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Note

The production of homogeneous extrudates of microcrystalline cellulose pastes

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Abstract

The homogeneity of water-based microcrystalline cellulose (MCC) paste extrudates was investigated during ram extrusion as a function of ram velocity. Variations in the water content of the extrudates were caused by liquid phase migration within the paste. The evolution in water content was measured by sectioning and drying the extrudate, and the subsequent homogeneity was quantified by the standard error in water content. The homogeneity of the extrudates was found to decrease as the ram velocity decreased. This result was also inferred from the rate of increase of the extrusion pressure. The extrudate homogeneity was significantly improved by compensating for water migration in the barrel during the compaction stage. This was achieved using a non-uniform initial paste billet, created by packing the barrel with layers of paste of different water contents. This technique also produced a smaller variation in extrusion pressure over the ram displacement range, and a reduction in water loss from the upstream paste compact into the extrudate and/or through the apparatus tooling.

Keywords: Paste extrusion; Liquid migration; Microcrystalline cellulose

The ram extrusion of pastes is frequently used in the pharmaceutical industry to produce extrudates that are further processed into spherical granules using a spheroniser (Vervaet et al., 1995). At an optimum extrudate water content, spherical granules are generated with a narrow size distribution, which are ideal for capsule filling. A major problem encountered in the ram extrusion of pastes is liquid phase migration, when the pressure developed on the liquid phase causes it to move relative to the solids. This phase redistribution can result in variations in the local liquid content within the extrudate and the barrel paste compact, leading to poor final product quality (Bains et al., 1991).

In a previous study by Rough et al. (2000), it was shown that liquid phase migration during the ram extrusion of an initially uniform water-based MCC paste created an extrudate of decreasing water content. For a given die geometry, the uniformity of the extrudate water content ultimately depended upon the ram compaction velocity prior to extrusion. During the compaction stage, a gradient of water content was generated down the barrel paste compact, with the wettest section occurring at the bottom next to the die entry. By studying the extrudate water contents measured for an initial 50 wt.% water-MCC bulk, it was surmised that the moisture gradient in the barrel paste prior to extrusion could be lessened by packing the barrel with

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layers of 48, 49, 50, 51, and 52 wt.% water content, starting with the 48 wt.% paste at the bottom of the barrel next to the die entry. Hence, during the compaction stage, it was assumed that the water would redistribute to produce a uniform 50 wt.% compact prior to extrusion, which would subsequently produce an homogeneous 50 wt.% extrudate.

Pastes containing MCC powder (Avicel PH101, FMC Corp., $d_{50} = 50.75\,\mu m$) and deionised water were prepared using a "Moulinex Ovatio 3" food processor (blade radius 80 mm) operating at 1350 ± 50 rpm for 120 s. Some examples of the granules produced are shown in Fig. 1. The extrusion experiments were carried out using a Dartec strain frame (Burbidge et al., 1995). Paste was loaded into a cylindrical stainless steel barrel (internal diameter, 25 mm) to produce a compact of initial height ~160 mm, and extruded through a concentric cylindrical square-entry die of 3 mm diameter and 48 mm length. The ram travelled 100 mm at a given velocity ranging from 0.833 to 0.0167 mm s^{-1}. The extrudate was collected at 10 mm ram displacement intervals, and the water contents determined by drying in an oven at 60 °C. After extrusion, the barrel paste compact (~60 mm high) was ejected and cut into 10 mm slices, and the water contents determined.

An example of some extrusion profiles is displayed in Fig. 2, and the range of extrusion pressures are listed in Table 1 as a function of ram velocity. For the initial 50 wt.% uniform bulk, the extrusion pressure increases with ram displacement for all velocities. At any given velocity, the layered pastes feature higher initial extrusion pressures, indicating a less wet extrudate exiting the die. More notably, the extrusion profiles for the layered compacts have lower pressure–displacement gradients. The rate of change of the extrusion pressure can be used as a measure of the extent of liquid migration (Rough et al., 2000). These gradients are plotted in Fig. 3, and the values for the layered pastes at any given ram velocity are all significantly lower than those for the uniform 50 wt.% paste.

