Powder Flow Studies II

Effect of Glidants on Flow Rate and Angle of Repose

By GERALD GOLD, RONALD N. DUVALL, BLAZE T. PALERMO, and JAMES G. SLATER

Glidants have often been selected by subjective or indirect methods such as measurement of the angle of repose. As a result, several materials have been empirically classified as glidants. The recording powder flowometer described in Part I of this series was utilized to evaluate various glidants. A comparison was made between the results obtained with this instrument and those by measurement of the angle of repose. The commonly used glidants, fumed silicon dioxide, magnesium stearate, starch, and talc were studied in combination with selected materials. Many of the more widely used glidants actually decreased the flow rate. Glidants which lowered the angle of repose did not necessarily increase the flow rate and marked changes in flow rate were not always detectable by angle of repose measurement. In addition, a comparison of the angle of repose and the flow rate of various commonly used raw materials indicated that the angle of repose was not a reliable method for evaluating the flow of these materials.

The flow properties of powders vary considerably from those of formulations which flow freely and continuously through a small orifice to those which are evident of noncontinuous flow under any circumstance. Poorly flowing powders or granulations present many difficulties in the pharmaceutical industry, especially in compressed tablet manufacturing, and considerable effort, therefore, has been directed toward overcoming flow problems. The most commonly used technique involves the addition of materials known as glidants in an attempt to improve the flow characteristics of the formulation. Selection of the glidant and the concentration thereof is often empirical since there is no generally accepted method for evaluating the effectiveness. As a result, glidant usage is often ascertained on a trial and error basis *via* subjective information.

One of the more objective methods, probably the most widely used, involves the measurement of the angle of repose. The powdered or granular material is allowed to fall freely through an orifice onto a flat surface to form a conical pile of the deposited material. The angle between the surface of the cone and the horizontal plane is known as the angle of repose. This numerical value is reproducible provided the conditions remain constant. Train (1), in a critical examination of four methods of determining the angle of repose with glass balls, lead shot, and silver sand, concluded that the type of method influenced the results, but that most methods of measurement would provide suitable data for comparison between samples during routine quality control tests. He did not mention relationship to flow. Although it is not clear to what physical property the angle of repose corresponds, it has often been assumed (2-4) that it relates to the flow properties of the material. A high angle reputedly indicates a poorly flowing material, while conversely a low angle indicates good flow. The magnitude of this angle is dependent on the conditions of measurement and the numerous methods by which this angle has been measured have added to the confusion. Pilpel (4), in summarizing the work of others, indicated that the value of the angle of repose depends not only on the way in which the cone is produced, but also on the nature of the powder, its preparation, particle size, and particle size distribution. In an attempt to study the problem of granulation flow in tableting as it is related to interparticle friction, Nelson (5) measured the angle of repose of a sulfathiazole granulation as a function of average particle size, the presence of lubricants, and admixed fines, but carefully avoided direct correlation with flow properties. Craik applied the supposed relation of flow to the angle of repose in an

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investigation of the flow properties of starch powders under laboratory conditions (2) and of three very different powders under humid conditions (3).

Timed delivery through an orifice is also of value in appraising the flow of materials. A stop watch is usually used to either time a certain weight of powder flowing through the orifice or to close the orifice after a given time so that the powder flowing through in that time period can be weighed. With this method, Hammerness and Thompson (6) showed that the addition of fines increased the rate of flow to an optimum level, after which the rate began to decrease. The addition of lubricant beyond 2% did not substantially improve the flow rate. Gunsel and Lachman (7) comparatively evaluated tablet formulations by the timed delivery technique and used the angle of repose measurement to substantiate similarities in flow rates. However, their data show nearly identical flow rates for two formulations having differences in angles of repose and a marked difference in flow rate for two materials having identical angles. Munzel (8), using a modified Emix powder dispenser and weighing the amount of material discharged in 10 sec. by manually opening and closing the flap, studied the influence of glidants on the flow properties of granules. He apparently was the first to employ the term "glidant" to designate agents which added in small amounts improve flow characteristics of granulations. Flow regulators and solid flow conditioners are two other terms used synonymously with flow glidants. The report of Munzel is the source of the Remington (9) differentiation of lubricants into three groups based on their ability to carry out respective functions: glidants, antiadhesives, or antisticking agents and lubricants.

Recently, Augsburger and Shangraw (10) rated some silica-type glidants in two microcrystalline cellulose systems based on tablet weight variation data. However, this method is tedious, time consuming, and not practical at the preliminary product development stage.

