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Granular Flow Fields in Vertical High Shear Mixer Granulators

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This study aims to elucidate some basic features of the granular flow field in a conical frustum-shaped vertical high shear mixer granulator by using the Positron Emission Particle Tracking technique. The tested range of particle size ($d_{50} = 23$, 43, and 60 μm) and shape as quantified by the angle of internal friction (26° and 38°) have a negligible effect on the granular flow field in the mixer granulator. Density of particle imposes great influence on the flow field where reducing density from 2600 to 700 kg m^{-3} increase the tangential velocity by 1.95 time. Decreasing fill level from 3.5 to 0.9 kg increases the tangential velocity by a factor of 1.19. No obvious discrepancy in the solids motion is seen between machines of 1 and 5 L capacity and both machines have similar mixing and dispersion with constant tip speed criterion. © 2007 American Institute of Chemical Engineers *AIChE J*, 54: 415–426, 2008
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Introduction

Wet massing granulation is a process where dry particles of various components are mixed and binder liquid is added to the powder mix to form granules. The wet massing granulation takes place in one of two types of closed granulating systems: fluidized bed granulators or high shear mixer granulators. The two techniques differ on the mode of solids agitation where the former uses a flow of air injected upwards to agitate the powder bed while the latter uses an impeller to maintain the powder in agitation. The high shear mixer granulators are concerned in this study. The advantages of the high shear granulation include producing spherical and well-compacted granules in a short operational time and its operation is very simple. These mixers are flexible and can be used to granulate materials of a wide range of properties. For these reasons, high shear mixer granulators are widely used in chemical, pharmaceutical, metallurgical, and food processing industries. Despite their extensive use, the flow of granular materials in high shear mixer granulators and the mechanisms by which particle motion is generated are still poorly understood. Flows of discrete particles are difficult to characterize because they exhibit a vast range of behavior, from solid-like, quasi-static flows to rapid fluid-like flows.[1,2] The complexity extends from the granular materials properties to various process parameters such as fill level and agitation speed. Further, various designs of the agitated devices have added difficulties in this field.

Iveson et al.[3] present a comprehensive review of the current state of knowledge based on the view that three processes occur during wet granulation: wetting and nucleation, consolidation and growth, and breakage and attrition. The properties of the granules such as strength, hardness, friability, morphology, and product size distribution are determined by a number of factors, such as the uniformity of distribution of the binder liquid, the rate of redistribution of the liquid between granules and the kinetics of the processes of granule breakage, and size enlargement.[4–10] The granules consolidate and grow through the coalescence in collisions and compaction in shear whereas the size reduction of the granules is

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due to the breakage, fragmentation and attrition in collisions and shear. Although there have been significant advances in understanding of the granulation mechanism,[3] the process is still difficult to monitor and control as fundamental progress has been inhibited by a lack of understanding of the solids motion induced by the impeller. Lekhal et al.[11,12] show that for agitated drying of crystals the final particle size is a function of attrition and agglomeration, which are very sensitive to the flow patterns within the particle bed. In his review, Knight[13] has identified one of the challenges in granulation process control requires an understanding of the motion of material within granulators and how the granulator interacts with the material being granulated. This knowledge leads to design mixers that inherently give a better control of granule size.

Earlier works in characterizing flow structure of an agitated powder bed were mainly conducted by photographing experiments through a transparent wall. A more detailed characterization of flow patterns is also possible by bed freezing (by solvent infiltration and setting) and dissection.[14–16] Granular flow over a flat blade has been studied in two dimensions. Bridgwater et al.[14] studied the horizontal and vertical displacements of single particle by one blade pass while Novosad[15] studied the motion of particle in a vertical cylinder stirred by a long flat blade by dissecting beds of different colored layers that had been frozen using paraffin. In these studies, the authors observed the formation of a heap of particles moving just ahead of the blade, and the presence of a wake region with high bed porosity behind it. It was also found that there is no vertical motion in the clearance between the blade and the wall.[14,16]

The advance in digital imaging technology and the invention of Positron Emission Particle Tracking (PEPT) have permitted more detailed quantitative investigation of granular flow. Many of the investigations have focused on the characterization of granular flow in vertical cylindrical mixers[17–26] and there are few studies on more complicated configurations such as the planetary mixers[27–29] and the vertical cone shape mixer.[30] Ramaker et al.,[17] Muguruma et al.,[18] Litster et al.,[21] Conway et al.,[22] Nilpawar et al.,[23,24] Lekhal et al.,[25] and Darelius et al.[26] have used the Particle Imaging Velocimetry (PIV) technique to characterize granular flow near the top surface in vertical cylindrical mixers. Ramaker et al.[17] have shown a toroidal flow structure moving along the impeller at the bottom of the mixer, rising at the periphery and falling in the center. The formation of this toroidal flow structure is attributed to a combination of two independent trajectories caused by centrifugal forces, impact forces and gravity. Similar flow structure is observed by other investigators in cyclindrical mixers[18,21,23,24] and also in a conical frustum-shaped mixer with helical blade.[30] The reversed toroidal flow structure is reported for planetary mixers.[27–29] Using PEPT technique, Hiseman et al.[27,28] and Laurent[29] show that the particles rise in the center and fall at the periphery. There is no satisfactory explanation to justify the contradicting findings and it is extremely difficult to compare and generalize the findings in the literature because of the differences in geometries and configurations of granulators, materials and operating conditions, which indicate the complexity and difficulties in understanding granular flows in mixer granulators.

The motion and flow structure of granular materials with relative to the blade was studied in 3D by Stewart et al.[19] through PEPT measurements in a cylindrical mixer equipped with two opposing flat blades. The motion of particles was similar to that observed by other workers[14–16] that at the wall the particles in the path of the blades are displaced upward forming a heap and then moved over the blades. Away from the wall, a fraction of the upwardly displaced material moved away from the blade down the faces of the heaps. This work has found significant radial motion and 3D patterns of recirculation. The granular flow structure is a strong function of blade location. Complicated 3D recirculation is observed by Stewart et al.[19,20] and the top surface PIV observation of Conway et al.[22] and Lekhal et al.[25] too indicate materials avalanche both ahead of and behind flat blades, coupled with subsurface replenishment flows.

Despite many works done over the pass decades, the motion of the powder within the mixer induced by the impeller is still poorly understood. Various factors, which may have influence on the granular flow in mixer granulators, have been attempted. These include the agitation speed,[17,19,21–23,28,30–33] fill level,[19,28,31–34] number and geometry of blade,[22,25,35] etc. Some of the observations are contradicting. No specific conclusion can be made considering the various differences in methodology, unit, materials, geometry and configuration of the mixers, flow regimes, etc.

The understanding of the solids flow patterns and velocity profiles of granular materials in mechanically agitated beds is crucial for scale-up and control of granular.[21,25] This study aims to elucidate some basic features of the flow patterns and velocity profiles of the granular materials in a specific type of vertical agitated mixer of conical frustum shape. This study is meant to be a step towards understanding granular flow that can be translated into advances in granulation process control technology. The findings are anticipated to have practical applications for machine and process design.

