Dynamics of axial segregation in granular slurries: Parallel experiments and influence of aspect ratio and periodic tilting

Stanley J. Fiedor,[1] Paul Umbanhowar,[2] and Julio M. Ottino[1]
[1]*Department of Chemical and Biological Engineering, Northwestern University, Evanston, Illinois 60208, USA*
[2]*Department of Physics and Astronomy, Northwestern University, Evanston, Illinois 60208, USA*
(Received 19 January 2006; published 20 April 2006)

An efficient technique for conducting rotating tumbler experiments in parallel is introduced and used to study the effect of tumbler length and periodic tilting of the tumbler on axial segregation. When rotated, bidisperse granular slurries segregate into what appear at the surface to be alternating bands of larger and smaller particles. The number of bands increases linearly with tumbler length while the fractional area occupied by each type of band is constant. Periodic tilting of the rotation axis induces a periodic axial flow of particles in the flowing layer. For the range of tilt angle amplitudes investigated (0°–3.5°), the number of bands decreases with increasing angle, but the rate of merging and the fractional area of bands rich in smaller particles are unaffected.

DOI: 10.1103/PhysRevE.73.041303 PACS number(s): 45.70.Mg, 05.65.+b, 45.70.Qj

I. INTRODUCTION

Flowing granular materials are ubiquitous in both nature and technological applications. Ubiquity, however, has not resulted in a comparable level of understanding, and many phenomena remain poorly understood. For example, flowing granular mixtures tend to segregate when particle properties differ [1,2]. In rotating tumblers partially filled with particles of different size or density, the flow drives radial segregation perpendicular to the axis of rotation; the segregation is due to percolation for size difference and "buoyancy" for density difference [3]. For bidisperse mixtures radial segregation typically occurs within a few rotations of the tumbler. In long tumblers and with $O(10^2)$ rotations, the core of smaller particles may grow in various locations and penetrate the surface which results in a configuration comprised of what appear—at the free surface and boundaries of the container—to be bands alternately rich and lean in one component. This is referred to as axial segregation.

Since the discovery of axial segregation by Oyama [4], several aspects of the onset of axial segregation have been investigated. Most of these studies focus on dry granular systems (DGS), where the interstitial fluid is a gas. Gupta *et al.* [5] found that the onset of axial segregation depends on the rotational speed for dry binary mixtures of grain sizes. They report that axial segregation occurs only for rotation speeds greater than a critical value when the difference between the repose angles of the pure components is sufficiently large. Hill and Kakalios [6] found a critical rotation speed above which axial segregation occurs for certain mixtures of glass particles, and that axial segregation is reversible. They also report that axial segregation occurs when a sufficient difference exists between the repose angles of the mixture and the larger particles. Choo *et al.* [7] found that transient waves of segregation may develop during the initial stages of band formation. Hill *et al.* [8] studied segregation onset in tumblers with circular and square cross sections for a narrow range of fill levels near half full. They found that remnants of radial segregation patterns seen in quasi-two-dimensional tumblers remain in the longer tumblers at corresponding fill levels, and that axial segregation is sensitive to fill level for square cross sections but much less so for circular cross sections.

Several experimental studies investigated the long time dynamics of segregation bands. Nakagawa [9] and Hill *et al.* [10] found merging of bands with increasing rotation. Fiedor and Ottino [11] observed both traveling waves and band merging depending on the rotation speed in systems with air as the interstitial fluid.

More recent research has focused on granular slurries or liquid granular systems (LGS) where the particles are completely immersed in a liquid [11–15]. In standard tumbling segregation experiments in DGS, particle interactions dominate and the viscosity of the interstitial fluid plays little or no role. In LGS, the viscosity and density of the interstitial fluid is typically many orders of magnitude greater than those of typical gases and cannot be neglected. There is no physical reason to expect similar results in LGS and DGS, yet axial segregation occurs in both. Jain *et al.* [12] found that axial bands form more quickly when the interstitial fluid is a liquid rather than air. Fiedor and Ottino [11] focused on the long time behavior of axial segregation in LGS and DGS and found that the number of bands in both systems decreases logarithmically with time. Arndt *et al.* [15] studied LGS in tumblers with a fixed tilt of the rotation axis so that the fill level varies from one end of the tumbler to the other. They found that the bands and core at a given axial position qualitatively correspond with those that form for the corresponding fill level in a horizontal cylinder.

