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Novel multistage solid–liquid circulating fluidized bed: liquid phase mixing characteristics

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ABSTRACT

Liquid phase axial mixing studies have been carried out in the novel solid–liquid circulating fluidized bed (SLCFB). The SLCFB primarily consists of a single multistage column (having an inner diameter of 100 mm i.d. and length of 1.40 m) which is divided into two sections wherein both the steps of utilization, namely loading (e.g., adsorption and catalytic reaction) and regeneration of solid phase, can be carried out simultaneously in continuous mode. Weak base anion exchange resin was used as the solid phase, whereas water as the fluidizing medium. The effects of physical properties of solid phase, superficial liquid velocity, and solid circulation rate on liquid phase axial dispersion coefficient were investigated. The dispersion coefficient increases monotonically with an increase in the size of solid particle, superficial liquid velocity, and solid circulation rate. The axial dispersion model (ADM) was used to model experimental residence time distribution (RTD) data. A good agreement was found between ADM predictions and the experimental measurements. A unified correlation has also been proposed to determine dispersion coefficient as a function of physical properties of solid and liquid phases, superficial liquid velocity, and solid circulation rate based on all previous and present experimental data on multistage SLCFB.

KEYWORDS : Liquid mixing; dispersion coefficient; solid–liquid fluidized bed; solid–liquid circulating fluidized bed; axial dispersion model

1. Introduction

Solid–liquid circulating fluidized beds (SLCFBs) are being consistently studied since last two decades owing to their unique eye-catching features over conventional solid–liquid fluidized beds (SLFBs) such as efficient liquid–solid contact, favorable mass and heat transfer, reduced backmixing of phases, and integrated reactor and regenerator design. The applicability of SLCFBs is fairly investigated for the diversified fields of industrial processes, such as production of linear alkyl benzene (Liang et al. 1995; Liang and Zhu 1997; Han et al. 2003; Xu et al. 2004), continuous recovery of fermentation products (Lan et al. 2000, 2002a, 2002b; Mazumder, Zhu,

and Ray 2010; Prince et al. 2012), removal and recovery of cesium from liquid radioactive nuclear waste streams (Feng et al. 2003), wastewater treatment (Cui et al. 2004; Islam et al. 2009; Chowdhury, Nakhla and Zhu 2008; Li, Nakhla, and Zhu 2012; Nirmala and Muruganandam 2013), and continuous enzymatic polymerization of phenol in bio-refining process (Trivedi, Bassi, and Zhu 2006).

SLCFBs studied so far mainly consist of riser column that is operated in the circulation fluidization regime and main column that is operated in the conventional fluidization regime. The loading operation is generally performed in the main column, whereas regeneration operation in the riser column. An integration of circulating and conventional fluidization regimes in the existing SLCFBs eventually gives rise to certain limitations, such as (1) proper pressure balance requirement for a stable operation with no intermixing between the riser and main sections, (2) the expectation of greater liquid phase mixing and solid phase mixing in the riser column because it operates in circulation fluidization regime, and (3) probable failure when loading/regeneration of solid phase is time intensive, demanding an enormous height of the riser section. Moreover, it has also been reported in the literature that the flow structure of the riser column is non-uniform with respect to bed voidage and liquid velocity in the radial direction (Liang et al. 1996, 1997; Roy and Dudukovic 2001; Zheng et al. 2002; Chavan, Kalaga, and Joshi 2009; Kalaga et al. 2012; Sang and Zhu 2012). The non-uniform flow structure adversely affects the driving force for transport processes and consequently reduces the overall performance of the SLCFB. Furthermore, owing to non-uniform flow structure, the empirical correlations developed on the basis of homogeneous fluidization concept cannot be applied for practical design and scale-up of SLCFB with sufficient degree of confidence. In view of this, the present work proposes a novel multistage SLCFB to circumvent the limitations of existing SLCFBs. The proposed SLCFB primarily comprises a single multistage column which is divided into two sections wherein both the steps of utilization or loading (e.g., adsorption and catalytic reaction) and regeneration can be carried out simultaneously in conventional fluidization regime. The operation of both loading and regeneration sections in the conventional fluidization regime offers several advantages over existing SLCFBs, such as efficient mass and heat transfer, reduced back-mixing, and adjustment of the desired residence time for time-intensive loading/regeneration operations. To standardize the proposed SLCFB, however, it is essential to investigate hydrodynamic, mixing, heat transfer and mass transfer characteristics as a function of system, operating, and geometrical parameters. The prime objective of the work was to investigate the effect of physical properties of solid phase, superficial liquid velocity, and solid circulation rate on the liquid phase dispersion coefficient. The stable operating window was established for smooth and uniform fluidization of a given solid phase. The axial dispersion model (ADM) was employed to predict liquid phase dispersion coefficients using experimental residence time distribution (RTD) data. Furthermore, an attempt has also been made to propose a unified correlation to predict dispersion coefficient using experimental data from the present and previous studies.

