米穀擠壓加工—

平均滯留時間及滯留時間分佈範圍模式之推導

Extrusion Cooking of Rice Flour—

Mean Residence Time and RTD Spread Modeling

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摘 要

比色法被用來測定米穀粉在雙軸食品擠壓機（APV Baker MPF 50/25）內之滯留時間分佈情形，本研究在實驗上之變數爲螺絲元件之排列組合，進料速率，及螺軸轉速。統計學上 3×3×3階乘實驗應用於此以分析滯留時間之分佈。

由實驗結果得知，實驗設計上之變數對平均滯留時間均有顯著之影響。統計軟體上之後退式漸進方法在本研究上用來推導及預估平均滯留時間及滯留時間分佈範圍之廻歸模式。結果顯示，一個二階之廻歸模式可滿意地預估平均滯留時間，另一個三階之廻歸模式亦可以滿意地預估滯留時間分佈範圍。

關鍵詞：比色法，滯留時間分佈，雙軸擠壓機，米穀粉

ABSTRACT

A colorimetric method was used to study the residence time distribution (RTD) in an APV Baker MPF 50/25 twin-screw extruder using rice flour as the feed material. The process variables were screw profile, feed rate, and screw speed. A 3x3x3 factorial experiment was designed to investigate the mean residence time and the RTD spread.

The effects of process variables on the mean residence time were significant. A Stepwise backward procedure in SAS was conducted to develop the regression models for the prediction of the mean residence time and RTD spread. A second order and a third order regression models were derived to satisfactorily predict the mean residence time and the RTD spread.

Keywords: Colorimetric method, residence time distribution, twin-screw extruder, rice flour.

INTRODUCTION

Extrusion technology has been used in plastic, food, rubber, metal; carbon and ceramic industries (Fellows, 1988; Harper, 1981; Mercier et al., 1989; Valentas et al., 1991). Single-screw cooking extruders were developed in the 1940's. The use of twin-screw extruders for food processing started in the 1970's, and the number of applications have been expanding in the 1980's (Mercier et al., 1989). Twin-screw extruders have better mixing, more uniform temperature distribution, better control of residence time, and more positive displacement action than single-screw extruders (Harper, 1981; Lin and Armstrong, 1988).

The most important factors affecting the extrusion systems are the operating conditions of the extruder and the rheological properties of the food (Fellows, 1988). The residence time distribution (RTD) of the feed material in an extruder is one of the most important parameters characterizing mixing and chemical kinetics that occur in extruders. It is also a useful tool in determining the optimal operation conditions for blending, dispersing, and polymerization applications (Bruin et al., 1978; Lin and Armstrong, 1988). Todd (1975a and b) stated that RTD data were most useful in diagnosing axial mixing phenomena in twin-screw extruders, providing the basis for scale up and guidance in improvements of equipment. Hsieh (1990) also described many applications of extruder RTD such as nutrient degradation, food safety, and product quality control.

RTD of the material in an extruder can be determined by using radioactive-tracer and dye-tracer techniques (Harper, 1981). A colorimeter is usually used in the dye-tracer technique because of its simplicity, accuracy, safety, and the least cost (Altomare and Anelich, 1988; Altomare and Ghossi, 1986; Badding, 1990; Likimani, et al., 1990; Onwulata, 1991; Peng, 1991; Peng et al., 1990, 1991a, and b).

Feed composition, feed moisture, feed rate, screw speed, screw profile, barrel temperature, and die size are the important operating variables for twin-screw extruders. Many of these variables have been shown to be important factors affecting the RTD in the extrusion systems (Altomare and Anelich, 1988; Altomare and Ghossi, 1986; Bruin et al., 1978; Davidson et al., 1983; Fellows, 1989; Kao and Allison, 1984; Lin and Armstrong, 1988; van Zuilichem and Stolp, 1983; van Zuilichem et al., 1973, 1975, 1988a, b and c, Wolf and White, 1976; Wolf et al., 1986). Of particular interest are the screw profile, feed rate, and screw speed.

