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Residence time distribution in a co-rotating, twin-screw continuous mixer by the step change method

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Abstract

Residence time distributions were determined for the continuous processing of chocolate in a twin-screw, co-rotating mixer, and modeled using as a series combination of piston flow and ideal mixing elements or as equal size tanks in series. Color (L-value) was measured after a step change from milk chocolate to white chocolate. Both models fit the data well, although the series combination of piston flow and ideal mixing fit better for short mean residence times, accurately predicting the observed deadtime. The series of tanks model appeared to fit data better under conditions where longer mean residence times were observed. The mean residence time was significantly influenced by feed rate, screw speed and gate opening. A high shear, low conveyance screw configuration was used that led to a high fill fraction (>0.85). Therefore, feed rate had the greatest effect on the mean residence time. The time of first appearance was affected only by the gate opening, and ranged from 0.44 to 0.68 times the mean residence time.

Keywords: Residence time distribution; Continuous mixer; Twin-screw; Co-rotating

1. Introduction

In the manufacture of chocolate, conching follows blending and refining (size reduction) of cocoa liquor, sugar crystals, cocoa butter and milk solids. Conching is a controlled shearing-heating process that liquefies the powdery, refined chocolate mass (known as refiner flake). Conventional conching is a batch process lasting 3–72 h depending on the manufacturer. However, conching time can be considerably reduced by using continuous equipment (Franke & Tscheuschner, 1991; Holzhäuzer, 1992; Ziegler & Aguilar, 1994). Cooker-extruders have been used for continuous chocolate conching because of their ability to mix, heat and shear simultaneously (Chaveron, Adenler, Kamoun, Billon, & Pontillon, 1984; Mange, 1987). Since some of the special processing capabilities of twin-screw extruders for cooking, forming, puffing and pressure generation are not needed for conching chocolate, extruders are unnecessarily complex and expensive.

Twin-screw, continuous mixers are widely used in the chemical and polymers industries for melting, mixing, coloring and compounding. Blending operations for incorporation of fillers, reinforcing agents, glass fibers, etc., are also carried out in twin-screw, continuous mixers (Miller, 1984). Typical applications of the mixer used in this study include compounding, kneading, cooking, drying, extruder preconditioning, crystallization and pelletizing (Anonymous, 1994).

Residence time is an important parameter in continuous processes because it determines the extent of chemical reaction (Levenspiel, 1972). During the conching of milk chocolate fat is melted, solid particles are dispersed and lactose is crystallized (Ziegler & Aguilar, 1994). In addition, caramel flavor is developed through the Maillard reaction, which is highly dependent on the time–temperature treatment given the chocolate mass (Aguilar, Dimick, Hollender, & Ziegler, 1995). In this study, we report on the residence time distribution during continuous conching of chocolate in a co-rotating, twin-screw continuous mixer. While several studies of the residence time distribution in co-rotating, twin-screw extruders have been published in the last decade (for the most recent see Unlu & Faller, 2002), they have focused on starch-based foods at relatively high mois-

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ture that are similar to polymer melts. In this study we describe the processing of a particulate suspension at high solids fraction. While most investigators have used the pulse method (Levine & Miller, 2002), we describe here the analysis of a negative step change in tracer concentration.

2. Materials and methods

2.1. Materials

Cocoa butter, chocolate liquor (mass), spray-dried whole milk powder, and sucrose were obtained from commercial sources. Milk chocolate and white chocolate refiner flake were prepared according to the formulations given in Table 1. Chocolate liquor and cocoa butter were heated at 60–65 °C until molten and mixed with the remaining ingredients using a Hobart mixer (model A-200, The Hobart Manufacturing Co., Troy, OH). The mixes were refined to a mean diameter over the volume distribution, $d_{4,3}$, of 10.5 μm (cumulative percentage undersize of 90% of the volume distribution, $d_{v,0.9} = 24.1$ μm) using a horizontal, three-roll refiner (Lehmann Maschinenfabrik, Germany) cooled with tap water (15–17 °C).

