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Granulation monitoring in a planetary mixer using a probe vibration analysis technique

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Summary

A method is described for monitoring wet massing or granulation in a planetary mixer, using a probe system. Movements or vibration of the probe during massing or granulation were monitored according to changes in displacement, velocity or acceleration using a vibration level monitor. It was found that gross changes occurring during massing and granulation could be monitored most sensitively using displacement or velocity measurements. Product sampled after different granulation times or after different volumes of granulating fluid were added was analyzed according to particle size and packing density as a method of assessing changes which could be related to specific regions of the vibration record. It was found that generally the granulation end-point corresponded with formation of a plateau region in vibration analysis; further mixing or addition of fluid caused a second increase in vibration signal as an over-wet mass was formed.

Introduction

Preparation of moist granules from a dry powder occurs by distribution of a granulating fluid between particles. This agglomeration or size enlargement is a rate process which is dependent on the efficiency of fluid distribution and effectiveness of particle impacts. Several variables have been suggested as influences on the formation of moist granules (Holm, 1984) and these include: process variables such as speed of agitation of the powder bed; load of the mixer; liquid addition method; liquid flow rate; wet massing time; and amount of liquid added. Product variables such as: particle size distribution; wettability of particles; solubility of particles in granulating liquid; type of binder material used; and binder concentration may also influence granule formation.

For many granulation processes, most of the above variables will be set constant through use of an unchanging process and a tightly controlled raw material specification. A specific process can therefore be monitored by measuring granule characteristics or granule-induced changes, relative to one of 2 variables: either granulation time or volume of granulating fluid added.

Several different induced changes have been used as methods of monitoring the granulation process and some are listed below.

(i) Power. The amount of power which drive motors consume as particles become agglomerated into granules can be measured using power meters

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or compensated power meters (Lindberg et al., 1974; Leuenberg, 1982).

(ii) Torque. The change in torque or resistance to the turning moment of a tangential force exerted by the granule bed on the drive impeller can be measured using a dynamometer system (Lindberg et al., 1974; Travers et al., 1975).

(iii) Moisture. The change in moisture levels and distribution can be measured using a capacitive sensor to detect power losses from a tuned RF circuit arising from changes in moisture levels which alter the dielectric of the sensor area (Fry et al., 1984).

(iv) Probes. A probe placed within the bowl of a high speed mixer granulator close to the impeller in a constant velocity region can be used to detect changes occurring during granulation. The probe arm is fitted with a set of strain gauges supported on a cantilevered shaft above the target area and records alterations in granule momentum (Kay and Record, 1978).

In the present study, a probe system was used which detected changes in vibration characteristics during granulation carried out in a planetary mixer.

Theory

Vibration signals have 4 major characteristics in addition to frequency these are: displacement (l); velocity ($l \cdot t^{-1}$) and acceleration ($l \cdot t^{-2}$). Displacement is the distance a body, in the present study a pivoted spring target, moves from a reference position. If the vibration was a pure sinusoidal wave form, the displacement, x, would be given by:

$$x = x_{peak} \cdot \sin(2 \cdot \pi \cdot f \cdot t) \quad (1)$$

where f is frequency, t is time, and x_{peak} is the maximum displacement from the reference position, since $2\pi f = w$, the angular frequency,

$$\text{then } x = x_{peak} \cdot \sin(w \cdot t) \quad (2)$$

However, the vibration profiles obtained in the present are more complex than simple sine waves and are more accurately described by a Fourier transformation of Eqn. 2:

$$F_{(t)} = x_0 + x_1 \cdot \sin(w \cdot t + Q_1) + x_2 \cdot \sin(2 \cdot w \cdot t + Q_2) + x_3 \cdot \sin(3 \cdot w \cdot t + Q_3) + x_n \cdot \sin(n \cdot w \cdot t + Q_n) \quad (3)$$

Velocity is the rate of change of this displacement and acceleration is the rate of change of the velocity.

