Physicomechanical Characterization of the Extrusion-Spheronization Process. I. Instrumentation of the Extruder

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Extrusion-spheronization is a popular means of producing spheres which can be coated to form a controlled-release system. In the extrusion process, stress is necessary to force a wet mass through small orifices, and as a result, frictional heat builds up at the screen. Therefore, the quantitative measurement of the screen pressure and screen temperature is described and shown to provide objective measures of extrudability. A strain gauge load cell was mounted tangentially to the screen of a Luwa EXDS-60 extruder with a specially fabricated holder. The load cell output was calibrated in terms of pressure inside the screen with a special rubber plug system. A fast-response thermocouple was used to measure the screen temperature. Experiments with 50/50 lactose/Avicel PH101 revealed that a linear relationship exists between the amount of water used in the granulation and the screen pressure, that the percentage open area of the screen determines the rank order of the screen pressure, and that the maximal yield of 18/25-mesh cut pellets was uniquely related to the screen pressure. Also, a high degree of correlation was observed between the screen pressure and the screen temperature.

KEY WORDS: extrusion; spheronization; marumerization; instrumentation; screen pressure; optimization; screen temperature.

INTRODUCTION

Extrusion-spheronization is being established as a popular means of producing spheres or pellets which can be coated to form a unique controlled-release delivery system (1–3). The success of this process has been judged subjectively (4–8) and by ram extrusion rheometry (9,10). Subjective assessment has the obvious drawbacks, and ram extrusion is certainly questionable in its ability to predict production-type screw extrusion.

A certain amount of stress (screen pressure) is necessary to force the wet mass (formulation) through the orifices of the screen or the die of the extruder. Frictional heat also builds up, resulting in a temperature increase. The ability to measure these parameters on a production-type screw extruder would provide new insights into the interplay between formulation and extrusion process variables and assist in the design of formulations by providing objective measures of extrudability.

One such attempt at measuring these parameters has been reported by Dietrich (11), who developed techniques to measure the extrusion pressure, extrudate temperature, and electric current consumption of a screw extruder with axial discharge. Relationships between these parameters and such process variables as the die aperture diameter, thickness of the die, and speed of rotation of the screw were investigated. An increase in screw rotation speed led to an increase in extrusion pressure, but the quality of the extrudate and of the resultant pellets was not adversely affected. This outcome was attributed to effective cooling, employed during extrusion, which prevented excessively rapid evaporation of water and preserved the plasticity of the extrudate required for spheronization. This observation is in contrast to that reported by Newton and his co-workers (12), wherein low extrusion rates were recommended for improving the quality of the pellets. These disparate observations may be explained based on the differences in feed mechanisms [screw feed (Dietrich) vs gravity feed (Newton)]; however, it is clear that the effect of formulation-dependent factors on extrusion pressure and related parameters needs to be examined.

The specific objectives of this research are (i) to instrument a typical twin screw extruder (EXDS-60 Luwa Corp., Charlotte, NC 28297) to measure screen pressure and temperature and (ii) to examine the possible relationship between the output of the instrumentation and the quality of the ultimate product, the pellets.

MATERIALS AND METHODS

Screen Pressure Instrumentation

The extruder screen was instrumented by mounting a strain gauge load cell tangential to the screen at site T in Fig. 1. Since this area of the screen is not reinforced or supported, it would be easily strained, thus providing a highly sensitive site for instrumentation. In addition, no physical bonding of strain gauges to any machine parts was required.

A 100-lb (444.8-N) capacity strain gauge load cell (Model 13/2444-03, Sensotec, Inc., Columbus, OH 43212) was selected. This load cell is 0.5 in. in diameter ("D" in Fig. 2) and its calibration factor is 0.1245 mV/lb.

A special holder was designed and fabricated to mount this load cell tangentially to the screen of the extruder (site T in Fig. 1). The various components are as shown in Fig. 2. Initially, the load cell is placed inside the holder ("F" in Fig. 2). Then the shim ("E" in Fig. 2) is placed on top of it and the entire assembly is put together as shown in Fig. 1. The wet mass during extrusion experiences resistance, and a force is registered on the load cell. The shim is used to distribute the force evenly onto the load cell and to dampen any machine vibration which may create noise in the output.