2.2. Twin-screw, co-rotating mixer

The refiner flake was processed in a 3.73 kW (5-h.p.) twin-screw, co-rotating continuous mixer with a 5 cm (2″) per shaft barrel diameter (Readco Manufacturing, York, PA). The screws were assembled from individual convex "lens-shaped" mixing paddles (Fig. 1(a)) similar to the kneading elements of twin-screw extruders (Dziezak, 1989). The shafts of the Readco processor can be fit with different arrangements of mixing paddles having forward pitch (right-handed paddles), non-conveying (flat paddles) and reverse pitch (left-handed paddles). The screw configuration used in this investigation imparts high shear with low conveyance (Fig. 1(b)). It comprises a short segment of conveying screw, followed by 24 flat, non-conveying mixing paddles and ending with a single left-handed paddle (Readco Manufacturing refers to this as paddle configuration #8). The "clam shell" barrel of the mixer is jacketed for temperature control. Barrel temperature was maintained at 70 °C with circulating hot water. The discharge opening consisted of a rectangular gate with a constant length of 89 mm and manually adjustable width.

The refiner flake was fed into the mixer with an Accurate (model 800) volumetric dry materials feeder (Accurate Inc., Whitewater, WI), individually calibrated for the milk and white chocolate formulations.

Table 1
Formulation of refiner flake

Ingredient	Milk chocolate	White chocolate
Chocolate liquor	15.9	0.0
Cocoa butter	12.9	19.2
Whole milk powder	23.1	26.2
Sucrose	48.1	54.6
Total/% fat	100/28.3	100/26.8

2.3. Experimental design

Response surfaces were generated using a central composite rotatable design with three variables, mass feed rate (g/min), discharge gate opening (mm) and screw rotational speed (rpm), at five levels (Table 2) (Montgomery, 1991). A total of 20 experimental runs were conducted, with six replicates at the center point (151.5 g/min, 250 rpm, 6.35 mm). Statistical analysis was conducted using ECHIP experimental design software (ECHIP Inc., Hockessin, DE). After regression analysis inclusive of all terms, any term with $p > 0.1$ was eliminated from the regression model and the data was reanalyzed.

2.4. Data analysis

Residence time distribution was measured by a negative step change corresponding to turning off the supply of an inert tracer. In this case the inert tracer was the chocolate liquor contained in the milk chocolate formulation, and the step change was accomplished by the immediate transition from milk chocolate feed to white chocolate feed. This is commonly referred to as a washout experiment (Nauman, 1985). For accurate determinations of residence time, the tracer should not affect the properties of the material being processed. For this reason, the milk and white chocolate refiner flakes were formulated and refined to match, as closely as practically possible, their physical properties. Samples were collected at the discharge end of the mixer at either 30-s or 1-min time intervals, and the L-value measured using a color meter (model CR-200, Minolta Camera Co., Ltd., Japan). A linear relationship between the proportion of white coating (from 0% to 100%) and the L-value was observed ($r^2 = 0.988$). A dimensionless concentration was calculated using Eq. (1):

$$\frac{L - L_0}{L_\infty - L_0} \qquad (1)$$

where L_0 is the initial lightness value (that of the milk chocolate mass) and L_∞ is the final lightness value (that of the white chocolate after complete washout).

The experimental response curve expressed in dimensionless concentration was fit directly to the following models using non-linear least squares and the Levenberg–Marquardt algorithm (Origin 6.1, Microcal