2. Previous work

Liquid phase mixing characteristics of conventional SLFB have been investigated extensively. Joshi (1983), Di Felice (1995), and Thombare et al. (2017) have presented excellent state-of-the-art reviews on the transport phenomena in solid–liquid fluidization. However, scanty information is available in the literature on liquid phase mixing aspect of SLCFB. Few reports are available

owing to Zheng (2001), Chen et al. (2001), Cho et al. (2005), Singh et al. (2008), and Kalaga et al. (2012).

Zheng (2001) has investigated the axial liquid phase characteristics in the riser column of SLCFB using conductivity measurement technique. Glass beads were used as solid phase, and tap water was used as the fluidizing liquid. The local axial dispersion coefficient was measured by traversing conductivity probe in a radial direction. The axial dispersion coefficients were higher at a center than near the wall of the riser. This non-uniformity in axial dispersion was due to the non-uniform distribution of superficial liquid velocity and bed voidage in radial direction of the riser column. Chen et al. (2001) have studied axial and radial mixing aspects of the liquid phase in the riser section of SLCFB using electrolyte tracer technique. Tap water and glass beads were used as the liquid and solid phases, respectively. The liquid phase mixing characteristics were adequately described using a two-dimensional diffusion model. It was found that the axial and radial Peclet numbers of liquid phase increased with an increase in the superficial liquid velocity. On the contrary, they decreased with an increase in the solid circulation rate. Cho et al. (2005) have investigated the effect of superficial liquid velocity, solid particle size, solid circulation rate, and liquid viscosity on radial liquid phase dispersion coefficient in the riser section of SLCFB using a salt-solution tracer technique. The radial liquid phase dispersion coefficient decreased with an increase in superficial liquid velocity and viscosity. However, it increased as the solid circulation rate and solid particle size were increased. This is due to the fact that the contact efficiency of solid and liquid phases decreases with an increase in superficial liquid velocity and viscosity, whereas reverse is true with respect to solid circulation rate and solid particle size.

Singh et al. (2008) have carried out RTD experiments in multistage SLFB to investigate the extent of liquid phase mixing on the stages using the step response technique. Tap water was used as a fluidizing medium, whereas glass beads were used as solid phase. Experiments were conducted by varying solid flow rate and superficial liquid velocity. It was observed that the axial liquid phase dispersion coefficient increased with an increase in superficial liquid velocity and solid flow rate. Kalaga et al. (2012) have investigated axial liquid phase mixing aspects of multistage SLCFB with prime emphasis on the development of empirical correlation to predict liquid phase axial dispersion coefficient. Ion exchange resins and glass beads were used as a solid phase and water as a fluidizing medium. RTD experiments for both the riser column and the multistage column of SLCFB have been conducted using pulse response technique. The experimental findings showed that the liquid phase axial dispersion coefficient increases with an increase in the liquid velocity, particle diameter, and particle density for the multistage column. In contrast, for the riser column, the liquid phase axial dispersion coefficient was found to decrease with an increase in the particle diameter and the particle density. Based on the experimental data available in the literature, the empirical correlations have been proposed to predict liquid phase axial dispersion coefficient in conventional SLFB and multistage SLCFB.

