

Study of Transitional Velocities of Solid-Liquid Micro-Circulating Fluidised Beds by Visual Observation

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Solid-liquid micro-fluidised beds (FBs), i.e. fluidisation of micro-particles in sub-centimetre beds, hold promise of applications in the microfluidics and micro-process technology context. This is mainly due to fluidised particles providing enhancement of mixing, mass and heat transfer under the low Reynolds number flows that dominate in micro-devices. Albeit there are quite a few studies of solid-liquid micro-fluidised beds, we are presenting the first study of a micro-circulating fluidised bed. The present experimental research was performed in a micro-circulating fluidised bed which was made by micro-machining channels of 1mm² cross section in Perspex. PMMA and soda lime glass micro-particles were used as the fluidised particles and tap water as the fluidising liquid to study flow regime transition for this micro-circulating fluidised bed. The results are in line with macroscopic observation that the critical transition velocity from fluidization to circulating regime is very dependent on solid inventory but once the inventory is high enough it is approximately equal to the particle terminal velocity. However, the transitional velocity is a weakly dependent on wall effect and surface forces confirming the importance of these two properties in a micro-fluidised bed systems. Similarly the transitional velocity to transporting regime is a strong function of surface forces. Finally, combining these results with our previous result on conventional fluidization indicated that map of solid-liquid fluidisation in a micro-circulating fluidised bed system is constructed showing conventional fluidisation, circulating fluidisation and a transport regime.

Keywords: *micro-fluidised bed, solid-liquid fluidisation, circulating-fluidised bed, fluidisation regimes, surface forces*

1 Introduction

Solid-liquid circulating fluidised beds are an important solid-liquid processing technology in various industrial processes (Liang, et al. 1995, Liang, et al. 1997, Natarajan, et al. 2009, Zhu, et al. 2000). At the moment, laboratory and pilot scale studies and development regarding solid-liquid circulating fluidised beds are costly and labour-intensive, and this is limiting their commercial utilisation (Natarajan, et al. 2015, Nirmala and Muruganandam 2015). In the past decade lots of attention have been paid to fluidised beds at the micro-scale due to their excellent operation abilities and fast screening for various applications (Liu, et al. 2008, Tang, et al. 2015, Xu and Yue 2009).

Micro-fluidised beds refers to a fluidised bed with the bed cross-section or inner diameter at micrometre or millimetre scale (do Nascimento, et al. 2016, Potic, et al. 2005, Zivkovic and Biggs 2015, Zivkovic, et al. 2013). In terms of applications, the micro-fluidized bed holds promise in the micro-process and micro-fluidic context. Besides fundamental studies on mixing and mass transfer enhancement (Doroodchi, et

al. 2013, Yang, et al. 2016, Zivkovic, et al. 2017), the current applications are mainly in high-throughput screening and testing of catalysts as it enables fast and reliable measurements of solid catalyzed reaction kinetics. (Yu, et al. 2010, Yu, et al. 2013). Some more specific applications are in a low-cost device for detection of infectious bacteria, granulation screening of pharmaceutical tablet fillers, photocatalytic reactors and compact carbon dioxide HVAC units (Kivikero, et al. 2009, Li, et al. 2017, Pereiro, et al. 2017, Wang, et al. 2017, Yang, et al. 2016). However, in many applications circulation of particles is required, e.g. continuous regeneration of catalyst or bio-solid particles for which circulating fluidised beds are excellent, in particular by improving solid-fluid contacting and reducing back mixing (Chavan, et al. 2009, Shilapuram, et al. 2009).

Albeit there are few reports on liquid-solid micro-fluidised beds as outline above, the paper presents the first experimental study of a solid-liquid circulating fluidised bed at the micro-scale. It is well known that particle handling in micro technology devices remains one of the big challenges in the field (Haeberle and Zengerle 2007, Jensen 2001, Mills, et al. 2007). In

order to successfully design a solid-liquid micro circulating fluidised bed system for future applications, e.g. novel micro-catalyst and bio-reactors, cooling of computers chips, it is essential to understand the hydrodynamics using techniques such as flow regime mapping as it allows to determine the bed performance and reveals the solid and liquid contacting (Shilapuram, et al. 2009, Zheng, et al. 1999).

