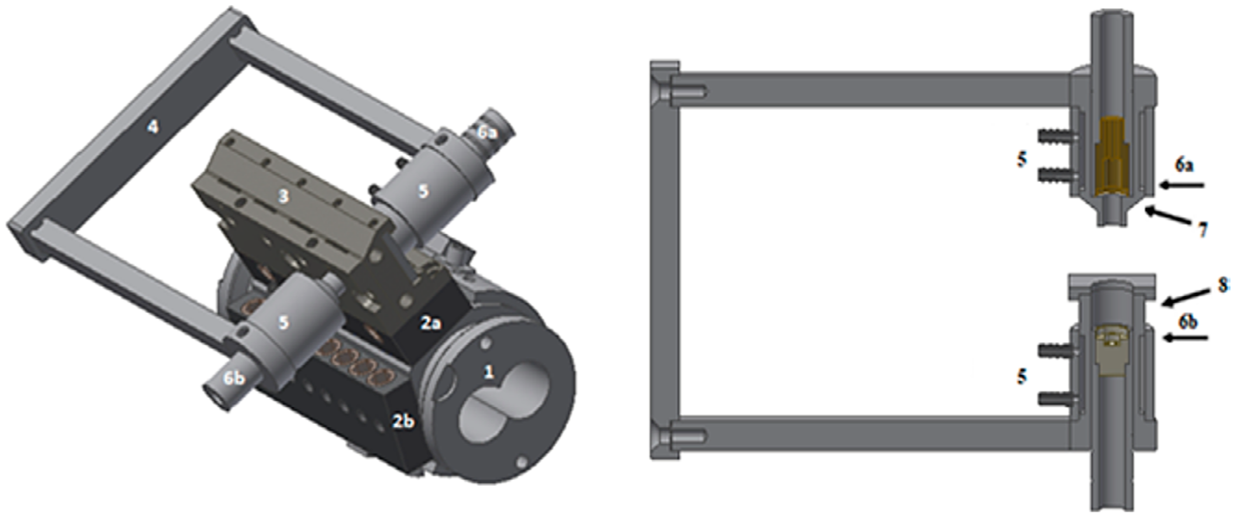

a) b)

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: LED (6a) with a polarizer (7) and two LDRs (6b) with a polarizing filter (8).

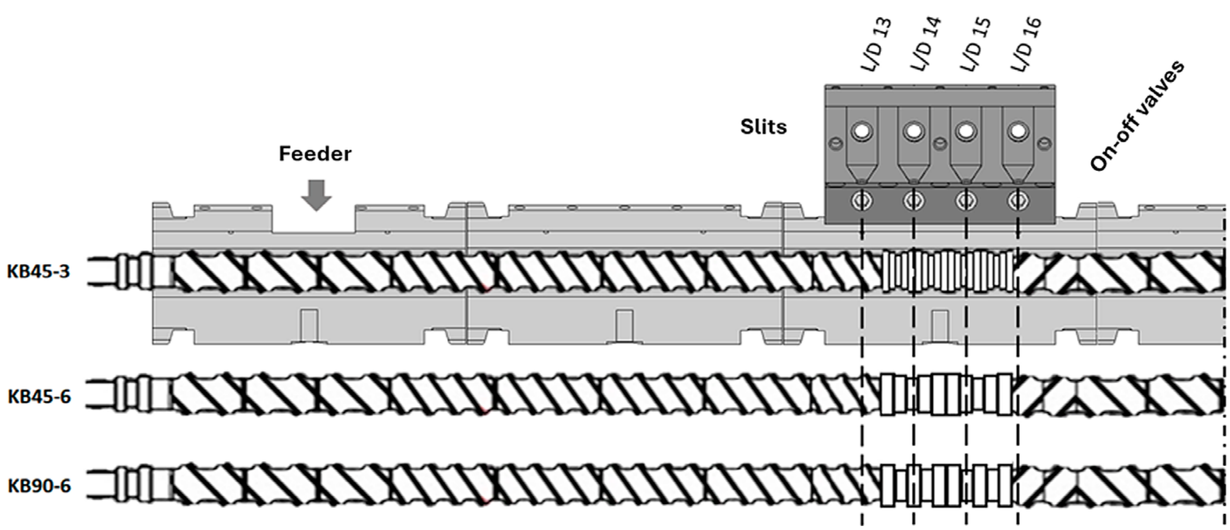

Figure 2. Screw profiles containing a 60 mm long mixing zone with different geometries: KB45-3–four kneading blocks with positive 45° stagger, each containing five 3 mm thick disks; KB45-6–two kneading blocks with 45° positive stagger, each containing five 6 mm thick disks; KB90-6–two kneading blocks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

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Three screw profiles were used and are presented in Figure 2.

