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# The rheology of microcrystalline cellulose powder/water mixes – measurement using a mixer torque rheometer

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## Summary

A mixer torque rheometer has been used to study the rheology of microcrystalline cellulose powder/water mixes. As the powder mixture became wetter the measured torque increased to a maximum (corresponding to the stage where the system was just saturated) thereafter decreasing as the mixture became a slurry. The torque and moisture content at saturation varied with both particle size and source of the microcrystalline cellulose.

## Introduction

Microcrystalline cellulose is an essential ingredient in the production of wet powder masses suitable for extrusion and spheronization (Reynolds, 1970; Conine and Hadley, 1970; Rowe, 1985). Indeed, for the optimum product, the amount of water added to the powder must also be strictly controlled (Miyake et al., 1973; Malinowski and Smith, 1975). Since both these observations imply a specific rheological requirement of the wet powder mass a method which measures a rheological parameter of the mass should be of use not only in understanding the process but also of distinguishing between samples of microcrystalline cellulose from different sources. Recently Harrison et al. (1985, 1987) have shown that it is possible to study formulation variables by using capillary rheometry but the method is time-consuming especially for studying batchwise variation of ingredients. In this study we have modified and instrumented a small, horizontal mixer and studied the rheology of powder/water mixes of microcrystalline cellulose obtained from several sources.

## Materials and Methods

Microcrystalline celluloses of various grades were obtained from several sources: Avicel PH101, PH102, PH103 and PH105 from FMC Corp., Philadelphia, U.S.A.; Emcocel (3 batches) and Emcocel 90M from Finnish Sugar Co., Helsinki, Finland; and Unimac MG 100 and MG 200 from Unitika Rayon, Osaka, Japan. All were used as received. Moisture contents of the powders were determined by thermogravimetry (Compu-trac, AZ, U.S.A.).

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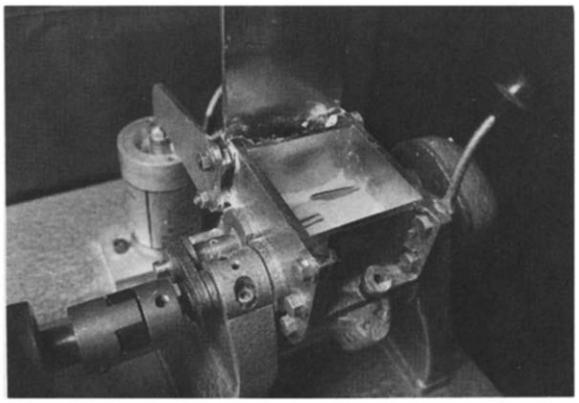

Fig. 1. Photograph of the mixer torque rheometer showing torque bar and dynamometer.

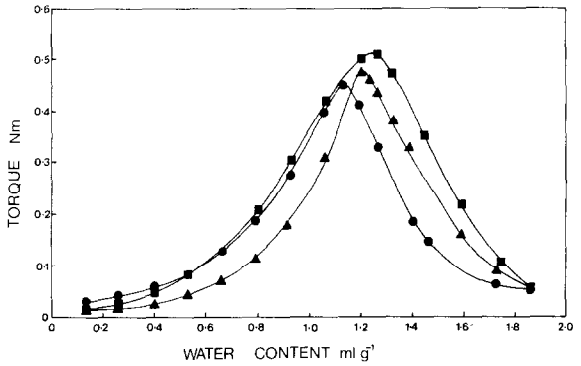


Fig. 2. The effect of increasing water content on the measured torque. ■, Avicel PH101; ▲, Emcocel Bx 2; ●, Unimac MG 100.

The mixer torque rheometer (Fig. 1) was similar to the commercial Plasti-Coder rotational torque rheometer used by Schildcrout (1984). It consisted of a 250 ml capacity jacketed Beken Duplex mixer (Beken Engineering, London) fitted with a 0.09 kW DC variable speed motor (Normand Electrical Co., London). The reaction of the mixing bowl was continually recorded via a torque arm fixed to the main body of the mixer and linked to a calibrated dynamometer. (Type UF2, Pioden Controls, Canterbury U.K.). Torque differences of $5 \times 10^{-3}$ Nm could easily be determined using a chart recorder. 15 g of the powder was added to the mixer followed by a measured amount of water. The mixer was left until steady state conditions occurred (usually 3–5 min) and the torque measured. The measured torque less the torque recorded with the empty mixer was plotted against the amount of water added. A shaft speed of 52 rpm was used throughout.

