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DETERMINATION OF THE RESIDENCE-TIME DISTRIBUTION IN INDUSTRIAL DRYER FOR THE PRODUCTION OF RECYCLED POLYESTER

Article Highlights

- The work consisted of an industrial scale technical investigation
- The industrial process had no impact on product quality or production rate
- The developed method can be applied on an industrial scale
- Numerical interpretation of the results provided important data for process flow characterization

Abstract

The study proposes the evaluation of the residence-time distribution (RTD) in situ in an industrial dryer for the production of recycled polyester fibers (PES) from colorless polyethylene terephthalate (PET) flakes without interruption of the production. A disturbance of the pulse type was employed, in which the tracer (blue PET flakes) had previously been crystalized and its concentration was obtained according to the time at the dryer outlet. Additionally, analyses of intrinsic viscosity and crystallization percentage of the PET flakes (colorless and blue) and PES intrinsic viscosity and color force were performed. By RTD, the mean residence time (322.8 min), the variance (1305.4 min^2), the standard deviation (36.1 min) and the relative error (1.5%) were obtained when compared to the theoretical residence time, indicating the absence of preferred paths or flake agglomerates in the equipment. Finally, the characterization demonstrated that there was no alteration in the parameters of product quality during RTD evaluation, confirming the potential of application of this methodology for diagnoses of continuous industrial processes.

Keywords: residence time, PET flakes, polyester fibers.

Polyester (PES) fibers are polymers with identical chemical structure to polyethylene terephthalate (PET) and have several applications in the textile industry. Given the similarity between the physicochemical properties of PES and PET, it becomes possible to transform the recycled PET materials into PES [1-3]. Initially, the PET bottles are decontaminated and reduced to the granulometry of flakes. Subsequently, they are taken to the processes of extrusion and wiring and, finally, to filament drawing and cutting [4,5]. Prior to extrusion, the flakes are treated in a crystallizer and then in a dryer, aiming at orienting the molecular chains and removing material moisture, since the excess of moisture favors PET hydrolysis and its agglomeration and adhesion to the equipment walls [6,7].

In order to guarantee PES quality, the residence time of the flakes in the dryer must be enough for the moisture to be reduced to levels inferior to 0.005 mass% [8,9]. Usually, residence times between 3 and 5 h are employed [10,11]. Nevertheless, if preferred paths are formed, which reduce the time of contact, or dead volumes, which increase the time inside the equipment, there might be expressive deviations in PES quality [3,4,7]. Furthermore, flake agglomeration in these units might cause process failures, requiring the stop of the production line to perform emergency

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clearing maintenance, with a consequent drop in productivity [12].

The existence of these phenomena and flake flow behavior in the dryer can be evaluated by determining the residence-time distribution (RTD), which is used to diagnose operational problems associated to the flow in continuous systems and to describe material displacement inside the equipment by parameters such as external age, mean residence time, variance and standard deviation [13-15].

The determination of the RTD occurs with the feeding of an inert component, named tracer or marker, inside the equipment, at a specific time, and further monitoring of its outlet concentration as a function of time [16,17]. To guarantee the efficiency of the assays, inherent characteristics of the marker must be observed, such as a proper quantification technique, physical and chemical properties similar to the raw materials of the process and the non-adsorption of the material on the inner walls of the equipment [13,18-20]. Additionally, in RTD *in situ* determination in industrial processes, it must be guaranteed that the presence of the tracer does not alter the final product characteristics [21].

There are few works in the literature that report RTD determination in industrial processes. Wolf [22] performed RTD studies of a twin-screw extruder on the industrial production line of poly(vinyl chloride) (PVC) with a tracer based on manganese dioxide (MnO_2), obtaining a mean residence time equal to 3.3 min without interruption of the process, maintaining the production without economical losses. Lepschi [13], studying RTD *in situ* in a pilot scale extruder for the production of polypropylene (PP), obtained results with a mean residence time of 20 s and indications of the influence of the equipment screw configurations on polymer polydispersity. The measurements *in situ* occurred with an equipment of fluorescence spectroscopy for the quantification of the tracer composed of a mixture of low concentration of tracking particles and the results varied depending on the operating parameters, which allowed the observation of the optimized conditions of polymer mixture in the equipment. On the other hand, Chevrel [23] studied the flow behavior and RTD of the polymerization of acrylic acid solution in a plug flow reactor with sodium nitrate tracer at the laboratory and pilot scales. The measurements *in situ* were conducted with online probes and the application of the photonic technique of Raman spectroscopy, which indicated a mean residence time around 45 min.