In a system where a target is acted on by a set of granules, the change of displacement, velocity or acceleration of the target will depend on the influence of granule properties on the external forces acting on the arm-target, shown in Fig. 1. The external forces include the arm-mass acting on the target mass, m, and the suspension force, s, acting on the arm at the point of support. The suspension force exerts no force in the direction of displacement since its point of application is on the axis of rotation. The target alone exerts a torque, but this is normal to the displacement and can be considered analogous to a torsional pendulum. The angular displacement, θ, of the torsional pendulum from its equilibrium position

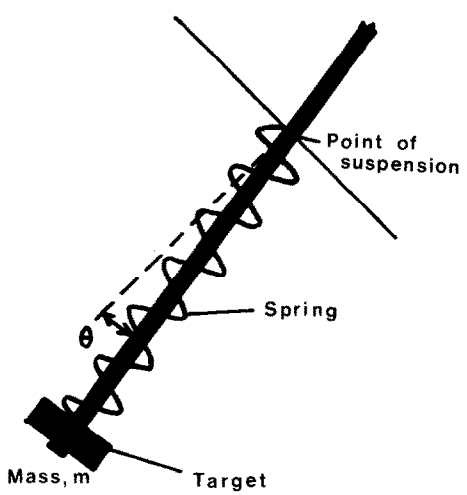

Fig. 1. Torsional pendulum arrangement consisting of helical spring and target mass, m. The angular displacement θ, is considered positive if the deflection of the target is to the right.

is small so that the restoring torque (τ) is proportional to this displacement, according to eqn. 4:

$$\tau = -\kappa \cdot \theta \tag{4}$$

where κ is the torsional constant of the spring ($Nm \cdot rad^{-1}$). The equation of motion of the probe target on which the torque acts is:

$$\frac{I \cdot d^2\theta}{dt^2} = -\kappa \cdot \theta \tag{5}$$

(for simple sinusoidal oscillation:

$$w = \sqrt{\kappa/I)}$$

Eqn. 5 shows that both acceleration and granule inertia, and hence granule mass, will influence the angular displacement of the probe target. Since the sprung arm acts as part of the target zone, displacement of the arm should be disproportionately increased as the viscosity of the granulation increases: the granules will experience greater difficulty in moving through the spring coils without exerting a significant drag force as granulation proceeds.

Methods and Materials

Moist granulation was carried out using: (i) 2.5 kg powdered crystalline α-stable lactose monohydrate, B.P. grade (Serolac, Dairy Crest, Surrey, U.K.), 250 ml distilled water was used as granulating fluid; and (ii) 1 kg microcrystalline cellulose (type PH 102, Avicel, FMC, Philadelphia, U.S.A.).

Different volumes of starch mucilage containing approximately 30% w/w maize starch in water were prepared and added to these granulations.

Wet massing and granulation was carried out using a planetary mixer (type AE 200, Hobart Mfg. Co., London, U.K.) specially adapted to allow the granulation process to be monitored using vibration analysis.

Vibration analysis was carried out using a piezoelectric accelerometer (Endevco, CA, U.S.A.) fixed to the end of a sprung lever swing arm, the other end of which extended a short distance into the mixing bowl (Fig. 2). The swing arm was constructed from steel studding having a diameter of 5 mm and an overall length of approximately 15 cm of which approximately 3 cm extended into the mixing bowl. The swing arm was held in place by a single washer and bolt on the outside of the mixing bowl acting against another washer and bolt on the inside of the bowl and separated by a stiff spring. The spring arrangement acted as a target area and also returned the swing arm to a neutral position following a given deflection. The maximum deflection possible was 10 cm during wet massing and granulation.

The probe was inserted through the mixing bowl so as to occupy the space in the middle of the lowest paddle element which had been cut away to prevent the probe target being struck by the moving blade.

The accelerometer was connected to a vibration level monitor, VLM (Derritron Electronics, Hastings, U.K.) which was in turn connected to a potentiometric recorder (Servoscribe 15, Venture, U.K.).

During mixing and granulation, the powder mass was moved around the bowl, striking the probe target. The movement of the target was measuring using the accelerometer. The current generated by the accelerometer when deflected by

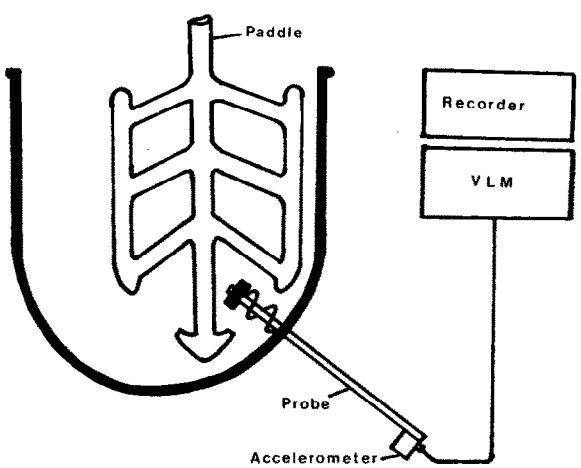

Fig. 2. Schematic diagram of the planetary mixer and vibration analysis equipment, showing the arrangement of the accelerometer probe arm and the modified mixing paddle.

a powder particle or granule was amplified and integrated in the VLM which output a signal corresponding to either peak velocity, peak-to-peak displacement or RMS acceleration. Vibration profiles were obtained using the chart recorder.

