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Effects of blender rotational speed and discharge on the homogeneity of cohesive and free-flowing mixtures

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Abstract

The roles of blender rotational speed and blender discharge on the homogeneity of free-flowing art sand and of a cohesive placebo formulation were investigated in the tote blender. For three practical operating speeds, 6, 10, and 14 RPM, spanning the entire range of commercial equipment, the homogeneity of the free-flowing mixture was independent of rotational speed and blender size. On the other hand, the homogeneity of the cohesive pharmaceutical powder mixture was dependent on vessel rotational speed in a complex fashion, with 10 RPM producing a better final mixture than either 5 or 15 RPM. The homogeneity of the free-flowing sand mixture was preserved when discharged into a vertical bin, while the homogeneity of the fine pharmaceutical powder mixture significantly improved after discharge from the tote blender.

Keywords: Discharge; Sampling; Tumbling speed; Powder mixing; Tote blender; Bin

1. Introduction

The mixing of dry solid particles is ubiquitous in industrial processes. Examples include the production of pharmaceuticals, foodstuff, building materials, cosmetics, petrochemicals, and ceramics to mention but a few. In many chemical processes, the resulting quality of an intermediate mixing process determines the success of subsequent operations. For example, the calcination of catalyst in a rotary kiln (Zablotny, 1965) takes place in refining operations where the catalyst is recycled back to the reactor with its quality influencing the conversion of the reaction. A more critical mixing step often occurs at the final stages of a process prior to packaging the final mixture. This is especially true in pharmaceutical manufacturing where 80% of the medicines produced are in a solid dosage form, e.g. tablets or capsules, where the drug content uniformity is heavily scrutinized by regulatory agencies.

The importance of uniform drug distribution in solid doses has long been recognized by the official compendia (The United States Pharmacopoeia, 1970; The National Formulatory, 1970) and later

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reinforced by court decisions (US vs. Barr Laboratories 812, F. Supp 458, D.N.J. 1993) to safeguard the public against active component variations in the final dosage. In the past, excessive inter-tablet dose variation in pharmaceutical products was not an uncommon occurrence Fusari (1973), Wagner et al. (1973). It can lead to massive product recalls, severe economic loses, and risk to patient's health, especially in high potency medications. In recent years, this issue has generated significant academic and industrial efforts aimed at understanding mixing and segregation of powders.

In recent investigations (Sudah et al., 2001a,b), we have characterized the homogeneity of mixtures of free-flowing and cohesive powders in pilot plant scale bin blenders as a function of important operating parameters, such as initial loading pattern, fill level, mixing time, particle size, component concentration, presence of baffles, and use of pre-blending. Comprehensive reviews on the mixing of powders in other type blenders can be found in (Fan et al., 1972, 1990; Poux et al., 1991; Brone et al., 1998; Brone and Muzzio, 2000). This paper extends previous results by probing into the effects of rotational speed and blender discharge on the homogeneity of free-flowing and cohesive powder mixtures.

Mixer vessel speed is a process variable that is widely believed to be important but that has not been systematically studied. It is well known that granular systems do not mix without agitation or input of kinetic energy. For most industrial processes, flow is induced either by rotating the vessel containing the powder, or by rotating an impeller, or both. Zablotny (1965) stated that the rate of rotation of rotary kilns directly influences the material's dynamic angle of repose and residence time. Castellanos et al. (1999) investigated the effects of rotational speed of a horizontal drum on the dynamic angle of repose of free-flowing beads and mildly cohesive powders. The authors were able to construct a phase diagram depicting the observed flow regimes as a function of particle diameter and speed. The flow profile of free-flowing larger size (180–350 μm) material evolved from a flat sloping surface at low rotation rate (4 RPM) to an s-shaped profile at higher speeds (20 RPM) with the dynamic angle of repose increasing with speed. For finer, more cohesive particles ($<$ 30 μm), the dynamic angle of repose decreased as the rotational speed increased due to fluidization effects and lower particle-wall interaction (friction). The Froude number for the experiments ranged between 1.88×10^{-4} and 0.042 (4–60 RPM).

Brone et al. (1998) and Brone and Muzzio (2000) investigated the effects of speed on the homogeneity of mono-disperse free-flowing grains in V and double-cone blenders. The authors found that for a fixed number of vessel revolutions, the degree of mixing was independent of speed of rotation for Froude numbers ranging between 5.8×10^{-3} and 0.057 (8–24 RPM). The flowing region's surface was observed to be flat at speeds below 10 RPM, S-shaped above 10 RPM, and cataracting close to 30 RPM. The authors stated that most industrial processes operate in the inertial regime (flat and S-shaped surface profiles) with the Froude number remaining below 0.2. On the other hand, segregation of bi-disperse (different size) free-flowing material is a strong function of rotational speed, as described by Hill and Kakalios (1994). The dynamic angle of repose varied with speed for mixed and segregated systems with the component having the lower angle of repose segregating out of the mixture first. However, at certain rotational speeds, the difference between the mixed and segregated dynamic angle of repose approached zero and segregation became reversible.