Because of the industrial PES production scale by a continuous process and the absence of works in the literature addressing RTD in this process, it becomes necessary to develop strategies that allow the real-time diagnosis of the units without altering the plant operating mode. In the face of this, this work studied the RTD *in situ* in an industrial dryer of recycled PES production from PET flakes without the interruption of the production and the alteration of final product quality, aiming at detecting the presence of preferred paths or material agglomerates in the equipment by the comparison of the theoretical and real residence times.

EXPERIMENTAL

Process description

The study was performed in a recycled polyester production factory, located in the city of Poços de Caldas-MG (Brazil), aiming at determining the RTD *in situ* of the production line dryer. The raw material (colorless PET flakes) and the tracer (blue PET flakes) were obtained from the industrial recycling plant, responsible for the processing of PET bottles from different PET distribution centers.

In the beginning of the process, the PET flakes are taken to the crystallizer and then crystallized at temperature of 160 °C and by shear stress promoted by the stirrer. At the end of this step, the polymer is transported to the dust removal system for the removal of impurities and, subsequently, to the dryer of 10 m^3, in which it flows by gravity, at a flow from 500 to 900 kg h^{-1} and it is dried at ~175 °C with atmospheric air (flow up to 900 m^3 h^{-1} and dew point of -35 °C). After drying, the material is conducted to the extrusion system, molten together with masterbatches (pigmented pellets) colored black (proportion of 95% of flakes and 5% of masterbatches) and physically conformed in a wiring machine to obtain the polyester filaments which are, at the end of the process, subjected to drawing for the adjustment of their mechanical properties and cut in a length according to the type of product (in the order of 56 mm). Figure 1 shows the simplified block diagram of the process, highlighting the dryer, the object of study of this work.

Crystallization and characterization of the tracer and the raw material

Prior to drying, the tracer (blue PET flakes) and raw material (colorless PET flakes) were crystallized to reduce their hygroscopy, which favors the drying of these polymers [10]. The raw material was crystallized in a crystallizer of 8 m^3 (brand Ineal) operated at 165 °C, flow of 600 kg h^{-1} and at atmospheric pressure before the RTD studies. Therefore, aiming at

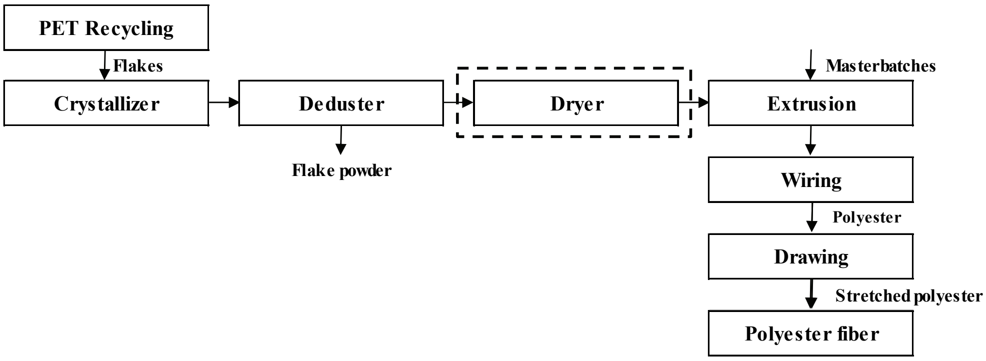

Figure 1. Block diagram of the manufacturing process of the recycled polyester fibers (PES), highlighting the dryer, the system in which RTD was studied.

maintaining the tracer properties similar, it was also crystallized at the same operational conditions for 4 h, feeding the crystallizer with 5000 kg of blue flakes. A fraction of the output current, 80 kg, was collected for the determination of the RTD in the dryer.