The effect of the agitation speed on the macroscopic flow fields of dry particles has been reported in Ng et al.[36] while the solids motion of both dry particles and wet granules were also compared.[37] This study extends the investigation to understand the change of granular flow field in a vertical high shear mixer granulator with size, density, particle shape, fill level as well as the machine scale. Two vertical high shear mixer granulators provided by Hosokawa Micron B.V. of the Netherlands are employed in the work. The PEPT technique is used to measure particle motion at the single particle level. The results obtained are in the form of particle location as a function of time, which upon processing, gives particle velocity distribution, occupancy of the tracer particle and the flow pattern under various conditions.

Experimental

The vertical high shear mixer granulator provided by Hosokawa Micron B.V. was the model Cyclomix 5L and the details of the machine can be found in the previous communication.[36] A smaller scale machine (Cyclomix 1L) with nominal capacity of 1 L was used to investigate the change of solids motion with the machine scale. Both machines are geometrically equivalent. Various factors which may influence the solids motion in the Cyclomix were investigated. The experiments were designed to investigate one factor at a time in sequence. The design of the experiments is summar-

Table 1. Design of the Experiments

Factor Studied	System	Granular Material	Tracer Particle
Mean bulk diameter	Cyclomix 5L	Durcal 15, 40, and 65	Apatite (180–300 μm)
Density	Cyclomix 5L	Hollow and solid glass ballotini	Polystyrene (60 μm)
Particle shape	Cyclomix 5L	Glass ballotini and Durcal 65	Polystyrene (60 μm) and Apatite (250–300 μm)
Fill level	Cyclomix 5L	Durcal 65	Apatite (250–300 μm)
Scale	Cyclomix 1L and 5L	Glass ballotini	Polystyrene (60 μm)

ized in Table 1 and the physical properties of granular materials used in the experiments are listed in Table 2. Except for the experiments of fill level, the vessel was filled up to 50% of the volume, corresponding to ~3.5 and ~1.2 kg of calcium carbonate and hollow glass ballotini, respectively. The mixer granulators were operated at a shaft speed to give the top impeller tip speed of 4.1 m s^{-1}, a speed which gives the optimum dispersion and vertical circulation in Cyclomix 5L.[36]

The Durcal 15 with d_{50} of 23 μm, Durcal 40 with d_{50} of 43 μm and Durcal 65 with d_{50} of 60 μm were used to compare the flow field of calcium carbonate of different mean bulk diameter. These granular materials were chosen to be polydisperse with size range that are comparable with the materials used in industry. Hollow glass ballotini with density of ~700 kg m^{-3} and glass ballotini with density of ~2600 kg m^{-3} were used to investigate the influence of density on the granular flow field while the comparison of Durcal 65 and glass ballotini of similar density provides some insights of the influence of particle shape as quantified by the angle of internal friction on the granular flow field. For the study of fill level, the vessel was filled at three levels with 0.9, 20, and 3.5 kg of Durcal 65, respectively. These fill levels were chosen to cover the second, the third and the top impellers at static condition. To compare the flow field of different scales, the equal tip speed criteria is adopted for a comparable shear field; Cyclomix 1L was operated at agitation speed of 9.5 Hz and Cyclomix 5L was operated at agitation speed of 5.8 Hz for a tip speed of 4.1 m s^{-1} at the top impellers.

The PEPT technique provided by the University of Birmingham (Birmingham, UK) was used to track particle motion. The principle of the PEPT technique and its capability can be found elsewhere.[38–40] In brief, PEPT technique makes use of a single radioactive tracer that carries positrons. Positrons annihilate with local electrons, which results in emission of back-to-back 511 keV γ-rays. Detection of the pairs of γ-rays enables the tracer location to be found as a function of time by triangulation. The detectors of the PEPT facility at Birmingham cover a field of view of approximately 2.40 $\times$ 50 cm^2, and have a maximum separation of ~80 cm.

In a typical experiment, particles were loaded into the vessel of the mixer granulator, which was then started and run for a couple of minutes to ensure that the steady-state was reached before starting the data requisition process. The data acquisition was performed for ~12–15 min for each run which gave at least 20,000 data points in the form of spatial locations in the Cartesian coordinate as a function of time. The data processing involved conversion of the Cartesian coordinates to the cylindrical coordinates, and calculation of the axial velocity (dy/dt), radial velocity (dr/dt), and the angular velocity (ω) by using the six-point method.[19,39] The results were further processed by grouping and averaging the data over pixels of 5 $\times$ 5 mm^2 in axial (y-axis) and radial (r-axis) directions. This simplified the way of presentation of the data in a two-dimensional axisymmetric system. To ensure the reliability of the data, pixels with less than five data points were discarded.

The tracer occurrence in each pixel was counted and normalized with respect to the maximum value. This time occupancy of tracer gives insights of the mixing and dispersion of granular materials in the vessel. The rate of vertical mixing can be quantified by a parameter *period* defined as the time taken by the tracer particle to move from a position higher than an upper boundary in the mixer vessel, reach a position lower than a lower boundary and return to the upper part of the mixer. The upper boundary is chosen as Y = 180 mm, which is about ~10 mm above the top impellers and the lower boundary is chosen as Y = 65 mm, which is at the level of second impellers.

Results and Discussion

General observations

As a general observation for all experiments, the tangential velocities are inline with the direction of impellers rotation and the vertical flows have a general pattern where there are two clockwise swirl flows bounded at the top impellers (Figure 1). The flow structure in the Cyclomix mixer granulator can be understood as a toroidal motion along the impeller as reported by Ramaker et al.[17] This toroidal flow structure can be broken into two distinct motions: an azimuthal motion inline with the direction of shaft rotation coupled with a vertical swirl. The formation of this toroidal flow structure is attributed to a combination of two independent trajectories caused by centrifugal forces, impact forces and gravity.[17] The particles are seen to rise in the center and fall at the periphery. The similar flow structure is also observed by other investigators in planetary mixers.[27–29] Nevertheless, the reversed toroidal flow structure, where the particles rise at the periphery and fall in the center, is observed in cylindrical high shear mixer granulators[17–18,21,23,24] and in a conical frustum-shaped mixer with helical blade.[30] The contra-

Table 2. Physical Properties of Granular Materials

Granular Materials	Mean Diameter (μm)*	Density (10^3 kg m^{-3})	Angle of Repose (°)
Hollow glass ballotini	27	0.7	26
Glass ballotini	41	2.6	26
Durcal 15	23	2.7	38
Durcal 40	43	2.7	38
Durcal 65	60	2.7	38

*d_{50} measured by Malvern Mastersizer 2000.