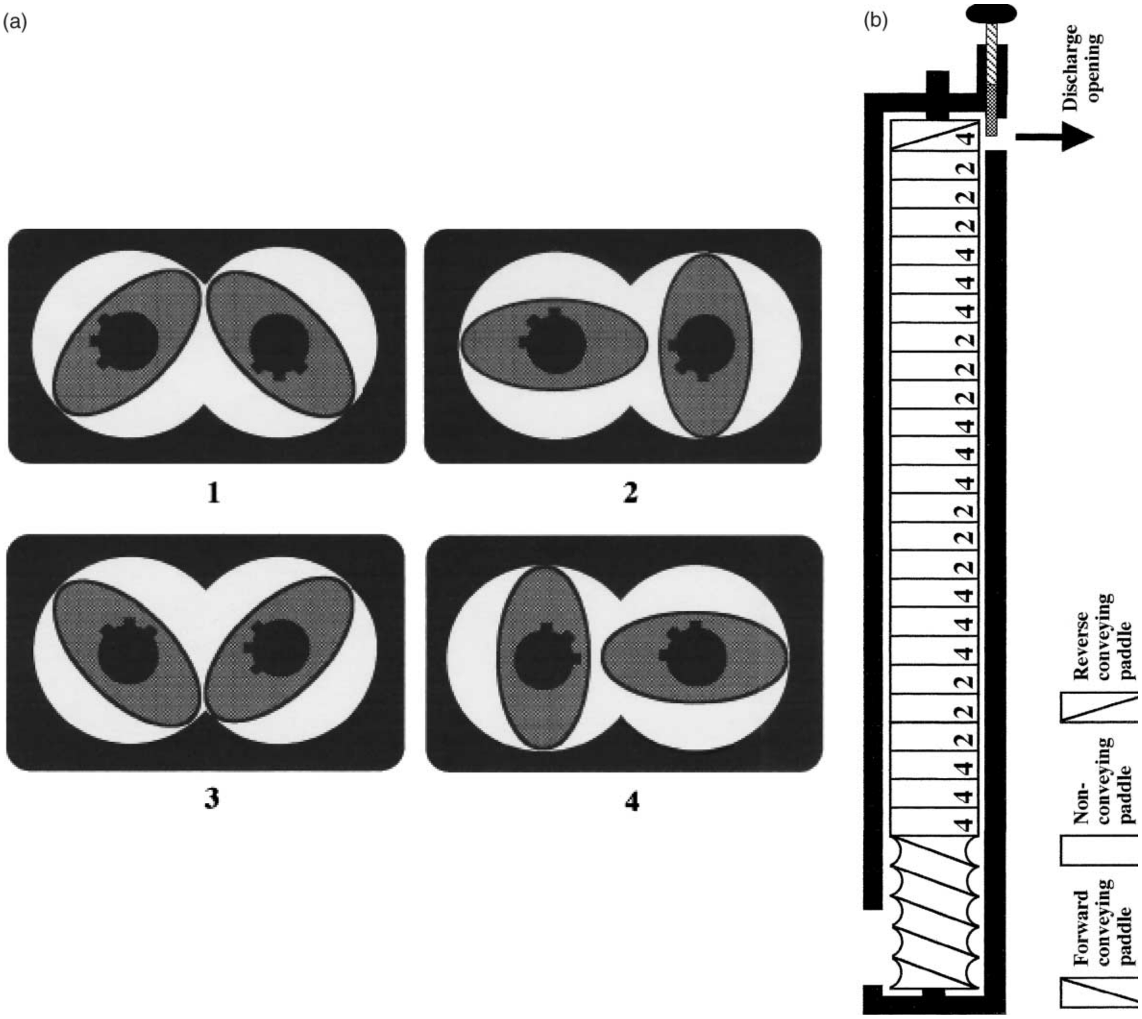

Fig. 1. (a) Paddle orientation 1–4 referred to in (b). (b) Paddle configuration along the barrel of the mixer.

Table 2
Experimental design

Variable	Level				
	−1.6	−1	0	1	1.6
Feed rate (g/min)	90.9	113.6	151.5	189.4	212.1
Screw speed (rpm)	210	225	250	275	290
Gate opening (mm)	3.8	4.8	6.35	7.9	8.9

Software, Inc., Northampton, MA). The first model was a series combination of piston flow and ideal mixing elements. The washout function for this model is (Nauman, 1985):

$$W(\tau) = \exp\left[\frac{-(\tau - \tau_P)}{(1 - \tau_P)}\right] \quad \tau > \tau_P \tag{2a}$$

$$W(t) = 1 \quad \tau \leqslant \tau_P \tag{2b}$$

where $\tau = t/\bar{t}$ and τ_P is a dimensionless parameter known as the fractional tubularity equal to the first appearance time/mean residence time. For Eq. (2) there are two adjustable parameters, $\bar{t}$ and τ_P. The second model employed was the theoretical residence time distribution for J equal sized tanks in series (Nauman, 1985):

$$W(t) = \exp[-J\tau]\sum_{i=0}^{J-1}\frac{J^i\tau^i}{i!} \tag{3}$$

In this case, each segment of three aligned paddles (Fig. 1(b)) was assumed to be a "tank". Therefore, J was set equal to 8 and the only adjustable parameter was then $\bar{t}$.

3. Results and discussion

3.1. Comparison of the models

The average regression coefficient (r^2) for the fit of Eqs. (2) and (3) for all 20 runs was 0.977 and 0.982, respectively. However, this difference was not significant ($P > 0.05$) by a paired *t*-test. Eq. (2) seemed to fit the data better for runs with short mean residence times (Fig. 2), while Eq. (3) fit better to data for runs with longer mean times (Fig. 3). This would seem reasonable since less dispersion and a greater approximation to piston (plug) flow could be expected for shorter residence time.