The vessel itself is not always the only component whose speed can be varied. In high shear blending and granulation operations, internal moving parts are typically rotated, rather than the vessel itself. Knight et al. (2000) varied the impeller speed from 450 to 1500 RPM ($24 <$ Froude < 270) in a high shear vertical mixer to granulate calcium carbonate powder mixed using polyethylene glycol as a binder. Furthermore, Vromans et al. (1999) studied the effect of impeller speed on the granulation of a multicomponent steroid hormone mixture. It was found that high shear (high impeller speed) can cause demixing and should be avoided in granulation whenever there is an initial agglomerate size difference. Impellers preferentially destroyed weaker agglomerates composed of larger particles, leading to poor content uniformity.

Blender discharge is another variable with potential influence over the quality of the final mixture. Many investigators have extensively studied this in the form of hopper discharge (for example, see Matthee, 1967; Carleton, 1972; Van Denburg and Bauer, 1964; Johanson, 1978; Alexander et al., 1999; Markley and Puri, 1999; Tuzun, 1999; Berry and Moore, 2002). Alexander et al. (1999) measured the intrinsic tendency of different size glass beads and fine powder to segregate upon repeated discharge from small scale hoppers with varying cone angles. It was reported that for bi-dispersed free-flowing materials, a steady state asymptotic discharge profile was achieved after approximately 30 discharges. In addition, it was concluded that hopper angle influenced segregation behavior of the material, and an optimum angle was reported above which (steeper slope) segregation becomes exacerbated. Matthee (1967) and Johanson (1978) examined bunkering of materials with different properties flowing out of industrial hoppers. It was concluded that the rate and degree of segregation depended on several parameters such as particle size, shape, density, flowability, angle of repose, cohesion, shape of bunker, diameter, etc., with the most influential parameter being particle size. Moreover, Van Denburg and Bauer (1964) stated that particle size is the most important parameter-influencing hopper-induced segregation. Acicular shaped material with a large aspect ratio minimized the degree of segregation due to limited flowability.

Markley and Puri (1999) investigated the effects of hopper filling method on the size segregation of material undergoing mass flow. Funnel fill, inverted cone fill and spoon-by-spoon fill methods were used to load hoppers with coarse sugar of a known particle size distribution. The particle size distribution of the discharge stream was found to be within a 95% confidence interval of the original distribution with particles ranging in size between 180 and 250 μm having the maximum percent difference (with respect to original distribution). The results were independent of hopper filling method.

A phenomenological two-phase system approach was employed by Carleton (1972) to predict granular flow out of hoppers assuming that wall friction was negligible and that the hopper discharge orifice was relatively large, greater than 2 cm. The author identified the forces acting at the discharge orifice to be inertial force, gravity, fluid drag, inter-particle normal force, and inter-particle shear force. It was stated that the fluid drag force becomes more significant for fine and dense particles that are less than 200 μm in size, while free-flowing large grains experience negligible air drag and normal force effects at the orifice. Similarly, Tuzun (1999) listed contact frictional forces, hydrodynamic drag and surface adhesion as the main effects causing the dissipation of kinetic energy in granular systems. Surface adhesion forces (short-range attraction forces) become much greater than gravitational and hydrodynamic forces when particles are smaller than 20 μm (presence of Van der Waals and electrostatic forces) causing arching and caking of powders. Vessels with a height to diameter ratio greater than 3 were also discouraged due to large compressive forces.

The primary focus of this paper is to study the homogeneity of free-flowing granular and cohesive powder mixtures as a function of blender speed, and compare blend homogeneity before and after discharge from a tote blender. The sampling and analysis techniques described in earlier studies (Sudah et al., 2001a,b) were utilized to characterize the homogeneity of the two mixture types in the tote blender (in situ sampling) and in the bin following discharge (post-discharge sampling).

2. Material and methods

2.1. Apparatus and material

Blending experiments reported in this study were performed in geometrically similar GEA Gallay bin-blenders of two sizes shown in Fig. 1. The larger device (Fig. 1a) is a pilot scale 56 l (2 cu. ft.) vessel previously described in detail in Sudah et al. (2001a), while the smaller tote (Fig. 1b) is a custom built transparent 14 l (0.5 cu. ft.) vessel.