LGS offer several advantages over DGS. Humidity, electrostatic charging of particles, abrasion, and cohesive forces due to moisture—problems that plague even the simplest DGS experiments—play little or no role in LGS. Consequently, experiments with small particles in small LGS are feasible (for example, we have observed axial segregation of 80 and 300 μm diameter particles in 4 mm diameter tumblers [16]). The control of body forces via buoyancy offers another advantage. Also, with suitable fluids and proper illumination, the interior of a granular bed can be visualized without recourse to x-ray or magnetic resonance imaging.

One challenge of studying band dynamics in axial segregation is the lengthy times needed to conduct experiments. A

second challenge is the large number of experimental parameters; these include particle properties (size, size distribution, shape, density, friction coefficient), tumbler geometry (length, width, cross section), control parameters (tilt, rotation rate), and interstitial fluid properties (density, viscosity). The combination of these two factors, long run times and a high dimensional parameter space where the relevant dimensionless numbers are yet unknown, makes experimentation time consuming. The band merger process is logarithmic with the number of rotations so experiments are time consuming. Bands form in $O(10^2)$ revolutions; thus, to study band merging for time intervals spanning two orders of magnitude requires $O(10^4)$ revolutions, almost 17 h for experiments running at 10 rpm (a typical rotation rate for our experiments). Besides having to perform multiple experiments to explore a range of experimental parameters, the relatively small number of segregation bands N and their integer nature demands additional runs to accurately determine $N(t)$. Initial band thickness is approximately the tumbler diameter D so a system with aspect ratio L/D generates $O(L/D)$ bands. The minimum number of bands is three since a band of large particles is always present at an end wall. Thus, both the maximum number of bands and the ratio of the maximum to minimum number of bands ($\approx(1/3)L/D$) are typically small. In most experiments to date $L/D=O(10)$. Taking all factors into account, comprehensive studies of band merger and other long duration phenomena are clearly prohibitive with serial experiments.

A concern often raised in regard to axial segregation experiments is the effect of tumbler length on band dynamics. In dry systems bands first appear near the end walls [11]. Particle tracking velocimetry experiments indicate that wall friction induces axial flow near the ends of the tumbler [17]. Donald and Roseman [18] concluded that the effect of the end wall friction is fundamental in starting axial segregation, and Khosropour *et al.* [19] found that if the end wall friction is large enough, axial bands propagate from the end walls. End wall friction is present in most axial segregation experiments, but its influence on band formation and dynamics in granular slurries is unknown. Typical experimental configurations prevent experiments with large L/D.

The purpose of our paper is twofold. The first objective is to introduce a method that allows multiple axial tumbler experiments to run in parallel resulting in considerable time savings; our experiments would have taken eight times longer to complete had they not been run in parallel. The second objective, which makes use of this parallel technique, is to examine how end wall effects and periodic axial flow influence axial segregation. We vary tumbler length to determine the effect of end walls on band formation and long time dynamics. We periodically tilt the tumbler rotation axis about horizontal to examine the influence of axial flows caused by nonuniform or nonlevel tumblers on coarsening dynamics.

II. PARALLEL TECHNIQUE AND EXPERIMENTAL METHODS

Axial segregation experiments conducted in parallel can be accomplished in various ways. A particularly efficient method is to attach multiple tumblers to the outside of a larger horizontal tube which is then rotated about its axis with angular velocity ω (see Fig. 1). The tumblers rotate at the same frequency as the center tube but with additional translation. There are many advantages of using such a system: there is only one axis of rotation so no additional programming or maintenance of motors is needed; the number of tumblers that can be rotated is variable; altering the ratio of the center tube diameter to the tumbler diameter changes the maximum number of experiments that can run simultaneously. Finally L/D can be large (41.5 in one of our experiments). Also, rocking motions can be induced by using a center tube of square cross section and mounting the tumblers at an angle θ_0 [see Figs. 1(c) and 1(d)] which results in a time periodic variation of the tumbler angle with respect to horizontal, $\theta=\theta_0\sin(\omega t)$.

