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Convergent flow analysis in the extrusion of wet powder masses

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The angle of convergent flow which occurs when a wet powder mass is extruded through a die has been assessed by the inclusion of dyes in the mass before extrusion. The angles obtained are dependent on the powder components and their proportion, the moisture level, and the ratio of the dead space to the area of open die. The method allows assessment of the type and ease of flow which occurs during extrusion.

Flow visualization studies in polymer systems have shown that when a material is extruded from a large diameter reservoir into a die of narrower cross section an angle of convergence is created as the material accelerates towards the die inlet (Tordella 1969). This angle of convergence has been shown to be dependent both on the selected material and on the chosen process conditions. A knowledge of the angle is useful in the design of tapered dies commonly used in practice, to reduce both the pressure necessary for extrusion (Beresnevet et al 1963) and the incidence of flow defects (Howells & Benbow 1962). With reference to die design, Cogswell (1978), for instance, has suggested that this reduction in extrusion pressure will only occur when the taper in the die is less than the angle of convergence. Most of the work in convergent flow analysis has been directed towards polymer systems and little has been undertaken with wet powder massed systems, despite the increase in the use of the extrusion of such systems as a performing process in the food and pharmaceutical industries. This study reports the measurement of the angle of convergence which occurs in such systems, by the controlled insertion of coloured dyes before the extrusion of wet powder masses.

MATERIALS AND METHODS

Two commonly used pharmaceutical excipients were chosen for this work—microcrystalline cellulose (Avicel PH101 - F.M.C. Corporation Marcus Hook, Pennsylvania, USA), a material derived from wood pulp, which has the ability to absorb large quantities of water and become readily deformable (Battista 1970), and lactose (Unigate, Regular Grade). The materials (alone or as blends) were mixed with varying amounts of water (2–55% w/w) in a planetary mixer (Hobart or Kenwood) and filled into a barrel (2·54 or 1·27 cm diameter) of a ram extruder (Ovenston & Benbow 1968). Layers (50 mg) of coloured material (microcrystalline cellulose stained with dyes) were placed at known intervals within the barrel. The material was extruded through dies of varying diameter (1–2 mm), length (2–20 mm) and die arrangement (singleholed or multiholed) using a hydraulic press (Model M1000/RE, Dartec Ltd., Stourbridge) at varying ram speeds (0·5–12 mm s^{-1}). The load required to extrude the material was monitored using a load cell mounted on the crosshead of the press and recorded against displacement on an X–Y recorder. As soon as colours were detected within the extrudate, the extrusion was stopped, the die removed, and the plug of material remaining within the barrel ejected by applying pressure to the piston. The ejected plug and extrudate was sectioned along its central vertical axis and dried at 60 °C. The patterns were either photographed as in Figs 1–3 or traced as in Figs 5–7.

RESULTS AND DISCUSSION

Sectioning of the extrudate (Fig. 3) revealed that a major component of the flow within the die was under plug flow conditions, a result that was consistent with extrusion studies by Ovenston & Benbow (1968) on clay-like materials.

The initial experiment undertaken was to determine the effect of the displacement of the piston, during ram extrusion, on the convergent flow patterns within the barrel. The results for a microcrystalline cellulose/lactose blend mixed with water (Fig. 4) show that the angle of convergence, as indicated by the angle of entry into the die, remains independent of the displacement, provided that the extrusion force remains constant (i.e. steady state

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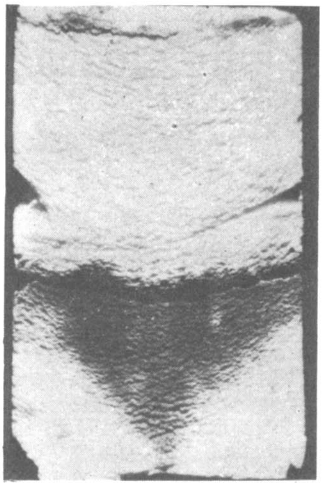

FIG. 1. Convergent flow pattern within the level of the ram extruder after extrusion of a 1 : 1 : 1 mixture of microcrystalline cellulose : lactose and water.

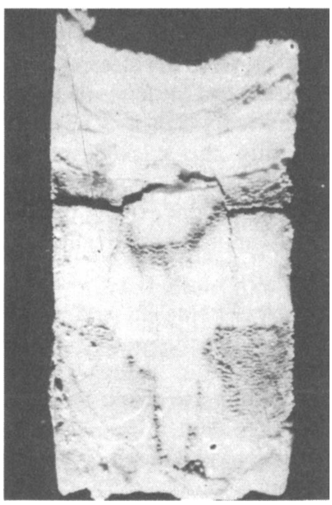

FIG. 2. Convergent flow pattern within the level of the ram extruder after extrusion of a mixture of lactose and water.

flow stage). However, when the extrusion force changes with displacement (i.e. forced flow stage) the constant angle is no longer maintained, presumably because of the close proximity of the piston to the die. Both stages were obtained for all microcrystalline cellulose : lactose : water and microcrystalline cellulose : water mixes but for lactose : water mixes steady state flow conditions could not be obtained and the convergent flow pattern for this system (Fig. 2) was similar to the patterns in the