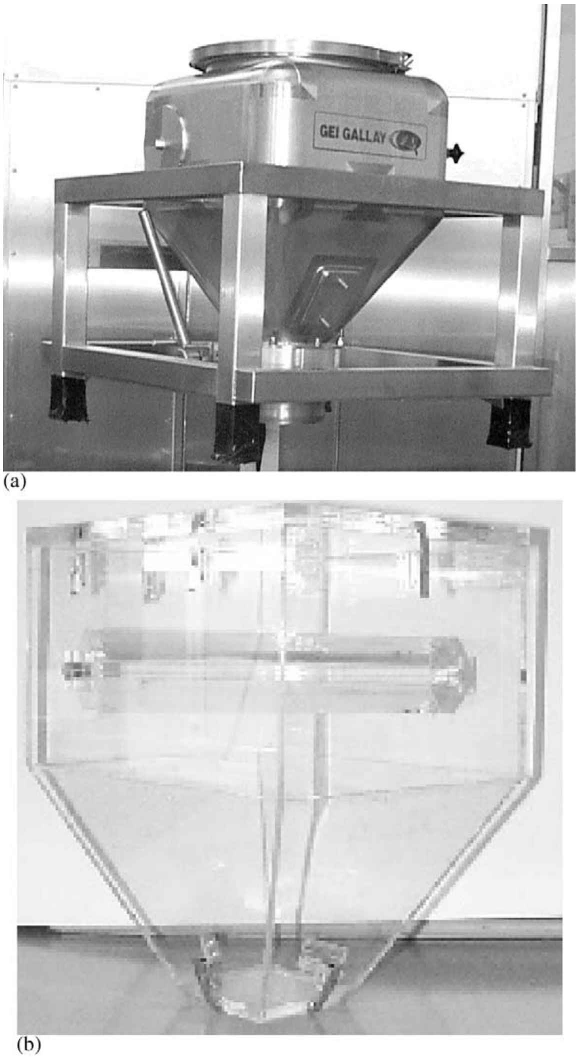


Fig. 1. Photographs of the two geometrically similar tote-blenders. (a) Side view of 56 l pilot scale tote blender mounted on the drive. (b) Side view of the 14 l tote vessel also used to conduct experiments.

The free-flowing material used in this investigation consisted of black and white art sand (crushed and surface-dyed silica particles) purchased from Clifford W. Estes Co. (Totowa, NJ, USA). The two components were used in equal amounts with resembling physical properties such as the average bulk density and mean grain size. The cohesive powder was a placebo blend composed of 96%wt microcrystalline cellulose (excipient), 3%wt NaCl salt (mock active ingredient), and 1% wt magnesium stearate (lubricant). Table 1 summarizes the material properties.

2.2. Experimental method

The experiments to determine the effect of rotational speed were performed in the 56 l tote for the free-flowing case and in the 14 l tote for the cohesive powder case. The 56 l blender was initially loaded with sand in a side-side fashion (see Fig. 2a) without the baffle, then operated at 6, 10, and 14 RPM ($0.0059 < \text{Froude} < 0.032$); 14 RPM is the maximum speed allowed by the manufacturer-supplied drive. The side-side loading pattern was chosen in order to examine the effect of speed on the rate-limiting axial mixing process (Sudah et al., 2001a). On the other hand, the 14 l vessel was loaded with powder using a hopper (Fig. 2b) with the microcrystalline cellulose poured in first followed by NaCl and then magnesium stearate. The 14 l transparent vessel containing the cohesive mixture was rotated at 5, 10, and 15 RPM ($0.0021 < \text{Froude} < 0.0192$). Similarly to the larger vessel, the 14 l vessel was sampled using core samplers through fixed sampling ports, shown in Fig. 2c. An average of 50 samples per experiment was collected from the 14 l vessel for both mixture types. As with all experiments presented in this paper, the mixture was discarded after sampling. Each time point in the mixing curves (Figs. 3–7) consisted of an independent experiment performed using fresh material.

For the discharge study, both the free-flowing sand and the cohesive powder were loaded into the 56 l vessel through a hopper. For the free-flowing case, the black sand was poured in first followed by the white sand. The placebo system was pre-blended (Sudah et al., 2001b) in order to minimize the agglomeration of the salt, and then poured into the vessel between two layers of microcrystalline cellulose; the magnesium stearate was the last component poured in. Blending experiments were performed at 10 RPM for various mixing times and fill levels as noted in the experimental grid (Table 2). At the end of each experiment, the mixture was sampled using nine core samplers spanning the vessel opening as described in

Table 1
Summary of the properties of the materials utilized in this study

Components	Free-flowing grains		Cohesive powder		
	White sand	Black sand	Microcrystalline cellulose	NaCl	Magnesium sterate
Proportion (wt.%)	50	50	96	3	1
Particle size (μm)	390	330	53	56	11
Bulk density (g/ml)	1.5	1.5	0.33	0.47	0.11

(Muzzio et al., 1999). The in situ sampled experiments were performed at a controlled humidity range of 30% RH ± 5%, consistent with the results presented in (Sudah et al., 2001a,b).