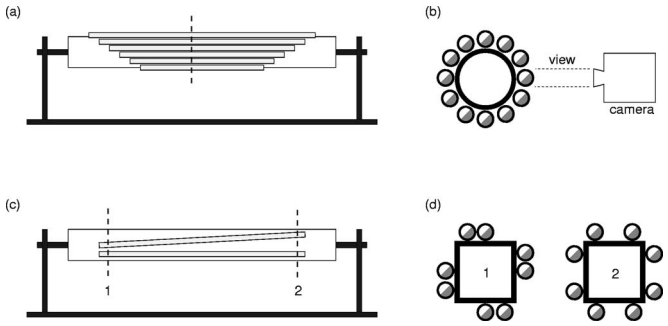

FIG. 1. Schematic of the experimental apparatus. (a) Side view and (b) cross section of the apparatus used to investigate tumbler length. Twelve tumblers are attached to the outer wall of a large center tube. (c) Side view and (d) cross sections of the apparatus used to investigate tumbler rocking. Eight tumblers are attached to the outer wall (two on each side) of a larger center tube with square cross section.

In both parallel and serial tumbler systems grains rotate about the rotation axis. At a distance r from the rotation axis a centripetal acceleration $r\omega^2$ exists. The centripetal acceleration is larger by approximately the ratio of the center tube diameter to the tumbler diameter (we assume the former is much larger than the latter). Perhaps a more relevant measure is the ratio of the centripetal acceleration to the gravitational acceleration at the Earth's surface g; the Froude number $r\omega^2/g$ has a maximum of $O(10^{-3})$ for our experiments. Also note that when grains in the flowing layer move with velocity v with respect to the stationary bed, an additional acceleration exists which is proportional to ωv corresponding to the Coriolis force when the motion is analyzed in the reference frame rotating at ω and with origin on the rotation axis. Since this acceleration does not depend on r, it is the same for tumblers operated singly or in parallel.

For all our experiments, acrylic tumblers capped with rubber end plugs and with inner diameter 1.59 cm and outer diameter 2.22 cm are filled halfway with a 1:1 volume ratio mixture of small and large glass beads. Water fills the remaining volume of the tumbler. The smaller black beads are 272 ± 24 μm in diameter, while the larger transparent beads are 882 ± 30 μm in diameter. The small tumblers are placed on the larger center tube and the beads are initially mixed by rotating the system at 100 rpm for 1 min. After mixing the

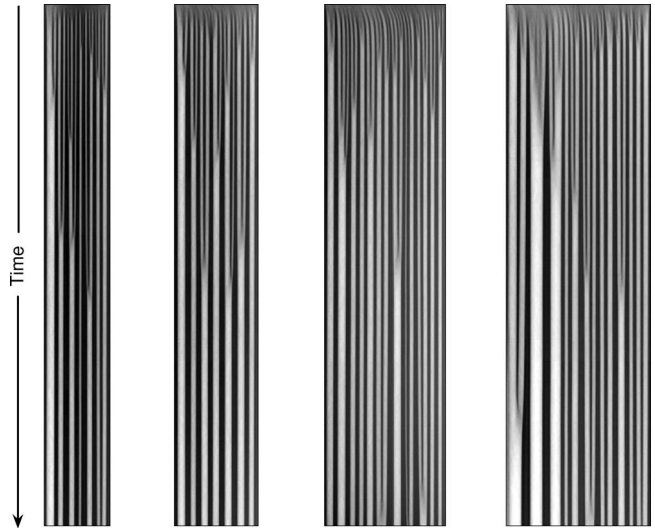

FIG. 2. Typical spatiotemporal images of experiments with tumbler lengths of 27.7, 35.2, 50.9, and 60.8 cm. Time increases from top to bottom (0 to 4200 revolutions or 7 h).

tumblers are rotated at 10 rpm for 4200 rotations; at this speed the flow is continuous (individual avalanches are not evident) and the free surface of the grains is flat. Experiments are repeated until at least 30 sets of experimental data for each tumbler length and θ_0 are acquired. A Kodak Megapixel 1.4i digital camera images the entire length of each tumbler from the side as the tumblers move up [see Fig. 1(b)]. Imaging the interface between beads and tumbler, rather than the flowing layer, ensures a clearer image. The camera is synchronized to the stepper motor, and images of each tumbler are acquired at the same angular position on each rotation.