It should be noted that most previous workers have studied liquid phase mixing in the riser section of SLCFB, wherein superficial liquid velocity is maintained above the terminal settling velocity of solid particle, to investigate the effect of non-uniformity of flow structure on dispersion coefficient (Chen et al. 2001; Zheng 2001; Cho et al. 2005). Singh et al. (2008) and Kalaga et al. (2012) have carried out liquid phase mixing study in multistage SLCFB with certain constraints. Singh et al. (2008) have used multistage SLFB rather than multistage SLCFB. They have collected solid particles at bottom of column and charged them via conveyor belt at top of column. Kalaga et al. (2012) have conducted liquid phase mixing study separately in the riser

and multistage columns due to practical difficulty in maintaining dynamic seal between riser and multistage columns. Furthermore, their studies predominantly reveal the effect of superficial liquid velocity and physical properties of solid phase on dispersion coefficient. In the present work, we have made an attempt to study the effect of solid circulation rate on dispersion coefficient along with the effect of superficial liquid velocity and physical properties of solid phase.

3. Experimental details

The weak base anion exchange resin (Tulsion A-8X MP) was used as a solid phase, whereas tap water was used as a liquid phase for experimentation. The resin particles were segregated into various sizes using standard SS sieves. The 0.60, 0.85, and 1.20 mm particle sizes were selected to study liquid phase mixing characteristics. The physical properties of the resin, supplied by the manufacturer are given in Table 1.

Table 1. The resin properties given by the manufacturer

Property	Weak base anion exchange resin, (Tulsion A-8X MP)
Particle size, mm	0.3- 1.2
Particle density, kg.m ⁻³	1100
Matrix structure	Styrene-divinyl benzene copolymer
Functional group	Ternary amine
Ionic form	Free base
Moisture content, (% kg water.kg wet resin ⁻¹)	47
Exchange capacity, (mo.kg dry resin ⁻¹)	2.6

3.1. Experimental set-ups

3.1.1. Solid–liquid fluidized bed

An acrylic column with an inner diameter of 100 mm and height of 1.20 m was used. A reciprocating pump of 250mL.s⁻¹ capacity was used to feed water to the column. To smoothen the liquid flow, a pulsation dampener was fitted to the pump. The dampener pressurizes the liquid when the pump is at full flow and discharges to the system when the pump outflow falls. This significantly smoothes the flow such that the outflow is practically uniform. The water flow rate was measured using rotameter. A calming section packed with glass beads of 0.30 m height was provided to homogenize the liquid flow before it reaches to the liquid distributor. The distributor was a perforated plate containing 315 holes of 2mm diameter on a triangular pitch, giving a free area ratio of 12.50%. A mesh of BSS 100 was attached on top and bottom of the distributor plate to restrict the movement of particles across it. To ensure that no air bubbles intruded into the column during operation, the necessary arrangements in the fittings and fixtures were made. A septum was provided at the bottom of the column below the liquid distributor for injecting predetermined amount of the tracer using a syringe (having 2 mm needle). The provisions were made for online measurement of tracer concentration using conductivity meter. The concentration of tracer was measured in terms of voltage with online data acquisition system at a frequency of 10Hz using a graphite electrode (probe), fixed at the bed surface.