The ability to position the load cell reproducibly at the same point was thought to be critical, and to that end a positioning system was fabricated. The load cell holder is mounted flush against the stationary aluminum block ("C" in Fig. 1), and the brass piece ("H" in Fig. 1) is held in place by a set of screws. This system provided a reasonable degree of confidence that the screen force is being measured at the same point every time.

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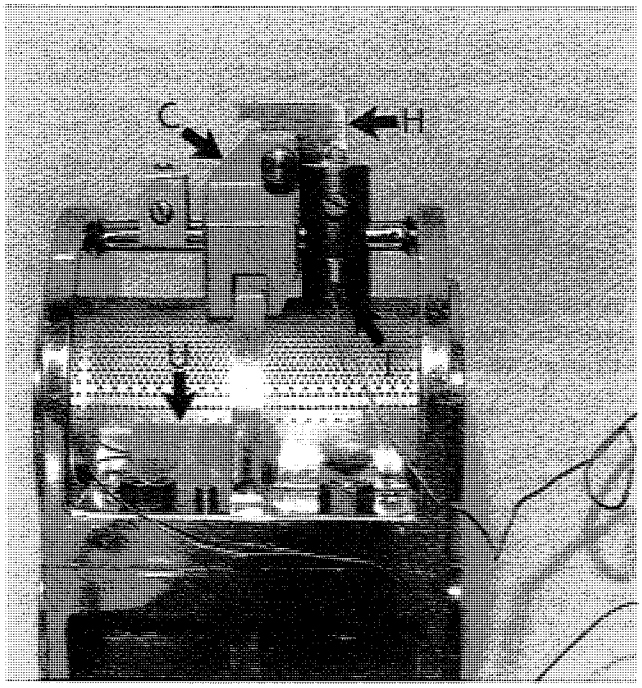

Fig. 1. Screen pressure and temperature instrumentation assembled on the screen housing of Luwa Extruder EXDS-60. C, aluminum block; H, brass piece; T, site of screen pressure instrumentation; U, thermocouple.

The excitation voltage (5 V) was supplied with a DC power supply unit (Model 6216A, Hewlett-Packard, Rockaway, NJ 07866), and the output from the load cell was fed to a wideband differential amplifier (Model 128, Neff Instrument Corp., Monrovia, CA 91016), where the signal was amplified 1000 times. The amplified signal was entered into a digital oscilloscope (Model 2090 III, Nicolet Instruments Corp., Madison, WI 53711), where the data were collected at a rate of 10 points/sec and were stored on a floppy diskette. Comparison with data collected at 20 points/sec for the same formulation revealed no difference in peak heights. Further analysis (calculation of mean screen force, as described later) was performed on a personal computer with digital data processing software VU-POINT (S-Cubed, LaJolla, CA 92038). The baseline was corrected for the preload, and the signal was converted to units of force (N).

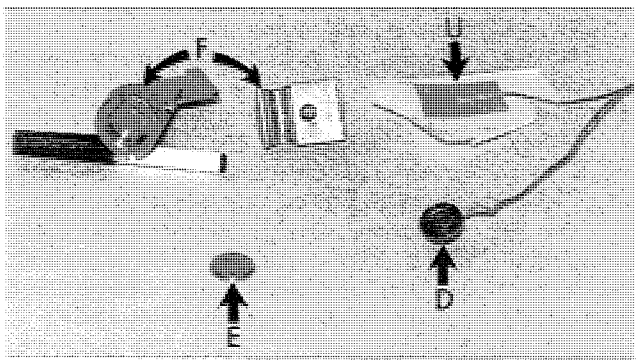

Fig. 2. Components of the screen pressure and temperature instrumentation. D, strain gauge load cell; E, shim; F, load cell holder; U, thermocouple.