To study the effect of tumbler length, 12 acrylic tumblers of inner length ranging from 27.7 to 66 cm long are used. The center tube is 7.6 cm in outer diameter and 76 cm long. It is rotated about its axis by a dc stepper motor with a planetary gear drive. Figures 1(a) and 1(b) show the side view and cross section of the system, respectively.

To study the effect of periodic tilting, eight tumblers of inner length 27.7 cm are attached to a larger 76 cm long horizontal center tube with square cross section (7.6×7.6 cm^2). The tumblers are arranged at angles ranging from 0° to 3.5° to the axis of rotation. Figures 1(c) and 1(d) show the side view and cross sections of the system, respectively.

The time evolution of the axial segregation process is studied by means of space-time sequences. Images of the entire tumbler are cropped to contain only the particles in the tumbler. Since there is no significant vertical variation in images of the segregation bands, images are vertically averaged to produce a one-dimensional (1D) array of the average intensity along the tumbler. 1D arrays are concatenated to form a spatiotemporal series of 4200 rotations of the tumbler, see examples in Fig. 2. Darker areas represent regions rich in smaller particles, while lighter areas are regions of mostly larger particles. The narrow, horizontal dark regions at the top of the spatiotemporal images in Fig. 2 represent mixed particles, although a core of smaller particles forms after a few rotations, and, as in other axial segregation studies [12,20], likely continues to exist beneath the bands of larger particles after axial segregation has occurred.

To extract quantitative data, further thresholding of the spatiotemporal images is necessary. First the spatiotemporal images are smoothed using a two-dimensional Savitzky-Golay filter to reduce lighting inhomogeneities. Next each line associated with a fixed time is independently thresholded according to the average pixel value of the line. The result is a binary spatiotemporal image from which data such as the number of bands at the surface and the band area are obtained.

III. RESULTS

Results are presented from two sets of experiments—aspect ratio experiments and periodically tilted experiments. In the aspect ratio experiments the tumbler length is varied while keeping the tumbler diameter constant to investigate how band dynamics vary with tumbler length. In the periodically tilted experiments the tumbler axes are inclined with respect to the rotation axis of the center tube to induce a periodic axial flow of particles.

Figure 2 shows representative examples of spatiotemporal images for experiments in four different tumbler lengths (27.7, 35.2, 50.9, and 60.8 cm). Superficially all four images show the same band dynamics. Bands form during an initialization period after which no further bands appear. Later, some bands merge resulting in wider but fewer bands. This scenario holds for all tumbler lengths studied. There is no evidence of waves or additional band formation after the initialization period which stands in contrast to previous studies with air as the interstitial fluid [11].

Figure 3(a) shows the evolutions of the number of surface bands rich in smaller particles (dark bands in the spatiotemporal images) for tumbler lengths L of 27.7, 30.3, 35.2, 42.8, 50.9, 55.6, 60.8, and 66.0 cm. Each data set is an average of 30 trials. The number of surface bands increases to a maximum after about 100 rotations and then decreases logarithmically with rotations in all cases. Longer tumblers on average have more surface bands. The number of bands decreases by about 30% from the maximum after $O(10^3)$ revolutions. Figure 3(c) shows the average number of surface bands rich in smaller particles versus the tumbler length after 100, 500, 1000, 2000, and 4000 revolutions. The number of surface bands varies linearly with the tumbler length at all times shown. Dividing the number of surface bands by the tumbler length produces some collapse in the data [see Fig. 3(b)]. The average number of surface bands per unit length when plotted against tumbler length is approximately constant [Fig. 3(d)], although a slight decrease with increasing L is evident.

Figure 4(a) shows that the fractional area of surface bands rich in smaller particles slowly increases during the band initialization period. However, for the remainder of the experiment (even during band merger) the fractional area remains constant for all tumbler lengths. Again, each data set is an average of 30 experiments, but individual runs show similar results. Bands rich in smaller particles occupy approximately half of the tumbler area, and increasing the tumbler