The operation regime map in a solid-liquid circulating fluidised bed has been reported previously by various researchers. Liang et al. (1997) classified the circulating fluidised bed into four operating regimes: fixed bed, conventional fluidisation, circulating fluidisation and the transport regime. Zheng et al. (2001) also studied the flow characteristics in a solid-liquid circulating fluidised bed. In their experiments they suggested an onset velocity (U_{cf}) which gives the lowest value of U_{cr} , critical transition velocity from the conventional to circulating fluidised bed regime.

In the current research investigation a new fluidisation regime, the solid-liquid micro circulating fluidisation regime, will be mapped for the solid-liquid fluidisation system based upon different particles sizes and materials (Polymethylmethacrylate (PMMA) and soda lime glass microspheres). The operation regime map is a function of liquid velocity inherently related to particle terminal velocity which is a function of solid-particle density and size, liquid density and viscosity. Previous studies showed that liquid density and viscosity have very little effect on the solid circulating rate, particle velocity and pressure drop, once taken into account in the particle terminal velocity (Liang, et al. 1997). Therefore, a new liquid-solid micro-circulating fluidisation regime will be studied primarily as a function of superficial liquid velocity for different sizes and densities of particles and bed sizes. The operation regime map in the micro-circulating fluidised bed is expected to be similar to the solid-liquid circulating fluidised bed regime map proposed by Liang and co-workers (Liang, et al. 1997). The major differences from their macro-scale counterparts are expected to be the surface forces, which can even prevent fluidisation, and inevitably the wall effects due to small bed size (do Nascimento, et al. 2016, Zivkovic and Biggs 2015).

2 Experimental details

2.1 Experimental setup and materials

The schematic representation of the research experimental set up is illustrated in Figure 1. The system consists of a syringe pump (AL-4000, WPI INC., US) to pump the water as a fluidising medium at the desired flow rate using a 5ml B-D Plastipak syringe, and Euromex Nexius trinocular digital microscope fitted with a USB digital camera (JB Microscopes Ltd, UK) to record the micro fluidisation behaviour. The images and movies were saved on a computer for offline studies.

The system used in the present experimental investigation was made by micro machining channels in Perspex as schematically shown in Figure 1. The micro-circulating fluidised bed consist of a riser column of 1 mm square cross-section and 100 mm in height, a solid-liquid separator, a down comer acting as a particle reservoir, a solid return pipe, and a solid feeding pipe. At the base of the riser is the distributor (a 1.5 mm thick porous plate distributor with mean pore size of 21 μm) which prevents particles leaving the bed at the bottom and provides uniform flow distribution and stable fluidisation. The solid-liquid separator is a simple diamond shaped expansion that enables the particles to be separated from the outflowing liquid.

Two different groups of particles were used as fluidised solid: (1) soda lime glass microspheres of five different diameters, $d_p = 26 \pm 1.5, 30 \pm 1.5, 35 \pm 3, 58 \pm 5, \text{ and } 115 \pm 9 \mu\text{m}$ whose density is $\rho_p = 2500 \text{ kg/m}^3$ and (2) PMMA particles of five different diameters, $d_p = 23 \pm 3.5, 35 \pm 3, 41 \pm 3.5, 58 \pm 5, 115 \pm 9$ whose density is $\rho_p = 1200 \text{ kg/m}^3$. Tap water (with average density of $\rho_f = 998 \text{ kg/m}^3$) was used as the fluidising liquid. All experiments were performed at room temperature of average $18 \pm 2 \text{ }^\circ\text{C}$.