Calibration of the Load Cell Output

The screen force signal was calibrated in terms of the pressure inside the screen using a rubber plug system. The procedure required a convenient way of applying a known pressure to the die wall. To this end, a specially machined rubber plug of 60 to 70 durometer hardness (Phelps Packing and Rubber Co., Baltimore, MD 21256) was fitted into one hemisphere of the screen holder. The open side was blocked off with an aluminum plate. Aluminum spacers were placed on each end of the rubber plug. A precalibrated piezoelectric load cell [Model 9712A5000; range, 5000 lb (22.24 kN); calibration, 0.966 mV/lb; Kistler Instrument Corp., Amherst, NY 14120] was placed on top of the spacer, and the entire system was placed between the platens (Fig. 3) of a servo hydraulic press (Fred S. Carver Inc., Menomonee Falls, WI 53051). The signal from the piezoelectric load cell was fed simultaneously to the digital oscilloscope, along with the screen force instrumentation. Upon application of pressure by the hydraulic press to the rubber plug, a corresponding force is registered on the screen. Due to the apparent hydraulic behavior of rubber (13), the radial pressure transmitted to the screen of the extruder may be assumed to be the same as that applied axially by the hydraulic press. The two outputs were plotted against each other to form a calibration curve ($r^2 = 0.996$). The equation of the calibration curve was Screen Force = $[(1.945 * 10^{-5}) *$ Rubber Plug Pressure$] - 18.61$. The significant Y intercept (-7.48 N) can be attributed to the imperfect fit of the rubber plug inside the screen housing. The intercept results because a minimum amount of pressure (384 kPa) is required before the plug starts to register a force against the screen. The equation of the line was later used to convert all screen force data to apparent pressure. This calibration was repeated five times and exhibited excellent reproducibility (%RSD = 5.89 for slope).

This calibration involves further assumptions, as follows:

(a) Pressure is distributed uniformly throughout the screen during extrusion.
(b) There is no loss of force to the die wall.

The rubber plug was lubricated with mineral oil before

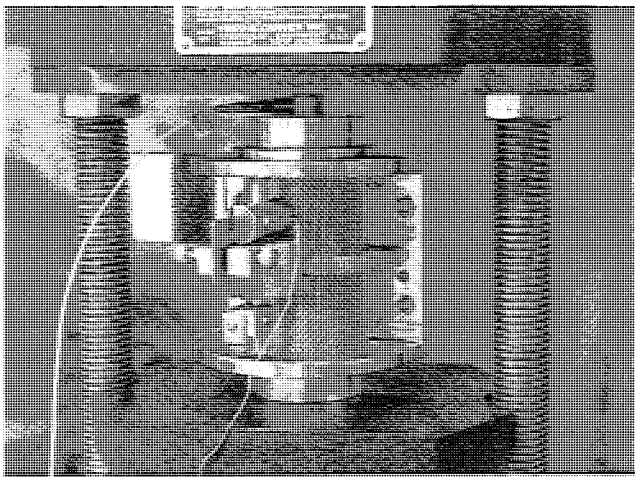

Fig. 3. Screen pressure calibration setup using a servo hydraulic press.

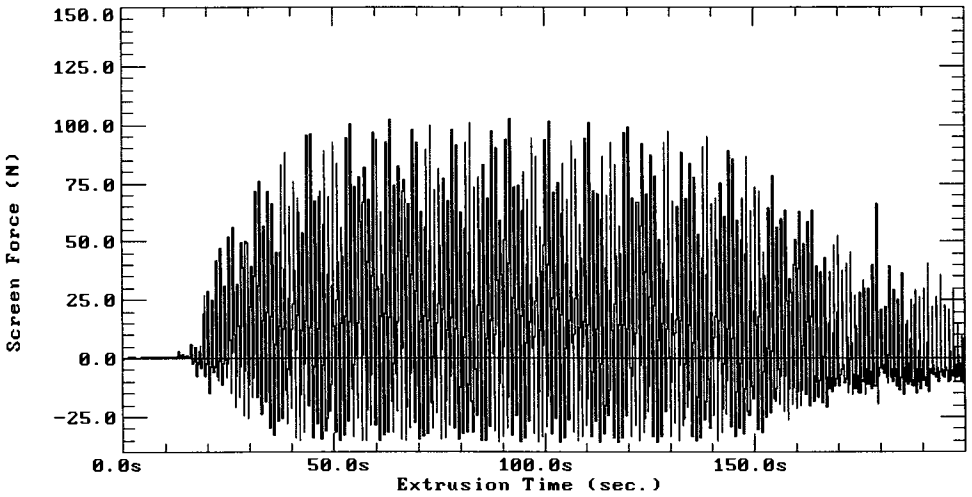

Fig. 4. Typical screen force profile exhibiting an apparent steady-state region.

each calibration run to ensure that the second assumption would hold.