For the discharge experiments, mixing experiments were duplicated, however, the sampling step was different. Upon mixing in the tote blender, the mixture was freely discharged through a butterfly valve into a cylindrical bin 13 in. in diameter and 2 ft high. The mixture was subsequently sampled using core samplers at 13 different locations (compared with nine locations for the in-blender case) spanning the whole bin opening. The 13 cores extracted from the discharge bin produced

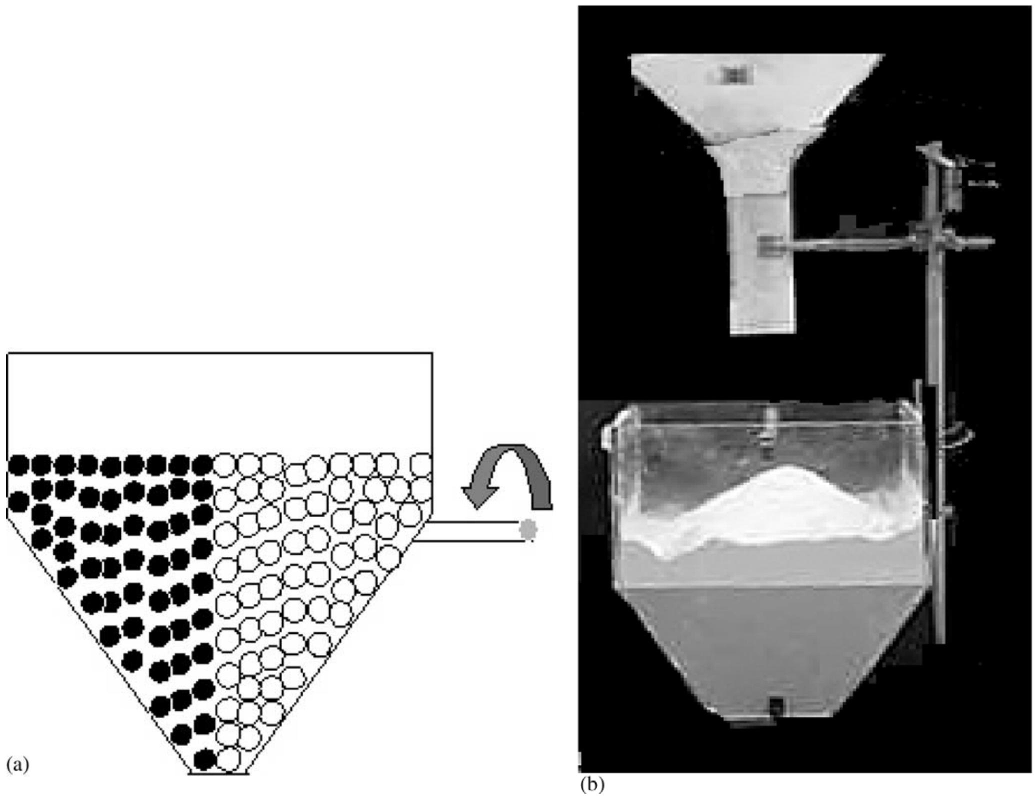
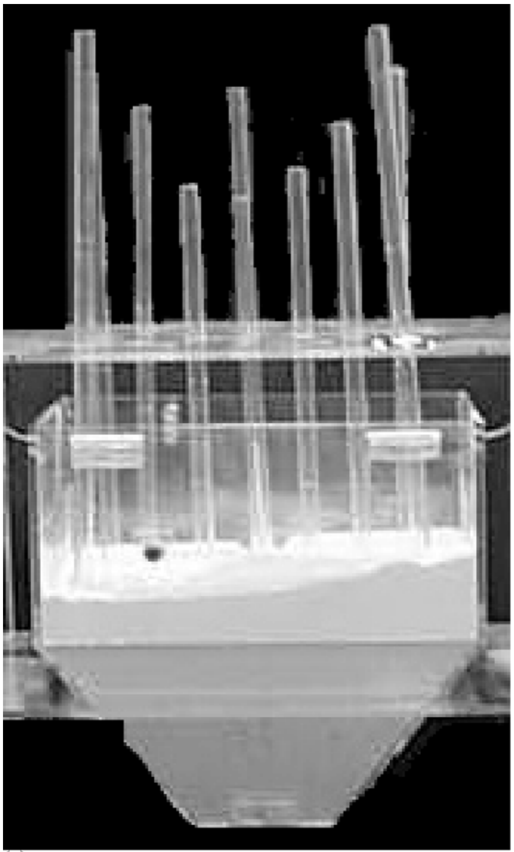


Fig. 2. (a) Side-side initial loading of white and black art sand in the 56 l tote Blender. (b) Initial loading condition of the pharmaceutical powder passed through a hopper into the transparent 14 l tote blender. (c) Core sampling scheme for the transparent 14 l vessel.