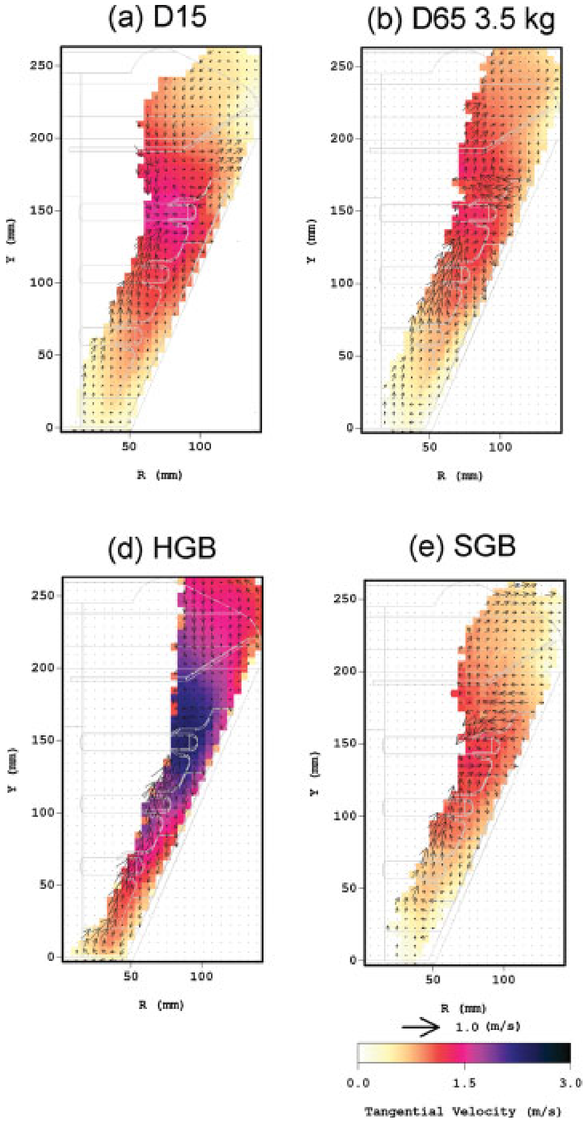

Figure 1. Vector plots of axial and radial velocities overlaid with the color plot of the tangential velocity.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

dicting findings can possibly be due to the differences in the geometries and configurations of the granulators as well as the granular materials, operating speed and conditions.

Previous communication[36] demonstrates the agitation speed as an important parameter in determining the toroidal structure for the current machine configuration. At 11.5 Hz, the granular materials were forced up along the vessel wall, rebound at the lid and then tumbled down towards the center of the vessel. The reversed swirl at lower speeds can be understood as insufficient upwards agitation to force the particles penetrate through the dense bed at the wall region as a result of the centrifugal force. Thus, the granular materials move upwards (due to impeller agitation) and outwards (due to centrifugal forces) at a small distance from the wall. This happens in the adjacent regions at the front and above the impeller (Figure 2a). At the wake of the impeller, the granular materials tumble down along the angled wall impeller (Figure 2b). The mechanism is verified and confirmed with high speed video recording, shown in Figure 2.

In agreement with the finding of Bridgwater et al.[14] and Malhotra et al.,[16] no obvious vertical motion in the clearance between the impellers and the vessel is seen (Figure 1). The tangential velocities are inline with the direction of impellers rotation. The highest values of the tangential velocity are found in the region near the end of the top impellers, in accordance with the highest tip speed. In this region, the bed moves at a tangential speed approximately equals to a quarter to two-third of the tip speed (Figure 3a). This implies a high shear region essential for granulation.

The overall average and standard deviation as well as the dimensionless maximum values of the velocities in the velocity plots (Figure 1) are calculated and shown as histograms (Figures 3a,b). In general, the range of the axial and radial velocities spread widely across the positive and negative values with an average close to zero. The tangential velocity is always positive and the ratios of the average tangential velocity to the mean of axial and radial velocities are ~7–

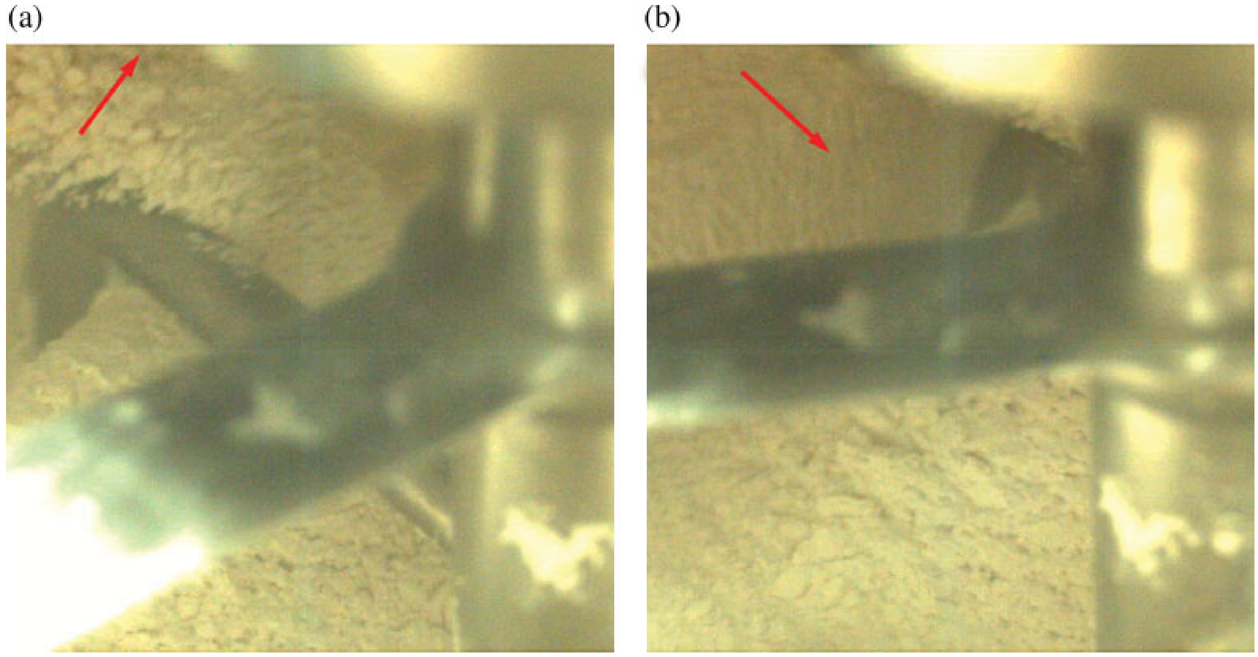

Figure 2. High speed video recording of the solids motion under agitation of the top impeller.