To keep the experimental effort within reasonable limits, all the experiments were performed with a feed rate of 2 ± 0.1 kg/h and a uniform screw rotation speed of 100 rpm, while temperature was varied between 220°C, 230°C, and 240°C. 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. The presence of the dispersed phase in the flow through the slit-die produces light scattering and retardation, which are recorded in real-time. Data comes out as a typical residence time distribution (RTD) curve. This procedure was repeated for the three remaining sampling positions and then the entire experiment was replicated for a different temperature and for the various screw profiles.

Results and Discussion

Turbidity Evaluation

Figure 3 below shows the normalized turbidity curves obtained with the addition of pulses under the measurement conditions for each L/D and for each of the 3 screw profiles. At L/D = 13, the local flow rate for screws KB45-3 and KB45-6 is either low or nil; for this reason, measurements on this position were not performed in this work.

Analyzing the data present in Figure 3, a different behavior is evident for each assembled thread profile. For the first profile of KB45-3, there is a greater discrepancy in the values found for the temperature 220°C, while for the other two thread profiles we have a few differences in values regardless of the temperature applied. Another evident point is the higher turbidity values found for the outlet valve of L/D = 14, something already expected as turbidity is expected to decrease downstream.

Mixing Evaluation

Once the obtained normalized turbidity values were analyzed, pulse function (Equation 3) was applied and the variance and area under the curve values were removed from each curve obtained. Thus, the mixing factors were quantified.

The data presented in Figure 4 confirms better mixing performance in elements with an angle between their kneading discs of 90°. Although the differences are low from one profile to another, they are present and easily identified graphically. For the analysis conditions with 45° elements varying the thickness of the discs, few differences were observed, therefore showing that the angulation of the elements stands out over their thickness.

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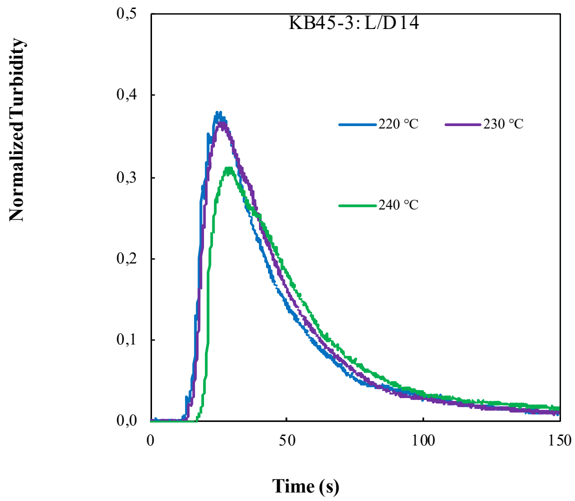

