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# Experiments with an instrumented twin-screw extruder using a single-step granulation / extrusion process

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## Summary

Preliminary studies with an instrumented, co-rotating twin-screw extruder were performed. The variation of water content of the extruded mass is dependent on the accuracy of both dry powder and granulation liquid feed rate. Using a given powder feed rate it is easy to vary the water content by changing the liquid feed rate. Operating the twin-screw extruder in this way the extruder barrel is not completely filled with wet mass. The steady state mass is dependent on both screw speed and mass flow rate. This leads to different behaviour of extrusion process variables compared to extruders with completely filled barrels. With a central composite design the influence of screw speed and water content on passage time, steady state mass and the process variables corrected power consumption, pressure and end temperature of extrudate were studied. Water content is the most important variable for the extrusion process variables. For further studies it is strictly recommended either to control the water content or to use it as a variable.

## Introduction

Extrusion is widely used for the production of pellets. Different types of extruders are used for this process. Generally the equipment for extrusion can be divided into three groups: ram extruders; radial screen and roll extruders; and screw extruders.

The simpliest type of extruder is the ram extruder. It is mainly used as a research instrument (Ovenston and Benbow, 1968; Harrison et al., 1985a,b; Fielden et al., 1989). Owing to its simple construction it is appropriate to study the relationships between process variables and the formulation (Rowe, 1986). Due to its construction it is not suitable for continuous production.

Radial screen extruders and roll extruders are used in the production of pharmaceutical pellets. They permit continuous extrusion of pregranulated powders. Dry powders cannot be used because no mixing takes place inside the extruder. Several authors have reported on this (Bianchini and Vecchio, 1989; Baert et al., 1991; Fielden et al., 1992; Hellen et al., 1992).

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The group of screw extruders can be divided into single-screw and twin-screw extruders. Most single screw extruders are equipped with an axial discharge, while twin-screw extruders are available with both axial and radial discharge of the extrudate. Screw-extruders offer the possibility of wet-mixing and extrusion in a single step although they also work with prewetted masses. Some authors worked with single-screw extruders (Goodhart et al., 1973; Dietrich and Brausse, 1988; Bataille et al., 1991) but the majority used the twin-screw type (Conine and Hadley, 1970; Reynolds, 1970; Jalal et al., 1972; Woodruff and Nuessle, 1972; Malinowski and Smith, 1974; Bodmeier et al., 1986, Lövgren and Lundberg, 1987; Elbers et al., 1992; Funck et al., 1991).

There is only little information available about the one-step granulation/extrusion process in a twin-screw extruder (e.g. Gamlen and Eardley, 1986; Lindberg et al., 1988; Hicks and Freese, 1989). Gamlen and Eardley (1986) reported first experiences with this production method. The short time from wetting to extrusion was described as the most significant advantage of this process. In these experiments the extrudates had very rough surfaces. According to Gamlen and Eardley, this phenomenon was caused by die plates, which were too thin. Lindberg et al. (1988) carried out more detailed investigations on the extrusion step itself. Using a statistical design the influence of powder flow rate, ethanol concentration, screw speed and type of die plate on the dwell time (passage time through the extruder) and the mean residence time was determined. Screw speed and powder flow rate were found to be the significant factors for the variations in mean residence time.

In this study we investigated the influence of screw speed and water content on the extrusion process using a central composite design. Extrusion was carried out on an instrumented twin-screw extruder using a mixture of lactose and microcrystalline cellulose. The predictor variables were screw speed and water content of the extrudate while the response variables were passage time, steady state mass, power consumption, end temperature and pressure of the extrudate at the die plate.

## Materials and Methods

### *Materials*

Lactose (Lactose D 20, Meggle, Wasserburg, Germany) with a specification for particle size of 33% < 200 μ and 98% < 400 μm, microcrystalline cellulose (Avicel PH 101, FMC Corp., Philadelphia, USA), charcoal (art. no. 2186, E. Merck, Darmstadt, Germany) were used as received. Demineralized water was used as the granulation liquid.