(a) At the front and above the impeller and (b) the wake of the impeller. The arrows indicate the direction of the particle motion. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

191, indicating that the dominating motion is in tangential direction. The results are in agreement with previously reported data.[18,23,24,27–29]

Quantitative comparisons of the axial, radial and tangential velocities are shown for $Y = 105$ mm (Figures 4a–c). In general, the axial velocities decrease almost linearly with increasing radial distance, with a magnitude change from ~15% to ~−10% of the local tip speed (Figure 4a). In most cases, the particles are moving upwards for radial positions less than ~80% whereas the average motion of particles close to the vessel wall is a downwards motion. The radial velocities also decrease with increasing radial distance; for radial positions less than ~70%, the particles are moving outwards as a result of the centrifugal force while beyond that point, low magnitude outwards motions and some inwards motions are seen (Figure 4b). The low magnitude of the radial velocity may be due to the high packing fraction in the close-to-wall region while the rebounded and falling particles at the angled wall produce the inwards motions.

An inspection of the tangential velocity distribution in Figure 4c indicates the existence of a maximum occurring at a radial position between 60 and 80% of the local radius of the vessel. A previous study[36] has shown that there is no significant variation in the trend of tangential velocity with elevation which is also true in this study. The conclusions drawn from the results of $Y = 105$ mm can be extended to other elevations. The drop of the tangential velocity at the wall region is mainly due to the particle-wall frictional interaction and particle-particle frictions attributable to the more dense packing of particles at the wall region as a result of the centrifugal force. The similar hump-shaped trend is also observed by Conway et al.[22] in their investigation of surface

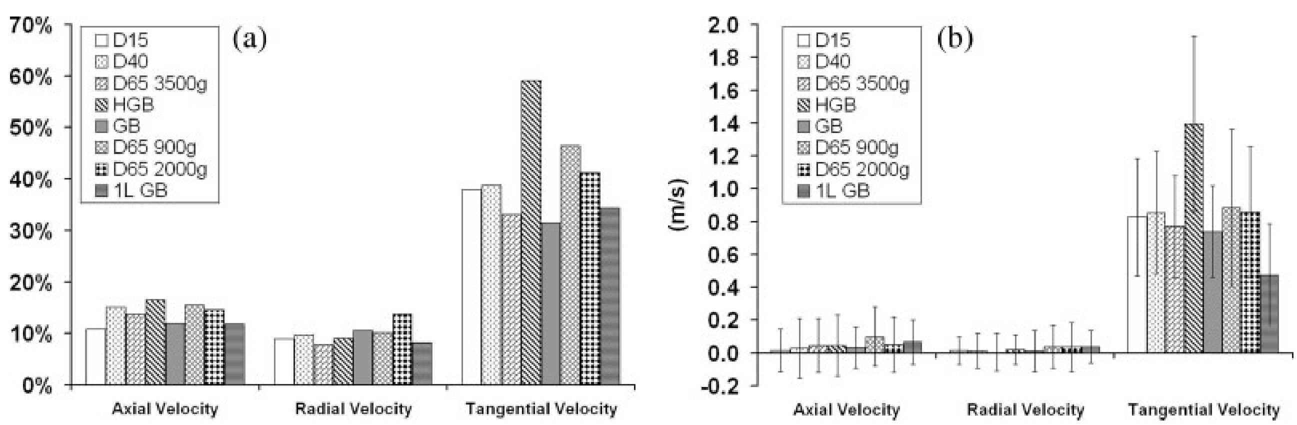

Figure 3. (a) Dimensionless maximum velocities and (b) average in axial, radial and tangential directions.

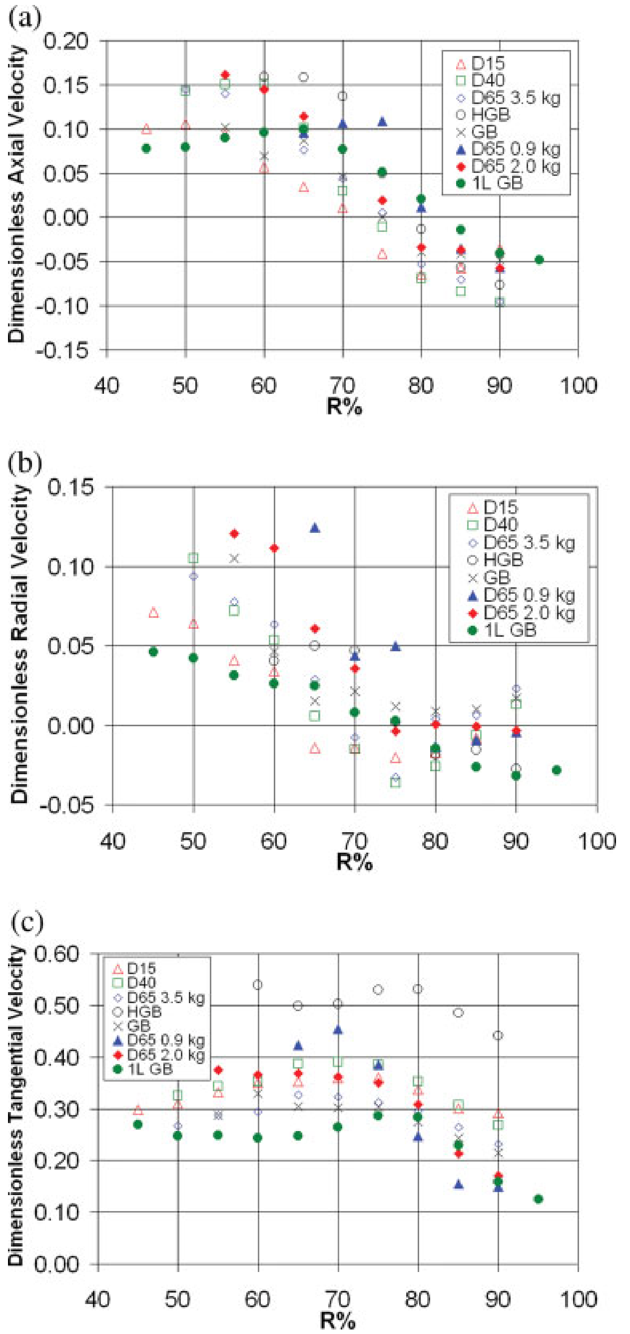

Figure 4. Quantitative comparisons of the (a) axial, (b) radial, (c) tangential.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

flow using PIV. A general decreasing tangential velocity distribution with increasing radial distance towards the vessel wall is also observed in other investigations,[18,23–26,28–29] concurring the hypothesis.

Solids motion due to the acute and obtuse types of impellers cannot be distinguished from the flow pattern as no obvious difference is seen between the axial velocity at planes where impellers lie, e.g. Y65 (acute motion) and Y105 (obtuse motion). This is mainly because the velocity distributions are obtained by tracking single particle motion over a period of time.

Particles are observed to circulate in both the horizontal and vertical directions. The period of horizontal circulation is short and is in the order of seconds, whereas that of the vertical circulation takes tens of seconds and consists of lots of higher frequency fluctuations. The ratio of vertical circulation between the lower and the upper impellers to the impeller rotation is around 1:25, which indicative of high material turnover in the vessel for efficient mixing.

