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Characterization of wet powder masses using a mixer torque rheometer. 4. Effect of blade orientation

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Abstract

A commercial mixer torque rheometer has been used to investigate the effect of blade orientation on the two contra-rotating shafts. Experiments have been performed on a model wet mass consisting of microcrystalline cellulose and water. Blade orientation has significant effects on the measured torque responses with respect to water content as well as on the yield stresses and kinematic viscosities calculated by applying the Casson model to mean torque values obtained on testing specific premixed wet masses. It is essential that, inter alia, blade orientation be kept constant if different wet masses are to be directly compared as in batch monitoring or scale-up.

Keywords: Wet granulation; Mixer torque rheometry; Blade orientation; Microcrystalline cellulose

1. Introduction

The rheological properties of pharmaceutical granulations can now be successfully monitored by mixer torque rheometry (Parker et al., 1990a; Rowe and Parker, 1994). This approach has enabled many effects in both the formulation, e.g. substrate source, binder type and concentration (Rowe and Sadeghnejad, 1987; Parker et al., 1990b, 1991, 1992; Parker and Rowe, 1991; Hancock et al., 1994), and processing, e.g. batch variation (Janin et al., 1990) and mixer scale-up (Cliff and Parker, 1990; Landin et al., 1996) of wet granulations to be studied. It is well known in rheology that the measured properties of a sample are highly dependent on the test conditions. Unlike liquid rheology with, for instance, concentric cylinders where a sample is sheared under controlled conditions with a constant gap, measurement in mixer torque rheometry is complicated by the fact that the sample is continuously being compressed and expanded between contra-rotating blades with a changing gap. It is not surprising, therefore, that instrument geometry, mixing kinetics, shaft speed and sample weight all have an effect on the output of mixer torque rheometers (Hancock et al., 1991, 1992; Landin et al., 1995). This study extends the original work by examining the effects of blade orientation, a factor made important by the recent development of a commercial instrument (Caleva MTR, Caleva Process Solutions Ltd., Sturminster Newton) supplied with shafts each with two blades of the same design but oriented differently.

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2. Materials and methods

A simple two-component system consisting of microcrystalline cellulose (Avicel PH101, FMC Corp) and distilled water was chosen since it has been extensively studied in previous work (Hancock et al., 1991, 1992; Landin et al., 1995).

The mixer torque rheometer used was a commercial instrument (Caleva MTR, Caleva Process Solutions Ltd., Sturminster Newton) similar to that described in detail by Parker et al. (1990a) but with an extended speed range and customised software for data acquisition and manipulation. The instrument has two shafts each with two blades of the same design. In one orientation the drive shaft has the blades offset by 30° and the subsidiary shaft blades offset by 90° (30/90 geometry) while in the other the drive shaft has the blades offset by 90° and the subsidiary shaft blades offset by 180° (90/180 geometry).

For one set of experiments 15 g of dry powder were mixed with varying amounts of water at 52 rev./min for 1, 2, 3, 5 and 10 min to obtain a graph of torque vs. water content. In another set of experiments a larger sample of wet mass was produced by mixing 400 g of dry powder with either 320 ml (0.8 ml/g), 400 ml (1.0 ml/g) and 480 ml (1.2 ml/g) of water in a laboratory mixer (Type AE200, Hobart) for 10 min. This was then stored in sealed containers for 24 h before being measured on the mixer torque rheometer at varying shaft speeds. For this measurement 35 g of wet sample were used with a premixing time of 30 s before data acquisition of 20 s.

3. Results and discussion

The blade orientation has a distinct effect on the graphical output of the instrument as shown in Fig. 1 for the same wet mass. The 90/180 geometry produces an output with clearly defined individual peaks in torque produced as the blades intermesh while the 30/90 geometry produces an output where the individual peaks become convoluted showing that the blades are at all times shearing the mass to some degree. The output from the 30/90 geometry is similar to that seen for the mixer torque rheometer used by Parker (1989) and Hancock (1991). Derived data show slight differences in the mean torque (1.31 Nm for the 30/90 geometry and 1.34 Nm for the 90/180 geometry) but large differences in the torque range or amplitude (0.99 Nm for the 30/90 geometry and 1.36 Nm for the 90/180 geometry both calculated for the average of 28 peaks within 60% of the maximum — see Parker et al., 1990a).