(c)

Fig. 2 (*Continued*)

an average of 300 samples per experiment for the free-flowing material compared with 100 samples obtained from 'in-blender' sampling. Similarly, post discharge sampling of the placebo blend produced an average of 450 samples per experiment compared with 200 for the respective 'in-blender' case. The discharged material was also discarded after each experiment. The samples from

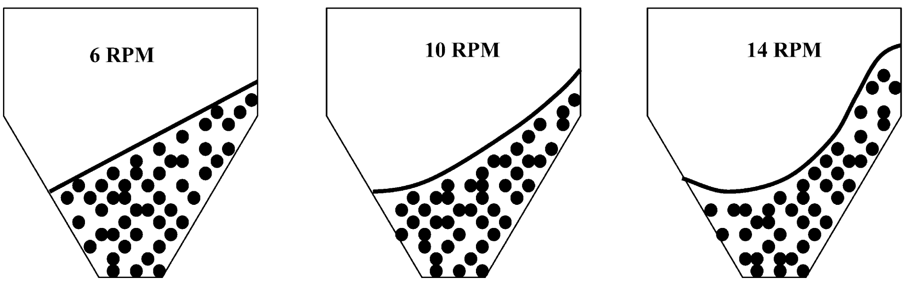


Fig. 3. Schematic representing the observed change in the flow profile of the free-flowing grains as a function of rotational speed. The upper surface was flat at 6 RPM and began to curve as the speed increased assuming an S-shaped profile at a speed of 14 RPM.

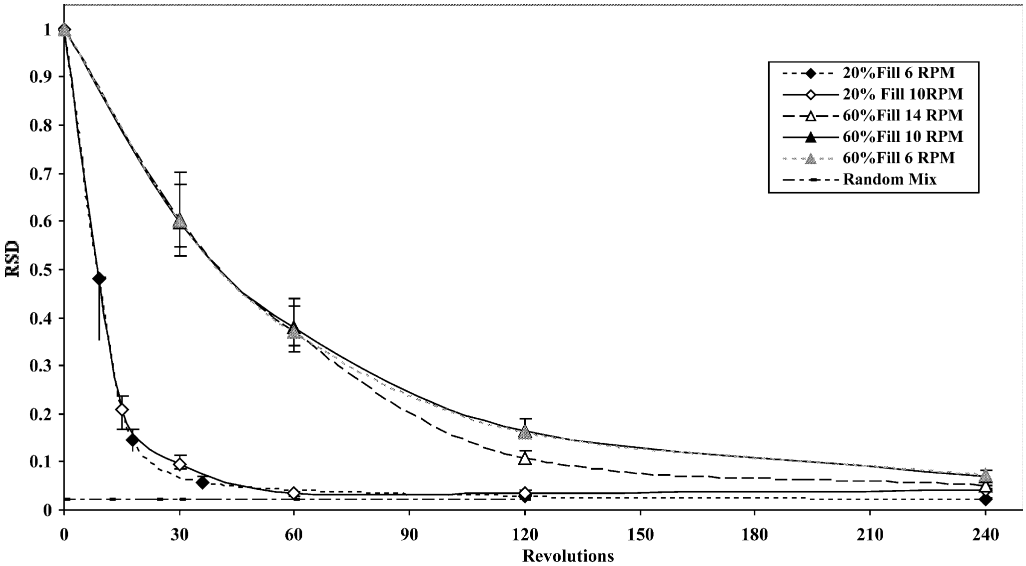


Fig. 4. Mixing curves for free-flowing material initially loaded in a side-side fashion in the 56 l vessel. The R.S.D. of the black component is plotted function of the number of revolutions for 20% fill at 6 RPM (closed diamonds, dotted line), 20% fill at 10 RPM (open diamonds, solid line), 60% fill at 14 RPM (open triangles, dotted line), 60% fill at 10 RPM (closed triangle, solid line), 60% fill at 6 RPM (gray triangles, dotted line). The dashed flat line represents the R.S.D. value for a random mixture.