It can be seen in the normalized tracer occurrence plots (Figure 5) that the granular material generally occupies a layer attached to the vessel wall. A high occurrence is seen a few millimeters away from the vessel wall and this active region stretches along the vessel wall. The tracer occurrence drops drastically to nearly zero at a radial position small than ~40% and larger than ~90% of the local radius of the vessel, suggesting the existence of an inactive zone near the wall and close to the vessel center. The inactive zone in the central part of the vessel is mainly due to the centrifugal effect which leads the tracer particles to move towards the wall region. The inactive region close to the vessel wall could be due to more dense packing of particles at the wall region as a result of the highest centrifugal force at a given elevation (The high packing fraction means low solids exchange in radial and axial direction at the wall region as evidenced from the low radial and axial velocities near the wall).

Some out-of-range data are seen in PEPT results (Figure 1). These erroneous data are conjectured to be the artifact of PEPT, which were resulted from the erroneous computation of the tracer location from the triangulation of γ-rays.[36]

Effect of particle size

The flow fields for the same type of granular materials, calcium carbonate, but with different mean sizes were investigated. The materials used are polydisperse particles with $d_{50} = 23$ μm (Durcal 15), 43 μm (Durcal 40), and 60 μm (Durcal 65), measured by Mastersizer 2000 (Malvern Instruments, UK). The flow fields of Durcal 15 and 65 are shown in Figures 1a,b while the flow field of Durcal 40 is a blend of both (not shown). In general, all materials have a similar flow field, in particularly the clockwise swirl flows in the region below the top impellers. There is a slight difference for the flow above the top impellers where downwards motions are detected at the inner region for Durcal 15 and 40 while Durcal 65 shows upwards motions. These downwards motions are possibly showing the falling particles after rebounding at the lid. A closer inspection on Figure 1a shows that there are clockwise swirl flows adjacent to the top impeller blade. The size of the swirl increases and more upwards velocities are seen with increasing mean particle size. The results suggest that bigger particles have a greater upwards agitation under the same action of impeller forces. Differences are seen in the tangential velocity, at which a decrease is seen for the highest velocities of Durcal 65 as compared with that of Durcal 15 and 40. These differences can be seen clearly from the average and maximum of the velocities (Figures 3a,b). There is no obvious difference seen in axial and radial velocities distributions (Figure 3b). The

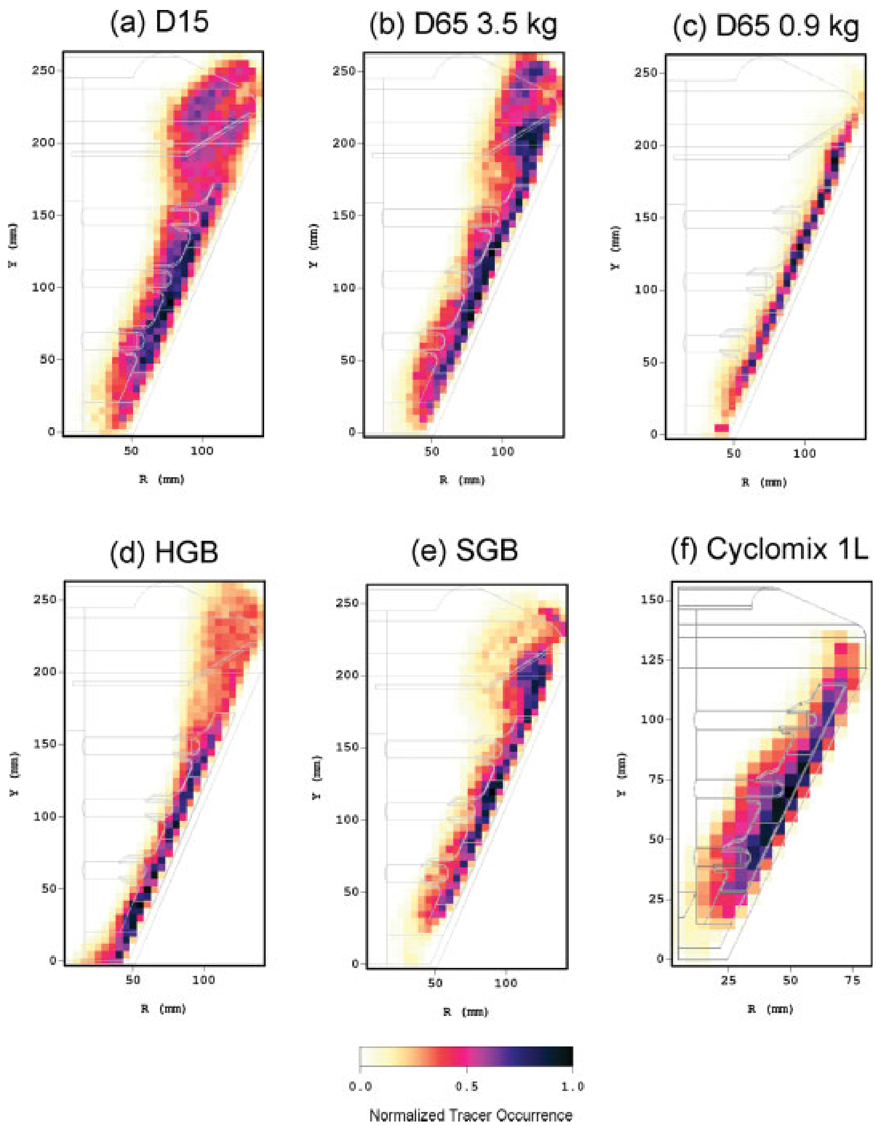

Figure 5. Normalized tracer occurrence.

The value of occurrence is normalized by the maximum value of the whole data set. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

dominating solid motion in the high shear mixer granulator is in the tangential direction with magnitude less than 38, 39, and 33% of the tip speed of the top impellers (4.1 m s^{-1}) for Durcal 15, 40 and 65, respectively. Taking all the values between elevation of 20 to 180 mm, the ratio of tangential velocity of Durcal 65 to Durcal 15 gives 0.81 $\pm$ 0.10. The regions outside Y = 20–180 mm were excluded in the analysis to eliminate the wall effect at the bottom and the free flow region beyond the impeller agitation. These results seem to indicate that granular materials with a smaller diameter perform better in transmitting the kinetic energy imposed by the impellers. Nevertheless, the difference in the tangential velocity between the Durcal 15 and 65 is not statistically significant (Student's t-test: P = 0.082). Quantitative comparison between the flow fields of Durcal 15, 40, and 65 is shown in Figures 4a–c. The distributions of axial, radial, and tangential velocities are almost identical for these particle sizes. Good correlation is seen between curves, with correlation coefficients greater than 0.935.