For the uniform 50 wt.% paste, it was found that the water content of the extrudate decreased during the extrusion process, and that the first section to exit the die was always of a higher water content than 50 wt.%. As the ram velocity decreased, the homogeneity of the extrudate in terms of water content decreased. Fig. 4 shows an example of the extrudate

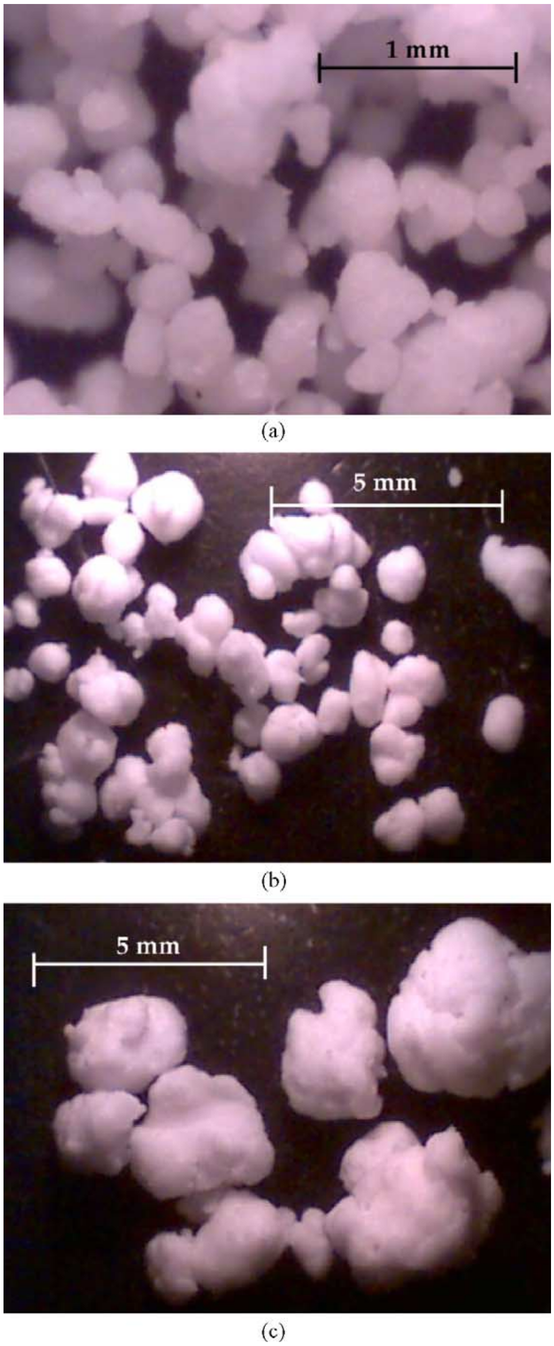

Fig. 1. Micrographs of water-MCC granules produced in a high shear mixer using water contents of: (a) 45 wt.%, (b) 50 wt.% and (c) 55 wt.%.

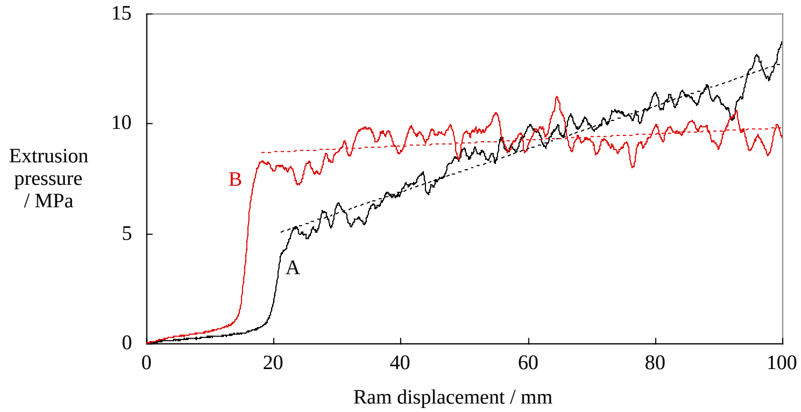

Fig. 2. Extrusion pressure–ram displacement profiles for MCC paste at a ram velocity of 0.0833 mm s^{-1} (48 mm × 3 mm die) with initial bulk water contents of: (A) 50 wt.% in one layer and (B) 48, 49, 50, 51, 52 wt.% in five layers. Dotted lines indicate best fit linear trendlines from onset of extrusion.

