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THE CONDUCTIVITY OF THE DAMP MASS DURING THE MASSING STAGE OF THE GRANULATION PROCESS.

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ABSTRACT

A Planetary mixer has been modified so that the conductivity of the damp mass could be studied during the process of wet-massing in granulation. The technique makes it possible to follow the distribution of the binder liquid and possibly also the changes in packing that occur during massing.

INTRODUCTION

A number of workers have made evaluations of the process of wet massing with respect to the granulation of powders in the Pharmaceutical Industry by measuring the torque developed during mixing in a planetary mixer.[1-3] This technique is an indirect method of following the change in density of the mass after the addition of water or binder solution. An alternative approach is to measure the conductivity of the damp mass during the massing

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process. This method gives a measure of the uniformity of liquid distribution and also, possibly, a measure of the change of packing density of the damp mass. Therefore, a Hobart planetary mixer was modified to enable the conductivity of the mass to be measured during massing.

EXPERIMENTAL

Modification of the Mixer

The bowl was isolated from the frame of the mixer by the addition of Tufnal insulation pads at the points of contact. The blade of the mixer was isolated from the frame by the replacement of the metal retaining clip by one made from nylon. Electrical contact was made to the bowl using a spring loaded contact point attached to the frame and to the blade via a copper ring attached below the nylon clip to a running contact. The potential difference across the powder bed was obtained using a Farnell constant voltage unit (Farnell Instruments Ltd., Wetherby, Yorks., U.K.) and a Servoscribe recorder was modified by the inclusion of a resistor between the input terminals so that the current flowing through the bed could be recorded as a potential difference.

Materials

Lactose (Serolac, Dairy Crest Industrial Division, Thames Ditton, Surrey, U.K.) either mean size 4.8μm (using Fisher sub-sieve sizer) or 170μm (50% weight by sieve analysis) was massed using water, specific conductivity $1.6 \times 10^{-4} ohm^{-1} m^{-1}$ or an aqueous solution of polyvinylpyrrolidone,PVP,(GAF,U.K.Ltd,K29-32) specific conductivity $1.38 \times 10^{-3} ohm^{-1} m^{-1}$.

Method

Lactose (1000g) was placed in the bowl of the mixer and stirred for 2min at the lowest speed, 1 rev s^{-1}. A potential difference of 5V was set between the blade and the bowl of the mixer, the recorder was set to a full scale deflection of 100mV or 200mV dependent upon the volume of liquid to be used and the liquid was added as a single amount of either $100cm^3$ or $150cm^3$, massing was continued until the system approached an equilibrium state.

RESULTS

The results were obtained in the form of recorder traces of mV against time and typical traces are shown in Figures 1 - 4. Due to the design of the recorder the traces read from right to left and show the increase in conductivity of the mass as the liquid is distributed and as the packing increases.

Figure 1 was obtained using $100cm^3$ water with the coarse lactose, Figure 2 represents the similar system but with $100cm^3$ PVP 5% as the massing liquid. Both traces show an initial rapid rise in conductivity, presumably as the water is distributed, followed by a lesser rise, possibly resulting from the mass packing to a greater density due to the prolonged massing. Figures 3 & 4 were obtained using the fine grade of lactose and indicate the slower distribution of water and also the greater time to achieve an equilibrium state. The rapid falls in conductivity found using the fine lactose were associated with the detachment of damp lactose which had adhered to the sides of the bowl, this occurred after 4 and 6min with water and after 6½, 7½ and 9min with PVP solution used as massing fluid.

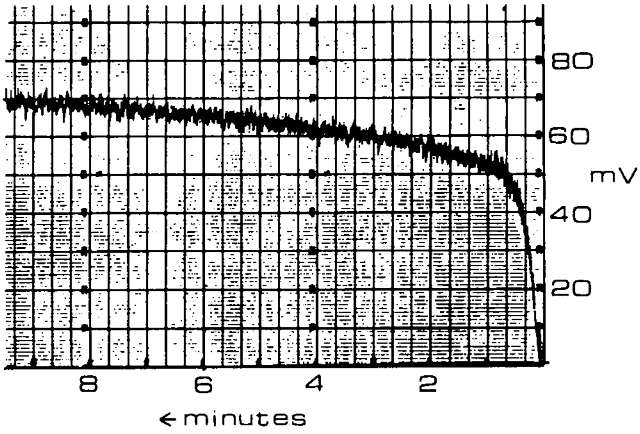

FIGURE 1

Conductivity, during massing, of the damp mass of coarse lactose following the addition of 100 cm^3 water.

