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Solids motion of calcium carbonate particles in a high shear mixer granulator: A comparison between dry and wet conditions

B.H. Ng [a], C.C. Kwan [a], Y.L. Ding [a,*], M. Ghadiri [a], X.F. Fan [b]

[a] *Institute of Particle Science and Engineering, University of Leeds, Leeds LS2 9JT, UK*

[b] *Positron Imaging Centre, School of Physics and Astronomy, University of Birmingham, Birmingham B15 2TT, UK*

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Abstract

An experimental study has been carried out on the solids motion in a conical frustum-shaped vertical high shear mixer granulator by using the positron emission particle tracking (PEPT) technique. The mixer granulator has a vertical shaft, to which four sets of impellers are attached at different elevations. The shaft is operated at 3.9 Hz, 4.9 Hz and 5.8 Hz, which corresponded to the top impeller tip speed of 2.8, 3.5 and 4.1 m/s. The motion of calcium carbonate particles with and without a liquid binder is evaluated. Particles are observed to circulate in both the horizontal and vertical directions. The macroscopic solids circulation in the vertical direction reduces after adding the binder. There is a dominant solids motion in the tangential direction under both the dry and wet conditions with the maximum tangential velocity of 2.2 to 12.6 times that of the maximum axial and radial velocities. No obvious change is observed in the average axial and radial velocities when the impeller speed is changed under both dry and wet conditions, while the ratio of the maximum tangential velocity to the tip speed decreases with increasing impeller speed, suggesting a velocity-dependent behaviour. The three velocity components decrease in the magnitude after adding the binder at all tested agitation speeds except for the tangential velocity at a shaft speed of 3.9 Hz. The main difference between the dry and wet operations is that the decrease of tangential velocity in the near-wall zone under the dry condition is not observed under the wet condition.

Keywords: Solids motion; Solids circulation; Granulation; High shear granulator; PEPT

1. Introduction

Size enlargement is one of the most important unit operations in the chemical, pharmaceutical, detergent, and nuclear industries for enhancing the flowability, appearance, strength and other physical and mechanical properties of particulate solids. There are a number of ways to enlarge particle size including granulation, rolling compaction and tableting. This work is concerned about granulation — a size enlargement method that has been a subject of numerous investigations [1–8]. The published studies on granulation can be categorised into the mechanism of granulation [9–12], optimisation of the granulation processes [13–23], effects of various parameters on the granular product properties [24–35], and the scale-up of granulators [36–51]. However, very little work has been done on the evolving granule product structure as the scale of a certain type of granulator changes. This forms the main motivation of an integrated research project being carried out at the University of Leeds. As the granule structure depends on the prevailing stress and strain status given other conditions, knowledge of the macroscopic flow field is crucial. This constitutes one of the tasks of the integrated project and is also the specific objective of this work.

Two techniques have been used to investigate the solids motion inside a granulator — the high speed imaging [33,52–58] and positron emission particle tracking [59–64]. Concerning the solids motion in a vertical granulator, the current literature of these two techniques appears to be different. The investigations through high speed imaging [53,55,56] are in general showing toroidal motion along the impeller with particles rise at the periphery and fall in the centre. While the studies using positron emission particle tracking technique

* Corresponding author. Tel.: +44 113 343 2747; fax: +44 113 343 2405.
E-mail address: y.ding@leeds.ac.uk (Y.L. Ding).