Table 1
Extrusion pressures and overall water contents of barrel paste for water-MCC at various ram velocities (48 mm × 3 mm die, 100 mm ram displacement)

Ram velocity (mm s^{-1})	Extrusion pressure range (MPa)		Barrel paste water content (wt.%)	
	Uniform bulk	Five layers	Uniform bulk	Five layers
0.833	10–17	17–13	49.4	51.7
0.333	6.9–13	8.3–10	48.6	50.6
0.167	3.7–12	9.3–8.6	48.2	50.9
0.0833	4.4–14	8.3–9.4	46.7	49.1
0.0333	4.7–16	6.5–9.2	44.7	47.1
0.0167	4.6–19	5.6–11	42.4	45.7

Uniform bulk = 50 wt.% initially 160 mm high; five layers of 48, 49, 50, 51, 52 wt.% each initially 32 mm high.

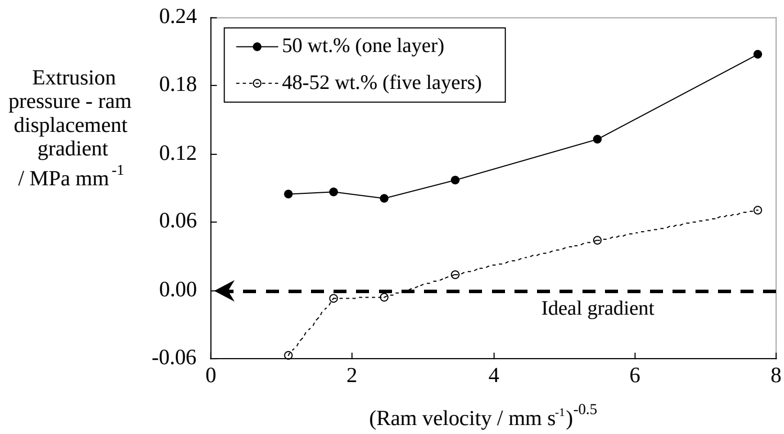

Fig. 3. Extrusion pressure–ram displacement gradient vs. (ram velocity)$^{-0.5}$ for MCC paste (48 mm × 3 mm die, 100 mm ram displacement).

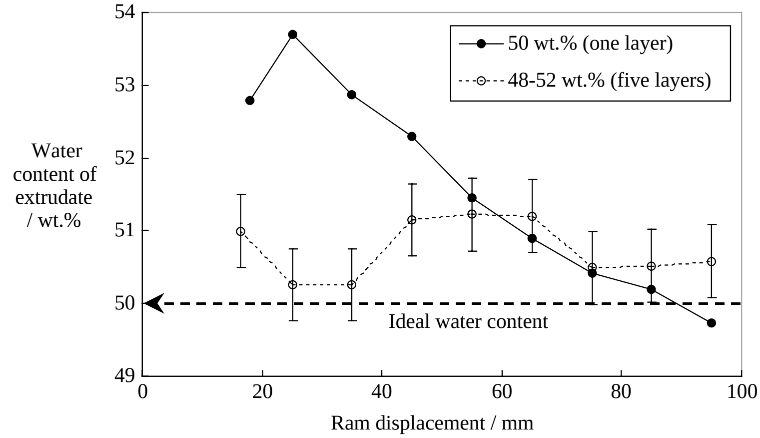

Fig. 4. Extrudate water contents for MCC paste at a ram velocity of $0.333\,mm\,s^{-1}$ ($48\,mm \times 3\,mm$ die). For clarity, error bars shown for one series only.