Avicel PH 101 has a water content of 4–6%, lactose about 1%. The water contents of the current batches were determined using an infrared balance (Sartorius GmbH, Göttingen, Germany). The water contents of the raw materials were taken into account to obtain the correct batch size of dry powder material.

All investigations were made with a standard mixture of 70% lactose and 30% microcrystalline cellulose (% w/w) calculated as dry substances. The powders were blended in a Turbula blender (Type T 10 B, Bachofen KG, Basel, Switzerland) for 15 min.

### *Equipment*

The extrusion equipment is shown schematically in Fig. 1. The dry powder blend was fed into the extruder (A) by a twin-screw-feeder (B) (Type T 25, K-Tron-Soder, Niederlenz, Switzerland). The granulation liquid was pumped by a membrane dosage pump (Type Gamma 4, ProMinent, Heidelberg, Germany) directly into the extruder barrel via a dosage valve (C).

Extrusion was performed on a laboratory scale, co-rotating, instrumented twin-screw extruder

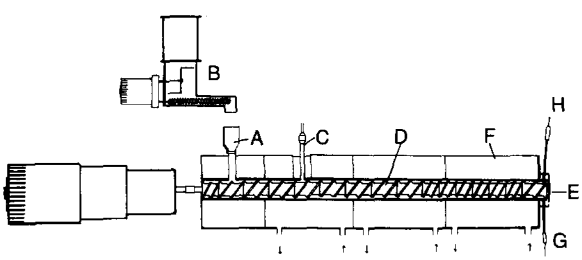


Fig. 1. Sheme of the extrusion equipment (explanation given in the text).

(Berstorff ZE 25 × 18 D, Berstorff GmbH, Hannover, Germany). The extruder is equipped with two screws (D) made of stainless steel with a diameter of 25 mm and a length of 45 cm. The axial mounted die plate (E) is made of 2.5 mm stainless steel and has 48 cylindrical holes of 1 mm diameter each. A side view of the extruder head is shown in Fig. 2. The die plate (E) is mounted directly in front of the screws (D). The gap between screws and die plate is minimized. Water at a temperature of 20°C circulates in the water cooling jacket (F, Fig. 1) of the extruder barrel. Temperature (G) and pressure transducers (H) are mounted in the head of the extruder (Figs 1 and 2). A frontal view of the extruder head (Fig. 3) shows the arrangement of the transducers and the holes (I) in the die plate.

Screw speed, voltage and current of the motor, temperature and pressure of the extrudate at the die plate are continuously monitored by a personal computer. The computer is connected to the extruder by an AD converter (DT 2811, Data Translation, Marlboro, U.S.A.). Using data acquisition sofware (Labtech Notebook, Labtech Inc, Wilmington, U.S.A.) the data are collected and displayed on the monitor. Data are stored into a file every 2 s. The power consumption is calculated by multiplying voltage and current (DC motor). Further calculations are performed with Lotus 123 (Lotus Dev., Berkshire, U.K.). Statistical evaluation of the data was carried out with the SAS Software (SAS 6.03, SAS Institute, Cary, USA).

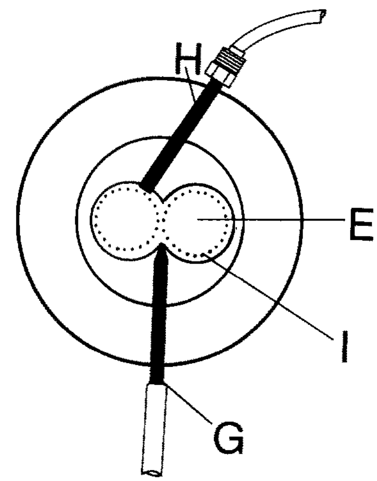

Fig. 3. Front view of the extruder.

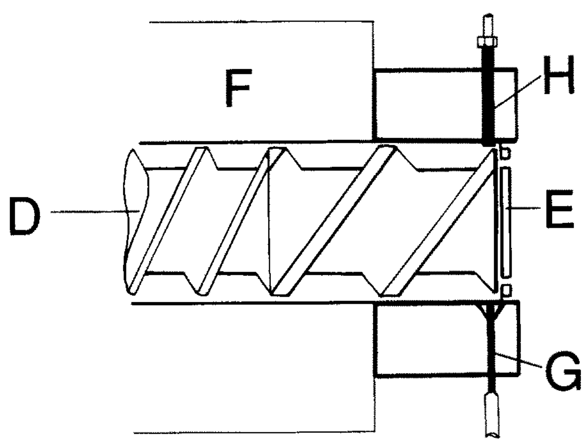

Fig. 2. Detailed side view of the extrusion zone.