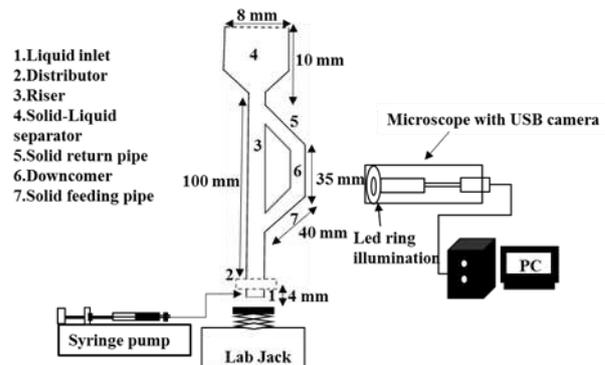


Figure 1 - Schematic of experimental set-up

2.2 Experimental methodology

The experimental procedure was as follows: initially the bed was filled with liquid (water) using a syringe pump. The bed was packed with solids to a known height and left to settle. The solid inventory, i.e. the amount of solids in the volume of the whole circulating fluidised bed system, was measured with the aid of ImageJ (Schneider, et al. 2012) and expressed in terms of percentage of the whole system. Liquid at varying velocity was forced by a syringe pump from the syringe to the bed inlet to produce the fluidisation liquid at the required velocity in the bed. Particles at the bottom of the riser were kept in motion by the upward liquid flow. When the liquid flow rate was high enough, particles were carried out of the riser and separated from the outflowing liquid by the solid-

liquid separator and recirculated back to the riser through the solid feed pipe. The experimental procedure was performed with both decreasing and increasing superficial liquid velocities. For each particle, this procedure was repeated at least three times to ensure repeatability and the measurement of experimental errors.

Calibration was performed with and without particles to determine the flowrate at which liquid moves through the system. The minimum fluidisation velocity was determined in our previous study using micro-fluidised bed by observing visually the bed height expansion and also by extrapolation of a linear relationship between the superficial liquid velocity and ratio of bed expansion (do Nascimento, et al. 2016). The theoretical minimum fluidization velocities, $U_{mf,theory}$ was calculated using the Ergun equation (Ergun 1952) with an estimated initial packed bed voidage of $\epsilon_{mf} = 0.40 \pm 0.01$ in line with previous experiments (Tang, et al. 2015) and trend of rectangular packed bed voidage with particle-to-bed ratios (Navvab Kashani, et al. 2016).

$$\frac{\Delta P}{H} = 150 \frac{\mu U_{mf}(1 - \epsilon)^2}{d_p^2 \epsilon^3} + 1.75 \frac{(1 - \epsilon) \rho_f U_{mf}^2}{\epsilon^3 d_p} \quad (1)$$

The critical transition velocity from conventional to circulating fluidisation regime was determined by visual observation as the maximum liquid velocity where no fresh particles were reintroduced back to the riser from the downcomer, shown in Figure 2, for both increasing and reducing liquid velocity experiments. This velocity was normalized by the particle terminal velocity using Stokes law equation as $Re < 1$ for all studied particles:

$$U_t = \frac{(\rho_p - \rho_f)gd_p^2}{18\mu} \quad (2)$$

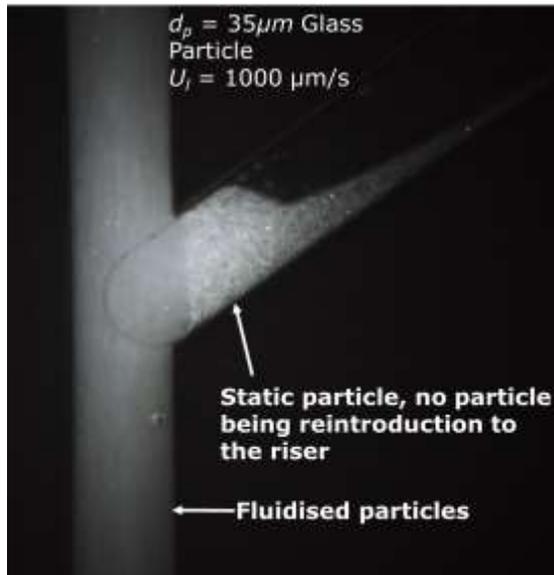


Figure 2 - The photo of the downcomer which was visually observed by a stereo-microscope for detecting solid particle circulation