In the previous report from this laboratory (11), a recording powder flowometer was described and represented a new approach to the study of powder flow. The authors believe that this technique more closely simulated and recorded flow as it occurs in a tableting operation. Although the angle of repose is controversial in the area of flow measurement, it has been widely used, and one objective of this study was to investigate the relationship between this angle and the flow rate with the recording powder flowometer. Numerous materials were evaluated in an effort to ascertain correlation, if any, between the two techniques. A second aspect of this work was to study the effect of materials generally known as glidants when added to selected powdered, crystalline, and granulated materials. The glidants used were fumed silicon

Table I.—Particle Size Analysis of Aspirin, Calcium Sulfate, and Spray-Dried Lactose

Screen	20	40	60	80	120	200	Pan
	Material on Screen, %						
Aspirin crystals	1	31	38	17	7	5	1
$CaSO_4 \cdot 2H_2O$ granules	5	49	16	9	6	8	7
S. D. lactose	0	0	0	3	16	43	38

Table II.—Effect of Magnesium Stearate, Fumed Silicon Dioxide, Cornstarch, and Talc on the Flow Rate and Angle of Repose of Aspirin Crystals, Calcium Sulfate Granules, and Spray-Dried Lactose

		ASA Crystals		$CaSO_4$ Granules		S.D. Lactose	
	% Glidant	Flow Rate	Repose Angle	Flow Rate	Repose Angle	Flow Rate	Repose Angle
Control	0	11.17	40.35	8.42	41.63	8.16	33.08
Mg stearate	0.25	12.62	36.77	8.75	41.30	8.49	32.62
	0.5	11.44	39.60	8.67	41.80	7.03	33.35
	1.0	11.48	40.73	9.14	41.40	7.21	33.90
	5.0	8.14	40.92	7.27	42.72	3.08	40.53
Fumed SiO_2	0.1	11.47	40.45	9.61	42.10	8.21	33.97
	0.25	12.08	40.17	9.27	42.32	7.63	32.42
	1.0	11.68	39.52	6.90	42.63	7.28	31.72
	5.0	5.71	40.53	3.84	43.03	1.34	35.73
Cornstarch	0.25	12.88	38.18	8.84	41.80	7.27	34.03
	1.0	12.10	37.85	8.29	42.52	6.63	33.28
	5.0	10.59	38.18	6.96	42.32	4.90	33.90
	10.0	8.93	39.33	4.83	43.15	4.11	38.62
Talc	0.25	10.24	41.42	8.26	42.02	5.78	33.77
	1.0	10.51	41.32	7.62	42.63	5.04	34.68
	3.0	9.78	42.42	7.06	42.42	3.44	36.43
	5.0	8.69	42.22	7.75	42.83	3.76	37.08

dioxide, magnesium stearate, cornstarch, and talc. Varying concentrations of these were evaluated for their effects on the angle of repose and flow rate of aspirin crystals, calcium sulfate granules, and spray-dried lactose. The numerous comparisons obtained from this study should help to clarify the status of angle of repose measurement as a measure of flowability.

EXPERIMENTAL

Materials.—The spray-dried lactose, crystalline aspirin, magnesium stearate, talc, fumed silicon

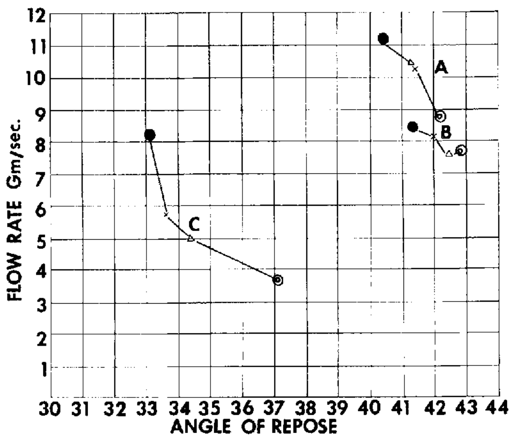

Fig. 1.—Relationship of angle of repose to flow rate for systems containing talc. Key: A, aspirin; B, calcium sulfate granules; C, S.D. lactose. Talc concentrations: ●, 0%; ×, 0.25%; △, 1.0%; ⊙, 5.0%.

