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COMPARISON OF METHODS FOR EVALUATION OF FLOW PROPERTIES OF POWDERS AND GRANULATES

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ABSTRACT

Powder mixtures and granulates with bulk densities ranging from 0.35-0.89 $g \cdot ml^{-1}$ were tested regarding flow properties using the following methods: the Hausner ratio (packed bulk density/loose bulk density), rate of packing on tamping, flow rate through a 30 mm orifice, orifice diameter allowing free flow, and "drained" angle of repose.

The Hausner ratio and the angle of repose could be measured with a relative standard deviation of about 2 %. The orifice diameter could also be determined accurately and these three methods correlated well with each other.

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The flow rate could only be measured on free flowing materials and the packing rate correlated poorly with the other methods.

INTRODUCTION

In the production of solid dosage forms, such as tablets, the ability of the materials to flow freely is often of great importance for the quality of the final product. Many of the methods used for evaluation of powder flowability have been tried for the evaluation of tablet granulates (1, 2, 3).

A reproducible fill in tablet production is important not only for the uniformity of the active drug content but also for the tableting pressure, which may affect the dissolution rate, the tablet strength, the appearance etc. However, the correlation between the reproducibility of the tablet weight and the estimation of the flowability of the tablet mass has often turned out to be poor (4, 5). This is not surprising as the flowability is only one of several factors which affect the dosage accuracy. Other important factors are, for example, the design of hoppers and feeding devices, the accuracy of the tooling, the machine speed, vibrations, and segregation. Only when the tablet mass flows poorly will the flow properties be the main factor determining the weight accuracy (3). Unfortunately, most methods of flowability evaluation give reproducible results only for noncohesive materials (3, 9).

There is a need for simple methods of evaluating tablet masses both in formulation work and in the process control during production of tablet granulates. Determination of weight variations during tableting is too tedious a method for this purpose and is difficult to standardize sufficiently well. Little has been published regarding the reproducibility of different methods, especially when applied to masses which are on the verge of giving problems in tablet production.

The purpose of this investigation was to compare some simple methods of measuring flow properties with respect to reproducibility and the correlation between them. For the studies we have chosen methods which determine flow rate, arching tendency, compressibility on tapping and angle of repose. The methods were applied to granulates and powder mixtures with a range of densities and cohesiveness to cover the range from free flowing to poorly performing tablet masses.

EXPERIMENTAL

Materials

Sodium chloride (dentritic type), microcrystalline cellulose (Avicel® PH 101, FMC USA) and acetylsalicylic acid type 7017, 7023, 7012 (Monsanto UK), were used without granulation. Lactose (DMV Holland), lactose-cornstarch and aluminium hydroxide-magnesium carbonate (codried gels F-MA 11, Armour Ireland) were get granulated in the conventional way. Different particle size distributions were obtained by variation of the dry milling procedure.

Methods

The particle size distribution was determined by sieving (Sonic Sifter, Model L3, Allen Bradley) and the mass median diameter was calculated. The loose bulk density (D_A) and the packed bulk density (D_P) were measured with a measuring cylinder and an Engelsmann Stampvolumeter according to the German Standards (DIN), 53192 and 53194. The Hausner ratio D_P/D_A (7) was calculated from the bulk densities. The rate of packing was measured by plotting bulk density against packing time.

The time to reach 75 % of the final packing density change was used to express the packing rate.

The flow rate from a cylinder with a 30 mm aperture was determined (8) immediately after filling the cylinder via a powder funnel. The same equipment was used to determine the apertures giving complete block or flow. The aperture could be varied from 5 mm to 65 mm in increments of 5 mm. Ten runs were performed at each aperture. When the material drained completely from the cylinder it was considered to flow. The results are given as the minimum diameter which allowed all 10 runs to flow as well as the maximum diameter which caused block in all 10 runs.