A die plate with only 48 holes was chosen to reduce the capacity of the extruder. A small batch size was desired while a certain experimental duration is necessary to reach steady state conditions.

The die plate is directly mounted to the screws (Fig. 2). This minimizes the dead volume between screws and plate. No further densification of the damp mass due to a hydraulic transport is put into the material (Hicks and Freese, 1989). In addition, this configuration reduces the possibility of water movement in the wetted powder as it was noticed in ram extruders (Fielden et al., 1989).

The thickness of the die plate was 2.5 times the hole diameter which results in a length-to-radius ratio ($L/R$) of 5. The plate was thick enough to produce a smooth extrudate in all experiments. A lower $L/R$ ratio may cause surface defects in the extrudate (Harrison et al., 1985b; Gamlen and Eardley, 1986).

*Precision of the powder feeder*

The precision of the powder feeder was evaluated using the dry powder blend. 4 kg was filled into the hopper and the speed of the screws was adjusted to a feed rate of 25 g/min. Every 10 min the feed rate was measured by collecting and weighing the powder discharged during 2 min.

*Calibration of the dosage pump*

The membrane dosage pump was operated at maximum stroke volume. The amount of water pumped through the dosage valve was only dependent on the pump frequency.

Calibration was performed at frequencies of 20, 40, 60, 80, 100 and 120 strokes per min on three different days with three replicates. Water was collected for 2 min and weighted. For each of the 54 measurements the mass per stroke was calculated.

*Power consumption of the empty extruder*

The loss of power consumption during warming up of the machine was measured by running the extruder without screws at a constant speed of 100 rpm for 30 min. All other investigations were performed after warming up for 30 min. The power consumption of the empty extruder was measured with the warm machine at speeds of 40–120 rpm in steps of 10 rpm (three replicates).

*Extrusion procedure*

The powder feed rate was adjusted to a constant rate of 25 g/min calculated as dry powder resulting in approx. 26 g/min powder blend. The maximum capacity to draw powder into the extruder results from the screw speed and the bulk density of the powder. Even at the lowest screw speed of 42 rpm the powder was easily drawn into the barrel. Under these conditions the volume of the extruder barrel is not filled completely.

To reach the desired total water content of the extrudate the required water pump rate was calculated and corrected for the water content of the powder blend. The pump frequency was adjusted to this calculated value using the determined calibration factor.

Extrusion was carried out after warming up the motor. Powder feeder, water pump, extruder and data acquisition were started simultaneously. The experimental run started when the power consumption and the pressure of the extrudate had reached a constant level (Figs 4 and 5). The

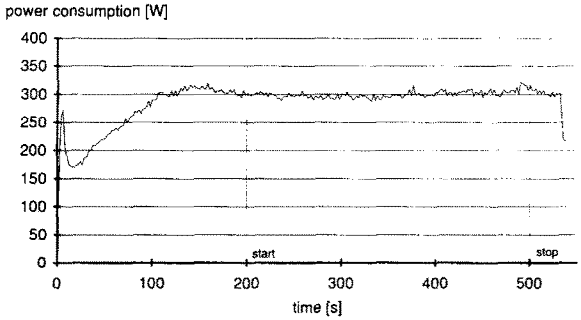


Fig. 4. Power consumption-time profile for run 17.

lag time was about 200 s. Now extrusion was carried out for 300 s. During this period the data of power consumption, pressure and temperature were collected for further analysis.

During the experimental run three samples of about 5 g of extrudate were drawn and dried at 105°C to determine the total water content. The total mass flow rate through the extruder was measured as an in-process control. It was determined by weighing the extrudate sampled during 1 min.

