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EXTRUSION OF AN EFFERVESCENT GRANULATION WITH A TWIN SCREW EXTRUDER, BAKER PERKINS MPF 50 D. INFLUENCE ON INTRAGRANULAR POROSITY AND LIQUID SATURATION.

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ABSTRACT

Anhydrous citric acid and sodium bicarbonate were granulated with ethanol in an extruder. The influence of the following process variables on intragranular porosity and liquid saturation was investigated: powder flow rate, ethanol concentration, screw speed, die plate and screw configuration.

Granule porosity was significantly influenced by screw configuration and ethanol concentration. Besides, the porosity was also correlated with response variables that affected the decomposition of sodium bicarbonate. A more intense screw configuration

enhanced the formation of carbon dioxide due to a temperature rise and consequently increased porosity. On the other hand, a higher ethanol concentration reduced the porosity.

There was a strong correlation between intragranular porosity and liquid saturation.

INTRODUCTION

A report regarding the extrusion of an effervescent granulation with a twin screw extruder was recently presented.[1] In this report, there was no presentation of porosity or liquid-saturation data. These variables are important in connection with granulation in high shear mixers[2,3]. Thus, both these factors will probably also be influenced in the process conditions when preparing the granulation by means of a continuous process in an extruder. Therefore, the earlier reponse variables - which were load of drive motor (LOAD); temperature of the extruded mass (TEMP); dwell time (TIME); output; granule fraction above 1.00 mm and below 0.125 mm of dried and unmilled granulation; geometric mean diameter by weight (MDGW) and carbon dioxide content (CO2) of dried and milled granulation - are now complemented by the further variables intragranular porosity, POR, and liquid saturation, SAT.

METHODS

The granule density, ρA, of 0.710-1.00 mm dried granules was measured by a pycnometric method where mercury was used as the penetration liquid according to Jaegerskou et al.[3]. Calculation of the porosity was performed according to:

$$\text{POR or } \varepsilon = \left(1-\frac{\rho A}{\rho T}\right)\cdot 100 \qquad \text{Eq 1}$$

where ρT is the density of the granules, measured with the aid of the Beckman pycnometer[4]. The porosity will be designed as POR in the text.

The liquid saturation of the granules was calculated according to Kristensen et al.[2].

$$\text{SAT or } S = \frac{H(1-\varepsilon)\rho T}{\varepsilon} \qquad \text{Eq 2}$$

where H is the percentage (v/w) of ethanol on a dry-weight basis and S stands for the liquid saturation. To avoid confusion with the process variable screw speed, liquid saturation will be indicated as SAT in the following text. The ethanol content was calculated from the flow rates of ethanol and powder.

The influence of the following process variables was investigated: powder flow rate (P); ethanol flow rate, which was expressed as ethanol concentration (E); screw speed (S); die plate (D); and screw configuration (C).

The influence of the process variables on each of the response variables was tested by analysis of variance and stepwise regression with all interaction terms included. Correlation coefficients between the response variables 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 be significant. To decrease this source of error, effects in the analysis of variance models were only considered significant at $P<0.01$.

It should be noted that even if there is a significant correlation between two variables, there need not be a causal relationship. The correlation may be caused by indirect effects.

The centre points of Table 1 were only used for estimation of the variance.

Extrusion Tests

The results of the experiments are summarized in Table 1, where the low-intensity screw configuration is marked -1 and the medium-intensity agitation is marked 1. Replicates of a factor combination are listed in the order that they occur for that particular combination.

Regarding the medium-agitation configuration, C=1, there was a reasonable variation within the replicates both in respect of porosity and liquid saturation. As for the low-intensity screw configuration, C=-1, the variation within the replicates of porosity was sometimes rather large. Of course this variation was also reflected in the liquid-saturation data. SAT-values larger than 100% are theoretically incorrect. It should be noted that the porosity values obtained by this pycnometric method are underestimated when the intragranular pores exceed about 20 μm. However, the introduced error is systematic, and the porosity data are therefore comparable. The high liquid-saturation values of the low-intensity screw configuration are explained by the underestimated porosity values. When comparing the impact of different factors on the effervescent granulation, this error is of less importance.

Porosity

The intragranular porosity was significantly influenced by screw configuration and ethanol concentration. There were some interactions, too; see Table 2.

The magnitude of the significant coefficients in Table 3 will directly indicate the degree of influence of the process variables. These assume the values ±1, and the sign in front of the coefficients indicates whether the response is increasing or decreasing.