Size analysis, friability testing and bulk density determinations were carried out on samples of granules removed from the planetary mixer bowl at different intervals, results from these analyses were compared with equivalent vibration profiles to allow correlation of changes in profiles with formation and growth of granules. Scanning electron micrographs of sample granules were obtained to allow quantitative comparison with vibration profiles.

Results and Discussion

During granulation, the lactose-based system showed an immediate increase in displacement values monitored after bulk addition of 250 ml water, reaching a maximum after the first 5–10 s massing (Fig. 3). This initial peak was occasionally followed by a fall in displacement, rising again to a plateau level after approximately 30 s massing. Fluctuations in displacement about the mean plateau level were caused by intermittent proximity of paddle and probe. This plateau level corresponded with a virtually steady displacement region which was maintained until the stage where the granule mass became over-wet. Significant

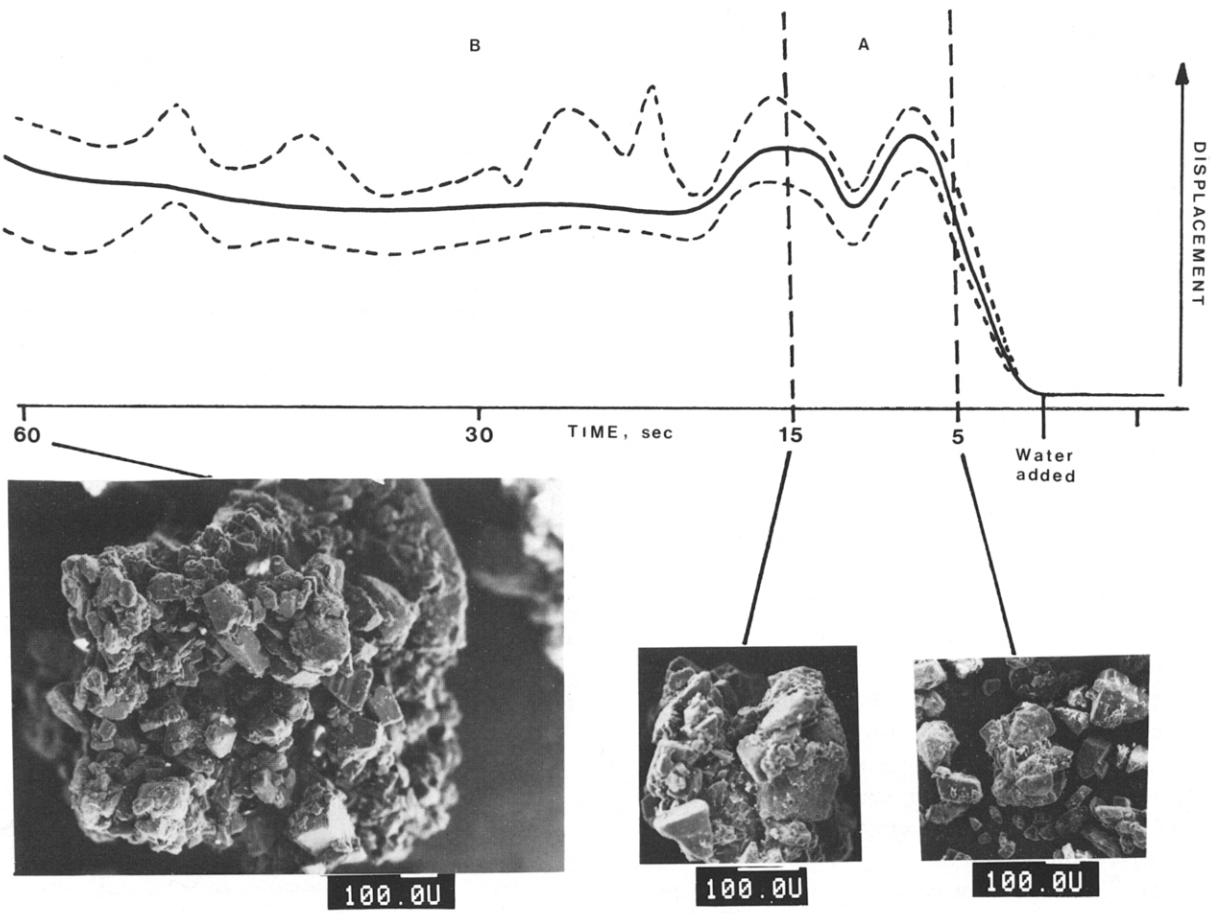

Fig. 3. Vibration profile for a lactose/water granulation. The recording shows average peak-to-peak displacement (———) and displacement envelope (- - - - -). Scanning electron photomicrographs show representative specimen product removed at different stages during granulation.

granule agglomeration or ball growth occurred in this region causing a second increase in mean displacement level following 60–120 s massing.