At the end of the run 0.2 g of charcoal was added at the inlet port for the powder. The time until the charcoal reached the die plate was determined as the passage time. During that time the extrudate was collected and weighed as the steady state mass.

*Experimental design*

The predictor variables in the central composite design were screw speed and water content of the extrudate. The levels of the variables are listed in Table 1. Each factor level combination

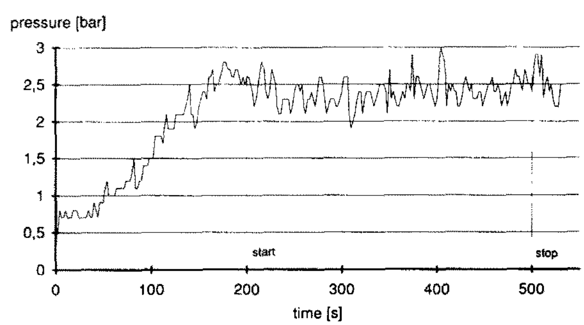


Fig. 5. Pressure-time profile for run 17.

TABLE 1

*Factor levels of the central composite design*

| Expt | Coded values | | Desired values | |
|---|---|---|---|---|
| | Water content | Screw speed | Water content (% (w/w)) | Screw speed (rpm) |
| A | −1.41 | 0 | 31.6 | 70 |
| B | −1 | −1 | 33.0 | 50 |
| C | −1 | +1 | 33.0 | 90 |
| D | 0 | 0 | 36.6 | 70 |
| E | 0 | 1.41 | 36.6 | 98 |
| F | 0 | −1.41 | 36.6 | 42 |
| G | +1 | −1 | 40.2 | 50 |
| H | +1 | +1 | 40.2 | 90 |
| I | +1.41 | 0 | 41.6 | 70 |

was performed twice. The resulting 18 experimental runs were carried out completely randomized.

The mean values for the power consumption and the pressure were calculated from the collected data. The power consumption was corrected for the power consumption of the empty extruder at the corresponding screw speed. Due to the warming up of the extrudate with progression of time in several runs, the temperature did not always reach a steady state (Fig. 6). This is specifically true for masses with a low water content, e.g., run 17. Therefore, the mean value of the last 10 values was calculated and designated as the (end-)temperature.

The response variables were corrected power consumption, pressure of the extrudate, temperature of the extrudate at the end of the run, passage time and steady state mass.

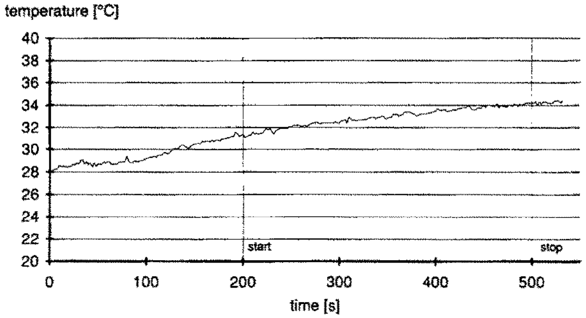


Fig. 6. Temperature-time profile for an extrudate that reached slowly steady state conditions (run 17).

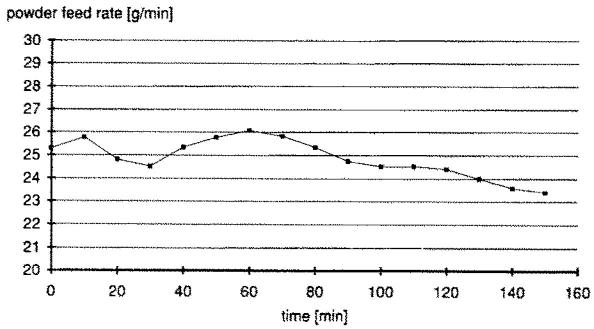


Fig. 7. Powder feed rate-time profile without an interruption of the process.

## Results

### *Precision of the powder feeder*

The powder feeder rate was observed over a period of 150 min (Fig. 7). For a constant screw speed of the powder feeder the dosage remains constant in a certain range. When the powder level in the hopper drops below a certain level the dosage rate decreases. To achieve a constant dosage rate with less powder it is necessary to adjust the screw speed of the feeder. According to this behaviour only the data from the first 120 min were used in calculating the precision. During this period the mean value of the feeder rate was 25.15 g/min with a standard deviation of 0.56 g/min (coefficient of variation of 2.25%). The range was 1.7 g/min (±3.3%).

