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EXTRUSION OF AN EFFERVESCENT GRANULATION WITH A TWIN SCREW EXTRUDER, BAKER PERKINS MPF 50D. DETERMINATION OF MEAN RESIDENCE TIME.

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ABSTRACT

Anhydrous citric acid and sodium bicarbonate were granulated with ethanol in an extruder.

A rough estimate of the dwell time was obtained by adding a small amount of colour in the inlet port and measuring the time until it reached the outlet. The mean residence time was calculated from results obtained when analyzing the colour content of the continuously sampled granulations after drying.

There was a significant relationship between dwell time and mean residence time. After elimination of the abnormal values of three of the factor combinations, the degree of significance for the slope of the line increased from P=0.003(**) to P=0.0001(***).

The mean residence time was significantly influenced by the process variables screw speed and powder flow rate. Higher levels of these two factors reduced the mean residence time.

INTRODUCTION

A report regarding the extrusion of an effervescent granulation with a twin screw extruder was recently presented.[1] Instead of measuring residence-time distribution, which is laborious, a simpler test was used, which gave a rough estimate of the dwell time. However, from residence-time-distribution functions - widely used in the field of chemical engineering - one can learn about the degree of mixing and life expectancy of the material in the extruder[2]. The response of the extruder to the pulse of a tracer at the inlet is given by a function representing the age distribution of the granulation in the extruder. The mean residence time, $\bar{t}$, is given by:

$$\bar{t} = \frac{\sum_{0}^{\infty} t\,C(t)\Delta t}{\sum_{0}^{\infty} C(t)\Delta t} \qquad \text{Eq 1}$$

where C(t) is the tracer concentration at time t.

Now the earlier report is completed by those mean residence times that were obtained when the medium-intensity screw configuration was employed.

MATERIALS AND METHODS

Approximately 1 g of patent blue[3] was added in the inlet port and the extrudate was collected at the outlet during 5-15 s intervals for 5 min. The samples were dried at 60°C and passed through a 1.0 mm screen.

An appropriate amount of the dried sample was weighed and dissolved in 0.1 M acetate buffer of pH = 5. The liberation of the carbon dioxide was accelerated in an ultrasonic bath. Then the absorbance of the solution was measured at 640 nm in a photometer, and the concentration of patent blue in the sample was calculated.

The mean residence time was calculated according to Wolf et al [2]. No replicates were made of $\bar{t}$.

It was not possible to note the dwell time while performing the sampling. The dwell-time measurements were made in connection with the previously reported tests[1], where replicates were made of some of the factor combinations. However, the tests employed in measuring $\bar{t}$ were performed in another experiment where the same factor levels were used.

The influence of the process variables on mean residence time was tested by analysis of variance with all interaction terms included. As no replicates were made of $\bar{t}$, the variance was estimated from the three- and four-factor interaction terms. Besides, the interdependence of the response variables was tested by means of stepwise regression. The other response variables, together with all the process variables (including their interactions), were used as independent variables. Besides, the correlation coefficient and the linear regression function between dwell time and $\bar{t}$ were calculated.

RESULTS AND DISCUSSION

Statistical Analysis

When studying the influence of many factors, there is always a risk that factors without effect on the response still happen to become significant. To avoid this source of error regarding the analysis of variance, effects were considered significant only at $P < 0.01$.

The estimated variance, s^2, obtained from the three- and four-factor interaction terms in the analysis of variance was 5.3.

Mean Residence Time

The results of the mean-residence-time measurements are summarized in Table 1.

Screw speed and powder flow rate significantly influenced $\bar{t}$; see Table 2. There were no significant interactions.

TABLE 1

Mean Residence Time

Factor combination				Mean residence
P	E	S	D	time, s
-1	-1	-1	-1	35.6
1	-1	-1	-1	29.4
-1	1	-1	-1	39.1
1	1	-1	-1	32.0
-1	-1	1	-1	28.0
1	-1	1	-1	22.9
-1	1	1	-1	31.2
1	1	1	-1	17.1
-1	-1	-1	1	35.9
1	-1	-1	1	32.9
-1	1	-1	1	42.2
1	1	-1	1	34.0
-1	-1	1	1	24.0
1	-1	1	1	26.3
-1	1	1	1	32.7
1	1	1	1	25.4

P = powder flow rate

E = ethanol concentration

S = screw speed

D = die plate

-1 is the low level, and 1 is the high factor level

TABLE 2

Mean Residence Time. Analysis of Variance, Significant Factors.