The residence time distribution is useful for the scale up and the optimization of process conditions. It affects the food safety and the product quality (Hsieh, 1990). Therefore, the mean residence time is a very important and useful index for extrusion cooking system, specially for industries. If the mean residence time can be satisfactorily predicted, the prediction will provide the important information for the better quality control of extruded products. Thus, it is important to predict the mean residence time in the extrusion cooking system.

The objectives of this study were to develop regression models to predict the mean residence time and RTD spread for twin-screw extrusion of rice flour.

RESIDENCE TIME DISTRIBUTIONS

The residence time distributions of the material in the extruder are usually described as E curve and F curve (Altomare and Anelich, 1988; Lin and Armstrong, 1988). The E curve gives the exit age distribution and was plotted as normalized concentration E(t) vs. residence time (t). The cumulative E(t), i.e. F(t), vs. normalized time (residence time/mean residence time) was plotted as F curve.

$$E(t) = \frac{c}{\int_0^\infty c\,dt} \cong \frac{c}{\sum_0^\infty c\Delta t} \qquad (1)$$

$$F(t) = \int_0^t E(t)\,dt \cong \frac{\sum_0^t c\Delta t}{\sum_0^\infty c\Delta t} \qquad (2)$$

where c is concentration and t is time.

The mean residence time ($\bar{t}$), which represented the mean time of the material spent in the extruder, was given by Smith (1981):

$$\bar{t} = \int_0^\infty tE(t)\,dt = \frac{\sum_0^\infty tc\Delta t}{\sum_0^\infty c\Delta t} \tag{3}$$

The variance (σ^2) which represented the square of the spread of the distributions was given by Levenspiel (1972):

$$\sigma^2 \cong \frac{\sum_0^\infty (t-\bar{t})^2 c\Delta t}{\sum_0^\infty c\Delta t} = \frac{\sum_0^\infty t^2 c\Delta t}{\sum_0^\infty c\Delta t} - \bar{t}^2 \tag{4}$$

MATERIALS AND METHODS

Materials

Rice flour (RL-100, RIVLAND, Stuttgart, AK) with moisture content of 20.0% (wet basis) was used as the feed material. Red dye (FD&C, #40, Warner Jenkinson, St. Louis, MO) was chosen as a tracer to measure the residence time distributions of the rice flour in a twin-screw extruder.

Extruder

A co-rotating and intermeshing APV Baker MPF50/25 twin-screw extruder (APV Baker Inc., Grand Rapids, MI), as shown in Figure 1, was used in this study. The power of this machine is 28.0 KW (kilowatt) and the total length: diameter ratio is 25:1. The barrel diameter is 50 mm. There are nine temperature controlled barrel sections, and six sections were used (Hsieh et al. 1990). The barrel temperature was set at 26.7°C (feeding zone), 51.7°C, 93.3°C, 121.1°C, and 121.1°C (80, 125, 200, 250, and 250°F) respectively throughout the experiments. The screw profile used in this study was shown in Figure 2. The maximum screw speed of this machine was 500 rpm. The die size was 3.175 mm (1/8 in.). Rice flour was fed into the extruder with a K-tron Type T-35 twin-screw volumetric feeder with a Series 6300 controller (K-tron Corp., Pitman, NJ). The adjustable cutter with four blades was operated at 325 rpm. A computer data acquisition system was used to record feed rate, barrel and product temperatures, die pressure and temperature, % torque, screw speed and cutter speed. It took about 15 minutes for the extruder to reach the steady state after start-up.

Experimental design

The process variables in this study were screw profile (7:5, 9:3, and 11:1, i.e., the number of forward screw paddles to reverse paddles in the extruder metering zone), feed rate (30, 40, and 50 kg/hr) and screw speed (200, 300, and 400 rpm). Two replications were used in this 3x3x3 factorial experiment. For each treatment, 33 samples were collected in 6 minte intervals to determine the RTDs of rice flour in the extruder.