TABLE 1

Intragranular Porosity and Liquid Saturation of the Different Factor Combinations

Factor levels					POR	SAT
P	E	S	D	C	%	%
-1	-1	-1	-1	-1	6.2	180
1	-1	-1	-1	-1	5.7	197
-1	1	-1	-1	-1	9.1	184
1	1	-1	-1	-1	11.1	147
-1	-1	1	-1	-1	9.7	111
1	-1	1	-1	-1	8.9	122
-1	1	1	-1	-1	11.6; 8.9	140; 179
1	1	1	-1	-1	9.4	177
-1	-1	-1	1	-1	10.2; 12.5	105; 79
1	-1	-1	1	-1	11.0	96
-1	1	-1	1	-1	9.7; 12.6; 13.0	171; 121; 117
1	1	-1	1	-1	9.4; 11.2; 9.4	177; 137; 167
-1	-1	1	1	-1	12.0	87
1	-1	1	1	-1	13.3; 12.5; 10.4	78; 78; 96
-1	1	1	1	-1	8.2;10.3;8.8;10.2	206; 167; 181; 138
1	1	1	1	-1	10.2; 6.7; 16.5	162; 241; 87
0	0	0	-1	-1	7.1; 10.8	198; 125
0	0	0	1	-1	12.1;9.0;8.5;10.2	110; 153; 162; 133
-1	-1	-1	-1	1	17.4	59
1	-1	-1	-1	1	21.1	46
-1	1	-1	-1	1	18.1; 17.4	87; 91
1	1	-1	-1	1	16.1; 17.3	100; 92
-1	-1	1	-1	1	22.3	43
1	-1	1	-1	1	22.9	42
-1	1	1	-1	1	12.8; 11.0; 10.3	131; 155; 167
1	1	1	-1	1	18.3	86
-1	-1	-1	1	1	14.3	74
1	-1	-1	1	1	20.1	49
-1	1	-1	1	1	20.9; 18.4	73; 85
1	1	-1	1	1	19.8; 17.3	78; 92
-1	-1	1	1	1	21.3	46
1	-1	1	1	1	25.2	37
-1	1	1	1	1	10.9; 11.7	157; 145
1	1	1	1	1	16.0; 12.6	101; 133
0	0	0	-1	1	20.7; 20.7	60; 60
0	0	0	1	1	19.6; 21.4	65; 60

P = powder flow rate
E = ethanol concentration
S = screw speed
D = die plate
C = screw configuration

-1 low level
0 centre point level
+1 high level

TABLE 2

Analysis of Variance. Significant Effects.

Response variable	Significant main effects and interactions $P\leq 0.001$	$0.001<P\leq 0.01$
POR	C, ES, EC	E
SAT	E, C	ES

TABLE 3

Regression Model for Significant ($P\leq 0.01$) Process Variables and Standard Deviation(s)

Model	s
POR = 14.1-0.96E+4.15C-1.44ES-1.32EC	2.03
SAT = 109.0+25.0E-32.2C+13.4ES	30.3

Regarding the main effects, a high level of screw configuration will increase the porosity, while a high level of ethanol concentration will reduce the response. A more intense agitation increased the decomposition of sodium bicarbonate[1]. Consequently, the formation of carbon dioxide may be expected to increase the porosity. An increase of the ethanol concentration will probably enhance the lubrication effect and reduce the viscosity of the mass and consequently reduce the decomposition of sodium bicarbonate. However, the impact of both screw configuration and ethanol concentration is complicated by the interaction terms.

The correlation coefficients between POR and the other response variables indicated a significant relationship with liquid

TABLE 4
The Largest Correlation Coefficients between Response Variables

Response variables	Correlation coefficient				
	SAT	TEMP	LOAD	TIME	CO2
POR	-0.88	0.84	0.81	0.77	-0.66
SAT	1	-0.84	-0.73	-0.61	0.60

saturation; see Table 4. This was expected from Eq 2. A reduction of the porosity will increase SAT, which is obvious from the sign of the coefficient. The carbon dioxide content, which is affected by TEMP, LOAD and TIME[1], was negatively correlated with POR and positively correlated with SAT as expected. A higher LOAD and a longer dwell time will mean an increased temperature with more decomposition of the sodium bicarbonate and consequently increased porosity. All the coefficients in Table 4 are significant at a level of $P<0.001$.

Liquid Saturation

This variable was also significantly affected by ethanol concentration and screw configuration; see Table 2. Besides, there was an interaction involving screw speed.

A high screw-configuration level will reduce SAT due to increased porosity; see Table 3. As was to be expected from Eq 2, a high level of ethanol concentration will increase the liquid saturation.

CONCLUSIONS

Intragranular porosity was significantly affected by screw configuration and ethanol concentration. A more intense screw configuration and a lower ethanol concentration increased the porosity. There were some interactions, too, including ethanol concentration and screw speed.

Mathematical models were fitted and the interdependence of the response variables was tested. Porosity and liquid saturation were strongly correlated; compare with Eq 2.

Likewise both variables were correlated with response variables increasing the decomposition of sodium bicarbonate, e.g. high levels of temperature, motorload and dwell time. Thus a more intense screw configuration increased the formation of carbon dioxide, which increased the porosity. A higher ethanol concentration decreased the temperature, which reduced the decomposition of sodium bicarbonate and thereby affected the porosity.

REFERENCES

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