Figures 5a,b show the normalized tracer occurrence of Durcal 15 and 65. In comparison, the normalized tracer occurrence of Durcal 15 forms a thicker layer attached to the wall but the high occurrence counts are limited to the region below the top impellers. The layer thickness decreases and the high occurrence counts extend to the upper region for Durcal 40 (not shown) and 65. Figures 5a,b seem to indicate that particles of bigger size have a better dispersion under same operating conditions.

The average and standard deviation of the vertical circulation period is shown in Figure 6. It can be seen that the granular materials of smaller particle size take longer time to complete one vertical cycle and have smaller number of cir-

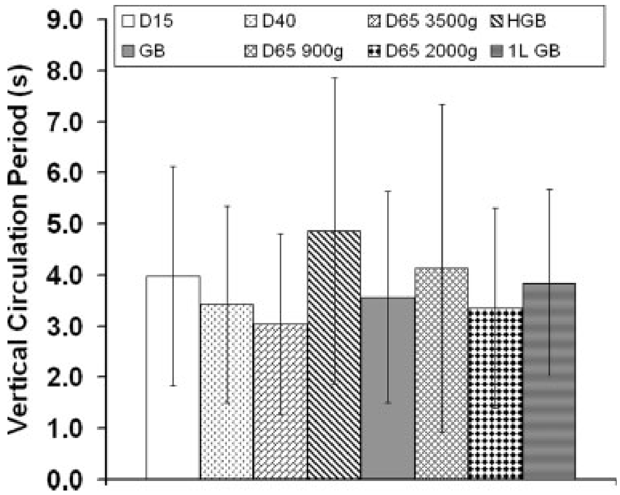

Figure 6. Period of vertical circulations between upper boundary at Y = 120 mm, Y = 180 mm and the lower boundary at Y = 43 mm, Y = 60 mm for Cyclomix 1L and 5L, respectively.

culation within the same operating time (15 min). The results imply that the vertical mixing can be sensitive to the particle size under the same operating conditions. The observations can possibly be related to the combined effect of size segregation during the agitation and angle-of-repose segregation due to avalanches. The investigation of Conway et al.[22] has shown that large particles segregate vertically towards the free surface in an operating cylindrical high shear mixer. This observation implies that larger particles have greater upwards velocity magnitude, concurring the results shown in Figures 1a,b and 3b where greater axial velocities are detected with Durcal 65. At the upper region, the particles rebound at the lid and tumble down towards the center and bottom of the vessel. Large particles avalanche more readily than small particles[22] and hence reach the bottom of the vessel in shorter time. The combination of both the size segregation during the upwards agitation and the angle-of-repose segregation during avalanche create a shorter vertical circulation period, proving a reasonable explanation for the results shown in Figure 6.

Effect of particle shape

The dynamics flowability of granular materials can depend on various physical properties of the primary particles such as the surface morphology, shape, coefficient of restitution, interparticle forces, yield strength, etc. In this study, assumptions have been made that the granular materials are completely cohesionless and that the effect of coefficient of restitution and yield strength are negligible. In this case, the effect of particle shape on the solids motion in a vertical mixer granulator can be seen by comparing the flow fields of solid glass ballotini to that of Durcal 65 (Figures 1b,e). The particle shapes as quantified by the angle of internal friction of both materials are different, with 26° and 38° for solid glass ballotini and Durcal 65, respectively. The shapes of the primary particles are shown in Figure 7, where solid glass ballotini has a general spherical shape while Durcal 65 is relatively irregular. Both granular materials have similar density of $\sim$2600–2700 kg m^{-3} (Table 2) to exclude the effect of density. Comparing Figures 1b and e, it can be seen that the granular flow field are very similar for both types of granular materials. This is also shown quantitatively by the average velocities in Figure 3a. Detailed comparison between the velocities of solid glass ballotini and Durcal 65 (Figures 4a–c) shows similarities in trend and magnitude (correlation coefficients: axial velocity, 0.955; radial velocity, 0.8; tangential velocity, 0.85). The statistics analysis shows that the differences in the axial and radial velocities are not significant between solid glass ballotini and Durcal 65 (Student's *t*-test: axial velocity, $P = 0.07$; radial velocity, $P = 0.583$). Figure 3b shows that Durcal 65 has a slightly higher average value in the tangential velocities (Student's t-test: $P = 2.79 \times 10^{-4}$), in opposition to the expectation deduced from the angle of internal friction. Taking all the values between elevation of 20–180 mm, the ratio of tangential velocity of solid glass ballotini to Durcal 65 gives 0.97 ± 0.13. The results indicate that, for the case of dry powder, the particle shape as quantified by the angle of internal friction has a negligible effect on the solids motion in the vertical high shear mixer granulator.

The normalized tracer occurrence obtained with the two materials are similar with both occupying a thick layer of a shape of an elongated comma attached to the vessel wall

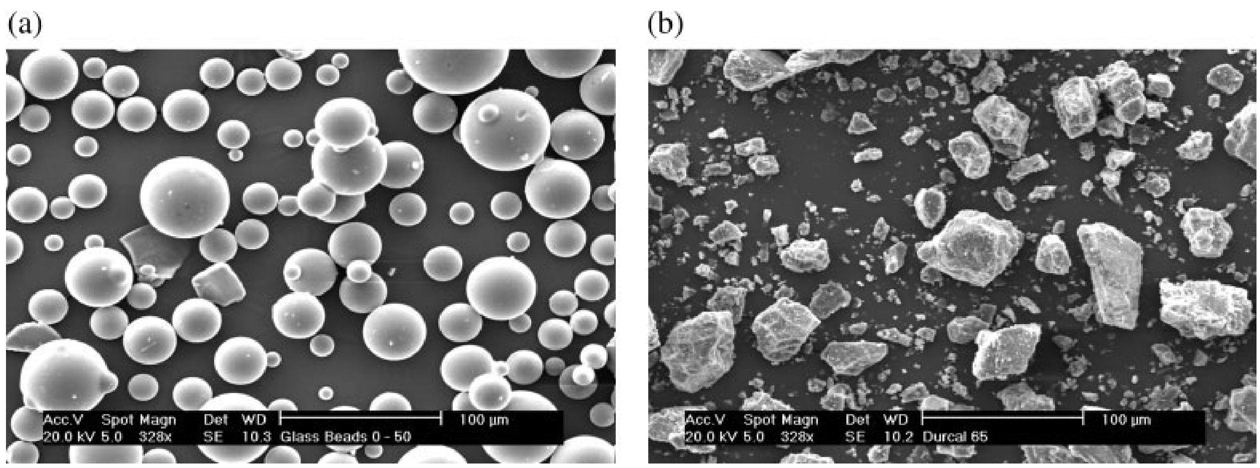

Figure 7. SEM of (a) solid glass ballotini and (b) Durcal 65.

(Figures 5b,e). The total area of inactive region in the vessel is larger for solid glass ballotini, especially in the bottom region of the vessel. The high occurrence region of Durcal 65 spread wider vertically and radially. These observations seem to indicate Durcal 65 has a better dispersion under same operating conditions as compared with the solid glass ballotini.

