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Flow defects in wet powder mass extrusion

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Surface impairment, in the form of the flow defects roughness and sharkskinning, has been shown to occur with microcrystalline cellulose/lactose/water mixes on extrusion under varying process conditions. These defects result from the imbalance of stresses at the outer wall of the extrudate on leaving the die and can be avoided by changing the process conditions that are known to affect these stresses, e.g. the length of the die, the velocity of throughput and the moisture content of the formulation.

Extrusion, a process that converts a suitable raw material into a product of specific cross-section by forcing it through an orifice (a die) under controlled conditions, is widely used as a preliminary preforming process in the production of spherical granules by spheronization (Reynolds 1970). The shape and surface finish of the extrudate can be impaired by defects that occur during the flow of the material through the extruder and these defects can affect further processing and mar the quality of the finished product. Whilst shape impairment has been previously reported for wet powder mass extrusion (Cheng 1970), surface impairment has not yet been demonstrated although it has been frequently reported to occur in polymer processing (Tordella 1969). This paper reports work on the identification and causes of such defects in wet powder mass extrusion.

MATERIALS AND METHODS

Two formulations commonly used for the manufacture of spherical granules by spheronization were chosen for this study. The first consisted solely of microcrystalline cellulose (Avicel PH101—FMC Corporation, Marcus Hook, Pennsylvania, USA), and the second of equal quantities of microcrystalline cellulose and lactose (Unigate Regular Grade). Each formulation was mixed with varying quantities of water (33·9–59·4% w/w) in a planetary mixer and filled into the barrel of a ram extruder (Ovenston & Benbow 1968). The materials were extruded through dies of varying diameter (1–2 mm) and lengths (2–20 mm) and varying die arrangements (single-holed or multi-holed), using a servo-hydraulic press (Model M1000/RE, Dartec Ltd., Stourbridge) at varying ram speeds (0·5–12 mm s^{-1}). The resulting extrudate was collected in a C-piece located beneath the ram extruder and random samples were visually inspected for surface defects. If the surface of the extrudate appeared smooth, its diameter was determined by measuring three random samples at 1 mm distance along each length using a travelling microscope. The degree of swelling, or die swell, was expressed as the swelling ratio, i.e. the ratio of the extrudate diameter to the die diameter.

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RESULTS

When both formulations were extruded through single holes, surface impairment invariably occurred under two distinct experimental conditions—extrusion using short-length dies (i.e. when the die length was less than or equal to the die diameter) and extrusion at high throughput rates. In the former, surface impairment was characterized by sharkskinning while in the latter it was by roughness, Fig. 1. Both defects are similar in appearance, being characterized by the presence of finely spaced, sharp regular circumferential ridges (Benbow & Lamb 1963), but they can be differentiated, since in sharkskinning the ridges are more pronounced, with the cracks between the ridges penetrating deeply into the core of the extrudate. No surface impairment was found when multi-holed dies were used.

Roughness was found to occur only at the widest die diameter (2 mm) and at the highest ram speed (6 mm s^{-1}) for the microcrystalline cellulose/lactose mix at its highest moisture content (41% w/w), but for the microcrystalline cellulose alone occurred at all die diameters, at all moisture contents greater than 51% w/w. Sharkskinning occurred in both formulations when short dies were used, regardless of all other process conditions.

Values for the die swell ratios for both formulations under varying process conditions are given in Tables 1 and 2. These show that the die swell increased with increasing moisture content and

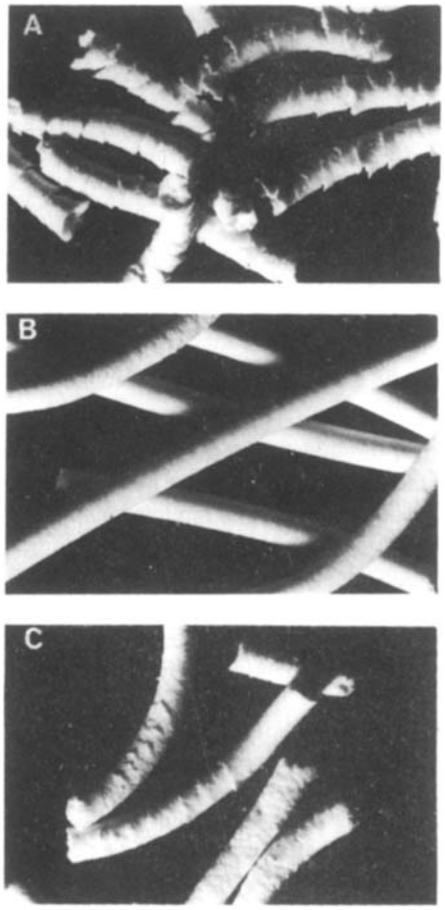

FIG. 1. Types of extrudate produced in this study. (A) product showing sharkskinning. (B) smooth product. (C) product showing roughness.

decreasing die diameter. However, the values themselves are small when compared with the experimental error (1–3%) in determining the diameter of the extrudate.

DISCUSSION

The swelling ratio has been described by Bagley & Schreiber (1969) as the measure of the deformation imposed on the material as it enters a die, which can be recovered by the swelling of the product as it leaves the die, i.e. its elastic recovery. This phenomenon occurs for viscoelastic materials and so rate processes are important. The small values of the swelling ratio (Tables 1 and 2) and their relative insensitivity to changes in processing conditions indicate that the elastic recovery of the formulations is minimal. The evidence of surface impairment, rather than large values of die swell, suggest that a further explanation is necessary.