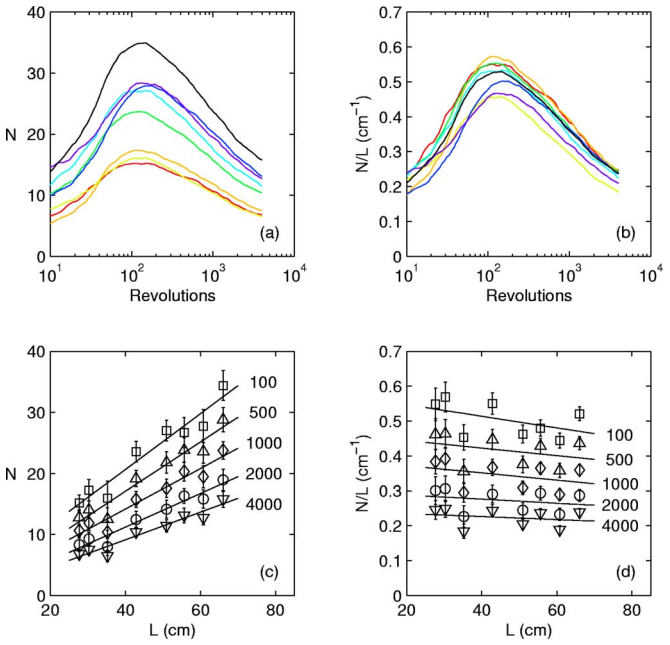

FIG. 3. (Color online) (a) Evolutions of the number of surface bands rich in smaller particles. (b) Evolutions of the number of surface bands rich in smaller particles per unit length. (c) Number of surface bands rich in smaller particles versus tumbler length after 100 (□), 500 (△), 1000 (◇), 2000 (○), and 4000 (▽) revolutions. (d) Number of surface bands per unit length rich in smaller particles versus tumbler length after 100 (□), 500 (△), 1000 (◇), 2000 (○), and 4000 (▽) revolutions.

length slightly increases the fractional area per unit length occupied by the bands [see Fig. 4(b)]. This small increase in area is most likely due to friction near the end walls; particle velocities and trajectories are different near the end walls [17] and may affect the degree of segregation.

Since the fractional area remains constant after the initialization period, no further segregation is occurring. Even with some bands merging to form larger bands, the fractional area occupied by each type of band is not affected. There is a direct connection between bands of small particles via the continuous core of smaller particles which runs beneath the surface of the bands rich in large particles. When bands of large particles merge the small particles from the dwindling band move to the core as, simultaneously, small particles in

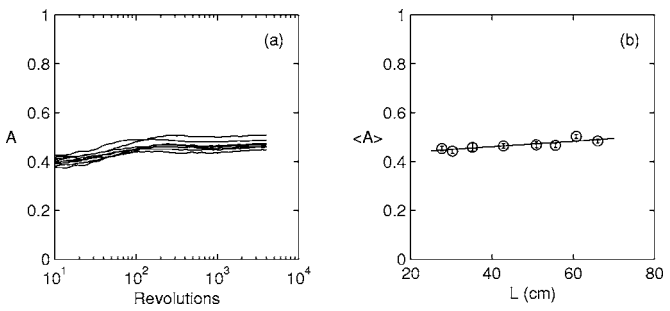

FIG. 4. (a) Evolution of the fractional area of surface bands rich in smaller particles. (b) Time averaged fractional area of surface bands rich in smaller particles versus tumbler length.

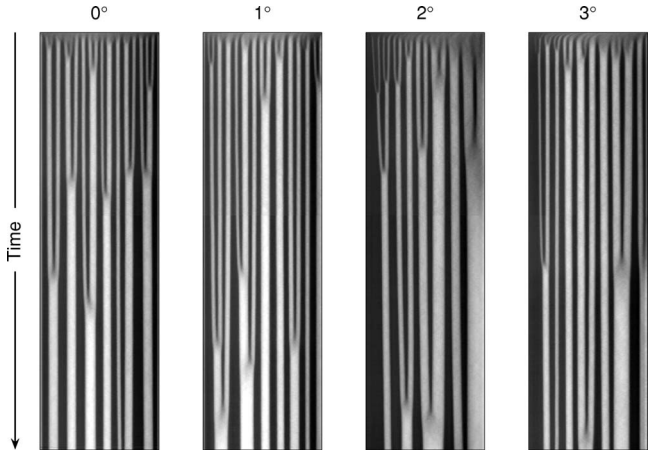

FIG. 5. Typical spatiotemporal images showing band dynamics over 4200 rotations (7 h) for experiments with maximum rocking angles from horizontal of 0°, 1°, 2°, and 3° in a 27.7 cm long tumbler.

the core emerge at the surface in the regions previously occupied by the bands of large particles. The intensity of bands of large particles in the spatiotemporal images depends on the band width. Wider bands of large particles have higher intensities than the narrower bands of large particles [see Fig. 2], and the intensity grows linearly with band width. The bands of small particles, on the other hand, have the same intensity independent of bandwidth.