Sieve analysis of the granules collected after 5 m massing (region A, Fig. 3) showed that the principle mode was between 125 and 250 μm, with over 35% of particles in this range. Fewer than 10% of particles had diameters greater than 710 μm (Fig. 4). After 15 s massing, region B of Fig. 3, corresponding to granule formation, the percentage of particles in the diameter range 0–250 μm fell from 70 to 25% of total frequency. In comparison, the percentage in the range 355–710 μm increased from approximately 11–25%. Similarly, for all granules greater than 355 μm, the change is from approximately 18% after 5 s massing to 62% after 15 s. Further changes in the fine particle and coarse granule frequencies occurred with longer mixing times, although these were not so marked as the changed noted in displacement profiles. It is considered that beyond a certain granule size, changes in particle packing or density may be more significant than changes in diameter. A method of characterizing particle packing using bulk density measurements is provided by determination of the Hausner ratio (H) for a given powder or granule system (Eqn. 6).

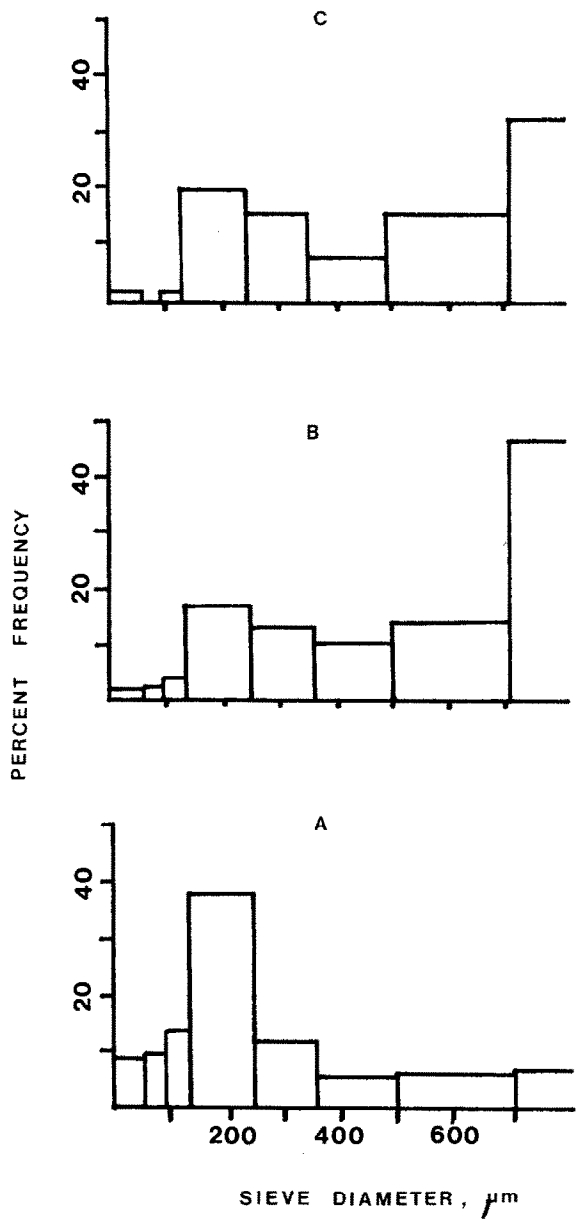

Fig. 4. Frequency size distributions for samples removed after: (A) 5 s. (B) 15 s, and (c) 60 s, granulation time.

$$H = \frac{P^B_{Eq}}{P^B_L} \qquad (6)$$

where P^B_{Eq} is the equilibrium consolidated bulk density and P^B_L is the loose bulk density. It was found that there was a large decrease in Hausner ratio values at approximately 15 s massing — the period indicated by probe displacement values to be the granule formation region. A smaller fall in Hausner ratio occurred over the region 60–120 s, identified as the region where over-wetting occurred.