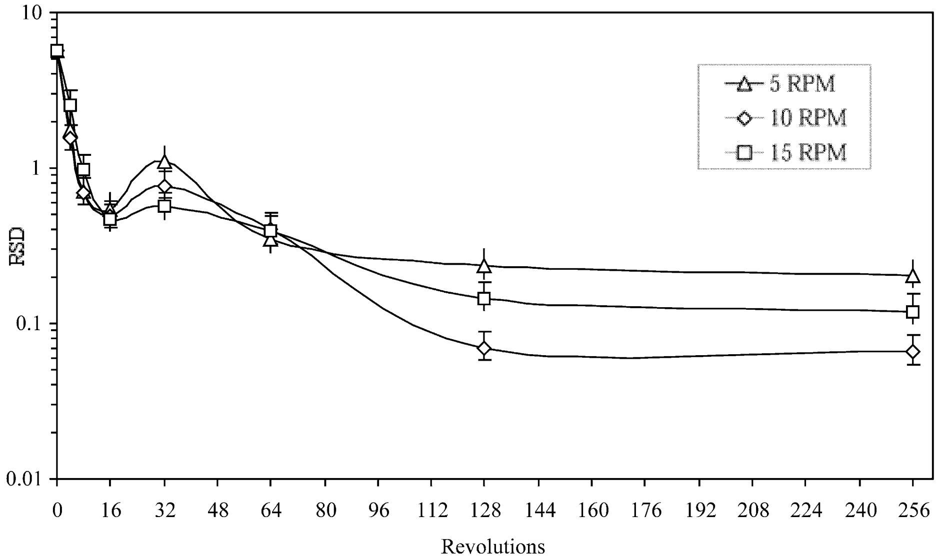


Fig. 5. Mixing curves for the cohesive powder system in the 14 l vessel at various rotational speeds. The R.S.D. of NaCl is plotted as a function of the number of revolutions for experiments at 60% fill 5 RPM (open triangles), 60% fill 10 RPM (open diamonds), and 60% fill 15 RPM (open squares).

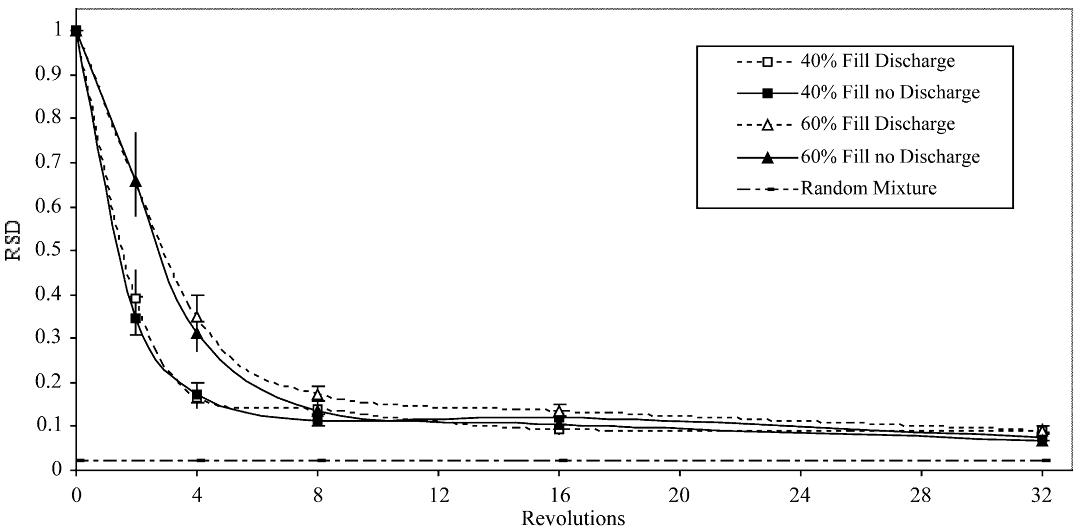


Fig. 6. Mixing curves for the discharged and non-discharged free-flowing system at 40 and 60% fill levels in the 56 l vessel. The R.S.D. of black component is plotted as a function of the number of revolutions at 10 RPM for discharged 40% fill experiments (open squares), discharged 60% experiments (open triangles), non-discharged 40% fill experiments (closed squares), and non-discharged 60% fill experiments (closed triangles).

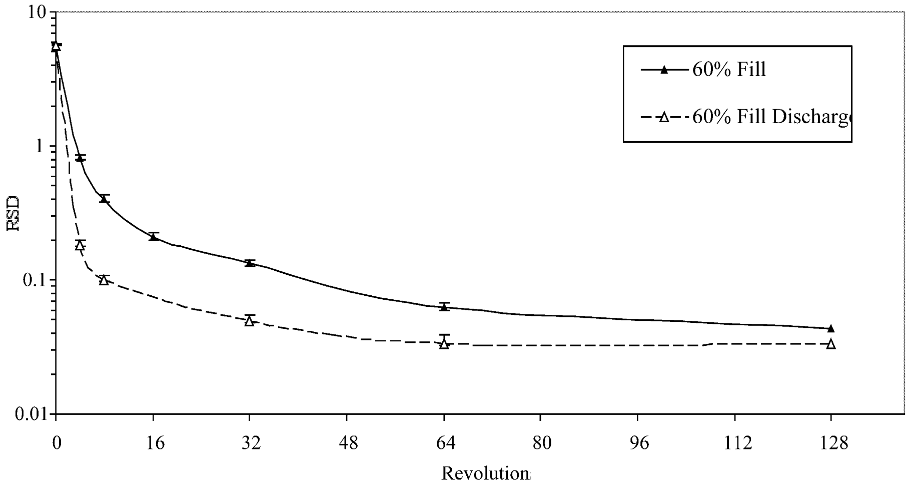


Fig. 7. Mixing curves for the discharged and non-discharged pre-blended cohesive powder system in the 56 l vessel. The R.S.D. of NaCl is plotted as a function of the number of revolutions at 10 RPM for 60% fill discharged case (dotted line), and the 60% fill non-discharged case (solid line).