## Results and Discussion

All the materials exhibited an increase in torque with increasing water content rising to a maximum (Fig. 2) thereafter decreasing as a slurry is produced. This behaviour is consistent with the different states of liquid saturation in an assembly of particles (Newitt and Conway-Jones, 1958). At low liquid levels discrete lens-shaped rings of liquid are formed at the points of contact of the particles (the pendular state). This persists until a continuous network of liquid interspersed with air is formed (the funicular state). Both these states will result in an increasing cohesiveness and hence increased torque on the mixer. At the limit of the funicular state all the pores become filled with liquid (the capillary state) and it is this state that corresponds to the maximum in Fig. 2. Further increase in water results in the formation of a slurry consisting of solid particles dispersed in the liquid. However, although all the materials exhibited similar phase changes, the values of both the maximum torque and amount of water added at this maximum varied with both source and particle size (Table 1).

In the Avicel range, with the exception of Avicel PH103, a specially produced 'low moisture content' grade, the maximum torque and water content at maximum torque decreased with decreasing particle size. This trend was not evident with the Unimac and Emcocel range although, in both these cases, no material of comparable size to Avicel PH105 was available. However, in all cases, when grades of similar size are compared, the Avicel range of materials exhibited both higher torques and higher water contents. The reason for the very high values recorded for Avicel PH103 is unknown although it must be noted that the apparently anomalous behaviour of this grade has also been confirmed by capillary rheometry (Raines, 1986, private communication). The apparent batchwise variation between the Emcocel

TABLE 1

*Data for the materials studied*

| Material | Moisture content (% w/w) | Average particle size * ($\mu$m) | Torque at max. (Nm) | Water content at max. torque | |
|---|---|---|---|---|---|
| | | | | Measured ($ml \cdot g^{-1}$) | Dry weight basis ($ml \cdot g^{-1}$) |
| Avicel PH102 | 5.09 | 90 | 0.512 | 1.26 | 1.32 |
| Avicel PH101 | 4.62 | 50 | 0.511 | 1.24 | 1.30 |
| Avicel PH103 | 2.68 | 50 | 0.533 | 1.33 | 1.37 |
| Avicel PH105 | 4.68 | 20 | 0.481 | 1.21 | 1.27 |
| Unimac MG 200 | 2.53 | 103 | 0.486 | 1.13 | 1.16 |
| Unimac MG 100 | 2.57 | 38 | 0.476 | 1.13 | 1.16 |
| Emcocel 90M | 3.40 | 90 | 0.469 | 1.26 | 1.30 |
| Emcocel Bx 1 | 4.37 | 56 | 0.472 | 1.13 | 1.18 |
| Emcocel Bx 2 | 4.18 | 56 | 0.488 | 1.20 | 1.25 |
| Emcocel Bx 3 | 3.40 | 56 | 0.498 | 1.20 | 1.24 |

* Manufacturer's literature data.

samples needs more explanation since it provides a clue as to the possible reason for the differences seen between the 3 sources of the material. Batch 1 was a batch prepared very early in the production campaign and not considered to be representative, Batch 2 was a sample of standard production and Batch 3 was a special batch produced using a different source of wood pulp. Processing conditions during production could therefore be at least a contributory factor to the variation seen between the sources.

It is interesting to note that the amount of water added at the maximum torque for Avicel PH101 is comparable with that found both for the plastic limit of this material when mixed with water – 1.23 $ml \cdot g^{-1}$ – and for the optimum production of spheroids during spheronization – 1.3 $ml \cdot g^{-1}$ – (Miyake et al., 1973).

The results illustrate that a mixer torque rheometer can be used to detect differences between materials from different sources. The measurements are relatively easy and rapid to undertake and are consistent with the known theories of granulation, extrusion and spheronization.

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