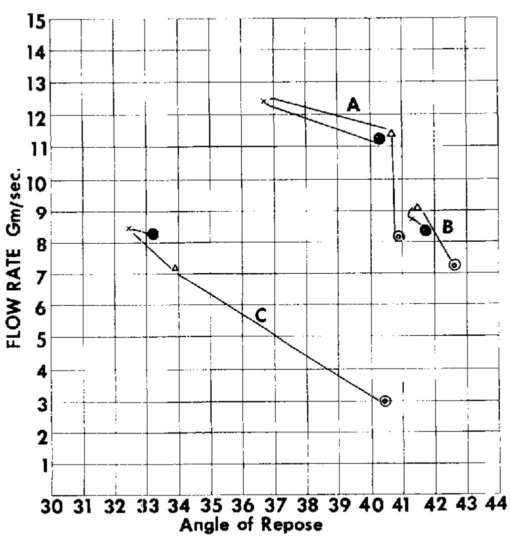

Fig. 2.—Relationship of angle of repose to flow rate for systems containing magnesium stearate. Key: A, aspirin; B, calcium sulfate granules; C, S.D. lactose. Magnesium stearate concentrations: ●, 0%; ×, 0.25%; △, 1.0%; ⊙, 5.0%.

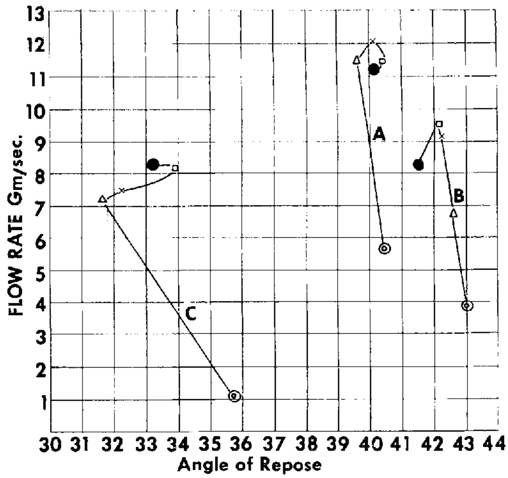

Fig. 3.—Relationship of angle of repose to flow rate for systems containing fumed silicon dioxide. Key: A, aspirin; B, calcium sulfate granules; C, S.D. lactose. Fumed silicon dioxide concentrations: ●, 0%; □, 0.1%; ×, 0.25%; △, 1.0%; ⊙, 5.0%.

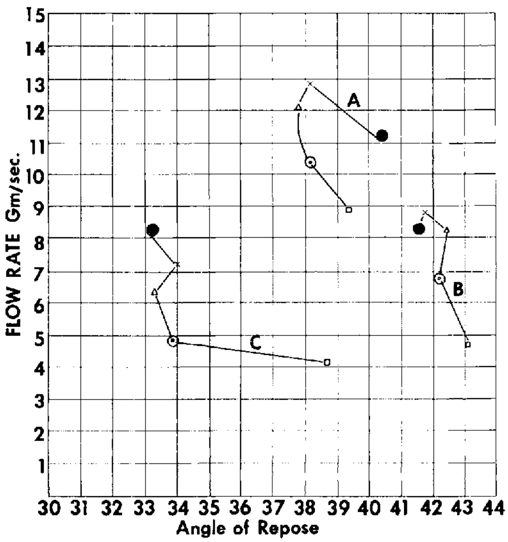

Fig. 4.—Relationship of angle of repose to flow rate for systems containing starch. Key: A, aspirin; B, calcium sulfate granules; C, S.D. lactose. Starch concentrations: ●, 0%; ×, 0.25%; △, 1%; ⊙, 5%; □, 10%.

dioxide,[1] and cornstarch were either U.S.P. or pharmaceutical grade materials. The calcium sulfate granulation was prepared by adding 5% gelatin solution to calcium sulfate dihydrate in a Blakeslee kitchen mixer and wet sizing through a No. 6 screen on an oscillating granulator. It was dried for 8 hr. at 140° F., and the dried granules were sized with an oscillating granulator and a No. 16 screen. Mesh analyses of the aspirin, spray-dried lactose, and calcium sulfate granulation used in the glidant studies

[1] Marketed as Cab-O-Sil by the Cabot Corp., Boston, Mass.

are shown in Table I. The cornstarch was dried at 140° F. for 24 hr., and all glidants were passed through a No. 40 hand screen prior to using. Glidants were used at concentration levels as indicated in Table II.

The materials used for the flow rate and angle of repose correlation study are listed in Table III and characterized according to the U.S.P. powder fineness classification for chemicals following particle size analysis.

Particle Size Analysis.—Particle size was determined with a Ro-Tap testing sieve shaker, using U. S. standard sieves in series 20, 40, 60, 80, 120, and 200 mesh sizes. A 100-Gm. sample was tested in the shaker for 5 min.