3 Results and discussion

3.1 Visual observation

Solid-liquid fluidisation in a micro circulating fluidised bed is reasonably uniform. In the circulating fluidisation regime with increasing liquid flow rate, a difference could be observed in the liquid flow rate in the riser, with higher liquid flow rate at the centre of the riser and lower liquid flow rate near the walls.

In the middle section of the riser, solids were carried up to the top of the column riser by a higher liquid flow, while a small downwards flow of particles occurred near the wall of the riser as shown in Figure 3. However, this downwards flow of particles near the wall of the riser column was insignificant and did not have an effect on regime transition. These observation are similar to that reported by Zheng and Zhu (2001) and Liang et al. (1997). Agglomeration of particles which was caused by the presence of bubbles in the system was also observed, especially with the PMMA particles which were extremely hard to separate using the solid-liquid separator, as these particle-bubble agglomerates usually float to the top of the solid-liquid-separator.

3.2. Influence of solid inventory on transition velocity

Figure 4 shows that the normalised critical transition velocity is highest when solid inventory is low (1-9%), but plateaus when the solid inventory is increased (10-25%) and stays roughly the same indicating that in solid-liquid micro circulating fluidised bed the critical transition velocity from conventional to circulating fluidised bed regime decreases with increasing solid inventory. It then levels off at a high enough solid inventory (10-25%). These trends were the same for all the PMMA and glass particles.

For beds with a solid inventory lower than 10 %, a higher superficial liquid velocity is required to achieve a circulating fluidised bed, and the transition from conventional to circulating fluidised bed is greater than the particle terminal velocity ($U_{cr}/U_t = 1.5$ to 4.5). However, for systems with solid inventory higher than 10%, the critical transition velocity from conventional to circulating fluidised bed regime occurs close to the particle terminal velocity, and the normalised transition velocity is approximately 1. These observation on the solid inventory trends are similar to that reported by Liang and co-workers (1997)., This is slightly different from the observation reported by Zheng and Zhu (2001) as their reported onset velocity (U_{cf}) which gives the lowest critical transition velocity from the conventional to circulating fluidised bed regime was found to be independent of the bed geometry, operating conditions and solid inventory due to the method applied.