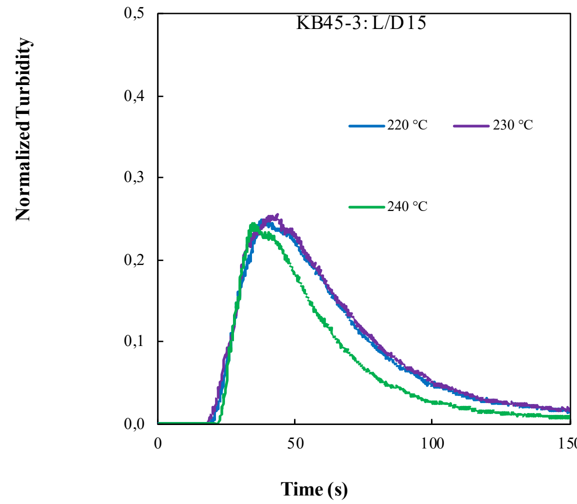

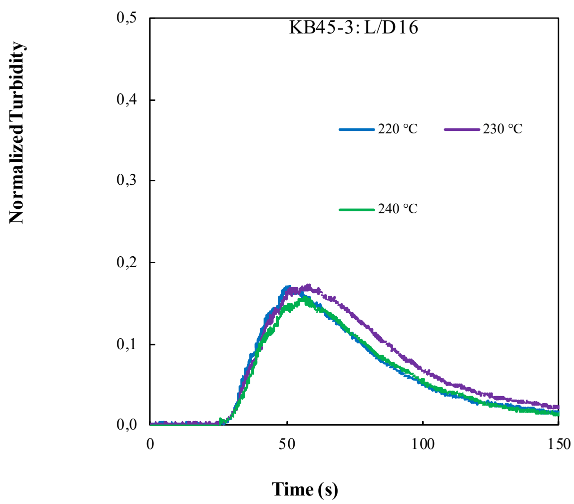

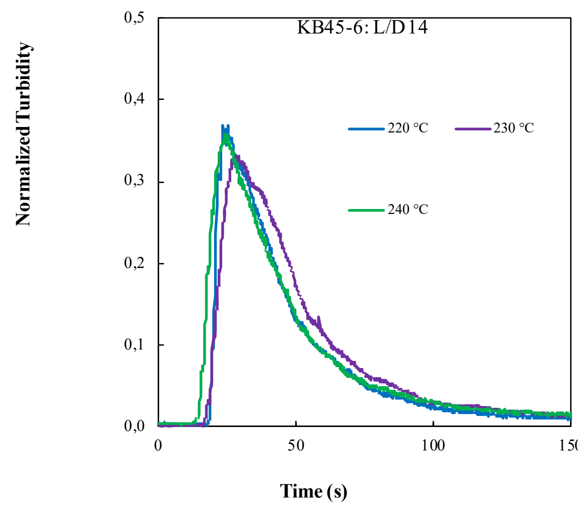

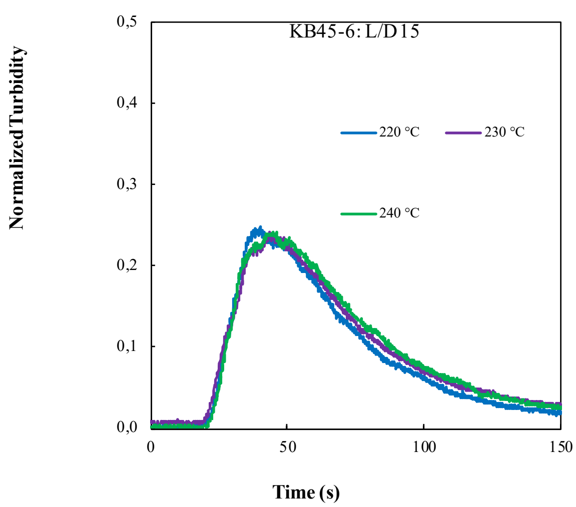

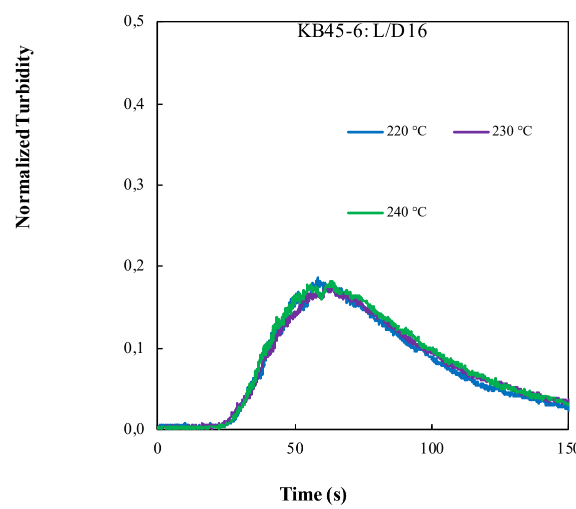

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