Standard Curve Experiment

A standard curve of this study was established by a separate experiment. Rice flour and red dye were mixed in a HOBART N50 mixer (Hobart Canada Inc., North York, Ontario, Canada) to different concerntrations of 0.0%, 0.01%, 0.025%, 0.05%, 0.075%, 0.1%, 0.15%, 0.2%, 0.4%, and 0.6% (w/w). The mixtures were extruded in a MPF 50/25 twin-screw extruder at 20% moisture (w.b), 40 kg/hr and 300 rpm. As the extruder reached the steady state, a timer was started. Samples for each mixture were collected in one minute intervals from the 4th to 6th minutes. The color values (L, a, and b) of each sample were measured according to the method described below, and the concentrations (g color/100g rice flour) vs. color values were plotted as the standard curve for the experiment.

Collection of Samples and Color Measurement

In the study of the 3x3x3 factorial experiment, samples of each treatment were collected as the extruder reached the steady state. One gram of red dye was suddenly dropped into the extruder as a tracer, and a timer was started at the same time. Meanwhile, the extrudate samples were collected for up to 6 minutes (10 s intervals in the first 30 s, 5 s intervals in he next 60 s, 10 s intervals in the following 90 s, and 20 s intervals in the last 180 s). Therefore, 33 samples were collected for each treatment. Each sample was ground by a Waring Blender (New Hartford, CT) and passed the Taylor standard screen (No. 8).

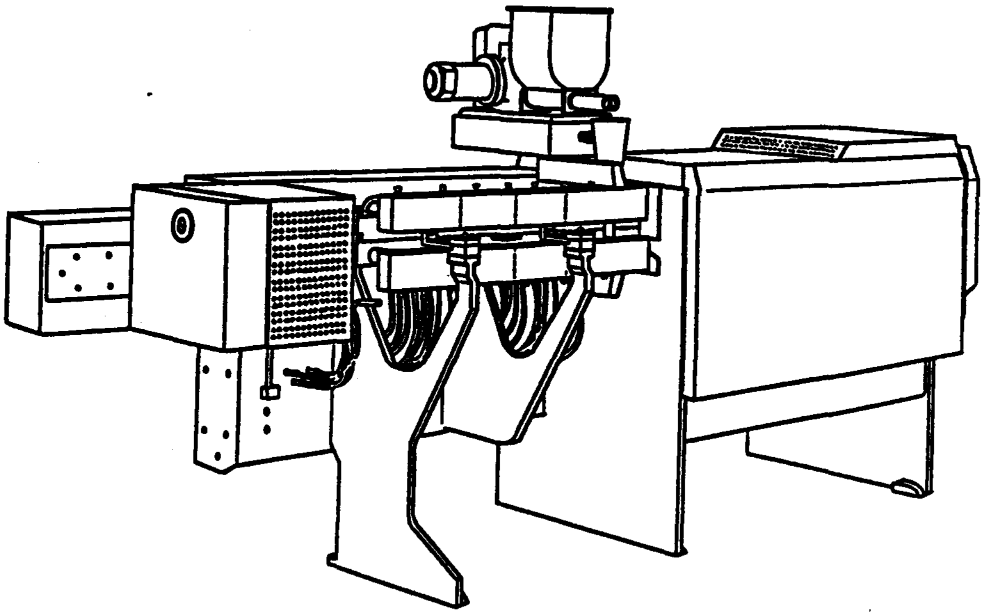

Fig 1. The APV Baker MPF 50/25 twin-screw extruder (Source: Hsieh and Hu, 1990. Agricultural Engineering Department, University of Missouri-Columbia, Columbia, MO)