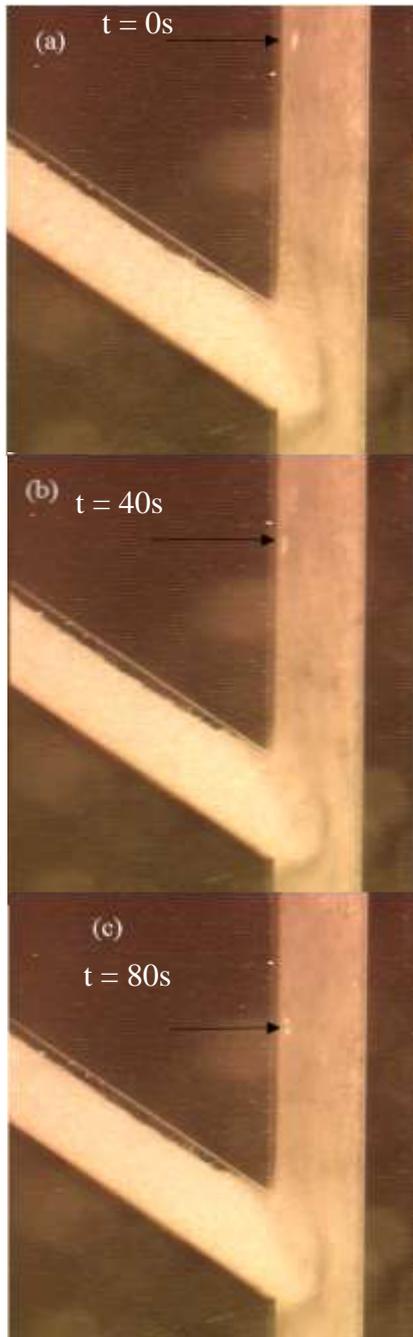


Figure 3 - The time sequenced images extracted from the movie of circulating fluidised bed using $38\ \mu\text{m}$ soda lime glass microsphere particle at $1.5\ \text{mm/s}$. The arrows shows small downwards flow of particles close to the riser wall. The letters a, b and c shows three consecutive images of downwards flow of particles taken in a short time interval (0, 40, and 80 second respectively)

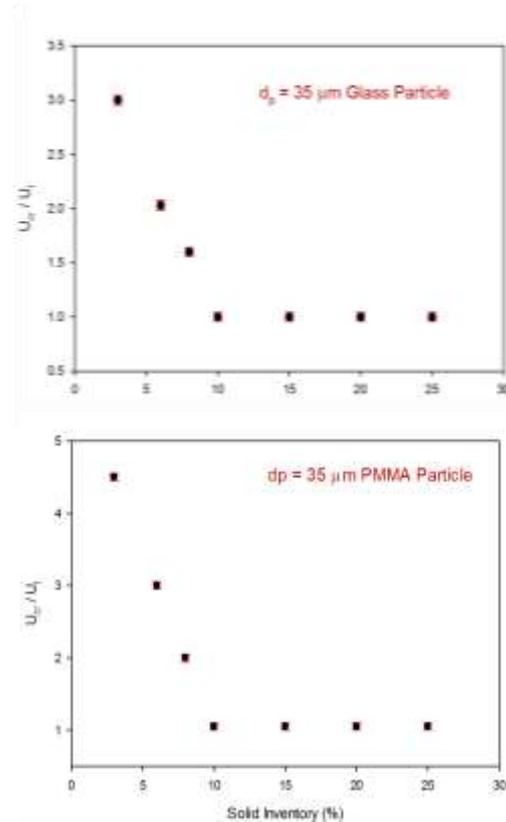


Figure 4 - Effect of solid inventory on normalised transition velocity for $35\ \mu\text{m}$ glass (upper graph) and PMMA (lower graph) particles.

3.3. Critical transition velocity

In the present experimental investigation the critical transition velocity (U_{cr}) which demarcate the transition from conventional to circulating fluidised bed is dependent upon the bed geometry, liquid and particles properties. Similar to macroscopic experiments, for all the particles U_{cr} is roughly equal to the particle terminal velocity (U_t), and the normalised transition velocity (U_{cr}/U_t) is close to 1 as shown in Figure 5. These normalised velocities are slightly smaller to those observed by Zheng and Zhu (2001) reported to be in the range of 1.1 to 1.5, but considerably larger than the observation made by Liang et al. (1997) where the critical transition velocity from conventional to circulating fluidised bed occurred at about only 60% of the terminal velocity. The Liang *et al.* (1997) circulating fluidised bed device had a third liquid stream close to the downcomer which was sufficiently high to fluidise the particles in the downcomer. This third liquid stream transported particles from the downcomer to the bottom of the riser and joined the other two streams (primary liquid flow rate and auxiliary liquid flow rate) to fluidised particles at the riser. Yet, Liang et al. (1997) did not take this third liquid stream into account when reporting the total liquid velocity and this could explain why their

reported critical transition velocity was smaller than the particle terminal velocity.