There is no obvious difference between the vertical circulation period of solid glass ballotini and Durcal 65 (Figure 6), although the average period of solid glass ballotini is slightly higher than that of Durcal 65. Glass ballotini has a smaller number of vertical circulations within the same operating time, implying an inferior vertical mixing under same operating conditions.

Effect of material density

The flow fields for the same type of granular materials, glass ballotini, but with different densities, 700 kg m^{-3} (hollow) and 2600 kg m^{-3} (solid), are shown in Figures 1d,e. Similar clockwise swirl flows in the region below the top impellers are seen for both hollow and solid glass ballotini. The obvious difference between these two granular materials lies in the tangential velocity, where the hollow glass ballotini yields higher magnitude with the same agitation speed. The average and maximum of the velocities in axial, radial and tangential directions are shown in Figures 3a,b. It can be seen clearly that the means of velocities distributions of hollow glass ballotini are greater than that of solid glass ballotini. Figures 3a,b indicate a strong influence of the density of granular materials on the solids motion in tangential direction. Figure 3a also shows that the dominating solids motion is in the tangential direction with a magnitude less than 59 and 31% of the tip speed of the top impellers (4.1 m s^{-1}) for hollow and solid glass ballotini, respectively.

Taking all the values between elevation of 20–180 mm, the ratio of tangential velocity of hollow to solid glass ballotini gives 1.95 $\pm$ 0.33, which indicates that hollow glass ballotini moves better than solid glass ballotini. The results indicate that density has a strong influence on the solids motion in the high shear mixer granulator in addition to the effect of mean particle size (27 vs. 41 μm, Table 2) as extrapolated from previous section. The difference in the tangential velocity is statistically significant (Student's *t*-test: $P = 1.7 \times 10^{-116}$) but the differences in the axial and radial velocities are not (Student's *t*-test: axial velocity, $P = 0.983$; radial velocity, $P = 0.504$). The differences are shown more clearly by the quantitative comparisons between the flow fields of hollow and solid glass ballotini in Figures 4a–c. The distributions of axial and radial velocities are close to each other in both trend and magnitude with correlation coefficients of 0.976 and 0.542, respectively. The tangential velocities have similar trend (correlation coefficient = 0.835) but the magnitude is greater for hollow glass ballotini as compared with that of solid glass ballotini (Figure 4c).

Figure 5d shows that the normalized tracer occurrence of hollow glass ballotini forms a relatively thin and long layer attached to the wall while the high occurrence counts are limited to the region below the top impellers. In comparison, the high occurrence of solid glass ballotini covers a wider region from the bottom impellers to the lid (Figure 5e). There is a low occurrence region at the bottom part of the vessel for solid glass ballotini that the tracer hardly reached throughout the whole experiment. This suggests there will be a relatively stagnant bed in the lower region of the vessel when using solid glass ballotini. Except for this, Figures 5d,e seem to indicate that solid glass ballotini has a better dispersion under same operating conditions than hollow glass ballotini (Figure 5d).

In Figure 6, the comparison between hollow and solid glass ballotini indicates that granular materials with a smaller density will take slightly longer time to complete one vertical cycle, implying a slightly lower vertical mixing rate.

Effect of fill level

Figures 1b,c show the flow fields of the same type of granular materials, calcium carbonate, but with different fill levels, 0.9 and 3.5 kg. No significant deviation is seen in the vertical flow pattern with different fill levels. The obvious differences are in the magnitude of tangential velocity near the top impellers, where increasing fill level will decrease the tangential velocity. The mean and standard deviation of the velocities show a general trend of decrease in velocity magnitude with increasing fill level (Figure 3b). Taking all the values between elevation of 20–180 mm, the ratio of tangential velocity of 0.9–2.0 kg gives 1.04 $\pm$ 0.20 while the ratio of tangential velocity of 0.9–3.5 kg gives 1.19 $\pm$ 0.39. The decrease of tangential velocity with fill level can probably be related to the solids pressure which is analogous to the hydrostatic pressure. It can be understood as the high solids pressure restricts the mobility of particles. The decreases in all the velocities are statistically significant when the fill level increases from 0.9 to 3.5 kg (Student's *t*-test: axial velocity, $P = 3.89 \times 10^{-7}$; radial velocity, $P = 5.22 \times 10^{-5}$; tangential velocity, $P = 1.44 \times 10^{-5}$). Contradicting observations are seen in the literature. Hiseman et al.[28] reported a limited increase in the average angular velocity when increase the fill level from 1 to 1.5 and 2 kg in a planetary mixer while the investigations of Broadbent et al.[32,33] and Laurent and Bridgwater[35] on horizontal mixers observed the angular velocity increases with the fill level. Aside from the differences in mixer geometry and operating conditions, there is no satisfactory explanation to justify the different observations.

Quantitative comparison in Figures 4a–c shows similar trends in axial, radial and tangential velocities for all fill levels. Except for radial velocity of 0.9 and 3.5 kg, the correlation coefficients of various pairs of fill level in Figures 4a–c are greater than 0.851. In general, the magnitude of axial, radial, and tangential velocities seem to decrease with increasing fill level (Figures 4a–c).

The normalized tracer occurrences look similar for all fill levels (Figures 5b,c) - the granular material occupies a thin and long layer attached to the vessel wall. This layer stretches from the bottom to the lid of vessel. The thickness of the layer increases with increasing fill level as a result of the increasing amount of materials available in the vessel.

Although it is not statistically significant (Student's *t*-test: $P = 0.6126$ for 0.9 vs. 2.0 kg and $P = 0.5134$ for 0.9 vs. 3.5 kg), the average vertical circulation period decreases with increasing fill level (Figure 6). A smaller number of circula-