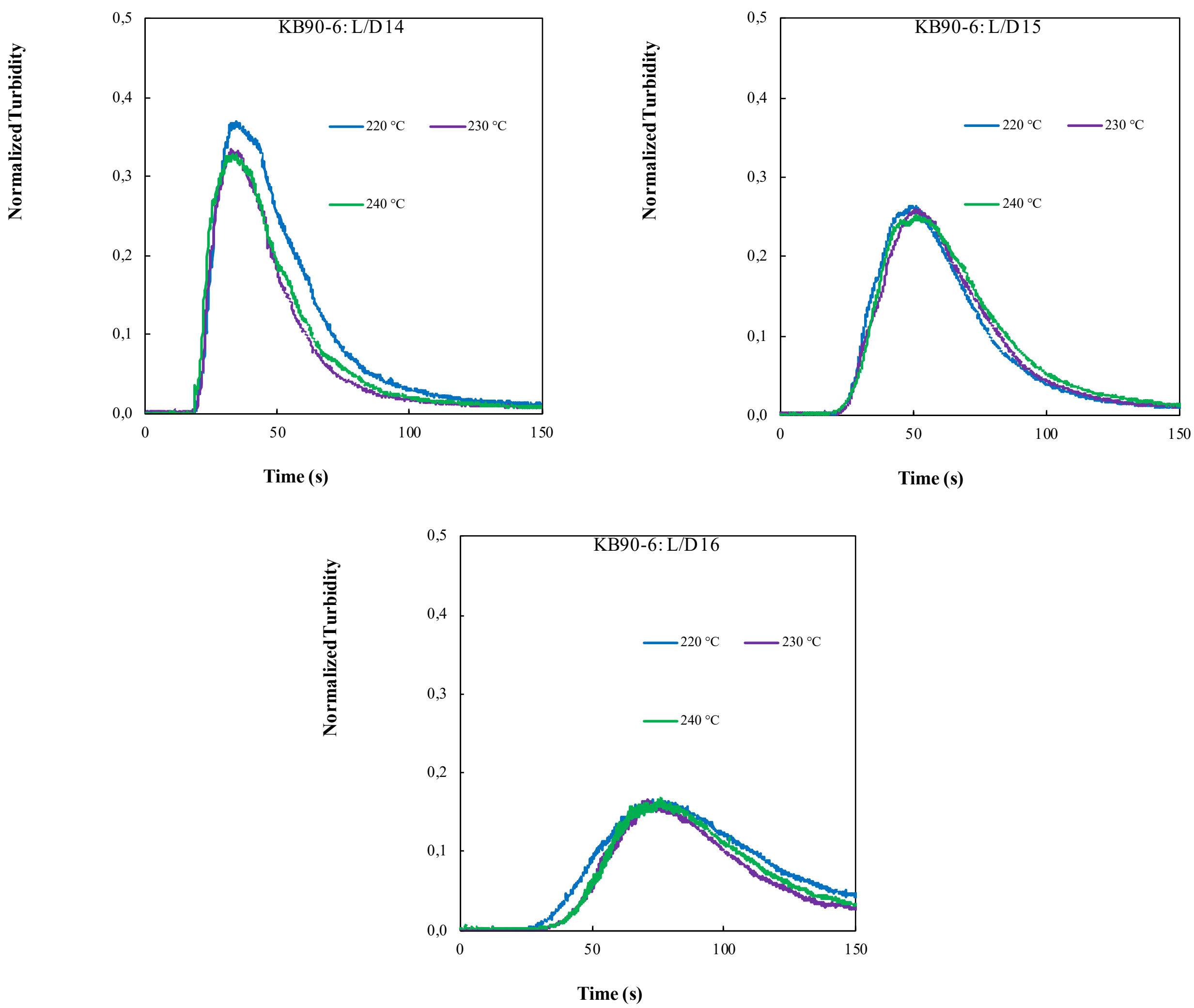

Figure 3. Normalized turbidity as function of time (s) for each screw profile: KB45-3, KB45-6, and KB90-6 for each measurement temperature. Screw rotation speed at 100 rpm for all measures.