Figure 5 shows spatiotemporal images in tumblers periodically tilted at four different maximum angles (0°, 1°, 2°, and 3°). All four images show the same type of band dynamics—bands form after an initialization period and then some merge. We observe that tilting of the tumbler produces a periodic axial flow of material, including the bands, as previously reported [21]. However, bands in the spatiotemporal images that are not merging show no axial movement since the images are acquired with the same period as the rocking.

We next investigate the evolution of the number of surface bands rich in smaller particles (N) as a function of revolutions for $\theta_0=\{0°,0.5°,1.0°,1.5°,2.0°,2.5°,3.0°,3.5°\}$. Figure 6(a) shows that N increases to a maximum and then slowly decreases for each θ_0. The maximum value of N is

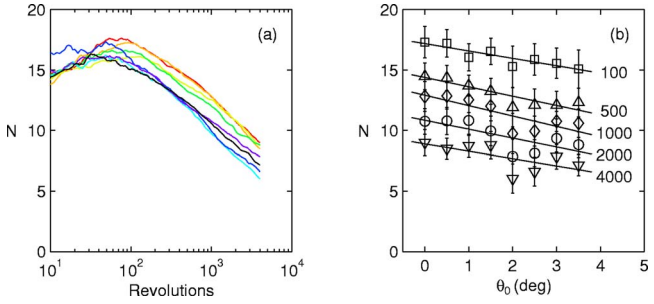

FIG. 6. (Color online) (a) Evolution of the number of surface bands rich in smaller particles for eight distinct maximum tilt angles θ_0. (b) Number of surface bands rich in smaller particles versus θ_0 after 100 (□), 500 (△), 1000 (◇), 2000 (○), and 4000 (▽) revolutions.

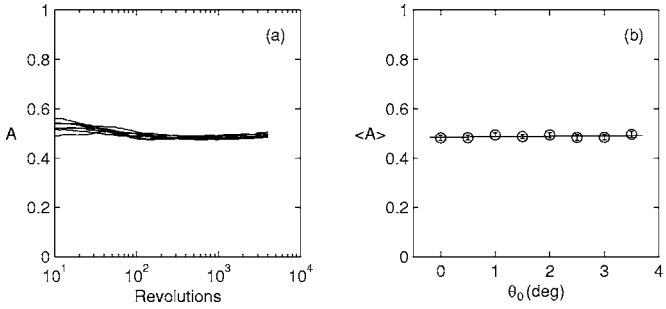

FIG. 7. (a) Evolution of the fractional area of surface bands rich in smaller particles versus revolutions for $\theta_0 = \{0°, 0.5°, 1.0°, 1.5°, 2.0°, 2.5°, 3.0°, 3.5°\}$. (b) Time averaged fractional area of surface bands rich in smaller particles versus θ_0.

again reached after about 100 revolutions in all cases. Figure 6(b) shows the average number of surface bands rich in smaller particles at various times during the experiments. The number of surface bands decreases slightly with increasing θ_0. The slope of the best fit lines for the data in Fig. 6(b) differ little, indicating that the merger rate is basically constant after band initiation, but the number of bands that initially form decreases with increasing θ_0. When bands form, their size and location depend on the initial conditions, which are random because the system is initially well mixed. We speculate that when two bands begin to form near each other, the periodic axial flow merges the bands before they reach the tumbler surface. The higher the rocking amplitude the more likely bands near each other will merge before reaching the surface. Further investigation is necessary to validate this picture.

Figure 7(a) shows a plot of the evolution of the fractional area of surface bands rich in smaller particles. The results are similar to the experiments with various tumbler lengths—after bands form the fractional area remains constant for the duration of the experiment, and the bands rich in smaller particles again take up approximately half of the tumbler area. Figure 7(b) shows that the added axial flow does not change the fractional area of the bands for the limited range of angles we have explored.