Table 2
Experimental grid illustrating all the experiments discussed in this paper

	Free-flowing grains		Cohesive powder	
	Discharge	Rotation rate	Discharge	Rotation rate
Fill%	40 60	20 60	60	60
Number of revolutions	2, 4, 8, 16, 32,2,4,8,16,32	9, 18, 36, 60, 120, 240, 30, 60, 120, 240	4, 8, 16, 32, 64, 128	4, 8, 16, 32, 64, 128, 256
RPM	10	6 10 14	10	5 10 15

the free-flowing and cohesive mixtures were quantified using image analysis and solution conductivity, respectively (Sudah et al., 2001a,b).

3. Results and discussion

3.1. Effect of speed on mixing performance

Although the free-flowing experiments presented in this paper were conducted in the 56 l vessel, a few experiments were also conducted in the 14 l for flow visualization purposes. Fig. 3 illustrates the observed change in the shape of the flow region of the free-flowing material as the rotation speed increased from 6 to 10 to 14 RPM. At 6 RPM the bed's surface was flat and cascading slowly with surface curvature evolving as the speed was increased to 10 RPM. At 14 RPM the dynamic angle of repose further increased and the surface became S-shaped. The curvature of the flowing region increased the surface area, hence the overall volume of the cascading region of the flow increased.

Fig. 4 illustrates the outcome of varying the rotational speed on the homogeneity of the free-flowing mixture in the 56 l vessel at 20% fill (diamonds), and 60% fill (triangles) levels. The relative standard deviation (R.S.D., i.e. standard deviation of a component's sample concentration divided by it average concentration) of the black component estimated for a random mixture is included as a comparison (dashed line) (Sudah et al., 2001a). The 95% confidence interval for the R.S.D. value for each experiment (R.S.D. of black component) was estimated using the χ Squared Critical value method with error bars included in the plot. A 60% fill was chosen since previous work (Sudah et al., 2001a) demonstrated that it was the optimum fill for the current tote blender configuration, while 20% fill represents an extreme case (example of a sub-optimal fill). The 6 and 10 RPM mixing curves for 60% fill perfectly collapsed at all times while the 14 RPM curve mixed slightly better at longer times. All three curves, however, converged at 240 revolutions with their R.S.D. values being nearly equal to that of a random mixture (shown by the black dotted line, see Sudah et al., 2001a). Similarly, the 6 and 10 RPM mixing curves at 20% fill perfectly collapsed. Further experiments (14 RPM) at 20% fill were deemed unwarranted since the R.S.D. curves quickly decreased for short times reaching a plateau at 60 revolutions. Experiments at high fill levels (such as 80% fill recommended by some blender manufacturers) were avoided since the rate of mixing is known to be extremely slow due to the lack of tumbling space and the potential presence of a large segregated core (Sudah et al., 2001a).

The effect of rotational speed on the homogeneity of cohesive powders is displayed in Fig. 5. The R.S.D. of NaCl is plotted versus the number of revolutions for the 60% fill placebo mixture. Experiments were conducted in a 14 l vessel at 5, 10, and 15 RPM denoted by triangular, circular and square symbols, respectively. Error bars are shown at each point demonstrating the 95% confidence interval of the R.S.D. value for each experiment. Taking into account the error bars at each point, differences between the curves were not significant up to 64 revolutions, and then became significantly different, each one leveling off at a different plateau, perhaps reflecting the role of the shear rate (which depends on RPM) on mixing of cohesive powders. The shape of the mixing curves in Fig. 5 resembled those observed in the 56 l tote for the same mixture (Sudah et al., 2001b), which did not decay monotonically with time and had a spike at short time (32 revolutions). This observation suggests that material flow in the 14 l vessel did not greatly vary from that in the larger tote. The high R.S.D. values observed at 32 revolutions are caused by the slow turnover of the center-loaded mixture. After just a few revolutions, the active returns to a position mainly in the central region of the blender, resulting in high salt concentration in some of the samples and very low concentration in the rest of the samples. This phenomenon caused the spike in R.S.D. values observed at 32 revolutions for all three rotation rates. This effect was especially exacerbated at 5 RPM since it had the slowest material turnover rate, followed by 15, and then 10 RPM.