Table 3
p-values from ANOVA for the influence of process variables on fit parameters

Parameter	τ_P	$\bar{t}_{\text{Eq. (2)}}$	$\bar{t}_{\text{Eq. (3)}}$
Feed rate	0.22	0.001	0.001
Screw speed	0.61	0.15	0.11
Gate opening	0.05	0.12	0.07

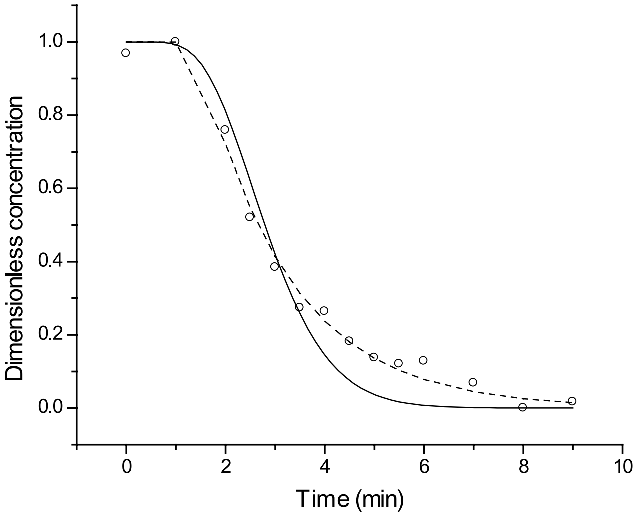

Fig. 2. Comparison of the fit of Eq. (2) (dashed line) to Eq. (3) (solid line) for an experimental run with a relatively short mean residence time.

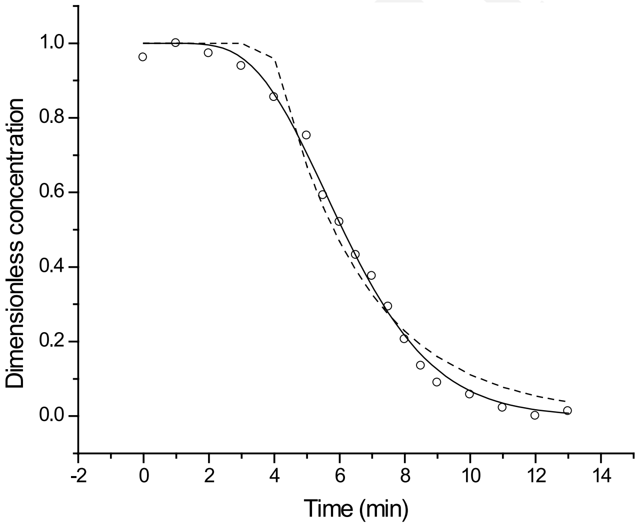

Fig. 3. Comparison of the fit of Eq. (2) (dashed line) to Eq. (3) (solid line) for an experimental run with a relatively long mean residence time.

3.2. Influence of process variables

The mean residence time, $\bar{t}$, extracted from the fit of Eqs. (2) and (3) to the experimental data was influenced most significantly by the feed rate (Table 3). The mean residence time was linearly and inversely related to the feed rate (Fig. 4) ($p = 0.001$). At first thought it would seem unreasonable that the screw speed would not have a stronger effect on residence time. However, the paddle configuration used was devoid of conveying elements, save the initial feed screw, and therefore, while screw speed may impart greater work to the material, it does not apparently propel it through the barrel much faster. The material is in effect pushed along by the introduction of new material. Yeh, Hwang, and Guo (1992) similarly found that feed rate had a more pronounced effect on mean residence time than did screw speed when processing wheat flour in a twin-screw extruder, an observation confirmed by Unlu and Faller (2002) for

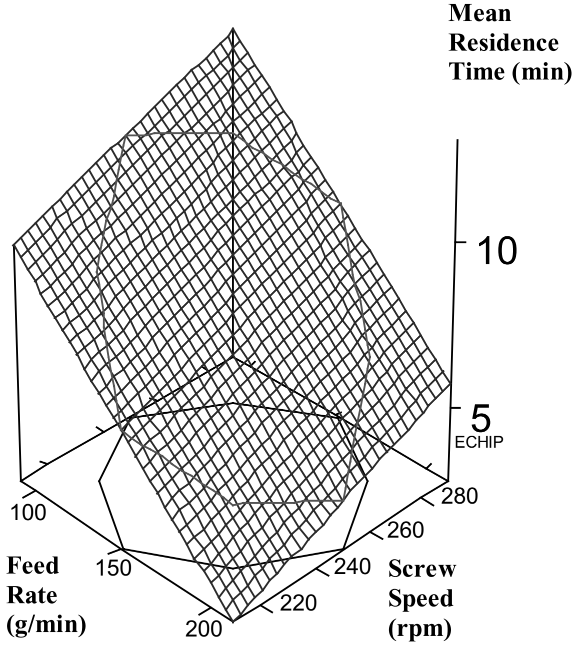

Fig. 4. Response surface for the effect of feed rate and screw speed (at constant gate opening of 6.35 mm) on mean residence time extracted from Eq. (3).