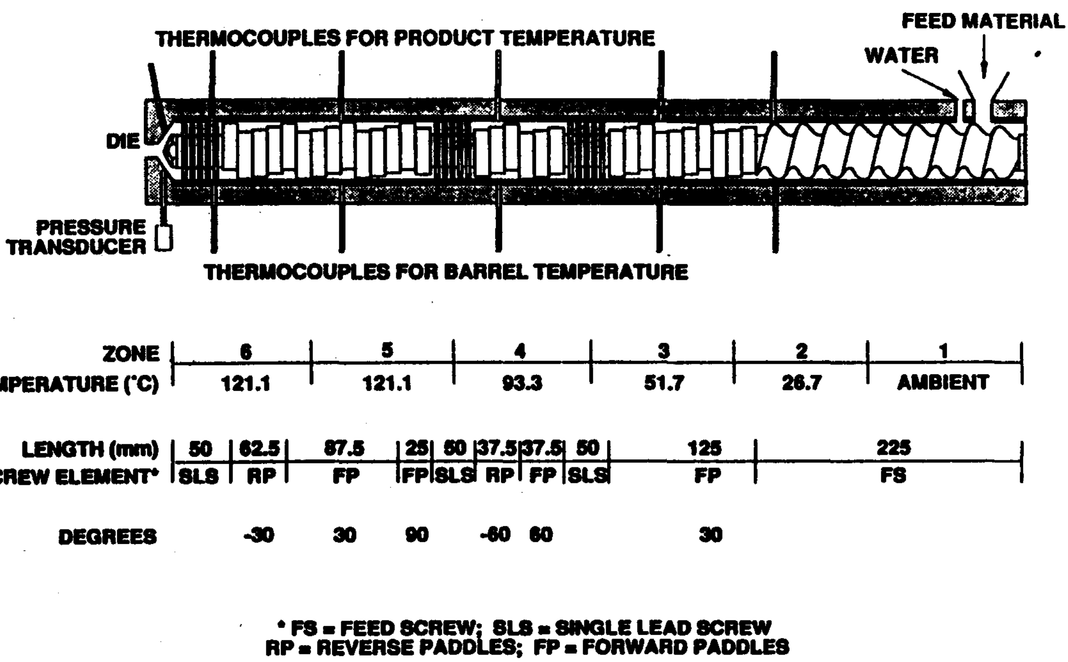

Fig 2. The screw profile of the APV Baker MPF 50/25 twin-screw extruder.

The color values (L, a, and b) for each ground sample were measured and recorded using a HunterLab D25 colorimeter (Hunter Associates Lab., Inc., Reston, VA). A white title (standard No. C2-28656) with values of L= 91.2, a= –0.9, and b= –0.7 was used to standardize the colorimeter. For each sample, two petri dishes with ground extrudates were meaured, and two color readings (L, a and b) were recorded for each dish. The second reading was obtained after a 90° rotation of the first reading. Therefore, four readings of color values L, a and b, respectively, were recorded for each sample.

Data analysis

A Fortran 77 program was written to convert the color values to concentrations. The LOTUS 1-2-3 computer software (Lotus Development Corporation, MA, 1986) was used to calculate the mean residence time ($\bar{t}$) and the spread of the residence time distribution (σ) for each treatment in the 3x3x3 factorial experiment. The regression models of the mean residence time and RTD spread were derived using Statistical Analysis System (SAS, Release 6.03, Cary, NC, 1989).

RESULTS AND DISCUSSION

Mean Residence Time

The mean residence time ($\bar{t}$) of each treatment of the 3x3x3 factorial experiment was shown in Table 1. A General Linear Model (GLM) procedure was conducted with SAS to analyze the significant differences of the mean residence time among screw profile, feed rate, and screw speed.