### *Calibration of the dosage pump*

An analysis of variance of the 54 data points showed no influence of the days of performance. The influence of the pump frequency was significant but very low. Changing the pump frequency from the lowest to the highest level results in a reduction of the mass per stroke by less than 0.2%.

### *Power consumption of the empty extruder*

The loss of power consumption due to the warming up of the motor is shown in Fig. 8. The

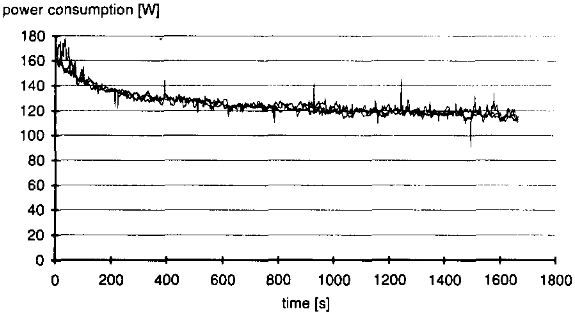


Fig. 8. Power consumption-time profile during the warming-up phase of the extruder (overlaid curves for three runs).

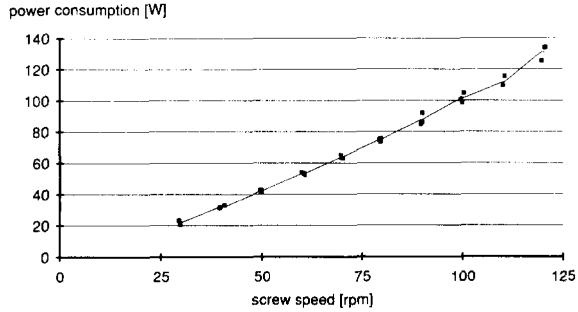


Fig. 9. Relationship between power consumption and screw speed.

power consumption of the cold machine is relatively high. During the warming up the highest reduction in power consumption occurs within the first minutes. After about 15 min the power consumption reaches a nearly constant level.

The power consumption of the empty extruder showed an almost linear relationship to the screw speed (Fig. 9). For the calculation of the power consumption at a specified screw speed level a linear interpolation between the corresponding mean values was performed.

*Central composite design*

The results of the central composite design are listed in Table 2. The statistical evaluation was performed using the coded factor values. This was necessary to avoid collinearities and for a better estimation of the standard errors. A quadratic response surface model in two predic-

TABLE 2

*Results of the central composite design*

| Expt | Run no. | Predictor variables | | | Response variables | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Desired water content (% (w/w)) | Screw speed (rpm) | Determined water content (% (w/w)) | Passage time (s) | Steady state mass (g) | Power consumption (W) | End-temperature (°C) | Pressure (bar) |
| A | 17 | 31.6 | 70 | 31.2 | 55 | 32.6 | 235.5 | 33.9 | 2.4 |
| A | 18 | 31.6 | 70 | 30.6 | 54 | 31.9 | 242.3 | 35.8 | 2.4 |
| B | 10 | 33.0 | 50 | 33.5 | 72 | 43.9 | 223.0 | 30.1 | 3.4 |
| B | 14 | 33.0 | 50 | 32.9 | 71 | 43.5 | 231.0 | 31.3 | 3.6 |
| C | 3 | 33.0 | 90 | 33.7 | 47 | 28.9 | 193.0 | 32.8 | 1.0 |
| C | 5 | 33.0 | 90 | 33.2 | 47 | 28.5 | 187.1 | 31.4 | 1.1 |
| D | 2 | 36.6 | 70 | 36.3 | 61 | 38.8 | 138.1 | 29.9 | 1.0 |
| D | 8 | 36.6 | 70 | 37.0 | 62 | 37.7 | 147.3 | 28.0 | 1.1 |
| E | 13 | 36.6 | 98 | 36.4 | 46 | 29.5 | 163.5 | 29.0 | 0.7 |
| E | 16 | 36.6 | 98 | 36.3 | 48 | 30.5 | 154.1 | 30.6 | 0.7 |
| F | 1 | 36.6 | 42 | 38.3 | 79 | 53.4 | 134.8 | 27.0 | 2.4 |
| F | 15 | 36.6 | 42 | 35.8 | 86 | 53.8 | 155.6 | 29.2 | 2.9 |
| G | 7 | 40.2 | 50 | 40.1 | 75 | 50.4 | 71.6 | 24.8 | 1.1 |
| G | 11 | 40.2 | 50 | 39.4 | 77 | 52.1 | 78.6 | 26.2 | 1.2 |
| H | 6 | 40.2 | 90 | 39.5 | 53 | 35.1 | 89.4 | 26.1 | 0.3 |
| H | 9 | 40.2 | 90 | 39.9 | 52 | 35.5 | 90.8 | 26.6 | 0.4 |
| I | 4 | 41.6 | 70 | 41.9 | 62 | 43.8 | 44.4 | 26.1 | 0.3 |
| I | 12 | 41.6 | 70 | 41.8 | 61 | 42.5 | 56.6 | 24.2 | 0.4 |