This trend, i.e. the measured torque for the 90/180 geometry higher than that for the 30/90 geometry was found for all mixes other than those that were very wet.

The effects of blade orientation in the measured mean torques at varying water contents are shown in Figs. 2 and 3. Both graphs show the same trends with an increase in measured torque as liquid levels were increased up to maximum and

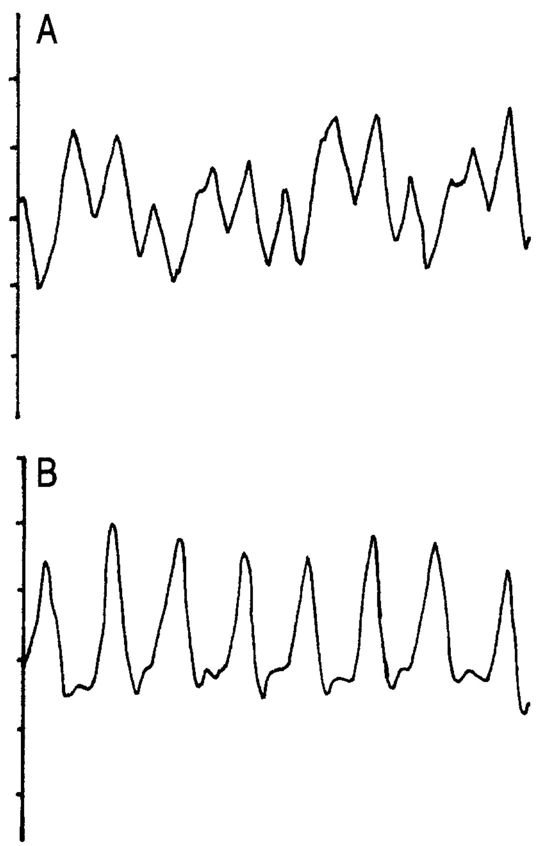

Fig. 1. Graphical outputs for (A) 30/90 geometry and (B) 90/180 geometry.

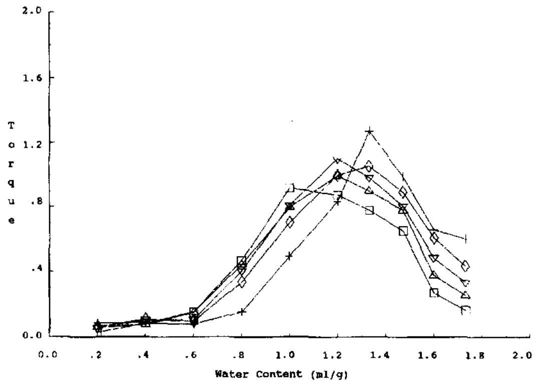

Fig. 2. The effect of mixing time/water content on the mean torque for the 30/90 blade orientation (□, 1 min; △, 2 min; ▽, 3 min; ◇, 5 min; +, 10 min).

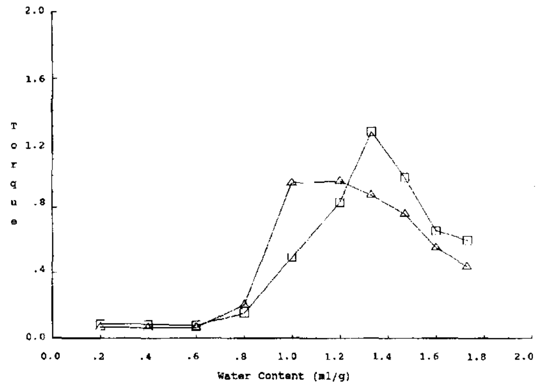

Fig. 4. The effect of water content on the equilibrium torque (□, mean; △, amplitude/range) for the 30/90 blade orientation.