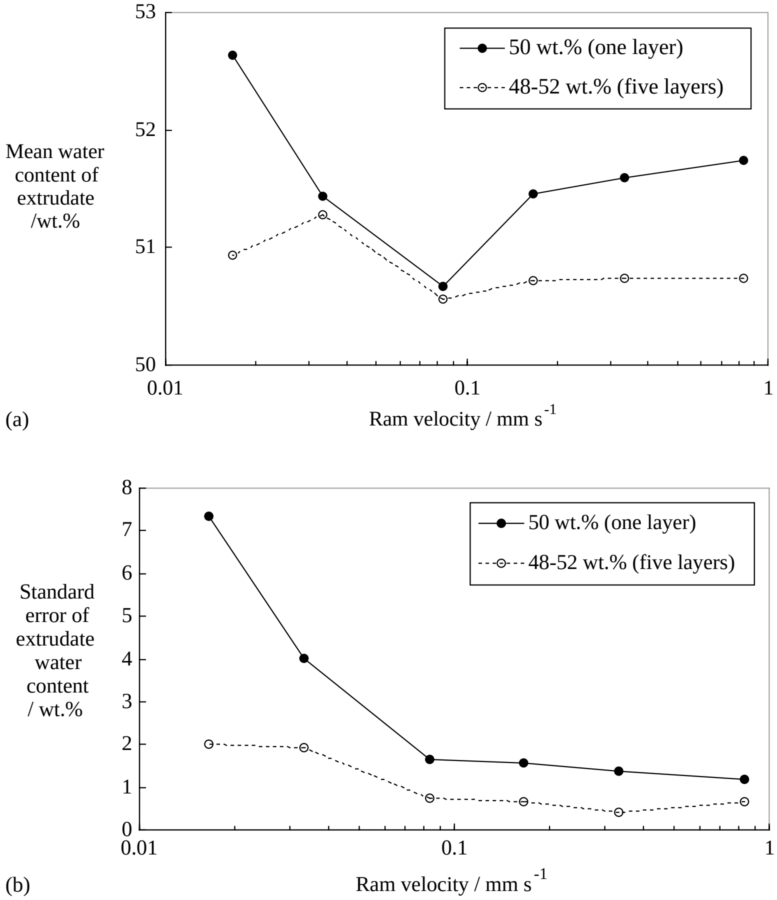

Fig. 5. Plot of: (a) mean and (b) standard error of extrudate water content against ram velocity for MCC paste ($48\,mm \times 3\,mm$ die, 100 mm ram displacement).

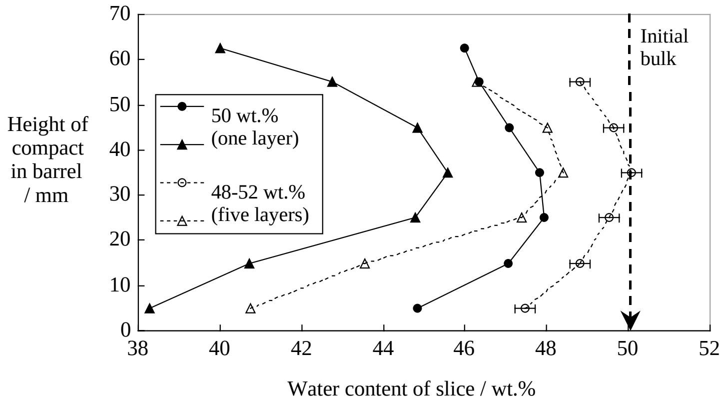

Fig. 6. Average water contents of 10 mm slices of barrel MCC paste for ram velocities of: (●, ○) 0.0833 mm s^{-1} and (▲, △) 0.0167 mm s^{-1} (48 mm × 3 mm die, 100 mm ram displacement). For clarity, error bars shown for one data series only.

water contents. For a given ram velocity, the initial extrudate water contents for the layered compact are lower and more consistent, and the final extrudate water contents are higher than those measured for the uniform 50 wt.% extrusion. The difference in the maximum and minimum water contents is a direct measure of the extrudate homogeneity, which can be further quantified by calculating the mean and standard errors. These values are shown in Fig. 5 as a function of ram velocity, and the results confirm that the layered pastes produce extrudates with mean water contents nearer to 50 wt.% and with smaller standard errors.

Examples of the water content profiles of the barrel paste after extrusion are illustrated in Fig. 6. For a given paste arrangement, the water content of a paste slice at a given height in the barrel decreases with decreasing ram velocity. At a given height, the slices for the layered compact all feature higher water contents than those for the 50 wt.% bulk extrusion. Table 1 lists the overall water contents of the barrel paste, and shows that the layered pastes consistently retain more water.

In summary, ram extrusion experiments incorporating paste layers of different water contents within the barrel, compared to those using an initial uniform paste bulk, provided:

(i) extrudates with a more homogeneous water content, which is an important requirement for downstream processing such as spheronising;
(ii) extrusion profiles with lower rates of pressure increase, which may be crucial in terms of equipment operability and maintenance; and
(iii) a more wet paste remaining in the barrel after extrusion, which may aid in the ejection of the unused compact and subsequent cleaning of the apparatus.

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