cornmeal. Gogoi and Yam (1994) also found a significant effect of screw speed and throughput on mean residence time, but with screw speed having the greater impact. De Ruyck (1997) found the effect of screw profile and screw speed on mean residence time of wheat flour in a twin-screw extruder to be more pronounced than that of feed supply. It is likely that the effect of screw speed increases with the introduction of more forward or reverse pitch elements. Gautam and Choudhury (1999) observed that the mean residence time decreased for screw profiles with kneading blocks (similar to the flat paddles used in this study) vis-à-vis those with reverse screw elements.

The mean residence time, $\bar{t}$, extracted from Eq. (3) can be estimated using Eq. (4):

$$\begin{aligned}\bar{t} = 7.84 &- 0.0592\ (\text{Feed rate} - 151.5)\\ &+ 0.0383\ (\text{Screw speed} - 250)\\ &- 0.7050\ (\text{Gate opening} - 6.35) \qquad (4)\end{aligned}$$

($p = 0.0000$, 0.0304, and 0.0145 for feed rate, screw speed and gate opening, respectively, $r^2 = 0.73$ and $p = 0.0001$ for the overall equation).

The fractional tubularity, τ_P, extracted from Eq. (2) was significantly affected only by the gate opening (Table 3). A quadratic relationship was observed between gate opening and τ_P (Fig. 5) described by Eq. (5):

$$\begin{aligned}\tau_P = 0.61 &- 0.0138\ (\text{Gate opening} - 6.35)\\ &- 0.0168\ (\text{Gate opening} - 6.35)^2 \qquad (5)\end{aligned}$$

($p = 0.22$ and 0.02 for the linear and quadratic terms, respectively, $r^2 = 0.310$ and $p = 0.04$ for the equation overall). τ_P averaged 0.58 with a range of 0.44–0.68, comparable to published values: 0.5 (Altomare & Ghossi, 1986), 0.55 (Curry, Kiani, & Dreiblatt, 1991), 0.41–0.55 (Lee & McCarthy, 1996), and from 0.4 to 0.6 (Todd, 1975).

Gate opening may be somewhat analogous to the die diameter in extrusion systems. Olkku, Antila, Heikkinen, and Linko (1980) reported that the residence time distribution depended on die diameter, flow resistance, screw speed and feed rate, and that the most important section of the extruder appeared to be the last 10–20 cm before the exit. This may apply to a greater extent in cooking extruders with screw-type elements where the barrel fill reaches 100% only near the die. However, with the screw configuration used in this study, barrel fill was observed to be nearly 100% along the entire length. The fill factor, f is defined as:

$$f = \frac{\bar{t}\dot{V}}{V} \qquad (6)$$

where V is the total volume of the barrel (in this instance 934.45 ml) and $\dot{V}$ is the volumetric flow rate. At 151.5 g/min and assuming 1.3 g/ml for the refiner flake (Beckett, 1999), $f = 0.98$. At the lowest feed rate used, 90.9 g/min, the predicted mean residence time is 11.43 min corresponding to a fill factor of 0.86.

Levine and Miller (2002) recommended plotting the dimensionless exit age distribution against dimensionless time in order to compare process changes. They suggested that not doing so is a common error. As an example, they describe a system whereby the feed rate is reduced resulting in a longer mean residence time and broader distribution, which is commonly misinterpreted as more mixing. However, when the data are compared in dimensionless form, the distributions appear identical, implying that the variance is proportional to mean residence time. This is illustrated in Fig. 6, in which the dimensionless concentration is plotted against the di-