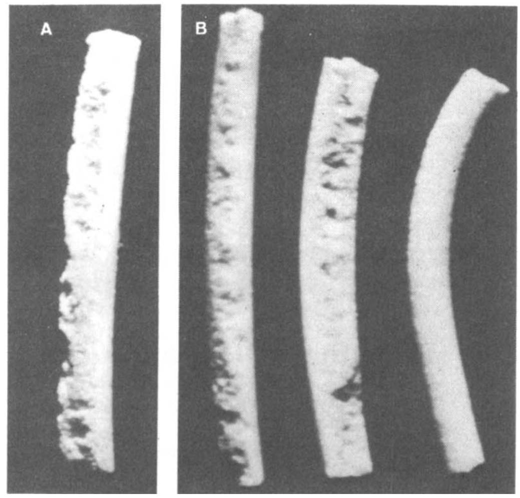

FIG. 3. Flow pattern within an extrudate—(A) Microcrystalline cellulose : lactose : water in a 1 : 1 : 1 mixture extruded through a 1 mm die. (B) Microcrystalline cellulose : lactose : water in a 5 : 5 : 6 mixture extruded through a 1 mm die.

forced flow stage in Fig. 4. The measured convergent angle under steady state flow conditions, for single-holed die arrangements (Figs 5–6), indicates that the angle is essentially independent of the process conditions (barrel diameter, die geometry, velocity of throughput and small changes in the moisture content of the material mix), but dependent on the formulation of the material blend. It can, in consequence, be suggested that there will be a yield plane created within the material before the flow and that this yield plane will be equivalent to the angle of convergence. The value for the angle of convergence found for a microcrystalline lactose : water mix can be seen (Fig. 6) to be greater than a microcrystalline cellulose/lactose/water mix. This would suggest that

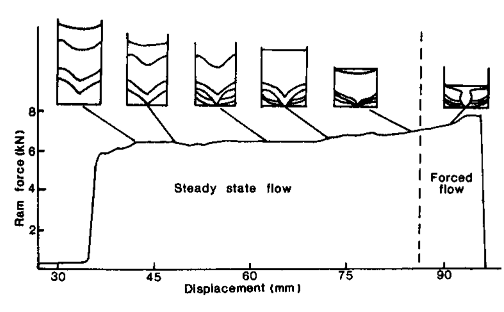

FIG. 4. Force displacement profile showing the convergent flow pattern into the die for microcrystalline cellulose : lactose : water (1 : 1 : 1). Conditions—Ram speed 4 mm s^{-1}: die diameter, 2 mm: die length to radius ratio 12.

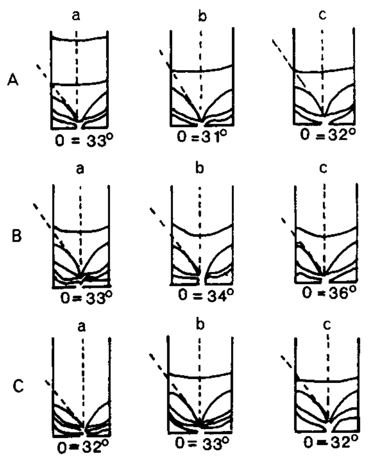

FIG. 5. Analysis of the convergent flow patterns during steady state flow under varying operation conditions for microcrystalline cellulose : lactose : water (5 : 5 : 5) mixtures in a 2·54 cm diameter barrel. (A) Ram speed 3·0 mm s^{-1}, extended through dies of length to radius ratios of 12 for dies of diameters (a) 1·0, (b) 1·5 and (c) 2·0 mm. (B) Ram speed 3·0 mm s^{-1}, extruded through a die of 1·0 mm diameter with a die length to radius ratio of (a) 4, (b) 12 and (c) 16. (C) Material extruded through 1 mm diameter dies of length to radius ratio of 12 at (a) 2, (b) 4 and (c) 6 mm s^{-1}.

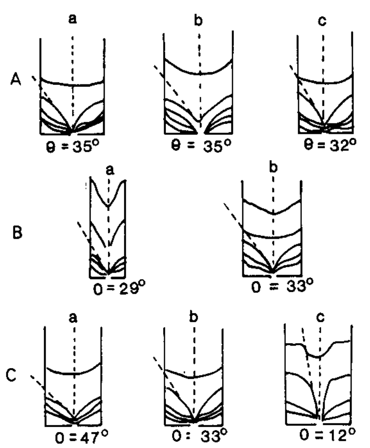

FIG. 6. Analysis of the convergent flow pattern during steady state flow under varying operating conditions for microcrystalline cellulose : lactose : water mixtures. Ram speed 3·0 mm s^{-1} and die length to diameter ratio 12. (A) At moisture contents of (a) 33·9, (b) 37·9 and (c) 40·5% w/v. (B) Barrel diameters of (a) 1·25 and (c) 2·54 cm. (C) Proportion of microcrystalline cellulose : lactose : water of (a) 9 : 0 : 11, (b) 5 : 5 : 6 and (c) 0 : 4 : 1.

the former would be more easily extruded than the latter, and actually found experimentally (Harrison 1982). Unfortunately it was not possible to record a value for the convergent angle for the multiholed die

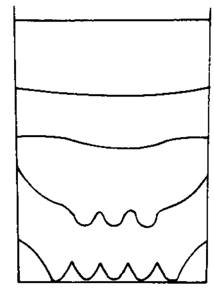

FIG. 7. Analysis of the convergent flow patterns for microcrystalline cellulose : lactose : water (5 : 5 : 6) mixtures extruded through multiholed dies of diameter 2·0 mm, length to radius ratio of 8 in a barrel of 2·54 cm at a ram speed of 8·0 mm s^{-1}.

arrangements because the height of convergent flow pattern created at each hole was severely reduced (Fig. 7). This clearly indicates that the extent of the convergent pattern produced is dependent upon the ratio of the area of dead space to the area of open die. In conclusion it can be seen that both the type of flow (i.e. steady state or forced) and the relative ease of flow can be assessed by determining the convergent flow pattern and the angle of convergence on any material mix for which it is possible to insert coloured dyes into the packed material before extrusion. This should be of use in the design of tapered dies to reduce the extrusion force required and subsequently improve the quality of the extrudate.

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