Samples of the tracer (collected after the crystallization step) and raw material were analyzed before (120 min), during (180 and 360 min) and after (1320 min) the beginning of the RTD study, aiming at determining the density, crystallization percentage and intrinsic viscosity of the materials.

The density was obtained in a pycnometer at 25 °C, using 5×10^{-3} kg of PET bottleneck flakes, previously crystallized, and calculated by Eq. (1) [24,25]:

$$\rho = 1000\frac{m_s}{(m_{pw}+m_s)-m_{psw}} \tag{1}$$

where ρ is the density (kg m^{-3}), m_s is the sample mass (kg), m_{pw} is the mass of the pycnometer with distilled water at 25 °C (kg) and m_{psw} is the mass of the pycnometer with distilled water and flake samples at 25 °C (kg).

Once the density was calculated, the crystallization percentage was calculated by Eq. (2) [25]:

$$\text{Crystallinity (\%)} = 100\frac{\rho-\rho_a}{\rho_c-\rho_a} \tag{2}$$

in which crystallinity is the crystallization percentage of the PET flakes (%), ρ_a is the density of the amorphous flakes (kg m^{-3}) and ρ_c is the density of the flakes with the maximum crystallization (kg m^{-3}).

The intrinsic viscosity $[\eta]$(dl g^{-1}), which is an indirect measure of the molar mass and one of the main parameters of PET rheological characterization, was determined according to the norm ASTM D2857 - Standard Practice for Dilute Solution Viscosity of Polymers [26]. The analysis was performed after the dissolution of (2.000±0.001) g of polymer, at (120.0±5.0) °C, in an Erlenmeyer flask with (25.0±0.1) mL of the solvent 2-chlorophenol (Sigma-Aldrich®, 99% purity), followed by the measurement of the flow time in a calibrated Cannon-Fenske viscometer at the standard temperature of (25.0±0.1) °C. With the flow time of the pure solvent, the relative viscosity was calculated, which is proportional to the intrinsic viscosity, according to Eqs. (3) and (4) [27,28].

$$[\eta] = \lim_{c\to 0}\frac{\ln\eta_r}{c} \tag{3}$$

$$\eta_r = \frac{t}{t_0} \tag{4}$$

where c is the concentration of the polymer solution (g dl^{-1}) and η_r is the relation between the flow times of the polymer solution (t) and of the pure solvent (t_0).

Determination of the RTD in industrial dryer

In the dryer, 80 kg of tracer with 99.51% purity (blue flakes, Figure 2), previously crystalized, were fed, by pulse-type disturbance, maintaining the constant flow of 600 kg h^{-1} at 165 °C. In the dryer flow field, 1 kg of sample (a mixture of colorless and blue flakes) was collected every 30 min for a period of 8 h. The mass concentration (% m/m) of the tracer was obtained after manual separation and weighing of this tracer present in 100 g of sample.

Figure 2. Tracer of blue PET flakes.

For the determination of the dryer theoretical residence time, operational data of the process (volume and level of the dryer and flow of PET flakes) were used, and values of the apparent densities obtained from the samples collected during the RTD assay.

The external age $E(t)$ was calculated by Eq. (5) for the calculation of the parameter external age [13,22] and the integral $C(t)$ was resolved numerically applying the five-point quadrature. This numerical integration model provides high robustness that minimizes possible analytical errors during resolution [19,29,30]:

$$E(t)=\frac{C(t)}{\int_0^\infty C(t)\mathrm{d}t} \tag{5}$$

in which $E(t)$ is the external age or RTD function (min^{-1}) and $C(t)$ is the concentration as a function of time (mass%).

With the $E(t)$ values, it was possible to calculate the mean residence time (first moment), Eq. (6), of residence of the PET flakes in the dryer [13,31,32]:

$$t_m=\int_0^\infty tE(t)\mathrm{d}t \tag{6}$$

in which t_m is the mean residence time (in min) and t is the time of analysis (in min).

Finally, the variance (second moment) and the standard deviation were calculated from Eqs. (7) and (8), respectively [13,19,33]:

$$\sigma^2=\int_0^\infty (t-t_m)^2 E(t)\mathrm{d}t \tag{7}$$

where σ^2 is the variance (min^2);

$$\sigma=\sqrt{\sigma^2} \tag{8}$$

where σ is the standard deviation (min).