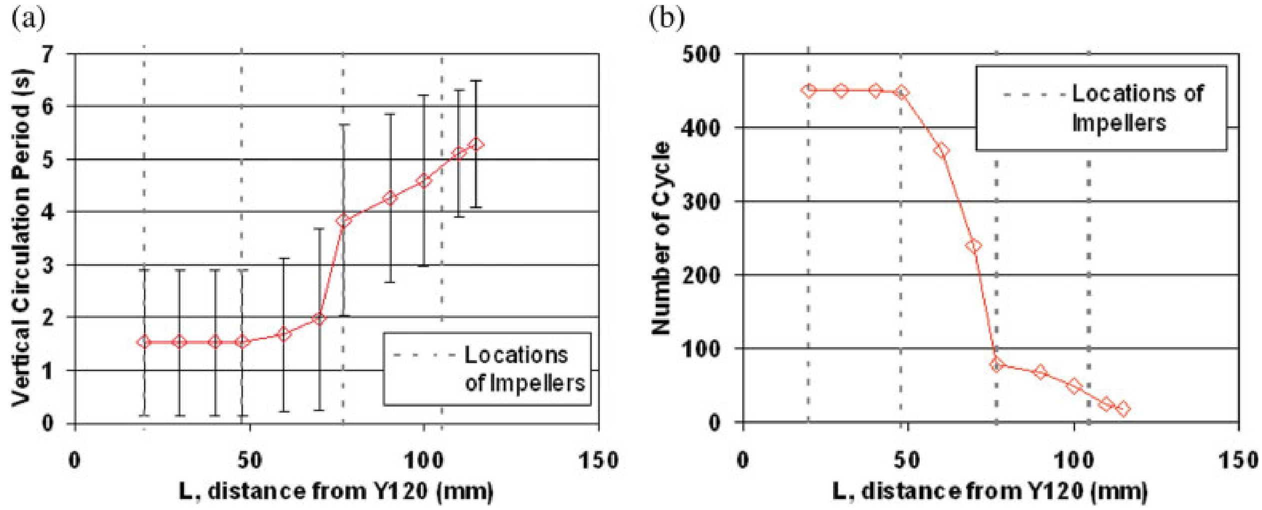

Figure 8. Period and number of vertical circulations as a function of distance (*L*) from *Y* = 120 mm for Cyclomix 1L.

[Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

tions within the same operating time (15 min) is seen at 0.9 kg fill level, 127, as compared with the 171 cycles of 3.5 kg fill level. It seems there is an optimum fill level for a maximum vertical mixing rate where the vertical mixing reduces at low fill level.

Effect of machine scale

Understanding the change of solids motion in machines of different scale is crucial to implementing a laboratory process to the full-scale industrial process successfully. In this very initial attempt to study the scale-up of high shear mixer granulator, the solids motion in two machine scales of identical geometry were investigated and compared. The granular materials used were the solid glass ballotini. While keeping all other operating conditions identical, the operating shaft speeds were set to follow the constant tip speed rule, which corresponded to the top impeller tip speed of 4.1 m s^{-1} for both machines.

The flow fields for both machine scales are shown in Figures 1e,f. The velocity fields obtained with two different machine scales vary notably. A more complicated flow is seen in y–r domain for Cyclomix 5L. There are two clockwise swirl flows bounded at the top impellers in Cyclomix 5L while there is only one swirl flow in Cyclomix 1L. Unlike the stagnant bed in Cyclomix 1L, the lower swirl in Cyclomix 5L involves the entire bed below the top impellers. The results imply that the number of toroidal flow structures is restricted by the limited space in the Cyclomix 1L and more toroidal flow structures are expected in machines of larger scale than 5L. Generally, the axial and radial velocity distributions are similar with a slightly smaller range for smaller machine scale. The difference in velocities can be seen clearly from Figure 3b, which also shows Cyclomix 5L has a higher average magnitude in the tangential direction. This is due to the larger area of highly agitated region around the top impellers (Figure 1e). The maximum tangential velocities of both machine scales are close to each other (5L; 31% vs. 1L; 34%, Figure 3a) as the result of constant tip speed.

The axial, radial, and tangential velocities are compared quantitatively at elevations of geometrical similarity (Figures 4a–c). The results are compared at 70 and 105 mm which correspond to the height of third impeller for Cyclomix 1L and 5L, respectively. Generally, the radial velocity and tangential velocity are greater while the axial velocity is lesser for Cyclomix 5L as compared with that of Cyclomix 1L. It can also be seen that the decline of the tangential velocity with radial location for $R > 75\%$ is stiffer for Cyclomix 1L. Nevertheless, the trends of axial and tangential velocity are similar in a broad sense. The correlation coefficients of the axial, radial, and tangential velocities curves are 0.926, 0.632, and 0.711, respectively. The results indicate that similar flow field can be obtained in different machine scales by using constant tip speed criteria while keeping other conditions.

In both scales, the granular material occupies a thick layer attached to the vessel wall (Figures 5e,f). However, the layer is relatively thicker near the bottom of the vessel for Cyclomix 1L, indicating that the granular material is concentrated in the lower region of the vessel. Figures 5e,f seem to indicate that both machine scales have similar dispersion with constant tip speed criteria.

In addition to the flow field, the vertical circulation period and number of circulation are used to compare the vertical mixing power between the two scales. The upper boundary of Cyclomix 5L is about 10 mm above the top impellers and the lower boundary is at the level of second impellers. The justifications of the upper (Y180) and lower (Y60) boundaries of the Cyclomix 5L were explained in previous communication.[36] The selection of the boundaries of the Cyclomix 1L is following the geometrical similarity where the upper boundary is chosen to be 10 mm above the top impellers (Y120) and lower boundary to be the level of second impellers (Y43). Figure 8 shows the period and number of vertical circulations as a function of distance (L) from Y = 120 mm for Cyclomix 1L. Little changes are seen in the period between $L = 20$ and 48 mm (Figure 8a). The number of circulation changes drastically between the second and third impellers ($L = 48 - 77$ mm) and little changes are seen for $Y <\sim 120 - 77 = 43$ mm (Figure 8b). The results indicates that selection of the upper boundary at $Y >\sim 120 - 48 = 72$ mm and the lower boundary at $Y <\sim 120 - 77 = 43$ mm should not affect the circulation period significantly under the conditions of this work. There is no obvious difference between the Cyclomix 1L and 5L in the vertical circulation period (Figure 6) and number of cycle (80 vs. 85), although the average period of Cyclomix 1L is slightly higher than that of Cyclomix 5L. The results imply that, in so far as the mixing is concerned, geometrically similar mixer granulators can be scaled by constant tip speed criterion. This is attributed to the finding that the tip speed rule gives comparable vertical mixing, both in period and number of cycle.

Conclusions

In collaboration with the University of Birmingham, the granular flow field in a conical frustum shaped high shear mixer granulator has been investigated by using the PEPT technique. The effects of the particle size, density, shape, fill level and machine scale were investigated. The following conclusions are obtained:

- The characteristic flow of Cyclomix machines is a toroidal motion along the impeller with particles rise in the center and fall at the periphery.
- The dominating solids motion in Cyclomix machines is in the tangential direction.
- Axial and radial velocities are strong functions of elevation but the distribution of the tangential velocity can be scaled by the local tip speed of each elevation.
- The tested range of particle size ($d_{50} = 23$, 43, and 60 μm) and particle shape as quantified by the angle of internal friction (glass ballotini, 26° and calcium carbonate, 38°) have a negligible effect on the granular flow field in the Cyclomix 5L.
- The density of particles has a strong influence on the solids motion in Cyclomix 5L where reducing the density increases the tangential velocity.
- The velocities magnitude and average vertical circulation period decrease with increasing fill level.
- Machine of a larger scale has a more complicated flow field as compared to the smaller scale machine. Similar flow fields are seen at constant tip speed and in so far as the mixing is concerned, geometrically similar mixer granulators can be scaled by constant tip speed criterion.

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