Flow Rate Determinations.—Flow rates, expressed in Gm./sec., were determined with the flowometer on 500-Gm. samples and with a conical stainless steel hopper measuring 20 cm. top diameter by 30 cm. in length and 10 mm. orifice diameter. Each value represents the average of nine measurements. To minimize variations in ambient conditions, three measurements were made on 3 different days. The average standard deviation was 0.47 Gm./sec.

Angle of Repose Measurements.—Repose angles were measured by the fixed funnel and free standing cone procedure described by Train (1). The fixed height of the funnel orifice was 2.54 cm., and the orifice diameter was 5.0 mm. The reported repose angles represent the average of five determinations. In no case was the standard deviation greater than 0.81°, which compares favorably with the average standard deviation of 0.74° reported by Fonner *et al.* (12).

RESULTS AND DISCUSSION

The effects of varying concentrations of glidants on the flow rate and angle of repose of aspirin crystals, calcium sulfate granules, and spray-dried (S.D.) lactose are shown in Table II. The concentrations of specific glidants included those recommended or generally used. Since S.D. lactose had the slowest flow rate, it might be assumed to respond more favorably to the addition of a glidant. However, with the exception of the lowest levels of magnesium stearate and fumed silicon dioxide, the flow rate was decreased substantially. Munzel (8) reported that the flow rate of sodium chloride was reduced by the addition of glidants, and attributed this to the fact that sodium chloride itself is an excellent glidant and that its flow properties were impaired by poorer glidants. This does not apply to S.D. lactose and suggests that other factors are involved. The S.D. lactose had a particle size distribution (Table I) much finer than the other two sample materials, a factor which may influence the effectiveness of glidants. Magnesium stearate and fumed silicon dioxide at their usual concentration ranges did increase the flow rate of both calcium sulfate granules and aspirin crystals. Cornstarch, at low levels, increased the flow rate of the same two materials but decreased the flow rate at concentrations normally employed. Talc appears to be a poor glidant for these sample materials since it decreased the flow rate at all concentration levels. The talc used in this study was designated as having "good slip," a designation determined subjectively by rubbing the material between the thumb and fingers. It is evident that a glidant which is effective with a given material may prove otherwise when used with a different material. The flowometer appears to be a convenient tool to determine the best glidant for specific material more objectively. This technique also appears to be valuable to ascertain the optimum concentration of a particular glidant. In those instances where the glidant increased the flow rate, the lower concentration was usually more effective. Two exceptions were magnesium stearate with calcium sulfate granules and silicon dioxide with aspirin crystals. The indiscriminate use of glidants should be avoided since beyond their optimum concentration they usually cause a marked decrease in flow rate.

In evaluating the effect of glidants by angle of repose measurement, an increase in magnitude of the angle should be associated with a decrease in flow rate and conversely, a decrease in the angle should result in an increased flow rate. This relationship seems to exist within the three talc-containing systems which were studied, as illustrated in **Fig.** 1. However, the angles of repose for both the calcium sulfate and aspirin series with talc were much higher than those for the S.D. lactose–talc series, yet the former two series had much faster flow rates. Figure 2 illustrates the relationship in the three systems containing magnesium stearate. The angle of repose did correlate generally with flow rate within these systems, although again no correlation between the three series was evident, and a marked drop in flow rate for aspirin–magnesium stearate was accompanied only by a negligible change in angle of repose. Figures 3 and 4, illustrating the three systems with silicon dioxide and cornstarch, respectively, indicate many exceptions to the angle of repose-flow rate theory. In many instances a decrease in the angle of repose was not associated with an increased flow rate, and marked changes in

TABLE III.—ANGLE OF REPOSE AND FLOW RATE FOR SELECTED RAW MATERIALS

Material	Classification[a]	Angle of Repose, Degree	Flow Rate, Gm./sec.
Acetaminophen	Coarse	37.00	11.20
Ascorbic acid	Coarse	41.92	11.26
Aspirin crystals	Coarse	40.35	11.17
Aspirin crystals	Moderately coarse	43.35	7.78
Aspirin granulation	Coarse	39.60	10.79
Aspirin (10% starch)	Very coarse	40.73	11.20
Calcium sulfate, dihyd.	Coarse	41.63	8.42
Dextrose, anhyd.	Coarse	34.62	18.90
S.D. lactose	Fine	33.08	8.16
Mannitol	Very coarse	41.02	8.10
Sodium ascorbate	Moderately coarse	41.92	9.91
Sorbitol	Fine	37.42	7.09
Sugar	Fine	38.53	14.24
Tartaric acid	Coarse	39.33	16.84
Thiamine mononitrate	Moderately coarse	38.18	13.72

[a] U.S.P. XVII classification of powders by fineness.