decreasing thereafter. This is consistent with the different states of liquid saturation in an assembly of powder particles as shown previously. If the mixing kinetics (i.e. the changes in torque with time at constant water content) are considered then the trends for both blade orientations are the same as those seen previously (Hancock et al., 1991), i.e. at low water levels there is an initial high level of torque generated at short times followed by a gradual fall at longer times but at high water levels the torque response increases slowly with continued mixing. Of interest are the effects blade orientation has on the equilibrium torque (mean and amplitude/range) responses (Figs. 4 and 5), specifically the relative location and magnitude of the maxima in the mean torque and torque amplitude, since these are considered to relate to the degree of substrate binder interactions occurring in the wet mass (Hancock et al., 1994). For both orientations the location of the maxima in the mean torque response is the same — 1.33 ml/g similar to previous work with microcrystalline cellulose. However, the location in the maxima in the torque amplitude is different occurring at a slightly lower water content for the 30/90 geometry (1.1 ml/g) as compared with the 90/180 geometry (1.2 ml/g). The shape of the torque amplitude response for the 30/90 geometry is similar to that seen previously (Hancock et al., 1991).

Based on the hypothesis put forward by Hancock et al. (1994) that the maximal values will be

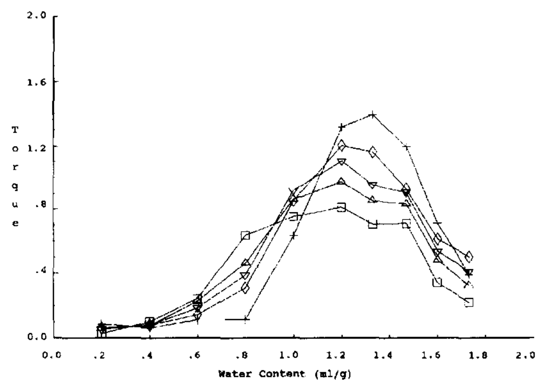

Fig. 3. The effect of mixing time/water content on the mean torque for the 90/180 blade orientation. Symbols as for Fig. 2.

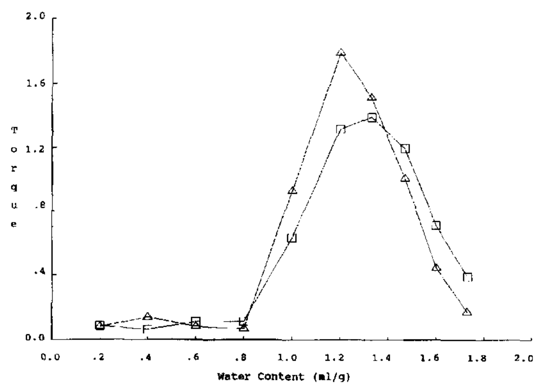

Fig. 5. The effect of water content on the equilibrium torque (□, mean; △, amplitude/range) for the 90/180 blade orientation.

furthest apart for a perfect wetting/spreading system and close together for a poor wetting/spreading system, it can be seen from Figs. 4 and 5 that different conclusions could be drawn for this simple two-phase system of microcrystalline cellulose and water. Obviously this cannot be the case and hence the differences seen in this study must be due to relative efficiencies of mixing of the two blade orientations affecting the rate controlled processes of wetting and spreading. Although the 90/180 geometry would appear to impart the higher shear as indicative of the higher mean torque values to the wet mass, the 30/90 geometry would appear to be the more efficient mixing orientation. This is not surprising since this geometry is similar to that used in Beken Duplex mixers claimed to produce homogeneity comparable with roller and ball mills.

In a recent paper Landin et al. (1995) suggested the adoption of specific rheological models to analyse the mean torque data from shaft speed studies. Three models were proposed:

(1) The logarithmic model:

$$\tau = \tau_y + k\,\alpha \qquad (1)$$

where τ is the measured shear stress calculated from the mean torque, τ_y is the yield stress of the wet mass (i.e. the stress required to initiate flow), k is the kinematic viscosity constant and α is the shear rate as calculated from shaft speed.

(2) The Herschel-Bulkley model (Herschel and Bulkley, 1926)

$$\tau = \tau_y + k\alpha^n \qquad (2)$$

where n is the power law constant proportional to the degree of non-Newtonian flow. This model can only be solved iteratively as it requires the solution of three unknowns from two variables.