With the information of operation and apparent density, the theoretical residence time was determined by Eq. (9):

$$t_R=\frac{V}{v}$$

where t_R is the theoretical residence time (min), V is the usable volume of the dryer (m^3) and v is the volumetric flow rate of the dryer (m^3 h^{-1}) defined by Eq. (10):

$$v=\frac{\dot{m}}{\rho_b} \tag{10}$$

in which $\dot{m}$ is the mass flow of the process (kg h^{-1}).

The apparent density of the samples collected during the RTD assay was calculated by Eq. (11), after the weighing of a container of 100 cm^3 (diameter of 4.0 cm and height of 8.0 cm) empty, filled with distilled water at 25 °C and filled with a flake sample (mixture of colorless and blue flakes) [34]. The addition of the flake samples in the container was performed with a funnel according to the specifications of method A of the norm ASTM D1895 - Standard Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials [35], with the superior diameter of the funnel being 9.0 cm, inclination of 20° for material flow, height of 11.5 cm, output diameter of 2.0 cm and distance from the collection container of 3.8 cm:

$$\rho_b=1000\frac{m_{es}-m_e}{m_{ew}-m_e} \tag{11}$$

where ρ_b is the apparent density (kg.m^{-3}), m_e is the mass of the empty container (kg), m_{ew} is the mass of the container with water (kg) and m_{es} is the mass of the container with sample (kg).

For the evaluation of the relative deviation (RD) from the mean residence time (t_m) in relation to the theoretical residence time (t_R), Eq. (12) was used:

$$RD=100\frac{t_m-t_R}{t_R} \tag{12}$$

Finally, after the investigations of the flow behavior, the Peclet number (Pe) can be calculated experimentally and used to understand the convective and diffusive transport rate by Eq. (13):

$$\frac{\sigma^2}{t_m^2}=\frac{2}{Pe}-\frac{2}{Pe^2}(1-e^{-Pe}) \tag{13}$$

PES characterization and quality control

As the RTD study was performed on an industrial scale and without interruption in the production, analyses of intrinsic viscosity and color force were performed for the evaluation of the influence of adding the tracer on the quality of the PES fiber after the extrusion, collected before (120 min), during (180 and 360 min) and after (1320 min) the beginning of the RTD study, and compared to a standard PES sample produced without the addition of a tracer.

The analyses of [η] were performed in conformity to the norm ASTM D2857 [26]. In the case of the PES fibers, before the dissolution with solvent, a preliminary step was performed for the removal of moisture and impurities from the filaments (such as the emulsions of anti-aesthetic oils added during wiring), in which the samples were washed with 250 mL of methanol, and the excess of liquid was removed, with metal tweezers they were cut into threads of 1 cm and dried in a convection oven at 120 °C for 60 min.

The color force was measured according to the model CIELAB of 1976 from the Commission Internationale de l'Eclairage (CIE) in a Hunter LabScan XE® spectrophotometer at a wavelength (λ) between 400 and 700 nm. The interpretation of the result from the equipment Hunter LabScan XE® is presented in Table 1 [36-39].

Table 1. Interpretation of the polymer color analysis in the spectrophotometer Hunter LabScan XE®

Result (%)	Interpretation on the color force [40]
100.0	Sample with coloration equal to the standard
> 100.0	Sample with darker coloration than the standard
< 100.0	Sample with lighter coloration than the standard

RESULTS AND DISCUSSION

Physicochemical properties of the PET flakes

The specifications of the process parameters had been previously defined by the department of engineering of the factory in order to obtain a higher performance and, mainly, to meet the requirements demanded by the consumer market. Therefore, crystallization percentages higher than 32% were adopted for the PET flakes at the crystallizer output and the values of intrinsic viscosity and color force of (0.640±0.020) dl g^{-1} and (100.0±7.0)%, respectively, were accepted for the PES fibers. The study occurred directly in the industrial production line, aiming at evaluating the tracer characteristics and the effects that occurred in the polymer after extrusion.