Increasing the feed rate from 30 kg/hr to 50 kg/hr, increasing the screw speed from 200 rpm to 400 rpm, and increasing the number of forward screw paddles in the extruder metering zone from 7 to 11 decreased the mean residence time ($p<0.0001$). All the extrusion process variables (screw profile, feed rate, and screw speed) significantly affected the mean residence time at the 5% level. The results were similar to those obtained by Altomaare and Anelich (1988), Altomare and Ghossi (1986), Kao and Allison (1984), Lin and Armstrong (1988), and Todd (1975a and b).

The shortest mean residence time in this 3x3x3 factorial experiment was 37.62 s at 11:1 screw profile, 50 kg/hr feed rate, and 400 rpm screw speed. However, the longest mean residence time 84.0 s at screw profile 9:3, feed rate 30 kg/hr, and screw speed 200 rpm.

Prediction of the Mean Residence Time

Since the residence time distribution is useful for the scale up and the optimization of process conditions, it affects the food safety and the product quality (Hsieh, 1990). Therefore, the mean residence time is a very important and useful index for extrusion cooking system, especially for industries. Thus, it is important to pedict the mean residence time in this study. In order to predict the mean residence time, the following regression function should be determined:

$$\bar{t} = f(SP,\ FR,\ SS) \qquad (5)$$

where $\bar{t}$: Mean residence time

SP: Screw profile

FR: Feed rate

SS: Screw speed

A first order Genral Linear Model (GLM) procedure was conducted in SAS to determine the regression equation. The R^2 was 0.91. In order to get a better prediction, the second order GLM procedure was used. The R^2 was improved to 0.98. However, some terms of the second order equation were not significantly different ($p>0.05$). For the prediction regression equation, the fewer the terms and the higher the R^2, the better it is. Therefore, a Stepwise SAS procedure was used to remove the insignificant terms. Three kinds of Stepwise procedures (forward, backward, and maximum R^2) are used by some investigators (Bhattacharya and Hanna, 1986 and 1988; Kumar et al., 1989; Lin, 1991). The backward approach is the best and only suitable procedure in this study, because it removes the least significant term step by step till all the remaining terms are significant at 5% level. In this study, the "step 0" of the backward procedure listed all 10 terms for the second order

Table 1. The mean residence times (s) of the 3x3x3 factorial design

Screw Profile	Feed rate (kg/hr)		Screw speed (rpm)		
			200	300	400
7:5	30	Rep1	83.49	75.33	68.22
		Rep2	80.61	70.98	70.19
		Ave	82.05±1.44	73.15±2.18	69.21±0.99
	40	Rep1	65.56	60.18	55.41
		Rep2	65.93	55.89	52.55
		Ave	65.75±0.19	58.03±2.15	53.98±1.43
	50	Rep1	56.63	50.30	46.17
		Rep2	58.57	48.05	45.77
		Ave	57.60±0.97	49.17±1.13	45.97±0.20
9:3	30	Rep1	84.71	72.19	64.50
		Rep2	83.30	70.19	65.39
		Ave	84.00±0.71	71.19±1.00	64.95±0.45
	40	Rep1	64.36	51.65	47.76
		Rep2	60.66	50.06	47.26
		Ave	62.51±1.85	50.86±0.80	47.51±0.25
	50	Rep1	55.03	45.30	41.42
		Rep2	52.32	43.17	37.79
		Ave	53.68±1.36	44.24±1.07	39.61±1.82
11:1	30	Rep1	73.11	60.45	51.31
		Rep2	75.99	59.46	55.62
		Ave	74.55±1.44	59.96±0.50	53.47±2.16
	40	Rep1	59.25	46.95	42.69
		Rep2	59.44	46.20	41.41
		Ave	59.35±0.10	46.58±0.38	42.05±0.64
	50	Rep1	56.27	42.65	37.89
		Rep2	56.57	43.41	37.34
		Ave	56.42±0.15	43.03±0.38	37.62±0.28

Ave = (Rep1 + Rep2)/2

regression equation. The "step 1" removed the least significant term ($p>0.05$). The "step 2" removed the least significant term after step 1. The "step 3" removed the least significant term after step 2. The procedure continued till all the remaining terms were significant ($p<0.05$), then it stopped automatically. Therefore, the regression model (equation) obtained from this procedure was acceptable for prediction. The second order Stepwise backward procedure was conducted to determine the regression model of mean residence time. The R^2 was 0.97 at the 5% significance level. The parameters and estimates for the second order regression model are shown in Table 2.