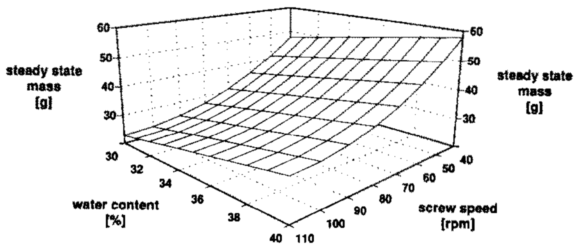


Fig. 10. Surface plot for steady state mass.

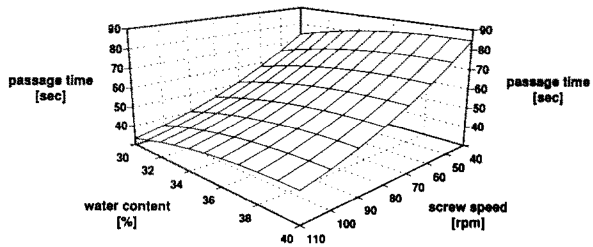


Fig. 11. Surface plot for passage time.

tor variables was fitted to the data (Eqn 1):

$$y = b_0 + b_1 x_1 + b_2 x_2 + b_{12} x_1 x_2 + b_{11} x_1^2 + b_{22} x_2^2 + e \qquad (1)$$

To select only the significant terms of the equation a stepwise regression was performed for each response variable. The resulting equations for the coded factor variables are listed in Table 3.

For the response variables passage time and steady state mass a re-specification of the model was performed. The factor screw speed was inverted (inverted screw speed = 60/rpm) and coded afterwards (Eqn 2).

$$\text{coded value} = \frac{\text{inverted screw speed–mean}}{\text{standard deviation}} \qquad (2)$$

The resulting equations for the respecified model are listed in Table 3. The equations of Table 3 were used to construct response surface plots (Figs 10–15).

## Discussion

*Precision of the powder feeder and dosage pump*

The water content of the extrudate depends on both powder feed rate and liquid feed rate. Variation in one of the feeding systems will result in different water contents. The more crucial part is the powder feeding system. The results are in accordance with those previously reported by Lindberg (1989).

For the measured range of $\pm 3.3\%$ the variation of the resulting water content can be readily

TABLE 3

*Predicting equations for the response variables based on the coded influence variables (standard errors in parentheses)*