Table 2 displays the values for density, crystallinity percentage and intrinsic viscosity obtained for the analyzed flake samples from the crystallizer output. It was observed that the results of crystallinity and viscosity presented variations inferior to 5% when compared to each other, indicating similar characteristics of the materials (raw material and tracer). It was also noted that the values of intrinsic viscosity obtained respected the tolerable limits for PET packages (0.700 to 0.850 dl g^{-1}) [3,27]. The values of density, obtained for the flakes under different conditions, were also consistent with the literature. Zhang and Ward [25], Fakirov [41] and Thompson and Woods [42] reported density values for amorphous PET and PET with maximum crystallization equal to 1335 and 1445 kg m^{-3}, respectively.

Residence time distribution in the dryer

During the RTD study, the drying air and the mass flow of the PET flakes were maintained, respectively, at a temperature around 165 °C and 600 kg h^{-1} (with negligible deviations). The samples were collected at the dryer output with a sampling device and stored in plastic bags. Figure 3 presents a photo of the sample collected with 30 min of RTD analysis and the photo of the sample with 330 min.

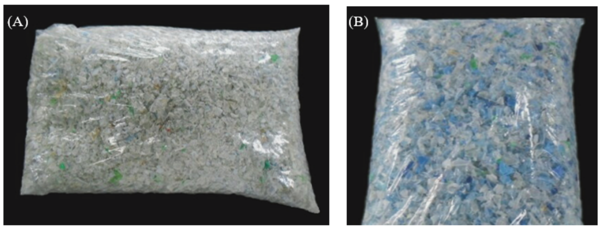

Figure 3. Photo of the sample with PET flakes from the dryer output after 30 min from the beginning of the RTD (A), and after 330 min from the beginning of the RTD (B).

By the separations of blue flakes in the samples, weighing of the material, implementation of the analytical procedure for the determination of the apparent density and Eq. (1), Table 3 was constructed, which shows the results found in the laboratory. The apparent density was subsequently used to obtain the flake volumetric flow in the dryer.

To improve the accuracy of the results, two considerations were made. The first was derived from the observation that at time t = 0 the content of blue flakes was 0.42 mass%. Commonly in PET recycling, the colorless flakes have residual contamination from the other colors of PET, such as green, yellow, pink or blue [43]. Thus, the content of 0.42% was considered as analytical blank and this value was subtracted from

Table 2. Results of the analysis of the PET flakes

Identification of the flake sample	ρ (kg m^{-3})[a]	Crystallinity (%)[a]	[η] (dl g^{-1})[a]
Crystallizer output (before the RTD test)[b]	1371.4 ± 0.2	33.1 ± 0.2	0.794 ± 0.002
Tracer	1371.6 ± 0.3	33.3 ± 0.3	0.788 ± 0.003
Crystallizer output (180 min of RTD test)	1371.1 ± 0.3	32.8 ± 0.3	0.791 ± 0.003
Crystallizer output (360 min of RTD test)	1371.2 ± 0.2	32.9 ± 0.2	0.797 ± 0.004
Crystallizer output (end of the RTD test)[c]	1371.0 ± 0.2	32.7 ± 0.2	0.801 ± 0.002

[a]Mean of 3 analyses ;[b]sample collected 120 min before the beginning of the test; [c]sample collected on the day after the test (1320 min from the beginning)

the other results. Concomitantly, tracer purity was analyzed by quantifying the concentration of blue flakes. Since the content was 99.51% of blue flakes, no weightings were performed in the results. In Table 3, the normalized results are also presented.

Table 3. Results of the tracer content and apparent density of the flakes

Time (min)	Content of blue flakes (mass%) - $C(t)$	Content of normalized blue flakes (mass%) - $C(t)$	ρ_b[a] kg m^{-3}
0	0.42	0.00	360
30	0.48	0.06	351
60	0.55	0.13	357
90	0.59	0.17	352
120	0.56	0.14	348
150	0.53	0.11	355
180	0.75	0.33	360
210	0.94	0.52	353
240	0.97	0.55	352
270	1.06	0.64	354
300	7.60	7.18	353
330	30.58	30.16	347
360	3.74	3.32	357
390	0.65	0.23	356
420	0.62	0.20	355
450	0.57	0.15	350
480	0.55	0.13	359

[a] Mean of 3 analyses (overall mean result of 354 kg m^{-3})

With the $C(t)$ values, the numerical integration of the five-point quadrature was used to calculate the integral $\int_0^\infty C(t)\,\mathrm{d}t$ in the interval between 0 and 480 min, in order to subdivide into four quadratures with a 30 min step. The result found was $\int_0^\infty C(t)\,\mathrm{d}t$ of 1519.9 % min (unit of concentration in mass%).