3.1.2. Multistage solid–liquid circulating fluidized bed

A schematic diagram of the multistage SLCFB is shown in Figure 1. The multistage SLCFB assembly primarily consists of a glass multistage column which was further divided into loading section (1) and regenerating section (2), a bottom solids return pipe connecting the riser and the multistage column at the base (3), a riser column (4), liquid–solid separator (5), and a top solids return pipe connecting liquid–solid separator and the multistage column. The loading and regenerating sections mainly consist of five stages (each having an inner diameter of 100mm and height of 100mm) assembled together with flange joints. The loading and regeneration sections were connected to each other by SS pipe having an inner diameter of 10mm and length of 200mm, giving total height of 1.40m. A stainless steel (SS) mesh with openings smaller than the solid particle size was fitted onto SS sieve plates that were sandwiched between every pair of glass stages using adjoining flanges. The holes of 2mm were provided on each SS sieve plate, providing 5.0% open area for water flow. The solid particles moved across on the stage to the next stage through a downspout, as the liquid flows upward through mesh openings. Two types of SS stages were arranged alternatively in multistage column using a pair of adjoining flanges. For one set of successive stages, first stage consisted of a downspout which was fitted at the center of the stage while the second stage comprised two downspouts located around periphery as circumferential downspouts. The SS pipe having an inner diameter of 10mm with 75mm length was used as downspout to encompass weir heights of 25mm. The schematic of SS stage configuration is given in Figure 2(A).

The flow of solid particles from loading to regeneration section was controlled by a butterfly valve (V2) appended on interconnecting SS pipe. There were two distributors at the bottom of each section as (1) specially designed conical distributor, providing 17.30% open area for primary liquid flow rate, and (2) secondary liquid distributor made of a SS stage with 10.40% opening area for auxiliary liquid flow. Figure 2(B) shows the geometrical details of specially designed conical distributor for primary liquid inlet. The total liquid flow to the section is a summation of primary and auxiliary flow rates. The auxiliary liquid flow rate mobilizes the solid particles underneath primary distributor to ease solid particles flow from one section to another.

The solid particles moved via solid return pipe (having an inner diameter of 25 mm and length of 500 mm) to the riser column and subsequently carried to the solid–liquid separator. The separator (having an inner diameter of 300 mm and length of 500 mm) was used to charge solid particles to loading section through solid transport line wherein solid transport was controlled by valve V1. Solid particles were kept in expanded state by liquid phase charged from bottom of the separator via calming section of 150 mm. The separator was provided with top outlet for discharge of liquid via mesh to avoid loss of fine solid particles. The solid particles moved from separator to multistage column in orderly manner via solid return pipe. The solid return pipe was provided with special type of valve to measure the mass flow rate of solid particles. Figure 2(C) shows dimensional detail of the specially designed valve. With the valve open, the solid–liquid mixture was collected for a known length of time. During the measurement, no solid–liquid flow was permitted to flow to multistage column.

Necessary arrangement was made to measure RTD of liquid phase. A septum was provided at the bottom of the loading column before the liquid distributor to inject predetermined the tracer using a syringe (having 2mm needle). The concentration of the tracer was recorded in term of conductivity of the solution at the outlet of the loading section as shown in Figure 1.

Figure 1. Experimental set up. 1. Loading section, 2. Regenerating section, 3. Solid return pipe, 4. Riser column, 5. Solid-liquid separator, 6. SS stage with SS mesh, 7. Down-comer, 8. Overflow, 9. Tracer injection syringe, 10. Conductivity probe, 11. Data acquisition system, 12. Computer. V: valve; D: diffuser/solid distributor (All dimensions are in mm).

Figure 2. Geometrical detail of auxiliary devices. (A) SS stage, (B) Primary distributor, (C) Specially designed valve for solid circulation rate measurement: (a) open position, (b) closed position (All dimensions are in mm)

3.2. Methodology

3.2.1. Expansion characteristics of solid particles

Expansion characteristics of solid particles of a given size were studied in SLFB. A known quantity of solid particles of a given size was charged in a column to encompass a predetermined height of 0.20 m. The fluidizing medium, water, was charged to the column at a predetermined flow rate using calibrated rotameter. The system was allowed to attain a steady state for 1 h for a given superficial liquid velocity and solid particle size. The expanded bed height was then noted. The average voidage was determined by taking solid phase mass balance at initial and final fluidized states for a solid particle size under consideration at a given superficial liquid velocity (Chavan and Joshi 2008).

$$\frac{\pi}{4} D^2 H_O (1 - \epsilon_{LO}) = \frac{\pi}{4} D^2 H (1 - \epsilon_L) \quad (1)$$

where the subscript “O” denotes the initial fixed bed conditions, H is bed height, D is diameter of column, and ϵ_L is voidage. All experiments were carried out three times and average values were noted.