| Response variable | Equation (standard error) | | | | | |
|---|---|---|---|---|---|---|
| Passage time | 61.50 | $+2.49\ x_1$ | $-12.29\ x_2$ | $-1.66\ x_1^2$ | $+1.73\ x_2^2$ | |
| | (1.13) | (0.40) | (0.40) | (0.67) | (0.67) | |
| Steady state mass | 37.84 | $+3.70\ x_1$ | $-8.05\ x_2$ | $+1.96\ x_2^2$ | | |
| | (0.26) | (0.18) | (0.18) | (0.23) | | |
| Power consumption | 146.48 | $-64.88\ x_1$ | $+12.99\ x_1x_2$ | | | |
| | (2.05) | (2.17) | (3.07) | | | |
| End-temperature | 29.11 | $-3.02\ x_1$ | $+0.65\ x_2$ | | | |
| | (0.26) | (0.28) | (0.28) | | | |
| Pressure | 1.05 | $-0.74\ x_1$ | $-0.75\ x_2$ | $+0.43\ x_1x_2$ | $+0.16\ x_1^2$ | $+0.31\ x_2^2$ |
| | (0.09) | (0.03) | (0.03) | (0.05) | (0.06) | (0.06) |
| Passage time | 61.56 | $+2.49\ x_1$ | $+12.08\ x_{2a}$ | | | |
| | (0.45) | (0.48) | (0.46) | | | |
| Steady state mass | 39.91 | $+3.70\ x_1$ | $+8.13\ x_{2a}$ | $-0.35\ x_{2a}^2$ | | |
| | (0.23) | (0.16) | (0.18) | (0.19) | | |

$x_1$, water content; $x_2$, screw speed; $x_{2a}$, inverse screw speed.

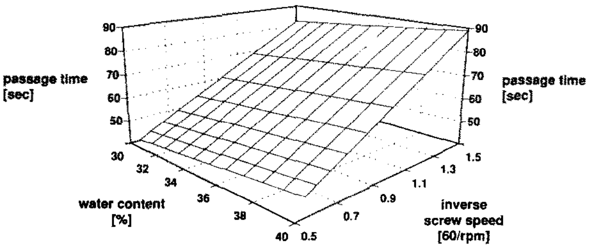


Fig. 12. Surface plot for passage time using the re-specified model.

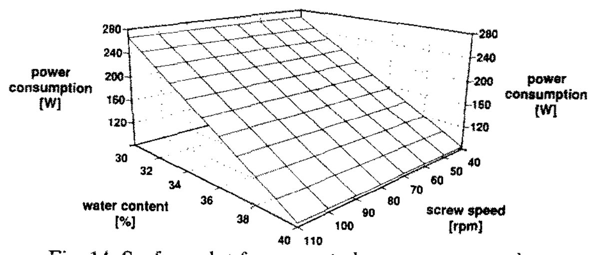


Fig. 14. Surface plot for corrected power consumption.

estimated. Given a constant water dosage of 25 g/min the water content will be in the range of 48.9–50.6% (w/w).

It is important to obtain information about the water content during a run. A fast in-process control is the determination of the mass flow rate through the extruder. Variations in one of the dosage systems can be seen immediately. The real water contents determined by drying samples at 105°C are listed in Table 2. The deviations between two replicated runs exceeds 0.7% only for one factor level combination (runs 1 and 15). The differences between the determined and the desired values result from the adjustment of the pump. The pump rate can only be varied in steps of 0.4 g/min. A finer adjustment of the desired pump rate is not possible.

*Central composite design*

The powder feed rate was maintained constant during the experiments. It was very simple to vary the water content of the extrudate by adjusting the water feed rate. Changing the water content at a constant screw speed level results in a different loading of the extruder barrel. This results in an increase of the steady state mass with increasing water content (Fig. 10). The steady state mass is a direct indicator for the degree of filling of the extruder barrel. The higher the screw speed, the greater is the transport capacity through the extruder. A higher screw speed results in a lower amount of material inside the extruder barrel at a constant feeding rate.

The passage time has the same qualitative dependences in the factor space (Fig. 11). This means that the passage time is inversely related to the screw speed. If this is true, a re-specification of the model including the inverse screw speed should result in a lower degree model equation. This is the case for the passage time, as shown in Table 3 and Fig. 12. The re-specification of the model for the steady state mass does not lead to a reduced model (Table 3).

Steady state mass and passage time are related by the mass flow rate (Eqn 3)

$$\text{steady state mass} = \text{mass flow rate} \cdot \text{passage time} \tag{3}$$

The most complicated model is necessary to describe the pressure of the extrudate (Fig. 13). This may be a result of the measuring technique for the pressure. The accuracy of the instrument in this measuring range (below 10 bar) is limited.