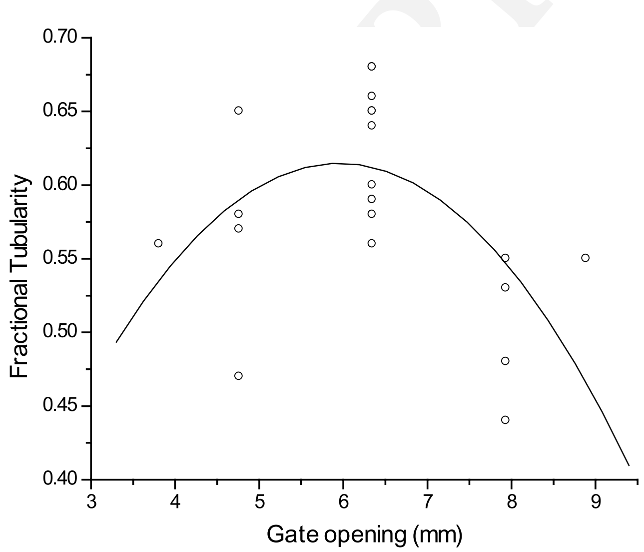

Fig. 5. Relationship of fractional tubularity estimated from Eq. (2) to gate opening.

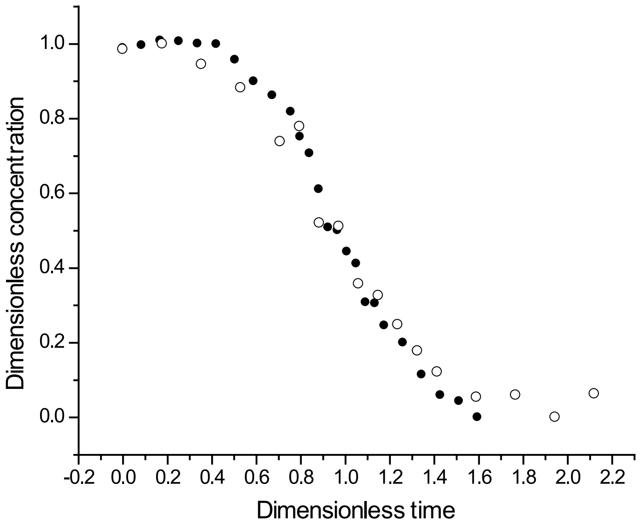

Fig. 6. Normalized washout curves for an experimental run at low feed rate (90.9 g/min, closed circle) and high feed rate (212.1 g/min, open circle).

mensionless time for two experimental runs differing only in the feed rate (90.9 vs. 212.1 g/min). The mean residence time extracted from Eq. (3) for these distributions was 11.91 and 5.66 respectively.

Mahungu, Drozdek, Artz, and Faller (2000) observed shorter mean residence times and narrower residence time distributions for pet food processed in a twin-screw extruder when feed rate and screw speed were increased (at a constant ratio of feed rate to screw speed). But from an analysis of the normalized RTD, they concluded that mixing was greater. Unlu and Faller (2002) found that the spread of the normalized residence time distribution increased with increasing screw speed and thus concluded that mixing was greater at high speed. We observed similar behavior. For example, screw speeds of 210–290 rpm (all other variables held constant) resulted in nearly equal mean residence times of 7.07 and 7.98 min, but with experimental variances of 4.19 and 7.60, respectively. The same two variances calculated from normalized curves (Levenspiel, 1972) are 0.084 and 0.119.

The theoretical variance, σ_τ^2, predicted by Eq. (3) is $1/J$ or in this case 1/8 (0.125). This matches to a reasonable approximation σ_τ^2 calculated for experimental runs 16 and 19 (depicted in Fig. 6), equal to 0.134 and 0.081, respectively. For Eq. (2), τ_P should equal $1 - \sigma_\tau$, where σ_τ is the dimensionless standard deviation (Nauman, 1985). If $\sigma^2 = 0.125$, then $\sigma_\tau = 0.354$, and τ_P could be expected to be approximately 0.65. For runs 16 and 19, τ_P was 0.59 and 0.68, respectively.

4. Conclusions

By the principle of Occam's Razor, we would be led to choose Eq. (3), since it was found to fit the data as well or better than Eq. (2), but with only one adjustable parameter instead of two. However, Eq. (3) predicts no sharp time of first appearance, i.e., some tracer moves through the system in zero time. In real extrusion-type systems a significant portion of the total residence time may elapse before any tracer emerges (Levine & Miller, 2002). Therefore, Eq. (2) has been more frequently used. The general behavior observed for chocolate mass was similar to starch-based products in a twin-screw extruder despite the difference in physical properties. Mean residence time was inversely proportional to feed rate and directly proportional to screw speed. Gate opening had a slight inverse relationship with mean residence time, and was the only variable that significantly influenced the time of first appearance.

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