(3) The Casson model (Casson, 1959)

$$\tau^{1/2} = \tau_y^{1/2} + k^{1/2}\alpha^{1/2} \qquad (3)$$

i.e. it assumes a power law constant of 0.5 (see Eq. (2)). Data on the effect of shaft speed on the mean torque of three premixed formulations of microcrystalline cellulose and water for the two blade orientations are shown in Figs. 6 and 7. Analysis of the data was carried out using the Casson model since the logarithmic model, although producing very high correlation coefficients, produced a negative intercept — a physical impossibility since it is known that such masses do have a yield stress (Harrison et al., 1987) — and the Herschel-Bulkley model was difficult to apply since the analysis would not produce sensible figures and was unable to converge. Results for the Casson model using the geometric constants given by Landin et al. (1995) are shown in Table 1.

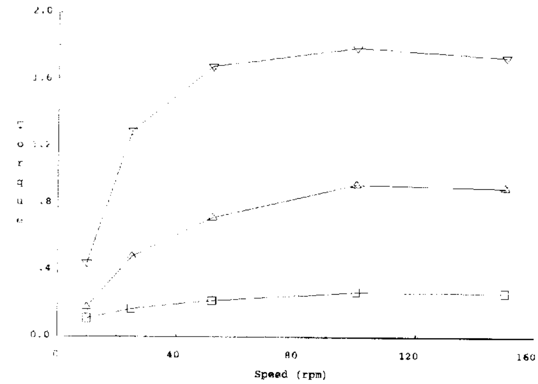

Fig. 6. The effect of speed setting on the mean torque for masses containing: □, 0.8 ml/g; △, 1.0 ml/g; ▽, 1.2 ml/g, using the 30/90 blade orientation.

It can be seen that as the water content of the wet mass is increased both the yield stress and kinematic viscosity increase for both blade orientations. However, apart from the wettest mass tested where there is no statistical differences be-

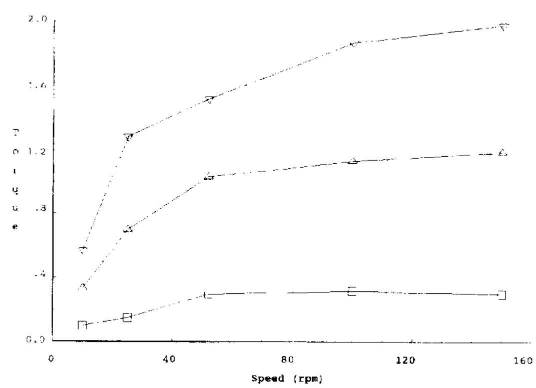

Fig. 7. The effect of speed setting on the mean torque for masses containing: □, 0.8 ml/g; △, 1.0 ml/g; ▽, 1.2 ml/g, using the 90/180 blade orientation.

Table 1
Values (± S.E.) for yield stresses (τ_y), kinematic viscosities (k) and correlation coefficients for three wet masses measured using two blade orientations

	Blade geometry	
	90/80	30/90
Mass 0.8 ml/g		
τ_y (Pa)	1350 ± 109	1664 ± 21
k (Pas)	742 ± 97	441 ± 19
r	0.8479	0.9422
Mass 1.0 ml/g		
τ_y (Pa)	4914 ± 258	2339 ± 268
k (Pas)	2990 ± 230	3456 ± 239
r	0.9014	0.9102
Mass 1.2 ml/g		
τ_y (Pa)	8683 ± 345	8579 ± 714
k (Pas)	4514 ± 345	3986 ± 689
r	0.9114	0.8115

tween the results produced by the two blade orientations, differences are seen for the other masses although with no trend except that indicated by the correlation coefficient. At very low water contents, i.e. with a dry mix, the 30/90 geometry has a higher correlation coefficient than the 90/180 geometry but whereas the correlation coefficient increases with increasing water content for the 90/180 geometry it decreases with the 30/90 geometry. It is interesting to note that Landin et al. (1995) using the 90/180 geometry obtained a correlation coefficient of 0.9672 for the Casson model for a wet mass of microcrystalline cellulose (Avicel PH101) containing 1.3 ml/g water.

It is apparent from the results that blade orientation can have a significant effect on the torque response and inferred behaviour of a wet powder mass. The results are not predictable and hence it is essential that, inter alia, blade orientation is kept constant if different wet masses are to be directly compared, e.g. as part of an experiment to investigate scale-up, batch/source variation of excipients, etc.

Acknowledgements

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