Screen Pressure Data Analysis

A screen force profile obtained with a representative formulation is shown in Figs. 4 and 5. In Fig. 4, an apparent steady-state region was observed wherein peak forces are almost constant. Within this region, upon expansion of the time axis (Fig. 5), three types of peaks having distinctly different shapes and peak heights were observed in a continuously repetitive sequence. These different peak heights and their repetitive nature are a function of the geometry and rotation of the three extrusion blades (as illustrated in Fig. 6). During rotation, as one of these blades approaches the force-sensitive area of the screen, there is an increase in the force signal until it reaches a peak. As the blade moves away from the force-sensitive area, there is a decline in the force signal. The angle of approach of the three blades to the force-sensitive area is different, which explains the differing peak heights and peak shapes. The mean of the peak height values of the five tallest peaks representing the maximum extrusion force generated at the sensor location upon each rotation of the blade is reported for each batch. This mean force value is converted to an apparent screen pressure based on the calibration factor and assumptions discussed above.

Screen Temperature Instrumentation

A fast-response thermocouple ("V" in Fig. 1) with a self-adhesive backing (Model TAC-80T, Omega Engineering Inc., Stamford, CT 06907) was used to monitor screen temperature during extrusion. The thermocouple was glued onto the screen ("U" in Fig. 1) at the same point for each batch. The signal from the thermocouple is fed into a strip-chart recorder (Model RD2020, Omega Engineering Inc.). During extrusion the screen temperature first rises and then levels off to a steady-state value. The difference between the steady-state value and the room temperature is reported as the extrusion screen temperature increase.

Preliminary Experiments

Preliminary experiments were performed with 50/50 lac-

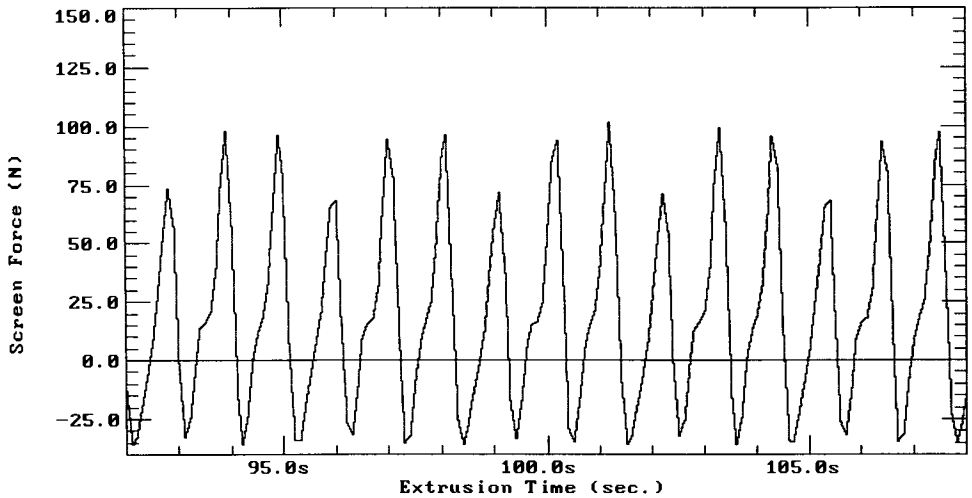

Fig. 5. Typical screen force profile upon expansion of the time axis.

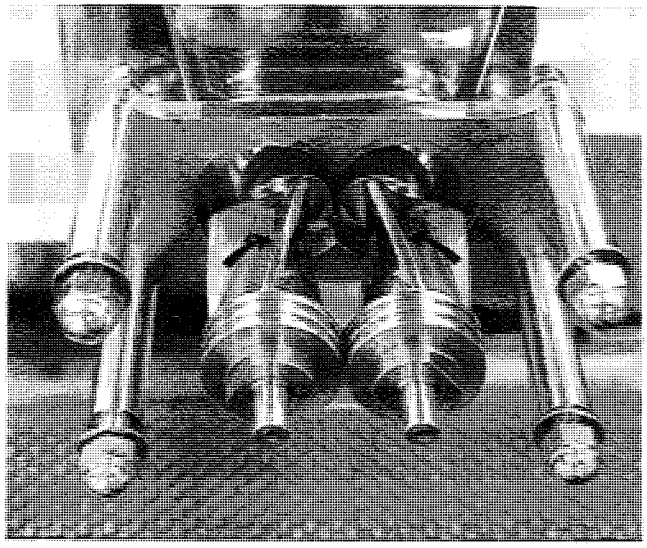

Fig. 6. EXDS-60 Luwa extruder screw blades.