For Avicel–starch mucilage granulations, vibration profiles were recorded as changes in displacement values with increasing volume of starch mucilage added by dumping 100 ml aliquots into the Avicel granulation.

The profiles of peak-to-peak displacement showed a steady increase in vibration displacement with increasing volumes of starch mucilage up to 700 ml (Fig. 5). Between 700 and 900 ml a plateau region was reached where displacement values fluctuated about an approximately steady mean. During this phase further addition of starch mucilage produced no significant change in displacement. Addition of volumes of 1 000 ml and over caused a second increase in displacement

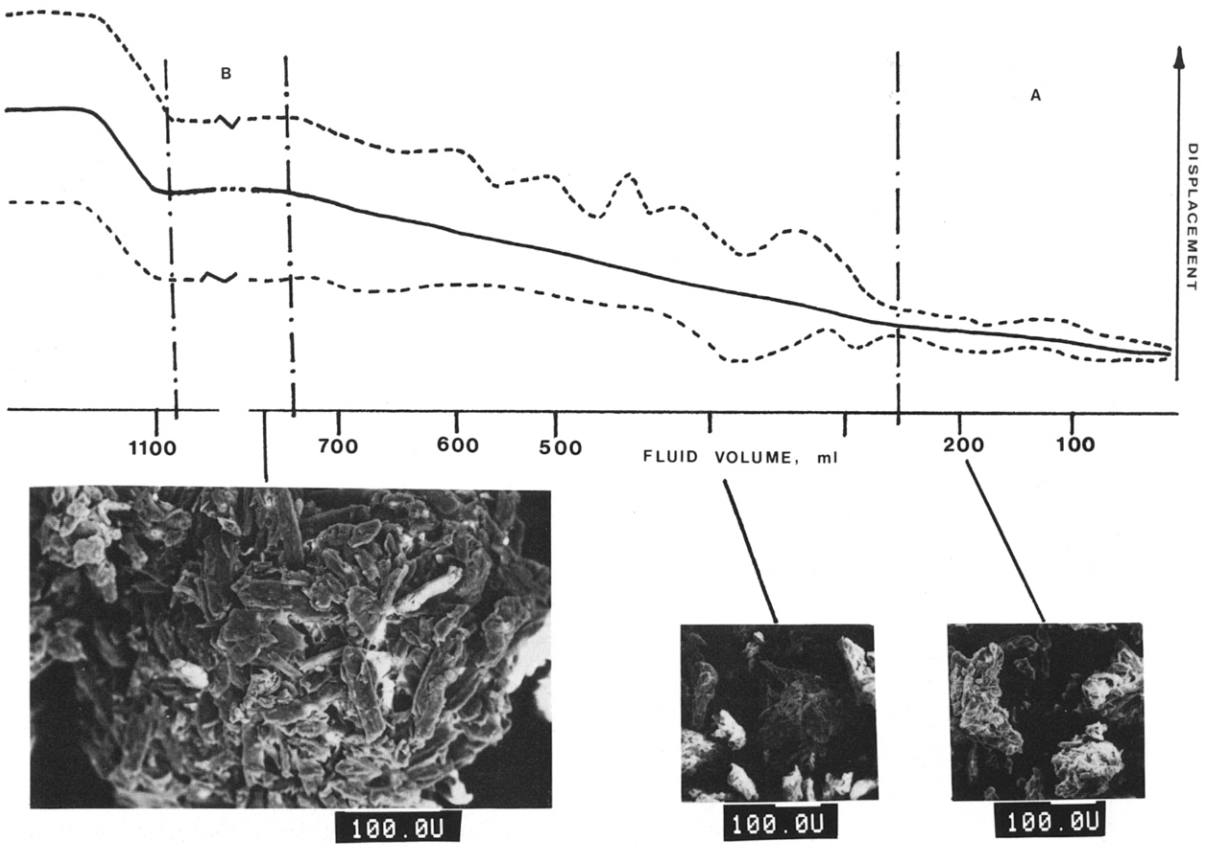

Fig. 5. Vibration profile for an Avicel/starch mucilage granulation. The recording shows average peak-to-peak displacement (———) and displacement envelope (- - - - - -). Scanning electron photomicrographs show representative specimen product removed at different stages during granulation.

values and on examination of the granulation it was found that an over-wet mass was formed.