TABLE IV.—CORRELATION COEFFICIENT AND PER CENT DEPENDENCE SHOWING RELATIONSHIP BETWEEN ANGLE OF REPOSE AND FLOW RATE

	Coefficient Correlation, r	% Dependence
Glidants with aspirin	−0.49	24.68
Glidants with calcium sulfate	−0.74	54.43
Glidants with S.D. lactose	−0.62	38.27
Selected raw materials	−0.32	10.33

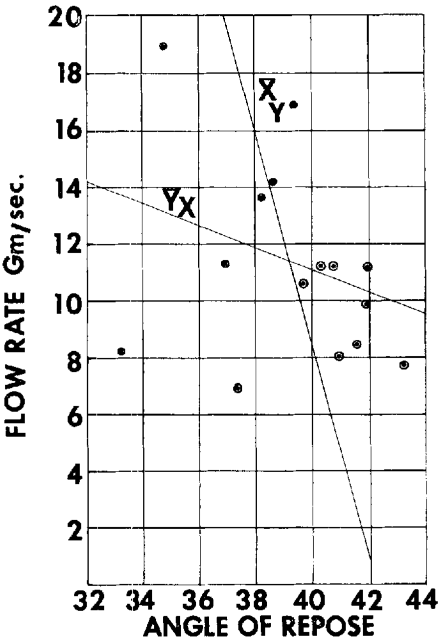

Fig. 5.—Relationship of angle of repose to flow rate for selected raw materials. Key: $\bar{X}_y$, regression line of the angle of repose on flow rate, $\bar{Y}_x$ regression line of the flow rate on the angle of repose.

the flow rate were not detectable by changes in the angle of repose.

Measurement of the angle of repose of a powder has been reported to yield information on flowability in comparison to that of other powders (2–4). Since this current work concerning the addition of various glidants to the three systems had cast considerable doubt on this relationship, the flow rate and angle of repose of 15 selected raw materials were determined. The results are shown in Table III. The materials represent wide ranges in angles of repose and flow rates. The coefficient of correlation (13) has been used to statistically measure the extent of the linear relationship between angle of repose and flow rate. A correlation coefficient close to 0 indicates a weak or nonexistent relationship, whereas a value close to +1 or −1 indicates a close relationship between the two variables. The per cent dependence (14), a measure of the percentage of the variation in flow rate related to variation in the angle of repose, has also been calculated. These statistics were used to evaluate the angle of repose–flow rate relationship for the aspirin, calcium sulfate, and S.D. lactose glidant series. Results are given in Table IV. Coefficients of correlation for the calcium sulfate and S.D. lactose systems are significant or high enough to assume some correlation between the two variables. However, the per cent dependence of these two systems are not sufficient to have high or reliable correlation. Correlation between the selected raw materials was poor. This is illustrated in Fig. 5. The difference in regression lines is indicative of the weak relationship between angle of repose and flow rate (r = −0.32). Although still not high, correlation was better within a given system. The over-all low correlation coefficients and corresponding per cent dependence values indicate that the angle of repose is not a reliable method to evaluate flow of materials.

SUMMARY

1. The effects of varying concentrations of the glidants, magnesium stearate, fumed silicon dioxide, cornstarch, and talc, on the flow rate of aspirin crystals, calcium sulfate granules, and S.D. lactose have been investigated with a recording powder flowometer. The angle of repose measurement was also used in a further attempt to evaluate flow and to investigate the relationship between this angle and flow rate.

2. The flowometer appears to be a valuable instrument to select the best glidant for a given material or granulation and to determine the optimum concentration of a particular glidant in a given system.

3. Magnesium stearate, fumed silicon dioxide, and cornstarch in low concentrations increased the flow rates of aspirin and calcium sulfate. However, effectiveness of the glidants with S.D. lactose was negligible. Talc appears to be a poor glidant for these materials since it decreased the flow rate at all concentration levels.

4. Optimum concentrations of glidants were 1% or lower. Excess of glidants should be avoided since beyond their optimum concentration they usually caused a marked decrease in flow rate.

5. Statistical tests were used to determine the relationship between angle of repose and flow rate. Low correlation was obtained within a specific system, whereas no correlation was found for selected raw materials. Consequently, angle of repose measurement does not appear to be a reliable method to evaluate flow of materials.

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