tose (anhydrous lactose, Lot No. ONA18-147, Sheffield Products, P.O. Box 630, Norwich, NY 13815)–microcrystalline cellulose (MCC; Avicel PH101, Lot No. 1018, Drum 1530, FMC Corp., Newark, DE 19711) formulations with varying water levels. The dry powders were mixed in a planetary mixer (Model A-200DT, Hobart Corp., Troy, OH 43276) for 2 min. The granulating liquid (water) was added to the dry powder, and wet massing was performed in the planetary mixer for 2 min. The wet mass was fed through a hopper into the twin screw extruder rotating at 19 rpm. The extruder was fitted with a 1-mm aperture screen, except in a study of the effect of screen aperture size on screen pressure. The extruded mass was fed in 350-g batches into a spheronizer (Model QJ 230, Luwa Corp., Charlotte, NC 28297) rotating at 830 rpm. Process time was 2 min. The spheronizer was fitted with a friction plate of groove size 2 mm. The wet pellets were collected and dried in a tray drier (Lydon Brothers Corp., Hackensack, NJ 07018) at 70°C for 2 hr.

The dried pellets were evaluated for particle size distribution and bulk density. Sieve analysis was performed on 100-g samples of pellets using a Fritsch analysette sieve shaker (Model 03-501, Fritsch GMBH Laborgeratebau, D-6580 IDAR-Oberstein 1, FRG). The test was run over 5 min at an amplitude setting of five. The percentage of pellets by weight retained on each sieve was determined to yield its size distribution.

The tapped bulk density of the 18/20 mesh cut of pellets was determined using a volumeter (Model 10700, Vankel Industries Inc., Edison, NJ 08820). In this method, a predetermined weight of the pellets was poured into a 100-mL volumetric cylinder. The bulk density was calculated after 500 drops from a height of 1.5 cm, as the ratio of the weight and volume of the pellets.

RESULTS AND DISCUSSION

Selection of Batch Size

The width of the steady-state region of the screen force signal is dependent on the batch size of the granulation to be extruded. Five experiments were conducted with the same formulation (i.e., at the same water level) to compare batch sizes ranging from 0.5 to 5 kg. The results (Table I) indicated that the 1.5-kg batch size exhibited a wide enough steady-state region in both screen force and screen temperature signals for further studies. The peak heights in the screen force signal of the 1.5-, 2-, and 5-kg batches were comparable. The remaining experiments were performed using a batch size of 1.5 kg.

Table I. Selection of Batch Size

Batch size (kg)	Screen temperature increase at steady state (°C)	Screen force mean (SD) of five peaks at steady state (N)	Width of steady-state region (sec)
0.5	5.7	79.4 (3.98)	17.0
1.0	7.9	124.7 (1.32)	31.5
1.5	9.0	96.5 (0.96)	99.2
2.0	9.5	89.8 (1.44)	156.0
5.0	9.2	92.6 (0.41)	351.2

Demonstration of Reproducibility

A 50/50 lactose/MCC formulation with a 52% (as percentage of total solids) water level was repeated five times, and screen pressure and temperature were recorded during extrusion. The results showed an excellent reproducibility, with %RSD less than 5 for both screen pressure and temperature.

Effect of Water Level on Instrumentation Output

After having demonstrated reproducibility, the instrumentation was used in the extrusion/spheronization of six batches of 50/50 lactose/MCC with varying water levels, ranging from 36 to 56%. The 36 to 56% water level range was determined to be the working range based on previous feasibility experiments. The screen pressure and temperature at these water levels are reported in Table II. The dried pellets were evaluated for particle size distribution and bulk density (Table III and Fig. 8).

Figure 7 shows a linear relationship between screen pressure and water level as percentage of solids. A similar plot was obtained for screen temperature increase vs water level. On average a change in screen pressure signal of about

Table II. Effect of Water Level on Instrumentation Output with 50/50 Lactose/MCC Formulation

Water (% of solids)	Screen temp. (°C)	Screen force (N), mean (SD)	Screen pressure (kPa)
56	7.5	75.2 (0.77)	4826
52	8.6	96.8 (1.57)	5934
48	12.3	138.4 (1.86)	8099
44	14.7	165.1 (3.42)	9445
40	17.1	202.4 (4.83)	11180
36	20.2	237.1 (3.32)	13180

Table III. Effect of Water Level on Dried Pellet Particle Size Distribution with 50/50 Lactose/MCC Formulation