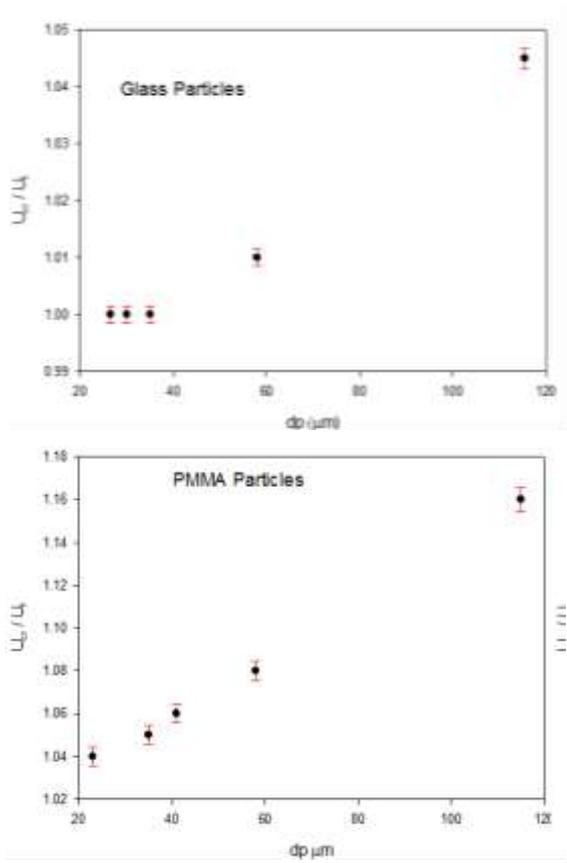


Figure 5 - Normalised critical transition velocity for glass (upper graph) and PMMA (lower graph) particles with solid inventory in the range of 10 - 25%.

Figure 5 shows that the transition from conventional to circulating fluidised bed regime in a solid-liquid micro circulating fluidised bed is influenced by particle properties such as size and surface properties beyond of the influence on the particle terminal velocity. First there is a trend of increased normalised critical transition velocity with an increase in particle size which is probably because of increased wall effects which are not usually present in large circulating fluidised beds.

From Figure 5 it can also be observed that the normalized transition velocity (U_{cr}/U_t) is perceptibly higher for PMMA particles compared to glass particles of the same size. This is probably due to difference in surface properties with PMMA particles being hydrophobic while glass particles are hydrophilic. Indeed, the particle agglomeration due to cohesion is observed for PMMA and particle adhesion to the walls as shown in Figure 6. Cohesion increases particle size as a result of agglomeration and that may postpone the critical transition velocity (U_{cr}) from the conventional to a circulating fluidised bed regime. Additionally, the wall adhesion present in the downcomer will also contribute to postponing of the transition.

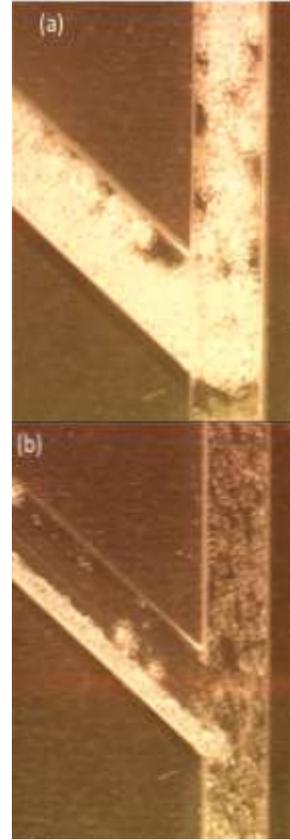


Figure 6 - Optical Microscope illustrating (a) agglomeration and (b) adhesion of 58 μm PMMA particle inside micro-circulating fluidised bed.