IV. CONCLUSIONS

Possibly the biggest hurdle to studying band dynamics in axial segregation is the amount of time required to collect enough data for analysis. The total time can be on the order of a few days to weeks for a single experiment. We have implemented a system—a method of conducting experiments in parallel—that allows for many experiments to run simultaneously with only minor additional setup time. The experiments reported here, if run separately, would have taken 140 straight days (3360 h) not including the time to setup each run. Instead, the time devoted to running experiments using the parallel setup was less than 18 days (420 h).

As mentioned above, the question of how long a tumbler must be to assure end effects are minimal arises frequently in the context of axial segregation experiments. We found that the number of segregation bands scale linearly with the tumbler length. Increasing the tumbler length allows for more bands but the intrinsic dynamics do not change; thus with suitable caveats all previous experiments in various tumbler lengths give meaningful results. A similar issue arises in the context of deviation of the tumblers with respect to the horizontal. Here we took a worst case scenario: the axis of rotation does not coincide with the axis of the tumbler leading to tumbler rocking. Our results indicate that the maximum number of bands decreases with higher rocking amplitude, but the rate of merging is not affected by the rocking. We find also that the fractional area of the bands rich in smaller particles is unaffected by the rocking motion. Taken together, these two findings are good news since they demonstrate that axial segregation is a robust phenomenon. But robustness has a price: There is now a clear challenge to identify dimensionless numbers to reduce the large parameter space and to explain why bands form and merge.

[1] H. A. Makse, S. Havlin, R. K. King, and H. E. Stanley, Nature (London) **386**, 379 (1997).
[2] K. M. Hill, D. V. Khakhar, J. F. Gilchrist, J. J. McCarthy, and J. M. Ottino, Proc. Natl. Acad. Sci. U.S.A. **96**, 11701 (1999).
[3] N. Jain, J. M. Ottino, and R. M. Lueptow, Phys. Rev. E **71**, 051301 (2005).
[4] Y. Oyama, Sci. Pap. Inst. Phys. Chem. Res. (Jpn.) **37**, 17 (1939).
[5] S. D. Gupta, D. V. Khakhar, and S. K. Bhatia, Chem. Eng. Sci. **46**, 1513 (1991).
[6] K. M. Hill and J. Kakalios, Phys. Rev. E **52**, 4393 (1995).
[7] K. Choo, T. C.A. Molteno, and S. W. Morris, Phys. Rev. Lett. **79**, 2975 (1997).
[8] K. M. Hill, N. Jain, and J. M. Ottino, Phys. Rev. E **64**, 011302 (2001).
[9] M. Nakagawa, Chem. Eng. Sci. **49**, 2540 (1994).
[10] K. M. Hill, A. Caprihan, and J. Kakalios, Phys. Rev. E **56**, 4386 (1997).
[11] S. J. Fiedor and J. M. Ottino, Phys. Rev. Lett. **91**, 244301 (2003).
[12] N. Jain, D. V. Khakhar, R. M. Lueptow, and J. M. Ottino, Phys. Rev. Lett. **86**, 3771 (2001).
[13] S. Courrechdupont, P. Gondret, B. Perrin, and M. Rabaud, Phys. Rev. Lett. **90**, 044301 (2003).
[14] N. Jain, J. M. Ottino, and R. M. Lueptow, J. Fluid Mech. **508**, 23 (2004).
[15] T. Arndt, T. Siegmann-Hegerfeld, S. J. Fiedor, J. M. Ottino, and R. M. Lueptow, Phys. Rev. E **71**, 011306 (2005).
[16] S. J. Fiedor and J. M. Ottino (unpublished).
[17] N. Pohlman, S. Meier, J. M. Ottino, and R. M. Leuptow, J. Fluid Mech. (to be published).
[18] M. B. Donald and B. Roseman, Br. Chem. Eng. **7**, 749 (1962).
[19] R. Khosropour, E. Valachovic, and B. Lincoln, Phys. Rev. E **62**, 807 (2000).
[20] K. M. Hill, A. Caprihan, and J. Kakalios, Phys. Rev. Lett. **78**, 50 (1997).
[21] J. F. Gilchirst and J. M. Ottino, Phys. Rev. E **68**, 061303 (2003).