Significant effects	Level of significance
Screw speed (S)	$P < 0.001$
Powder flow rate (P)	$0.001 < P \leq 0.01$

TABLE 3

Mean Residence Time. Regression Model with Significant Process Variables and Standard Deviation (s)

Model	s
$\bar{t} = 30.5 - 4.59S - 3.04P$	2.30

S and P take the values -1 and +1.

The magnitude of the significant coefficients in Table 3 will directly indicate the influence of the process variables, and the sign in front of the coefficients indicates whether the response is increasing or decreasing. High levels of both screw speed and powder flow rate reduced $\bar{t}$.

Stepwise regression on all process variables and all response variables resulted in the same model as the one presented above.

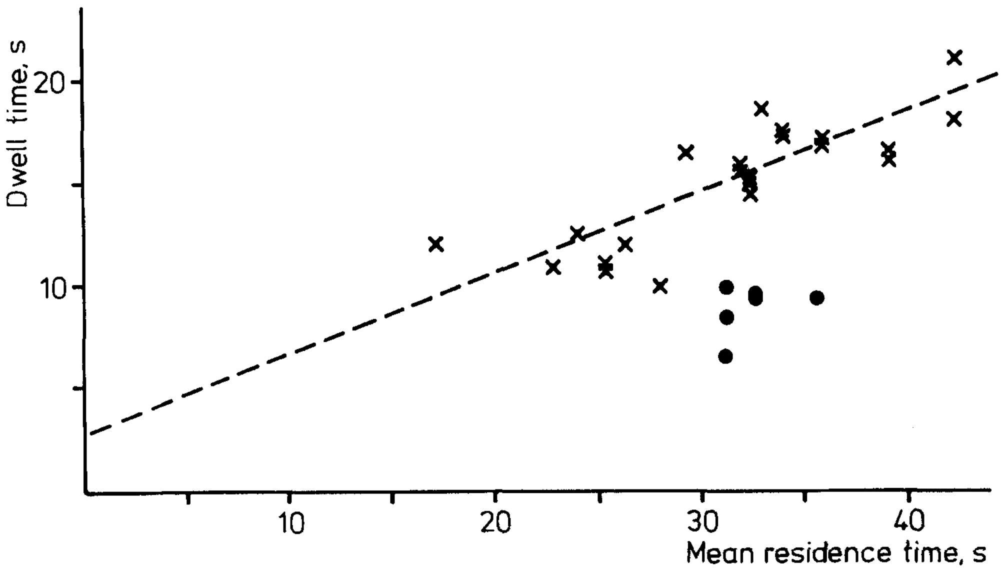

FIGURE 1

Dwell time, s, (y-axis) versus mean residence time, s, (x-axis). Abnormal dwell-time values are marked with circles. The regression line refers to data excluding the abnormal values.

TABLE 4

Linear Regression between Dwell Time (TIME) and Mean Residence Time ($\bar{t}$), both expressed in seconds.

Model	Level of significance for slope of line
TIME = 2.56 + 0.35 $\bar{t}$	$0.001 < P \leq 0.01$

Thus, there was no significant influence from the other response variables.

Dwell time was plotted versus mean residence time; see Fig. 1. All values are plotted in this figure. Regarding the values of three of the factor combinations, marked with circles, the dwell time was obviously abnormal. The regression line refers to data excluding the abnormal values. From the linear regression analysis a model of the dependence between the two factors was obtained; see Table 4.

The correlation coefficient between dwell time and mean residence time was 0.54 at a level of significance of P = 0.0029 (**) regarding all data, and 0.82 with P = 0.0001 (***) excluding the abnormal values of three of the factor combinations. Consequently, there was a significant correlation between dwell time and $\bar{t}$.

CONCLUSIONS

Mean residence times were determined respecting the screw configuration of the medium-agitation type. They varied between approximately 17 and 45 s depending on the factor combination.

There was a significant relationship between dwell time and mean residence time.

The process variables screw speed and powder flow rate significantly influenced mean residence time. A reduction of the response was obtained by increasing the screw speed and/or the powder flow rate.

REFERENCES

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