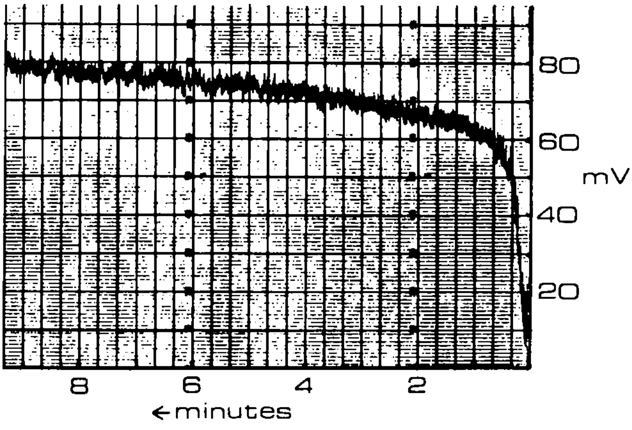

FIGURE 2

Conductivity, during massing, of the damp mass of coarse lactose following the addition of 100 cm^3 5% PVP solution.

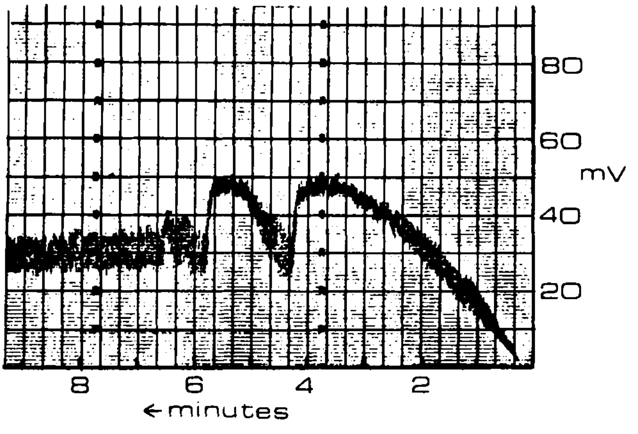

FIGURE 3

Conductivity, during massing, of the damp mass of fine lactose following the addition of 100 cm^3 water.

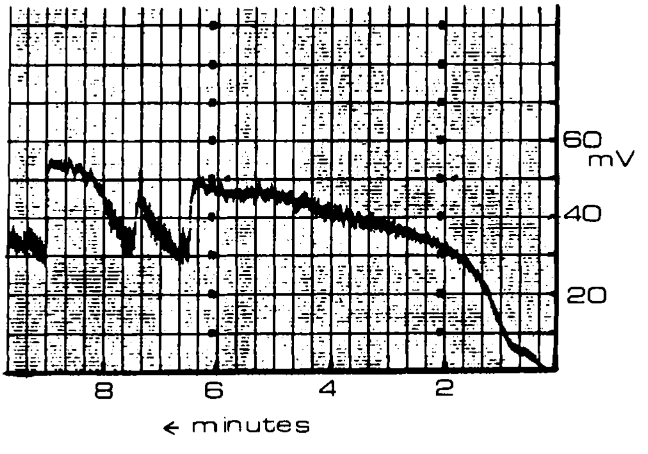

FIGURE 4

Conductivity, during massing, of the damp mass of fine lactose following the addition of 100 cm^3 5% PVP solution.

Further massing did not result in further build-up of lactose on the sides of the bowl and the trace continued at a constant level. In both cases the rapid rise and fall in conductivity associated with the rotation of the blade is seen in the width of the trace. The addition of PVP results in a small rise on maximum values of conductivity due to the greater conductivity of PVP solutions compared with that of pure water.

Addition of greater volumes of water resulted in similar traces with greater maximum conductivity, addition of the water in aliquots produced a series of stepped traces rising with each addition of water to a value that depended upon the total volume added and which was not dependent upon the number of aliquots.

The technique of measuring the conductivity of the damp mass has great potential, hence this preliminary communication. Work is continuing to establish the effects of the many variables and to evaluate the method as one for the determination of the end-point of a wet massing granulation.

REFERENCES

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