When a material flows through a die, it will do so with a steady-state velocity gradient where the material nearest the wall will either be static (in the condition of no slip or stick) or moving at a finite velocity (in the condition of slip where a lubricating layer exists) and the material within the centre of the die will be travelling at maximum velocity. On leaving the die, the material on the outside of the

Table 1. Die swell (D/D_o) as a function of operation conditions for microcrystalline cellulose systems.

Die diameter = 1 mm	Moisture content = 45·6% w/w	
Ram speed ($mm\ s^{-1}$)	L/R = 3·99	L/R = 11·74
6·0	1·03	1·03
3·0	1·03	1·03
Die diameter = 2 mm	**Moisture content = 45·6% w/w**	
Ram speed ($mm\ s^{-1}$)	L/R = 3·80	L/R = 12·00
6·0	1·02	1·03
3·0	1·02	1·02
Die diameter = 1 mm	**Moisture content = 50·9% w/w**	
Ram speed ($mm\ s^{-1}$)	L/R = 3·98	L/R = 11·74
4·0	1·06	1·05
2·0	1·05	1·04
Die Diameter = 2 mm	**Moisture content = 50·9% w/w**	
Ram speed ($mm\ s^{-1}$)	L/R = 3·80	L/R = 12·00
4·0	1·05	1·03
2·0	1·04	1·04

L/R = Length to radius ratio.
D_o = Die diameter.
D = Extrudate diameter.

Table 2. Die swell (D/D_o) as a function of operating conditions for microcrystalline cellulose/lactose systems.

Die diameter = 1 mm	Moisture content = 34·0% w/w			
Ram speed ($mm\ s^{-1}$)	L/R = 3·98	L/R = 7·90	L/R = 11·74	L/R = 15·6
4·0	1·04	1·03	1·04	1·04
2·0	1·04	1·03	1·03	1·03
Die diameter = 2 mm	**Moisture content = 34·2% w/w**			
Ram speed ($mm\ s^{-1}$)	L/R = 3·80	L/R = 7·80	L/R = 12·00	L/R = 20·00
4·0	1·03	1·02	1·03	1·01
2·0	1·02	1·02	1·02	1·02
Die diameter = 1 mm	**Moisture content = 40·8% w/w**			
Ram speed ($mm\ s^{-1}$)	L/R = 3·98	L/R = 7·90	L/R = 11·74	L/R = 15·6
4·0	1·08	1·08	1·08	1·06
2·0	1·09	1·09	1·06	1·04
Die diameter = 2 mm	**Moisture content = 41·0% w/w**			
Ram speed ($mm\ s^{-1}$)	L/R = 3·80	L/R = 7·80	L/R = 12·00	L/R = 20·00
4·0	1·05	1·06	1·05	1·05
2·0	1·03	1·04	1·05	1·04

L/R = Length to radius ratio.
D_o = Die diameter.
D = Extrudate diameter.

extrudate will no longer be contained and will accelerate while the central core will decelerate to compensate, until the extrudate, at some distance downstream of the die exit, assumes uniform velocity. It has been suggested (Nickell et al 1974) that this causes a compression in the central layers and a tension in the outer layers of the extrudate. If the tension exceeds the forces required to maintain the smooth surface and cylindrical shape of the extrudate, splitting in the wall of the extrudate will occur, resulting in roughness or in severe cases, sharkskinning.

The effect seen in short dies, where the length of the die is similar to or less than the die diameter, is due to the non-steady state condition within such a die. When a material enters a die, there must be a distance between the die entry and the point at which a steady state flow is created. If this so-called entrance length is greater than the length of the die, then steady state conditions will not apply. In the formulations studied, it has been shown that there is a uniform lubricating layer close to the wall of the die (Harrison 1982) and hence there will be a finite length before this uniform lubricating layer can be formed. If this layer is not of uniform thickness, then the situation 'stick-slip' could arise at the exit of the die where, at zero thickness, there will be a condition of stick while at the maximal thickness there will be a condition of maximal slip. At the condition of stick the material at the die wall will be static and therefore, according to Nickell et al (1974), the tension created as the material exits from the die will be maximal, resulting in catastrophic surface impairment.

Other factors which can affect the balance of the tension in the outer surface of the extrudate and the forces required to maintain the cylindrical shape of the extrudate are (i) high ram speed, which can result in higher wall shear stresses in the die (Harrison 1982), thus increasing the tension; (ii) moisture content which results in a reduction in the forces required to maintain the smooth surface, and (iii) the presence of lactose which, it is thought, reduces the deformability of the microcrystalline cellulose, strengthening the surface of the extrudate. All these factors affect the incidence of roughness which was found to be more prevalent at high ram speeds, high moisture contents and in the formulation containing microcrystalline cellulose alone. There does appear to be a critical velocity above which roughness occurs. For example, for microcrystalline cellulose containing 54·7% w/w water, roughness was induced at all velocities exceeding 3·87 mm s^{-1}. The reason why there was no evidence of any surface impairment when multi-holed dies were used is the reduction in the wall shear strain and the velocity of throughput (associated with an increase in the total area available for extrusion) in multi-holed dies compared with single-hole dies at similar ram speeds (Harrison 1982).

Unfortunately, the ram speed of a system could not increase towards these critical velocities when extrusion is through multi-holed dies.

Conclusion

Both shape impairment in the form of die swell, and surface impairment in the form of sharkskinning and roughness, have been shown to occur in the extrusion of formulations commonly used in the preparation of spherical granules by spheronization. The die swell that does occur is small and unlikely to be detrimental to further processing. However, both types of surface impairment will severely mar the qualities of the product and should be avoided wherever possible. Sharkskinning can be avoided by reducing the wall shear stress within the die by decreasing the ram speed or increasing the number of holes in the die, and reducing the deformability of the extrudate by decreasing moisture content, or changing the formulation in terms of the solid components.

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