Table 4 presents the values of the function $E(t)$ calculated by Eq. (5) and other values associated to the RTD analysis.

For the determination of the residence time, the $tE(t)$ values (Table 4) were applied in Eq. (6) and subdivided for the numerical integration with the five-point quadrature at the interval between 0 and 480 min. A mean residence time (t_m) of 322.8 min was obtained. The variance and deviation were also calculated using Eqs. (7) and (8) applying the numerical integration of the five-point quadrature. Variance was of 1305.4 min^2 and the standard deviation of 36.1 min. The low values of standard deviation and variance are derived from a narrow residence time distribution, with PET flakes flowing evenly through the dryer, as observed in Figure 4.

Table 4. Values associated with the RTD

Time (min)	$E(t)$ (min^{-1})	$tE(t)$	$(t-t_m)^2E(t)$ (min)
0	0.00000	0.00000	0.00000
30	0.00004	0.00118	3.38450
60	0.00009	0.00513	5.90741
90	0.00011	0.01007	6.06206
120	0.00009	0.01105	3.78854
150	0.00007	0.01086	2.16119
180	0.00022	0.03908	4.42780
210	0.00034	0.07185	4.35360
240	0.00036	0.08685	2.48122
270	0.00042	0.11369	1.17415
300	0.00472	1.41720	2.45690
330	0.01984	6.54833	1.02713
360	0.00218	0.78637	3.02191
390	0.00015	0.05902	0.68325
420	0.00013	0.05527	1.24308
450	0.00010	0.04441	1.59666
480	0.00009	0.04106	2.11351

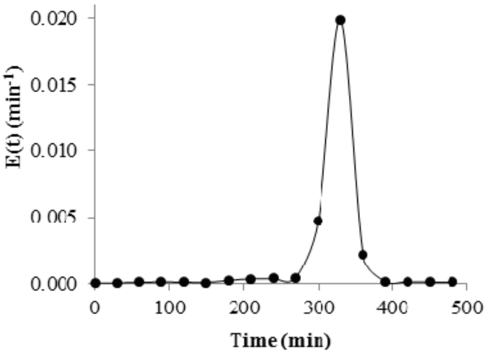

Figure 4. E(t) profile of the dryer.

To evaluate the existence of preferred paths or agglomerates in the dryer, the t_m was compared with the theoretical residence time (t_R), calculated by Eq. (9). The volumetric flow of the flakes was calculated by Eq. (10). The mean apparent density of the samples (Table 3) was of 354 kg m^{-3}. The total volume of the dryer was of 10 m^3, but its operation was conducted in 9 m^3 with mass flow of 600 kg h^{-1}. The t_R of the PET flakes in the dryer was of 318.0 min. This value presents a relative deviation of 1.5% (Eq. (12)) in comparison with t_m, indicating that there was no pronounced formation of agglomerates or material bypass during drying. Nevertheless, the ratio $t_R/t_m < 1$ indicated a slight trend of the PET flakes to remain for more time in the dryer, which can be attributed to the friction among the flakes or sintering, in which the particles adhere to each other and stay adsorbed on the inner walls of the equipment, promoting an inhomogeneous transport profile of the PET flakes [19,44].

The absence of preferred paths proved the importance of the partial removal of the fine particles in a dust removal system between the steps of crystallization and drying, since excessive amounts of fine particles cause the formation of preferred paths, the increase in load loss in the drainage flow and product contamination by degradation and carbonization [45,46].