3.4. Fluidisation regime map

Based on this experiment and our previous experiments in the simple micro-fluidised bed (do Nascimento, et al. 2016), the fluidisation regime map is constructed for both PMMA and glass particles as shown in Figure 7. We plotted both experimental results on the same plot using non-dimensional quantities of dimensionless liquid velocity, U_l^* and dimensionless particle diameter d_p^* as introduced by Liang (1997):

$$U_l^* = U_l \left(\frac{\rho_f^2}{\mu g \Delta \rho} \right)^{1/3} \quad (3)$$

$$d_p^* = d_p \left(\frac{\rho_f g \Delta \rho}{\mu^2} \right)^{1/3} \quad (4)$$

The most striking trend of the plot is that the minimum fluidisation velocity ($U_{mf,exp}$) for both PMMA and particles deviates strongly from the theoretical prediction of the Ergun equation (1952), Eq 1, with proportionally higher deviation for the PMMA particles. This is due to the strong influence of surface forces on the minimum superficial liquid velocity at which particle fluidisation is achieved as found in our recent study (do Nascimento, et al. 2016). For PMMA particles fluidised by water in a PMMA micro-

fluidised bed, adhesion forces are 3-5 orders of magnitude higher than the drag forces, while for glass particles adhesion forces are only 1-3 orders of magnitude higher than the predicted drag force due to weaker adhesion forces and larger particle densities. These particle-wall adhesion forces are transferred as frictional forces to the particle ensemble inside the bed (Mabrouk, et al. 2008, Oke, et al. 2015). Consequently, this increased wall friction force results in an increase of the experimental minimum fluidization velocity (Rao, et al. 2010).

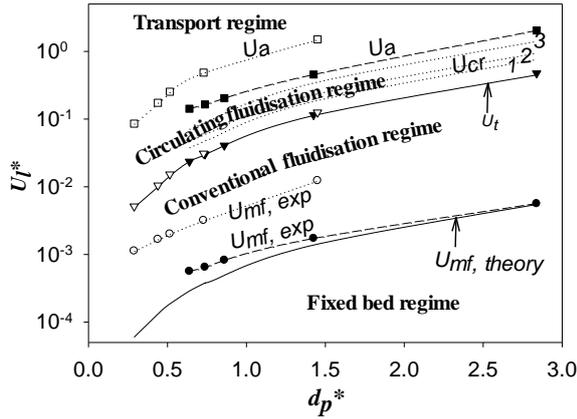


Figure 7 - Flow regime map of solid-liquid micro-circulating fluidised bed for glass (filled symbols) and PMMA (empty symbols) particles. The solid lines are the theoretical prediction for minimum fluidization velocity and terminal velocity. The dashed (glass particles) and dotted (PMMA particles) lines connecting experimental points for the minimum fluidization velocity (circles), the critical transitional velocity (triangles) and the transport transition velocity (squares) are a guide for the eye only. Three dotted lines labeled 1, 2 and 3 are U_{cr} of glass particles at solid inventories of 8%, 6% and 3% respectively.

The minimum fluidisation is postponed for both types of particles but the proportional increase is much bigger for PMMA micro-particles (8-20 times bigger experimental U_{mf} to the theoretical prediction depending on the size of particles) in comparison with glass beads (only about 2 times bigger experimental U_{mf} compared to the theoretical prediction for the smallest size particles). Our experimental data shows that the increase in the minimum velocity scales linearly with the product of the adhesion/drag force and particle-to-bed diameter ratios (do Nascimento, et al. 2016), but mostly is influenced by the surface forces. Therefore, the proportional increase is the highest for the smallest particles of both glass and PMMA, decreasing with the increase in particle size, and becoming unity for the biggest glass particles as can be seen in the plot.

However, the surface forces did not have a major influence on the critical transition velocity (U_{cr}) which demarcate the transition from conventional to

circulating fluidised bed regime as shown in Figure 7. For both types of particles the normalised transition velocity, U_{cr}/U_t , is roughly 1 at solid loading above 10%, with a weak increased U_{cr}/U_t , with an increase in particle size which is probably because of an increased wall effect as already discussed. In addition the relative transition velocity is slightly higher for PMMA particles due to influence of cohesion which forms bigger agglomerates and wall adhesion observed in the downcomer as already discussed. This is not visible in the plot as the increase is only 5 to 10% (smaller than the symbol used). Three additional dotted lines labelled on the plot as 1, 2 and 3 shows the U_{cr} of glass beads at solid inventories of 8%, 6% and 3% respectively. These shows that the solid loading influences significantly this transitional velocity further limiting operating range of velocities for the circulating fluidized bed at the micro-scale. This is not given for PMMA particles due to clarity reasons but a similar trends are also present.