Table 2. The parameters and estimates for the second order regression model of the mean residence time

Parameter	Estimate
Intercept	241.00293907
FR	–5.75295352
SS	–0.19168401
SPxFR	0.08472632
SPxSS	–0.00886011
SPxSP	–0.16685230
FRxFR	0.04813056
SSxSS	0.00032131

SP: Screw profile (7, 9, and 11), i.e., the number of forward screw paddles in the extruder metering zone

FR: Feed rate (30, 40, and 50 kg/hr)

SS: Screw speed (200, 300, and 400 rpm)

$R^2 = 0.97$

The second order regression model shown in Table 2 can be used to predict the mean residence time satisfactorily for rice flour in the APV Baker MPF 50/25 twin-screw extruder in the range of the variables studied. This method can also be applied to other experiments for the mean residence time prediction.

The Spread of the Residence Time Distribution

The residence time distribution spread (σ) of each treatment in the 3x3x3 factorial experiment is shown in Table 3. An GLM procedure was conducted with SAS to analyze the significant differences of the distribution spread among screw profile, feed rate, and screw speed.

The knowledge of the spread of the residence time distribution (RTD spread) in an extruder is important, however, it has been seldom studied. The RTD spread indicates how long a certain part of the material is exposed to a certain heat load and shear (Janssen, 1978; Jannssen et al., 1979). Olkku et al. (1980) showed that a reverse mixing element near the end of the machine caused a significant spread in the residence time distribution.

In this study, increasing the feed rate decreased the spread of the residence time distribution. There was no significant difference in RTD spread between 40 kg/hr and 50 kg/hr, but significant differences did occur from 30 kg/hr to 40 kg/hr and 30 kg/hr to 50 kg/hr ($p<0.0001$).

The screw speed had results similar to the feed rate, but the RTD spread at 300 rpm was slightly lower than at 400 rpm.

For the screw profile, when increasing the number of the forward paddles in the extruder metering zone from 7 to 11, no significant difference occurred between 7 and 9 forward paddles, however, significant differences did occur between 7 and 11 or 9 and 11 paddles ($p<0.0001$). Increasing the number of forward screw paddles from 7 to 9 slightly decreased the spread of the residence time distribution (not significant). Based on the SAS results, feed rate was the most important factor affecting the spread of the residence time distribution among all three variables studied; screw profile was the least important factor. The results were in agreement with Janssen (1978), van Zuilichem et. al. (1988a, b, and c), and Olkku et al. (1980). For screw profiles 9:3 and 11:1, the longest RTD spread occurred at the lowest feed rate and screw speed (30 kg/hr and 200 rpm), and the shortest was obtained at the highest feed rate and screw speed (50 kg/hr and 400 rpm).

The shortest spread of the residence time

Table 3. The spreads of the residence time distribution (s) of the 3x3x3 factorial design