Water (% of solids)	% (by weight) retained on each sieve							
	<45	35/45	25/35	18/25	16/18	14/16	10/14	>10
56	0.0	0.0	1.1	19.0	25.1	13.3	35.7	5.8
52	0.0	0.0	0.5	66.5	22.4	3.6	7.0	0.0
48	0.0	0.0	5.0	85.0	8.0	1.0	1.0	0.0
44	0.5	0.6	6.0	82.9	9.9	0.1	0.0	0.0
40	7.4	2.7	10.7	75.9	2.3	0.9	0.1	0.0
36	33.1	8.6	14.4	43.3	0.4	0.1	0.1	0.0

14% is observed for each 4% change in water level, which indicates that the instrumentation is sufficiently sensitive to pick up changes in the input material. The data showed a high degree of correlation (r^2 = 0.996) between screen pressure and temperature.

Normally, the target diameter of the dried pellets is in the range of 80 to 100% of the aperture diameter of the screen used during extrusion (14). Thus, for a 1-mm aperture screen, the yield of 18/25 (1000- to 710-μm) mesh cut was chosen as the parameter reflecting the yield within the target range and a narrow size distribution. The relationship between the yield of the 18/25 mesh cut of the dried pellets and the corresponding screen pressures is in the form of a "U"-shaped convex curve with a maximum around 8000 to 9000 kPa (Fig. 8). This curve demonstrates that screen pressure potentially can be used to optimize a given formulation. Clearly, screen pressure is a function of the rheological character of the mass and the formulation parameters that affect rheology.

While the yield 18/25 mesh cut was chosen as the parameter reflecting the yield within the target range and a narrow size distribution, the 18/20 mesh cut was found to be more appropriate for bulk density measurements. Figure 9 shows the relationship between bulk density of dried pellets and screen pressure. Surprisingly, lower bulk densities (from 0.824 to 0.676 g/mL) were associated with higher screen pressures. This finding is in contrast to expectations that higher screen pressures indicate greater densification of the wet mass and, hence, denser pellets. Instead, the bulk density of the 18/20 mesh cut is apparently influenced by the "rheology" of the wet mass in a different manner. For this formulation, wetter, more easily extruded masses apparently resulted in denser pellets.

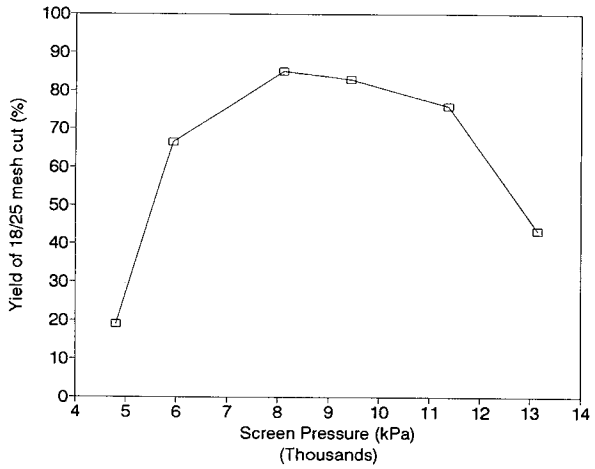

Fig. 8. Relationship between yield of 18/25 mesh cut and screen pressure (kPa).

Effect of Screen Aperture Size

The effect of screen aperture size on screen pressure was studied by extruding a 50/50 lactose/MCC formulation with 52% water through screens ranging in aperture sizes from 0.5 to 1 mm. An interesting observation made from the results (Fig. 10) was that the screen pressure was higher for the 0.9-mm screen than for the 0.7-mm screen. In an attempt to explain this observation, the number of apertures per square centimeter of screen area was calculated for each of the four screens. The percentage open area of each screen was also calculated and plotted against screen pressure (Fig. 11). The results indicate that the percentage open area of the screen is what determines the rank order of screen pressure, and not the screen aperture size.

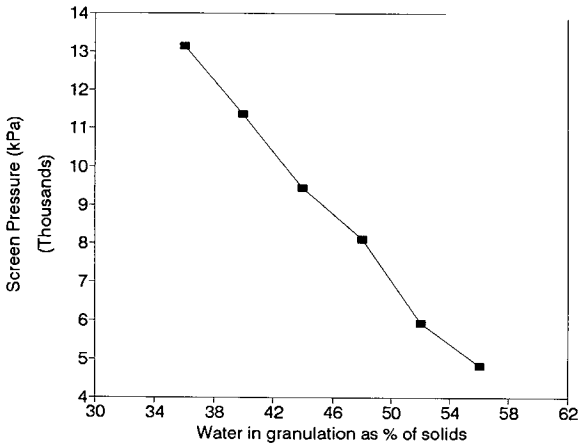

Fig. 7. Effect of varying water levels (% of solids) in 50/50 lactose/MCC formulations on screen pressure (kPa).