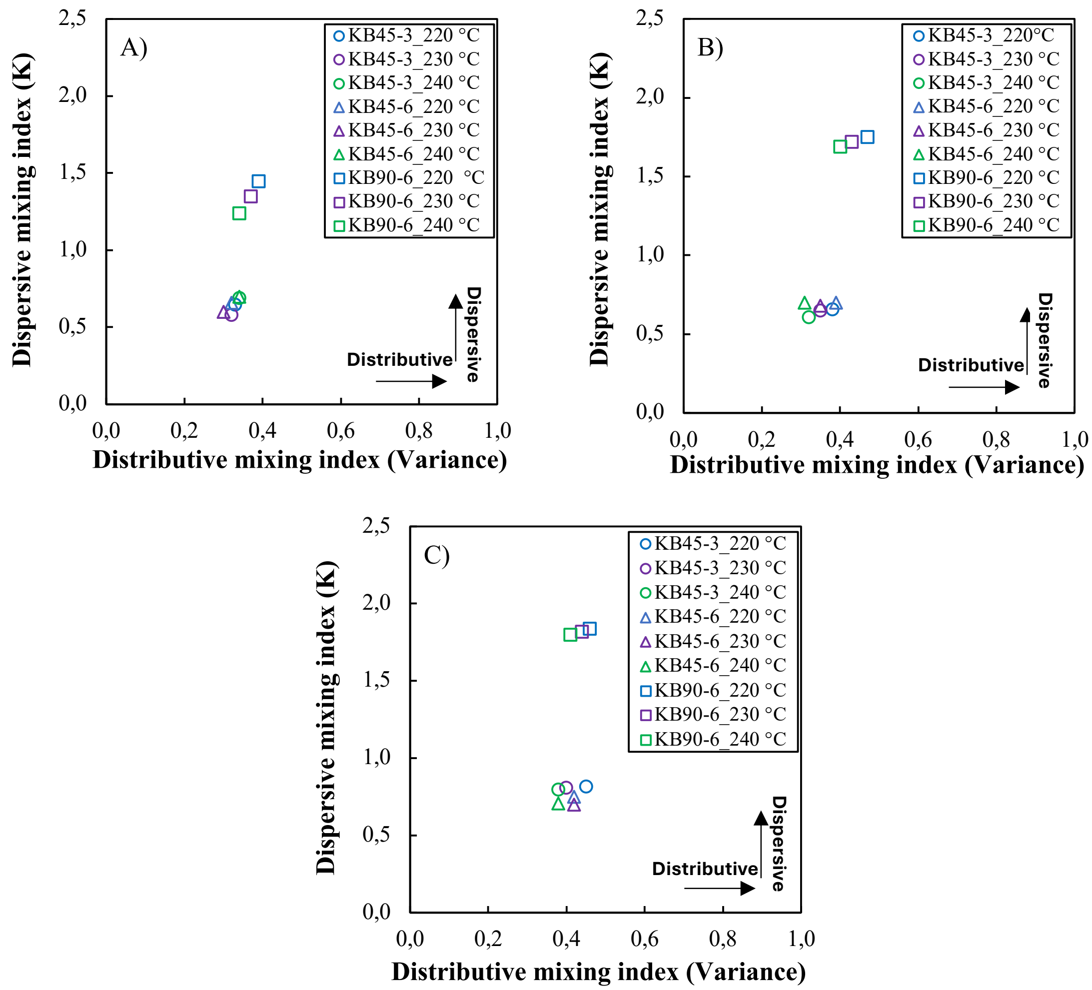

Figure 4. Mapping the mixing performance: Dispersive mixing index (K) versus Distributive mixing index (Variance) for different kneading blocks (KB45-3, KB45-6, and KB90-6) and temperatures 220°C, 230°C, and 240°C at ports: A) L/D = 14, B) L/D = 15, and C) L/D = 16.

Conclusion

In this study, a real-time optical detection system was employed to monitor the melt mixing behavior of a diluted polymer blend along a kneading section of a co-rotating twin screw extruder. The system utilizes light scattering and retardation caused by dispersed phase particles, providing information on particle number. By introducing the second phase component as a pulse at the extruder entrance and tracking its exit at various axial positions along the mixing zone, residence time distribution (RTD) curves were generated. The parameter K (a constant in the pulse curve, corresponding to the area under an RTD curve) and the variance of the RTD curves were utilized as dispersive and distributive mixing indicators, respectively. Analysis of K and the variance of the RTD curves revealed that higher dispersive mixing occurred in zones comprising disks staggered at 90°, which are associated with longer residence times compared to zones with smaller angles (45°). Thus, the experimental set-up and methodology employed in this work offer a way to rapidly evaluate the mixing performance of screw zones. This capability is valuable for optimizing screw configuration, establishing processing parameters, or designing new screw elements for twin-screw extruders.

Acknowledgments

The authors acknowledge the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior -Brasil (CAPES) for funding this work as well as the Programa de Pós-Graduação em Ciência e Engenharia de Materiais (PPG-CEM) of Federal University of São Carlos and the Institute for Polymers and Composites (IPC) of the University of Minho for providing access to the laboratorial facilities.

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