Besides the evaluation of the relative deviation between t_R and t_m, Eqs. (7) and (8) were also used for the calculation of t_m variance and standard deviation. As displayed in Table 4, $(t-t_m)^2E(t)$ was obtained. With the numerical integration of the five-point quadrature, variance σ^2 resulted in 1305.4 min^2 and the standard deviation σ in 36.1 min. In summary, the flake particles might have diverted in ±36.1 min from the mean residence time.

Using the experimental results of mean residence time and variance (Eq. (13)), it was also possible to evaluate the convective and diffusive transport rates with the Peclet number (*Pe*), which resulted in approximately 150. This value indicates that the predominant transport in the flow of the PET flakes is of the convective type [19].

It is important to highlight that, in the industrial dryer studied, it was not possible to perform samplings at intervals lower than 30 min, since the withdrawal of material in short periods caused process destabilization, loss of extrusion efficiency and consequent wiring machine power failure, whose normalization implied complex operational adjustments and long periods of plant downtimes.

The pulse-type disturbance was efficient for the dryer RTD analysis, mainly by the easy implementation *in situ* to the productive system. This type of disturbance is more economical in comparison with step disturbance, in which the tracer concentration in the feeding is kept constant until its concentration in the equipment flow field is equal to that of feeding [33], which would imply a greater amount of tracer, as well as a higher energy expenditure in the crystallization step. Furthermore, step disturbance uses data differentiation equations, which algebraically lead to numerical errors that increase uncertainty in the measurement of the results [19].

PES physicochemical properties

The analyses of the recycled PES produced from the PET flakes were performed with samples taken from the wiring (sequent step to extrusion, Figure 1), aiming at observing the effects of the tracer on the final product. Figure 5 illustrates the filaments of the recycled polyester fibers with pigmentation of black masterbatches after 360 min of RTD test (wiring sample).

Figure 5. Photo of the filaments of the recycled polyester fibers with pigmentation of black masterbatches after 360 min of the beginning of the RTD test (wiring sample).

In accordance with the literature, textile fibers, such as PES, have intrinsic viscosity [η] around 0.600 dl g^{-1} [3], and may vary between 0.400 and 0.700 dl g^{-1} [27]. According to the industrial specifications, PES fibers must present values of intrinsic viscosity and color force close to (0.640±0.020) dl g^{-1} and (100.0±7.0) %, respectively. Table 5 presents values of intrinsic viscosity and color force obtained for the standard PES and the PES collected before, during and after the RTD tests. It was observed that the addition of the tracer in the process feed current did not alter the PES quality parameters, which guaranteed that the subsequent product commercialization would be accomplished without technical and economic losses.

Comparing Tables 2 and 5, an average decrease in the intrinsic viscosity of 0.147 dl g^{-1} of the PET flakes was observed in relation to the produced

Table 5. Results of PES characterization after the extrusion

Sample identification	[η] (dl g^{-1})^a	Color force (%)^a
Recycled polyester standard	0.642±0.003	99.9±0.1
Recycled polyester (before the RTD test)^b	0.649±0.003	98.3±0.2
Recycled polyester (180 min of RTD test)	0.645±0.002	98.5±0.4
Recycled polyester (360 min of RTD test)	0.646±0.003	98.8±0.4
Recycled polyester (end of the RTD test)^c	0.650±0.003	98.2±0.3

^{a}Mean of 3 analyses ;^{b}sample collected 120 min before the beginning of the test; ^{c}sample collected on the day after the test (1320 min from the beginning)

PES. According to the literature, the PET flakes present intrinsic viscosity of 0.800 dl g^{-1} and after the extrusion, in a system without vacuum adjustment, there might be a loss in the intrinsic viscosity of 0.200 dl g^{-1} in the wired PES because of thermal and oxidative reactions. Nevertheless, in cases of high moisture, an additional decrease of 0.010 dl g^{-1} happens for each 0.002% of moisture, which impairs the quality of the final product (PES) mainly because of the adjustments in the polyester fibers mechanical properties of traction and toughness [3,5,8,47-50]. This low loss of intrinsic viscosity of the PES fibers during the fusion of the molecules agrees with the low deviation of the times of the flakes in the dryer in relation to t_R, since the excess of moisture, which causes problems such as hydrolytic degradation and sintering, was eliminated in the drying step [4,44,48]. Therefore, moisture removal was not affected by the addition of the tracer, indicating that it was possible to perform the RTD analysis of the dryer with the industrial plant under continuous operation without impairing the quality of the PES fibers.