The transport transition velocities, U_a from circulating fluidised bed to transport regime are very similar in magnitude for PMMA and glass particles of the same size which indicate it might be a property of the system geometry only for a given particle size. Therefore, there are two distinctive lines on the map as the relative transition velocity for PMMA particles is around 20 times particle terminal velocity while it is only 5 times for the glass beads which indicate that transition to the transport regime is influenced by cohesion. Further study is needed to elucidate this further.

As a result of observed trends, the fluidization regime is proportionally bigger for the glass beads in comparison with PMMA particles. The operational velocities for the conventional fluidization regime are approximately over two orders of magnitude of velocities (ratio U_{cr}/U_{mf} is approximately 100 to 1) for glass particles whilst it is only one order of magnitude or less for PMMA micro-particles (ratio U_{cr}/U_{mf} is approximately 10 to 1). The trend is opposite for the circulating fluidisation regime where the PMMA region is more than double in size in comparison with the glass particles case due to the transition velocities trends which are proportionally higher for PMMA beads.

4 Conclusions

A research investigation on solid-liquid fluidisation in a micro circulating fluidised bed was performed to map different regimes with a special interest in the transition from a conventional fluidised bed to circulating fluidised bed regime. Likewise in macroscopic counterpart, four operating regimes of fixed bed, conventional fluidisation, circulating fluidisation, and transport regime were mapped using

23-115 μm glass and PMMA particles. The surface forces and wall effects influence strongly the minimum fluidisation velocity which can be up to 20 times bigger for the smallest PMMA microparticles while the increase is only minor for glass particle (less than 2 times for the same size smallest glass microparticles). As in macroscopic circulating fluidised bed, the transition velocity from conventional to circulating fluidised bed decreases with solid inventory before levelling off at high enough solid inventory. The transition velocity is comparable to the particle terminal velocity, i.e. the normalised transition velocity is approximately 1 in line with previous macroscopic studies. However, there was a weak increase in the normalized transition velocity with particle size which is probably due the wall effects (higher particle to bed ratio). In addition the normalised velocity is slightly higher for PMMA particles due to stronger adhesion and cohesion forces but this influence is minimal in comparison with the influence on the minimum fluidisation velocity. The transition velocity from circulating fluidised bed to transport regime is proportionally higher for PMMA particles maybe due to particle cohesion. Consequently, the conventional regime is proportionally bigger for the glass beads in comparison with PMMA particles, whilst the situation is opposite for the circulating fluidisation regime as it is bigger for PMMA particles.

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Nomenclature

d_p	= Particle diameter	[m]
d_p^*	= Dimensionless particle diameter (Eq. 3)	[]
g	= Gravitational acceleration	[m/s ²]
H	= Bed height	[m]
	= Transport transition velocity	U_a [m/s]
U_{cf}	= Onset velocity	[m/s]
U_{cr}	= Critical transition velocity	[m/s]
U_{cr}/U_t	= Normalised transition velocity	[m/s]
U_l^*	= Dimensionless superficial liquid velocity	
U_{mf}	= Minimum fluidisation velocity	[m/s]
$U_{mf,t}$	= Theoretical minimum fluidisation velocity by Ergun equation (Eq. 1)	[m/s]
U_t	= Particle terminal velocity (Eq. 2)	[m/s]
μ	= Liquid viscosity	[Pa·s]
ΔP	= Pressure drop of bed	[Pa]
$\Delta\rho$	= Density difference of fluid and particles	[Pa]
ε	= Bed voidage	[]
ρ_f	= Fluid density	[kg/m ³]
ρ_p	= Particles density	[kg/m ³]

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