3.2.2. Operating window of SLCFB

The solid transport from loading section to regenerating section, and regenerating section to the riser was closed initially using valve V2 and valve V3, respectively. The primary and auxiliary liquid streams were started in loading and regenerating sections at a predetermined flow rate using calibrated rotameters and subsequently the fresh solid particles were charged using valve V1 at the top of the loading section via diffuser in order to distribute solid particles uniformly. The solid bed on the stage was allowed to expand up to the weir height and subsequently flowed to the next lower stage because solid particles continued to pour from the adjacent upper stage through downspout, creating difference between bed depths from the center to the periphery of the stage or vice versa, depending upon the type of the downspout in that stage (center or circumferential). Thus, the state of fluidization on every stage was cross-current although the overall flow of the solid and liquid phases was in the counter-current direction. The similar flow pattern was achieved in the regenerating section when the valve V2 was gradually opened and solid particles were allowed to enter into the section for given liquid flow rates in the section. When solid particles reached to the bottom of the regenerating section, valve V3 was gradually opened to transport solid particles to the top feed tank for continuous operation. The system was allowed to attain steady state for 2 h before all experimental measurements were carried out.

In the proposed system, the loading and regenerating sections were interconnected by a solid transport line through which solid particles flowed from one section to another. The dynamic seal between these two sections is of critical importance to avoid intermixing of two liquid streams of fairly different properties for successful operation. The dynamic seal was realized by maintaining a particle plug in the solid transport lines, allowing solid particles to flow in fixed bed mode.

3.2.3. Liquid mixing study

The pulse response technique was employed to investigate the axial mixing characteristics of SLFB and multistage SLCFB. A septum was provided at the bottom of the each column before the liquid distributor for injecting the tracer. A pulse input of 2 mL NaCl solution (3.5 M) was injected through a septum provided at the bottom of SLFB and loading section of multistage SLCFB using a syringe (having 2 mm needle). The time taken for the tracer injection in all the

experiments was less than one third of a second. The provisions were made for online measurement of tracer concentration using conductivity meter. To measure the conductivity, graphite electrode (probe) was fixed at the outlet of the loading section of multistage SLCFB and bed surface of SLFB. To record the online conductivity measurements, a conductivity meter was fabricated and the concentration in terms of voltage was measured at a frequency of 10 Hz using the online data acquisition system. The conductivity data was converted to salt concentration using calibration equation based on separate experiments.

4. Mathematical model

In the present work, a one-dimensional ADM was applied to characterize axial liquid mixing. The key assumption in this model is that axial liquid dispersion coefficient is constant along the bed length. Taking mass balance for a tracer over a control volume, one can get the following unsteady state one-dimensional equation:

$$\frac{\partial C}{\partial t} = D_L \frac{\partial^2 C}{\partial z^2} - U \frac{\partial C}{\partial z} \quad (2)$$

with the boundary conditions

$$C(z, 0) = 0 \quad \text{for } 0 \leq z \leq H \text{ and } t = 0$$

$$C_o = C - \frac{D_L}{U} \frac{\partial C}{\partial z} \quad \text{for } z = 0 \text{ and } t \geq 0$$

$$\frac{\partial C}{\partial z} = 0 \quad \text{for } z = H \text{ and } t \geq 0$$

where D_L is axial dispersion coefficient, C is concentration of tracer at a given time (t) and position (z), C_o is inlet concentration of tracer, and U is interstitial liquid velocity. Equation (2) was numerically solved using “PDEPE” solver in MATLAB (2016b). The axial dispersion coefficient was estimated by calculating the mean squared error function (Equation (3)) between experimental and predicted dimensionless RTD curve

$$\text{Error} = \frac{1}{m} \sum_{i=m}^m \frac{E(\theta)_{\text{exp}}^i - E(\theta)_{\text{ADM}}^i}{E(\theta)_{\text{exp}}^i} \quad (3)$$