Finally, it was highlighted that during the assay performed, the tracer continued with the process flow, in other words, it went through the extruder and wiring for polyester conformation, since the blue PET is chemically similar to the colorless PET. Additionally, since the black masterbatches were added according to the product of interest [51], fiber color and viscosity did not suffer significant impacts, as presented in Table 5. As presented by Wolf [22], the work developed was efficient, since RTD characterization was possible to execute without impairing the characteristics of the process, of the product and with the industrial plant under continuous operation. Thus, the dryer RTD was characterized and, simultaneously, the product of interest was obtained.

CONCLUSION

In this work, it was possible to determine the RTD of an industrial dryer for the production of PES from PET flakes without interrupting the continuous operation of the plant. The blue flakes used as tracer demonstrated similar physical properties to those of the raw material and were easily separated and quantified at the dryer output. The ratio between the theoretical residence time (318.0 min) and the mean residence time (322.8 min) indicated a slight trend of flake retention in the dryer, but not big enough to characterize preferential flows or PET flake agglomerates that might lead to the obstruction or loss of dryer performance. The variance (1305.4 min^2) and standard deviation (36.1 min) obtained showed a narrow residence time distribution, indicating that all flakes stayed for similar times inside the dryer, having their moisture homogeneously reduced before extrusion. Finally, PES fibers were produced with intrinsic viscosity and color force inside the market specifications, demonstrating that PES quality was not affected by the addition of the tracer in the RTD analysis. Therefore, it was proved that it is possible to perform the detection of transport problems in an industrial PET flake dryer, by the RTD analysis.

Nomenclature

ASTM	American Society for Testing and Materials
$C(t)$	concentration [% m/m]
$C_0(t)$	initial concentration [% m/m]
CIE	Commission Internationale de l'Eclairage
RTD	residence time distribution
$E(t)$	external age [min^{-1}]
$\dot{m}$	mass flow [$kg.h^{-1}$]
PES	polyester
PET	polyethylene terephthalate
PP	polypropylene
PVC	poly(viny1 chloride)
t	time [min]
t_R	residence time [min]
t_m	mean residence time [min]
v	volumetric flow [$m^3.h^{-1}$]
$[\eta]$	intrinsic viscosity [$dl.g^{-1}$]
η_r	relative viscosity
ρ	density [$kg.m^{-3}$]
ρ_b	apparent density [$kg.m^{-3}$]
σ^2	variance [min^2]
σ	standard deviation [min]
λ	wavelength [nm]

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NAUČNI RAD

UTVRĐIVANJE RASPODELE VREMENA ZADRŽAVANJA U INDUSTRIJSKOJ SUŠARI ZA PROIZVODNJU RECIKLIRANOG POLIESTERA

Rad predlaže procenu raspodele vremena zadržavanja (RTD) na licu mesta u industrijskoj sušari za proizvodnju recikliranih poliesterskih vlakana (PES) od bezbojnih ljuspica polietilen-tereftalata (PET) bez prekida proizvodnje. Korišćen je pulsni poremećaj, kod kojeg je traser (plave PET ljuspice) prethodno kristalisan, a njegova koncentracija je dobijena prema vremenu na izlazu iz sušare. Pored toga, izvršene su analize unutrašnje viskoznosti i procenta kristalizacije PET ljuspica (bezbojne i plave) i unutrašnje viskoznosti PES. Na osnovu RTD dobijene su srednje vreme boravka (322,8 min), varijansa (1305,4 min^2), standardna devijacija (36,1 min) i relativna greška (1,5%) u poređenju sa teorijskim vremenom zadržavanja, što ukazuje na odsustvo željenih putanja ili aglomerati ljuspica u sušari. Na kraju, karakterizacija je pokazala da nije došlo do promene parametara kvaliteta proizvoda tokom RTD procene, što potvrđuje mogućnost primene ove metodologije za dijagnosticiranje kontinualnih industrijskih procesa.

Ključne reči: vreme zadržavanja; PET pahuljice; poliesterska vlakna.