Screw profile	Feed rate (kg/hr)		Screw speed (rpm) 200	300	400
7:5	30	Rep1	26.52	19.48	17.19
		Rep2	22.57	20.04	28.24
		Ave	24.55±1.98	19.76±0.28	22.71±5.53
	40	Rep1	14.24	19.58	17.76
		Rep2	14.01	13.61	15.56
		Ave	14.12±0.12	16.60±2.99	16.66±1.10
	50	Rep1	11.83	14.84	15.07
		Rep2	13.48	14.31	18.87
		Ave	12.65±0.83	14.58±0.27	16.97±1.90
9:3	30	Rep1	28.33	23.45	22.37
		Rep2	24.99	23.11	21.42
		Ave	26.66±1.67	23.28±0.17	21.90±0.48
	40	Rep1	14.86	11.83	11.26
		Rep2	15.10	12.31	18.19
		Ave	14.98±0.12	12.07±0.24	14.73±3.47
	50	Rep1	12.08	12.18	11.05
		Rep2	11.54	9.37	8.73
		Ave	11.81±0.27	10.78±1.41	9.89±1.16
11:1	30	Rep1	20.12	16.46	13.20
		Rep2	24.95	15.19	11.20
		Ave	22.54±2.42	15.83±0.64	12.20±1.00
	40	Rep1	13.96	9.47	11.32
		Rep2	13.71	10.12	9.49
		Ave	13.84±0.13	9.80±0.33	10.41±0.92
	50	Rep1	11.69	8.97	9.03
		Rep2	14.62	9.57	10.82
		Ave	13.16±1.47	9.27±0.30	9.93±0.90

Ave = (Rep1 + Rep2)/2

distribution in this 3x3x3 factorial experiment was 9.27 s at 11:1 screw profile, 50 kg/hr feed rate, and 300 rpm screw speed. However, the longest spread of the residence time distribution was 26.66 s at screw profile 9:3, feed rate 30 kg/hr, and screw speed 200 rpm.

Prediction of the Residence Time Distribution Spread

Te procedure used to predict the spread of the residence time distribution was the same as the mean residence time. Also, the following regression function needed to be determined.

$$\sigma = f(SP, FR, SS) \qquad (6)$$

where σ: Spread of the residence time distribution

SP: Screw profile

FR: Feed rate

SS: Screw speed

First, a first order GLM procedure was conducted in SAS to find out the suitable regression model. The R^2 was 0.63. Then, a second order GLM procedure was used, and the R^2 was 0.81. However, some terms of the second order model were not significant ($p>0.05$). Thus, the Stepwise procedure was introduced, and the R^2 reduced to 0.78. In order to obtain the higher R^2, the third order GLM procedure was conducted in SAS. The R^2 was improved to 0.88. Then, the third order Stepwise backward procedure was used to remove the insignificant terms. The R^2 was 0.87 at the significance level of 5%. The parameters and estimates for the third order regression model using the Stepwise backward procedure is shown in Table 4.

The third order regression model shown in Table 4 can be used to predict the spread of the residence time distribution for rice flour in the APV Baker MPF 50/25 twin-screw extruder in the range of the variables studied.

Table 4. The parameters and estimates for the third order regression model of the spread of the residence time distribution

Parameter	Estimate
Intercept	−122.77520749
FR	63.84973647
SS	−0.16899879
SPxFR	−1.41856340
SPxSS	−0.01893726
SPxSP	−3.42549357
FRxFR	0.06546187
SPxSPxFR	0.07986477
FRxFRxSS	−0.00012229
SSxSSxSP	0.00001627

SP: Screw profile (7, 9, and 11), i.e., the number of forward screw paddles in the extruder metering zone

FR: Feed rate (30, 40, and 50 kg/hr)

SS: Screw speed (200, 300, and 400 rpm)

$R^2 = 0.87$

CONCLUSION

A colorimetric method was used to study the residence time distribution (RTD) in an APV Baker MPF 50/25 twin-screw extruder using rice flour as the feed material. The process variables were screw profile, feed rate, and screw speed. A 3x3x3 factorial experiment was designed to investigate the mean residence time and the RTD spread.

The effects of process variables on the mean residence time were significant. A stepwise backward procedure in SAS was conducted to develop the regression models for the prediction of the mean residence time and RTD spread. The second order and third order regression models developed in this study can be used to predict the mean resdence time and RTD spread satisfactorily for rice flour in the APV Baker MPF 50/25 twin-screw extruder in the range of the variables studied. This method can also be applied to other experiments for the mean residence time and RTD spread prediction.

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