Sieve analysis of the granules collected from region A (Fig. 5) shows a positively skewed tri-modal distribution with 95% of material having diameters less than 250 μm (Fig. 6). At this stage there is insufficient granulating fluid available to form strong particle agglomerates. As further volumes of granulating fluid are added the size distribution gradually changes from being positively skewed to a bi-modal distribution corresponding to region B (Fig. 5) with significant proportions of larger granules (Fig. 6). Finally, after addition of 1100 ml of granulation fluid a uni-modal negatively skewed distribution was produced (Fig. 6) with approximately 70% of particle agglomerates having diameters > 710 μm. The change in diameter corresponding to the principle mode with volume of granulating fluid is summarized in Fig. 7. It was considered both from qualitative assessment of the wet mass and from the sieve analysis data that granulation was complete after addition of 700–900 ml starch mucilage, the region (B, Fig. 5) corresponding to the plateau displacement levels obtained during vibration analysis.

Bulk density determinations carried out on Avicel–starch mucilage granules showed a rapid decrease in Hausner ratio over the region 600–800 ml (Fig. 8). Decreasing Hausner ratios correspond to denser particle packing, a further indication of granule formation over this region of volume addition.

Use of velocity monitoring in place of displace-

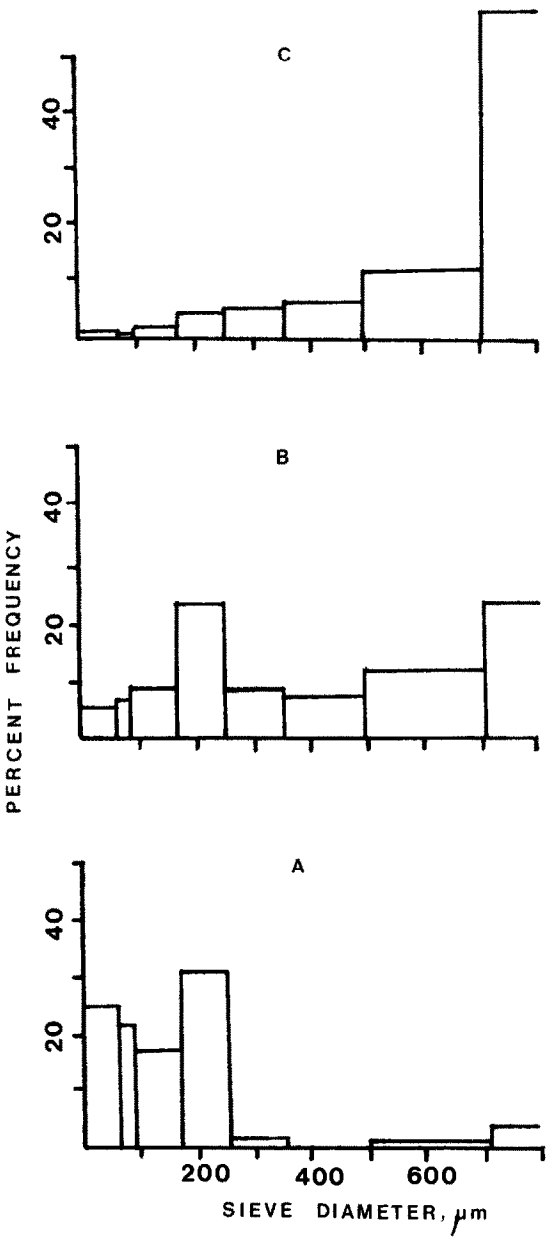

Fig. 6. Frequency size distributions for samples removed after addition of: (A) 100 ml, (B) 700 ml, and (C) 1100 ml starch mucilage during granulation.