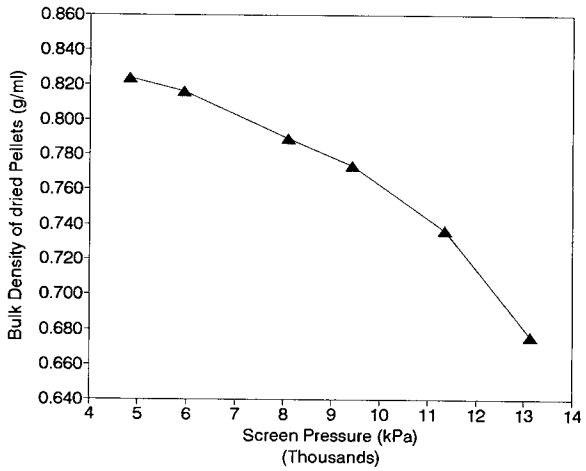

Fig. 9. Relationship between bulk density (g/mL) of 18/20-mesh cut dried pellets and screen pressure (kPa).

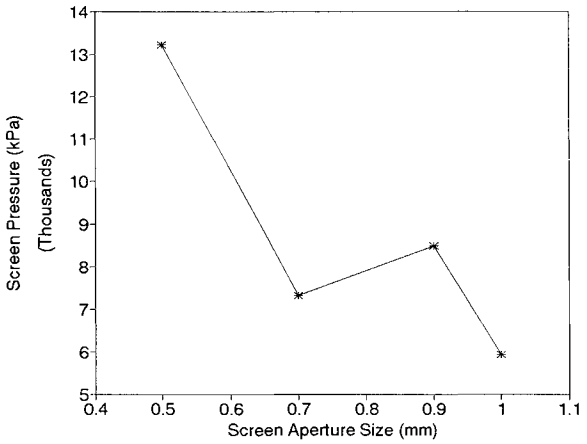

Fig. 10. Effect of screen aperture size (mm) on screen pressure (kPa).

CONCLUSIONS

A production-type screw extruder EXDS-60 was successfully instrumented to measure screen pressure and screen temperature. Experiments with 50/50 lactose/Avicel PH101 revealed a high degree of correlation between screen pressure and screen temperature. A linear relationship observed between the amount of water used in the granulation and the screen pressure showed that the instrumentation was sufficiently sensitive to changes in input materials. The "U"-shaped convex relationship between the yield of the 18/25 mesh cut of the dried pellets and the corresponding screen pressures provides an approach to optimize the product yield within the target mesh cut.

In the extrusion process, stress is necessary to force a wet mass through small orifices of a screen. Since the screen pressure and screen temperature have been shown to provide a quantitative measure of the ease with which the wet mass is forced through the small orifices of a screen, these instrumentation outputs can be used to determine the extrudability of a given formulation. The extrusion process can be monitored by observing the instrumentation signals. A safety feature using the instrumentation could be generated to prevent damage to the screen and the machine which may result from excessive pressure buildup.

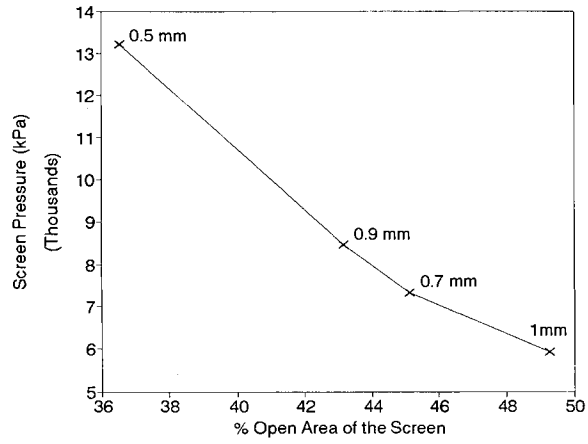

Fig. 11. Effect of percentage open area of the screen on screen pressure (kPa).

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