It seems surprising at first that mixing fine powders in tumbling blenders at 10 RPM should perform better than at 15 RPM. However, visual observation of the flow in the transparent vessel shed light on this phenomenon. At 5 RPM, the surface flow was made up of discrete avalanches of powder that did not slide from one end of the blender to the other, but rather dissipated shortly after they began. At 10 RPM, the frequency of avalanches per rotation increased, nevertheless remaining discrete in time. On the hand, the 15 RPM case was different. The powder located at the highest region of the sloping surface simply slid down the entire slope to the opposite walls of the blenders, in larger ‘glacier like’ avalanches, and exhibited fewer avalanches per time than the 10 RPM case. The increase in the number of avalanches per revolution signified a higher dispersive rate of mixing observed after 64 revolutions (the 10 RPM curve had the lowest R.S.D. value), while fewer avalanches of larger powder portions resulted in a greater radial (convective) mixing component after 32 revolutions (the 15 RPM experiments had the smallest spike).

3.2. Effects of discharge on blend homogeneity

The results of sampling after discharge for free-flowing material out of the 56 l vessel, compared with those of the in situ sampling case, are displayed in Fig. 6, where the R.S.D. of the black component is plotted versus the number of revolutions. Experiments were conducted at 10 RPM for both 40 and 60% fill levels, denoted by square and triangle symbols, respectively. The open symbols denote the R.S.D. for the discharged mixture, the closed symbols represent the R.S.D. for the in situ mixture, and the dashed line represents the estimated R.S.D. of a random mixture (Sudah et al., 2001a). The mixing curves for the 40% fill level nearly collapsed and were practically identical, indicating that at low fill percentage, where the mixing in the tote is efficient (insignificant segregated core), the homogeneity is unaffected by discharge. Although the 60% fill mixing curves did not collapse as closely as the 40% fill case, they were statistically similar (within the 95% confidence interval) and mixed slightly slower (Sudah et al., 2001a).

Different from the free-flowing case, the discharge of fine cohesive pharmaceutical powders resulted in a significant improvement in mixture homogeneity. Fig. 7 (R.S.D. of NaCl vs. number of revolutions), illustrates the mixing curves for discharged (open triangles) and non-discharged (closed triangles) pre-blended mixtures of microcrystalline cellulose (96%), NaCl (3%), and magnesium stearate (1%) at 60% fill. The difference between the two mixing curves was much wider than what the 95% confidence interval error bars can account for. The improvement in the homogeneity of these fine powders is attributed to the shearing of powders upon exiting the tote-blender, and is consistent with the improvement in mixing performance observed at higher RPM. The flattening of the discharge curve at a plateau of 3% R.S.D. was probably due to the limited amount of shearing of the powder at the blender exit.

4. Conclusions

Previously developed techniques for sampling and analysis were employed in this study to characterize the effects of blender rotational speed and blender discharge on the homogeneity of a free-flowing mixture and a cohesive placebo formulation.

In the inertial regime (practical mixing speeds), the homogeneity of free-flowing granular material with similar physical properties mixed in a pilot plant scale tote blender was found to be independent of tumbling speed. This agreed with the findings of previous studies (Brone et al., 1998; Brone and Muzzio, 2000) indicating that the mixing performance in bench scale blenders was independent of rotational speed for free-flowing material, despite a 56 fold increase in capacity (56 l tote vs. 1L V-blender and 5 l double-cone blender). Therefore, for most industrial applications processing similar free-flowing grains, picking the proper tumbling speed should in general be regarded as a non-issue.

However, the homogeneity of cohesive powders in the inertial regime ($0.0021 < F_r > 0.0192$) is dependent on tumbling speed. Surprisingly, mixing at a speed of 10 RPM out-performed mixing at 5 and 15 RPM, presumably due to the complex nature of the flow; the size and frequency of avalanches per rotation seemed to determine the overall rate and degree of mixing. The 10 RPM experiments had more avalanches per rotation than the 5 RPM case, and smaller but more frequent avalanches per turn than the 15 RPM case. Although mixing was best at a speed of 10 RPM, if a higher degree of blend homogeneity is desired (well below 10% R.S.D.), it is recommended to use a blender equipped with intensifier bars or introduce a high shear pre-blending step (Sudah et al., 2001b). In view of the complex dependency on blender speed, scale-up of cohesive powder mixing processes will remain a major manufacturing issue. Clearly, more work is needed to further understand this issue.

Moreover, another critical manufacturing issue is whether to rely on in-blender or post-discharge sampling for the assessment of final blend homogeneity. In the case of free-flowing material with similar particle properties (especially particle size), the mixture can be reliably sampled in situ where the homogeneity of discharge from the pilot scale tote blender was found to be statistically equivalent to that prior to discharge. On the other hand, cohesive powders with similar physical properties appreciably mixed upon being discharged from the tote blender. It is likely that the powder was sheared while exiting the blender thus mixing further. Thus, since the best mixed state for cohesive powders is difficult to estimate, it is recommended that post-discharge sampling be performed whenever cohesive material is mixed.

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