The angle of repose was determined in the apparatus shown in figure 1. The diameter of the circular table was 65 mm and it was surrounded by a longitudi-

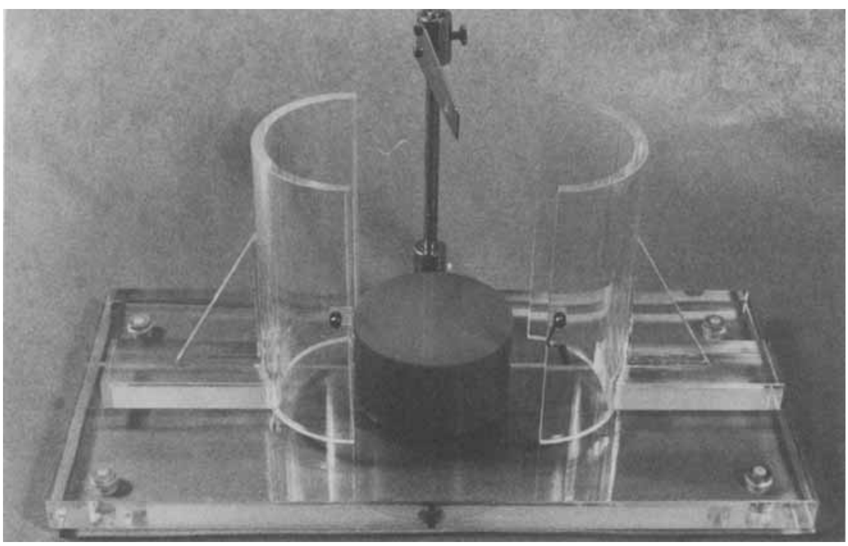

FIGURE 1
Apparatus for determination of the angle of repose

nally divisible cylinder. The materials were filled into the cylinder through a funnel and the two parts of the cylinder were slowly separated. The angle of repose was calculated from the height of the powder cone remaining on the table.

The mean values and the relative standard deviations were calculated from ten determinations of the Hausner ratio, the flow rate and the angle of repose.

RESULTS AND DISCUSSION

Table 1 summarizes the results for the flow properties tested. It is obvious that the angle of repose measured in this special way and Hausner ratio can be determined with good reproducibility even for fairly cohesive materials. The relative standard deviations were about 2 %. The flow rate from the cylinder could not be determined for the more cohesive materials and the relative standard deviation for the flowing materials range from 3.7 to 25.3 %. The diameter allowing complete block or flow from the cylinder could be determined accurately for all materials.

Determination of the packing rate was fairly tedious and the results did not correlate with other measures of flowability. The angle of repose, Hausner ratio and flow rate correlated well with each other. All materials having an angle of repose less than 52^{o} flowed freely through a 10 mm aperture and for less free flowing materials the aperture correlated reasonably well with the other methods.

CONCLUSION

Our results indicate that the flow rate from a cylinder is of limited value for testing tablet granu-

TABLE 1

Properties of the investigated masses

	Median diameter µm	Packed bulk density (D_p) g/ml	Hausner ratio (D_p/D_A)	Time for 75 % compaction s	Flow rate g/min	Max aperture for block mm	Min aperture for block mm	Angle of repose °
$Al(OH)_3$-$MgCO_3$ gran I	105	0.35	1.59 (1.3)[a]	318	block	50	60	63.5(2.4)[a]
- " - II	350	0.40	1.33 (1.1)	87	block	35	55	59.4(1.6)
Avicel PH 101	<60	0.45	1.32 (1.7)	118	block	25	45	61.2(2.9)
Acetylsal. acid 7012	70	0.61	1.49 (1.0)	16	block	50	65	62.6(1.7)
- " - -"- 7023	95	0.67	1.32 (1.4)	47	22.7 (25.3)[a]	25	55	57.9(2.2)
- " - -"- 7017	440	0.83	1.09 (1.3)	47	172.5 (3.7)	<5	5	39.2(1.5)
Starch-Lactose (2+1)	285	0.70	1.18 (1.4)	22	84.5 (12.0)	<5	10	50.5(2.3)
- " - (1+2)	230	0.72	1.12 (1.1)	32	101.5 (7.9)	<5	5	45.7(2.0)
Lactose granulate I	410	0.73	1.15 (2.1)	34	94.3 (5.4)	<5	10	46.4(1.2)
- " - II	195	0.76	1.20 (1.2)	28	46.0 (14.0)	<5	10	51.2(2.4)
- " - III	105	0.84	1.25 (0.8)	20	33.7 (16.0)	15	30	54.7(1.9)
Sodium chloride	245	0.89	1.06 (0.9)	13	160.6 (7.9)	<5	5	36.4(2.4)

a) relative standard deviation.

lates because the method is only applicable for materials which flow well. However, it is possible to characterize even fairly cohesive tablet granulates with good reproducibility using the drained angle of repose, Hausner ratio and opening diameter for free flow. These methods are rapid and easy to use and deserve further studies on their suitability for control of tablet granulates during formulation and production.

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