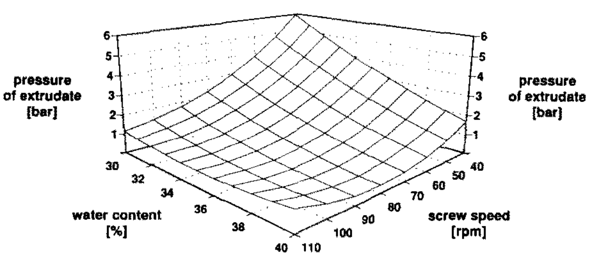


Fig. 13. Surface plot for pressure of the extrudate.

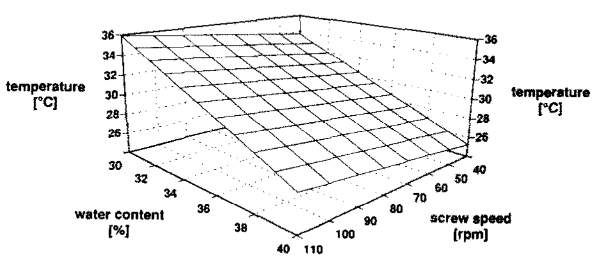


Fig. 15. Surface plot for end temperature.

An over-interpretation of the pressure data should be avoided. However, the general tendencies in the data are clear. With increasing screw speed the pressure drops according to the lowering of the steady state mass. In contrast, Dietrich and Brausse (1988) reported for a single screw extruder that an increase in screw speed increases pressure at the die plate. According to the feeding system of a single screw extruder the barrel is always filled completely with wet mass resulting in constant steady state masses. An increase in screw speed will raise both throughput through the die plate and pressure. In the case of the twin screw extruder the transport capacity is not fully used. The throughput is independent of screw speed at fixed powder and liquid feed rates. It is possible to achieve a desired pressure by selecting an appropriate screw speed. It is important to understand this different behaviour for the interpretation of experimental results obtained with the single-step granulation/extrusion process. The water content is also an important factor for the pressure. The pressure decreases with rising water content although the steady state mass will increase. This happens due to a strong lubricant effect of the water at the die wall (Ovenston and Benbow, 1968; Harrison et al., 1985b).

The corrected power consumption is only dependent on the water content of the extruded mass (Fig. 14): the greater the water content the lower the power consumption. The steady state mass seems to have no influence on the power consumption. The power consumption is only dependent on the rheological behaviour of the extruded mass. A higher water content results in a smoother material and less power is necessary to knead the mass.

The temperature is mainly influenced by the water content (Fig. 15). A minor influence of the screw speed is obvious. The lowest temperatures are obtained using low screw speeds and high water contents. The temperature is an important variable in pharmaceutical extrusion. In terms of drug stability the combination of high moisture content and high temperature may cause problems. On the other hand, high temperatures may lead to uncontrolled loss in water content after extrusion. In pellet production the water content should be held constant, since it is an important factor for the following spheronization step. It is only possible to prepare successful products in a certain range of moisture content (Lövgren and Lundberg, 1989; Bains et al., 1991).

## Conclusions

This study demonstrates that the water content of the extrudate has the greatest influence on the important extrusion parameters. Higher water contents result in smoother masses which lead to lower pressure, lower power consumption and lower temperature.

The screw speed is an important variable for the steady state mass and the passage time through the extruder. It also influences the pressure but has only a minor effect on power consumption and temperature.

When using a twin screw extruder in a one-step granulation/extrusion process it is most important to control the water content. This is only possible on the premises of accurately adjusted feeding systems. On the other hand, the system allows easy variation in water content of the extrudate without changing the powder feeding rate. In-process controls of the water content are an essential tool in this process. The single-step granulation/extrusion process leads to a different behaviour compared to an extruder with completely filled barrel. For the interpretation of experimental results it is important to be aware of this fact.

For further studies including different formulation variables it is necessary either to control the water content strictly or to include the water content as a variable. Compared with this it may be appropriate to fix the screw speed at a certain level in these studies.

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