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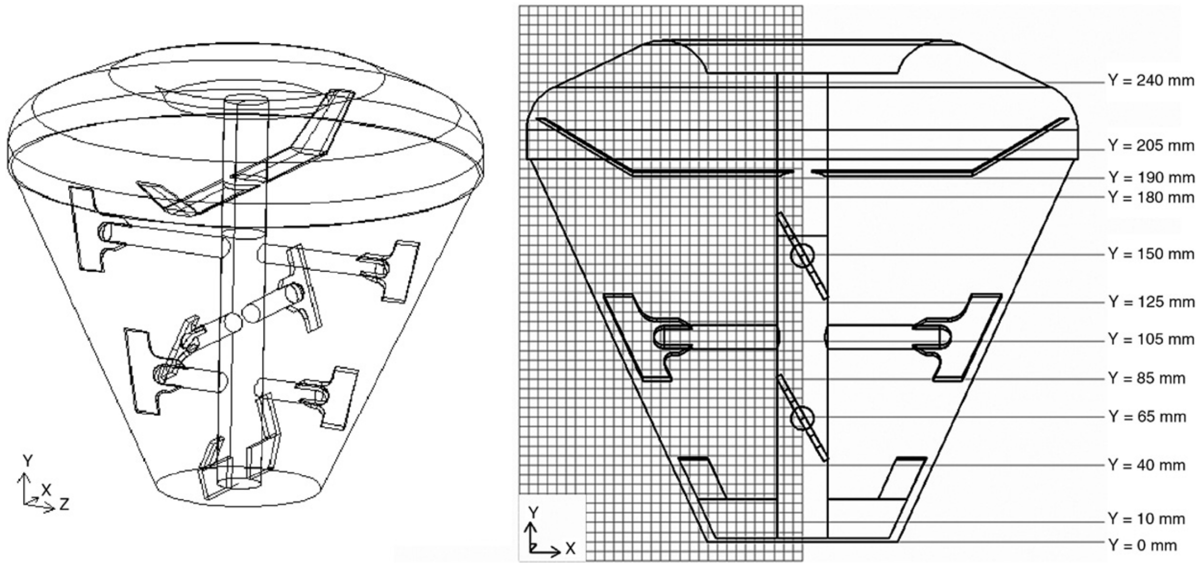

Fig. 1. Schematic diagram of the Cyclomix 5 L mixer granulator: the angle of the flat blades is 30°; the angle of the lowest and the third lowest pairs of impellers is opposite to the angle of the top and the second lowest pairs such that when the shaft is turned clockwise, the lowest and the third lowest pairs of impellers give obtuse motion while the top and second lowest pairs of impellers give acute motion.

[60,61,64] show that the particles rise in the centre and fall at the periphery. The contradicting findings can possibly be due to the difference in the geometries of the mixers as well as the operating speed and conditions.

The macroscopic flow fields of dry particles and wet granules are investigated in the study. A vertical high shear mixer granulator provided by Hosokawa Micron B.V. of the Netherlands is employed in the work. The positron emission particle tracking (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.

2. Experimental

The vertical high shear mixer granulator provided by Hosokawa Micron was the model Cyclomix and had a nominal capacity of 5 L. The mixer had four pairs of flat-bladed impellers; see Fig. 1 for a schematic diagram. These impellers are hereafter named one to four from the bottom to the top. The angle of the flat blades is 30° to the vertical axis; the angle of the first (bottom) and the third pairs of the impellers is opposite to that of the second and fourth (top) pairs such that when the shaft is turned clockwise, the first and the third pairs give obtuse motion (upwards agitation) while the second and fourth pairs give acute motion (downwards agitation). The gaps between the impellers and the vessel wall are 5 mm at the side and 2 mm at the bottom. The lid, the impellers and the shaft were constructed with stainless steel, while the vessel was made of transparent Perspex. All the interior surfaces in contact with particles were fully polished.

Calcium carbonate (Durcal 65) with an average diameter of 60 μm and a density of ~2700 kg/m^3 were used in the investigation. The vessel was filled to 50% of its volume (3.5 kg) and the mixer granulator was operated at three speeds of 3.9 Hz, 4.9 Hz and 5.8 Hz, which corresponded to the top impeller tip speed of 2.8, 3.5 and 4.1 m/s. Four hundred gram of 65% V/V aqueous solution of polyethylene glycol 4000 (PEG4000) was used as the binder. The positron emission particle tracking (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 [65,66,67]. In brief, PEPT technique makes use of a single radioactive tracer that carries positrons. Positrons annihilate with local electrons, resulting 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 40 × 50 cm^2, and have a maximum separation of ~80 cm. Apatite (calcium phosphate) beads with 250–300 μm diameter and a density of ~3190 kg/m^3 were used as tracers, which were activated by an ion exchange method with radioactive water produced in a cyclotron.

In a typical experiment, batch of calcium carbonate was 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. Following the data acquisition of dry particles, the speed of the mixer granulator was reduced to pour in binder. The speed of the mixer granulator was then increased to a higher speed for 1 min to disperse the binder before the speed was reduced back to the operating speed and starting the data requisition process. The binder introducing speeds were 1.9, 2.5 and 2.9 Hz, and the binder dispersing speeds were 7.5, 9.8 and 11.5 Hz for the operating speeds at 3.9 Hz, 4.9 Hz and 5.8 Hz, respectively. The data acquisition was performed for 15 min for each run which