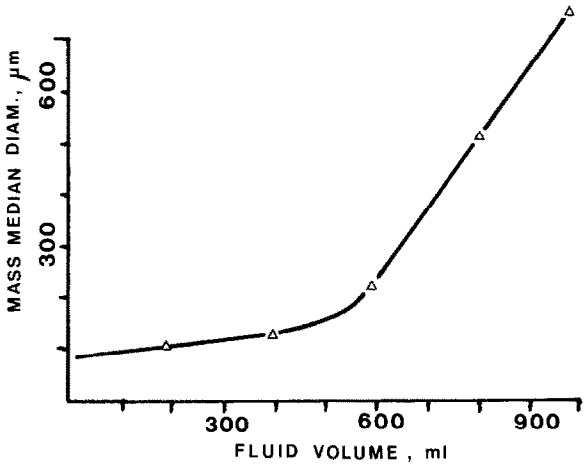

Fig. 7. Relationship between granulating fluid volume and mass median diameter of granules for Avicel/starch mucilage granulation.

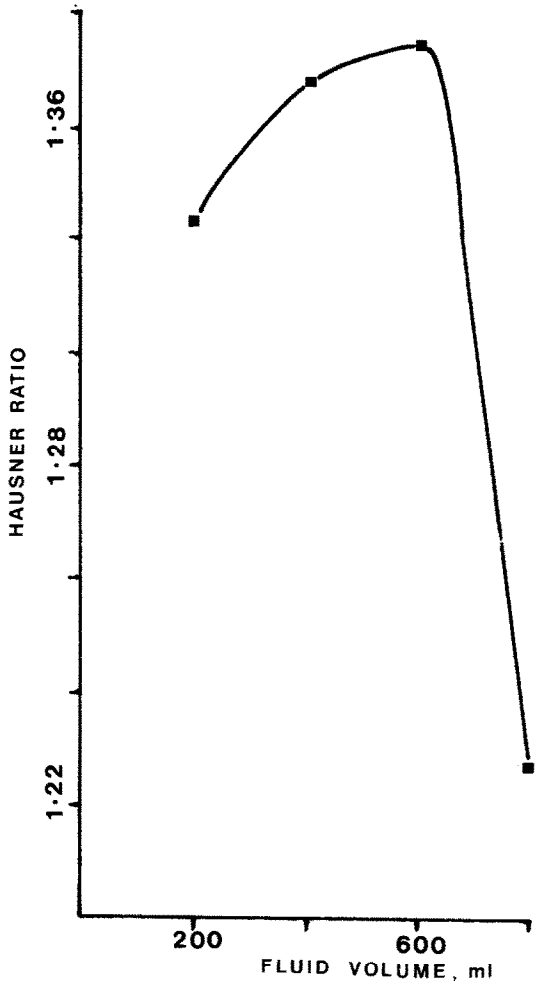

Fig. 8. Relationship between Hausner ratio and granulating fluid volume for Avicel/starch mucilage granulation.

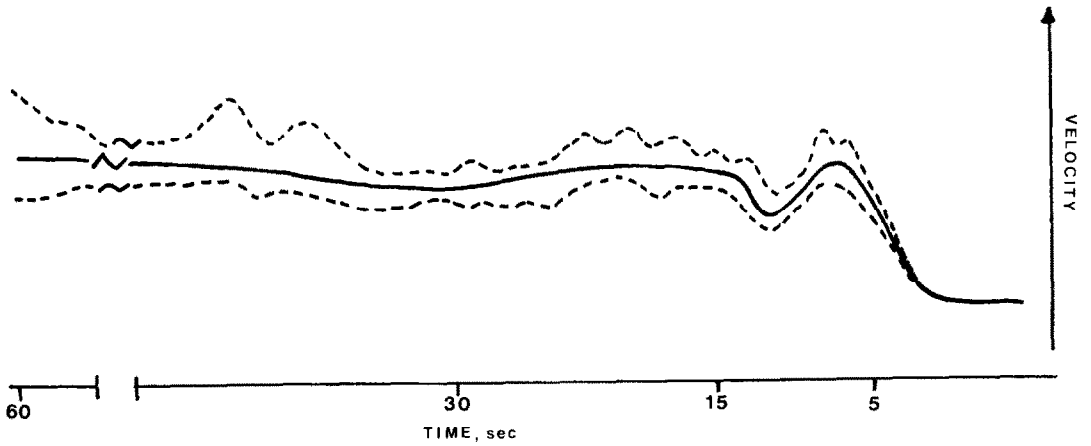

Fig. 9. Vibration profile for a lactose/water granulation. The recording shows average velocity (———) and velocity envelope (- - - - - -).

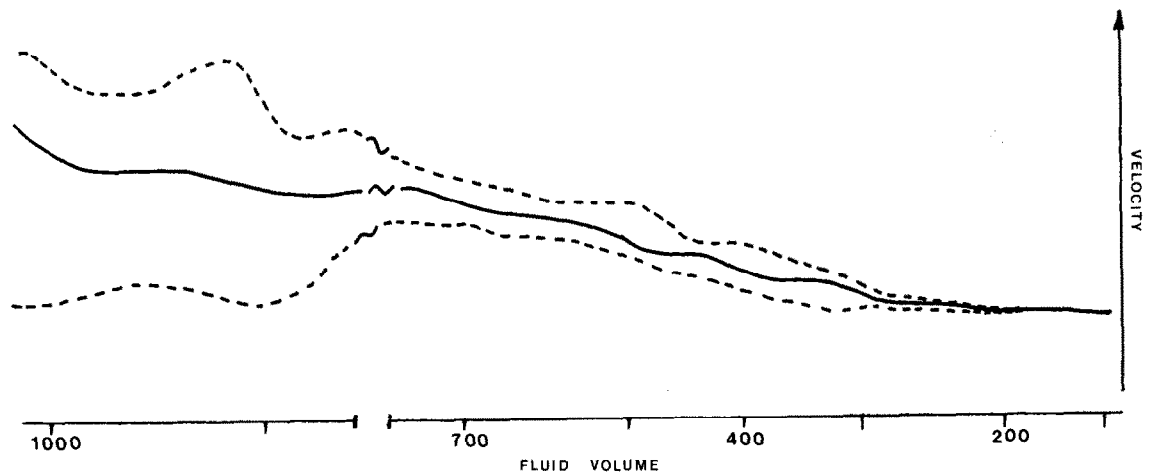

Fig. 10. Vibration profile for an Avicel/starch mucilage granulation. The recording shows average velocity (———) and velocity envelope (- - - - -).

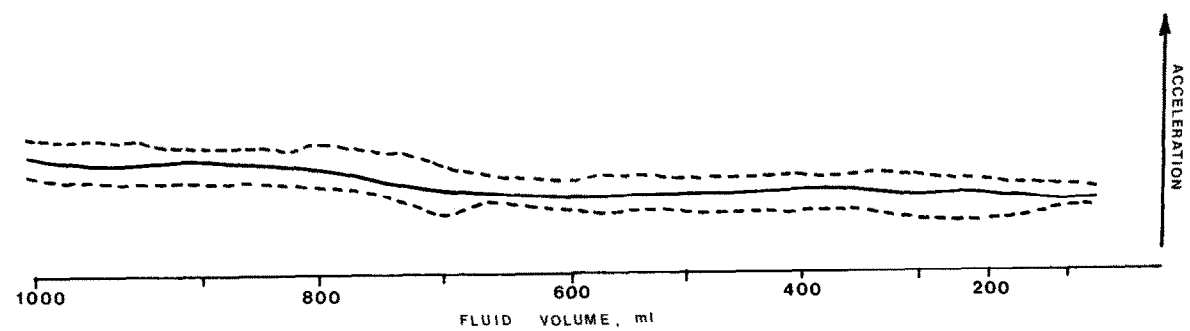

Fig. 11. Vibration profile for an Avicel/starch mucilage granulation. The recording shows average acceleration (_____) and acceleration envelope (- - - - -).

ment monitoring produced similar results for each granulation system. In the lactose system, a large increase in velocity was found after 5–10 s massing, and as with displacement profiles, this was followed by a decrease in velocity, rising again to an almost constant level after 15–20 s massing (Fig. 9).

With the Avicel–starch mucilage granulation, a more marked increase in peak velocity with further addition of starch mucilage was noted. As with displacement measurements, increasing the volume of starch mucilage added between 700 and 900 ml caused no further increase in velocity (Fig. 10). A total volume of more than 900 ml starch mucilage added to the granulated mass, caused overwetting and a corresponding increase in recorded peak velocity.

Recording peak acceleration (Fig. 11) gave a less distinct profile of vibrations occurring during the granulation process even when the sensitivity of the recording instruments was increased 2- or 3-fold.

Scanning electron photomicrographs (Figs. 3 and 5) show the growth of granules for both lactose and Avicel systems and provide some qualitative indication of the changes in particle association at different regions in the granulation process.

Conclusions

It is possible to monitor some of the changes which occur during granulation using vibration analysis of the movement of a swing-arm probe mounted in a planetary mixer bowl.

Displacement or velocity monitoring were found to provide better sensitivity to granulation changes than use of acceleration values.

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