where $E(\Theta)$ is a dimensionless RTD function, defined as follows:

$$E(\Theta) = t_m E(t)$$

$E(t)$ is a RTD function and t_m is a mean residence time, estimated as follows:

$$E(t) = \frac{C}{\int_0^\infty C dt} \quad (4)$$

$$t_m = \frac{\int_0^\infty t C dt}{\int_0^\infty C dt} \quad (5)$$

5. Result and discussion

The physical properties of liquid and solid phases and superficial liquid velocity primarily affect the liquid phase mixing for a given geometrical parameters in conventional SLFB. Needless to

mention that in conventional SLFB, solid phase is in batch mode, whereas liquid phase in continuous mode. In SLCFB, however, the solid particles are maintained in a circulation mode between the riser and main columns in order to accommodate both the steps, namely loading and regeneration, in a continuous mode. Therefore, it is essential to consider solid particle circulation rate as one of the operating parameters while discerning the liquid phase mixing characteristics. In liquid fluidization, the factors that cause the axial mixing in the liquid phase are (1) the presence of particles, (2) the motion of particles, (3) the continuous formation and separation of particle wakes, and (4) the liquid phase radial velocity profile. The effect of these factors on liquid phase axial mixing can be implicitly elucidated by ADM, wherein axial dispersion of the liquid is considered to be composed of the molecular diffusion, the turbulent diffusion, and the convective diffusion.

5.1. Solid–liquid fluidized bed

5.1.1. Expansion characteristics

The velocity–voidage relationships were established by fluidizing solid particles at different superficial liquid velocities in the range of 1–15 mm s⁻¹ for a given solid particle size in SLFB. The solid mass balance method was used to estimate average voidage of a bed at given superficial liquid velocity. Figure 3 shows experimental expansion data for three different particle sizes (0.60, 0.85, and 1.20 mm). The terminal settling velocity of solid particles was determined by fitting the Richardson–Zaki equation to the experimental data.

$$\frac{V_L}{V_{S\infty}} = \epsilon_L^n \quad (6)$$

where V_L is superficial liquid velocity, $V_{S\infty}$ is terminal settling velocity of solid particle, ϵ_L is bed voidage, and n is the Richardson–Zaki parameter. The solid lines in Figure 3 show the fitting of Richardson–Zaki equation to experimental data with maximum deviation of 1.60%. The Richardson–Zaki parameters for a given particle size are reported in Table 2.

Figure 3. Expansion characteristics of solid particles. Experimental: ($\diamond$) 0.60 mm, (Δ) 0.85 mm, ($\square$) 1.20 mm; Richardson-Zaki equation: (—) 0.60 mm, (- - -) 0.85 mm, (—) 1.20 mm.

Table 2. Expansion parameters of solid phase

Particle diameter, d_p (mm)	Terminal settling velocity, $V_{s\infty}$ (mm.s ⁻¹)	Richardson- Zaki parameter, n (-)	Reynolds number, Re (-)	Galileo number, Ga (-)
0.60	14.04	3.51	8.42	211.90
0.85	17.93	3.34	15.24	613.15
1.20	21.32	3.24	25.58	1695.16

5.1.2. Liquid mixing

Figure 4 shows the typical dimensionless RTD curves measured experimentally and those derived from the ADM at different superficial liquid velocities for 0.85mm solid particle size. All the symbols in this figure represent experimental values of the dimensionless tracer concentration and the solid lines represent curves predicted from ADM. Figure 4 demonstrates good agreement between the experimental RTD curves with those predicted by ADM. The similar experimental dimensionless curves have been seen for 0.60 and 1.20mm solid particle sizes. Figure 5 demonstrates the effect of superficial liquid velocity on axial dispersion coefficient for a given solid particle size. Figure 5 also depicts the comparison between experimental data and ADM simulations where the agreement was found to be within a standard deviation of 10%. The superficial liquid velocity strongly influences the axial liquid phase dispersion coefficient. The dispersion coefficient increases fairly linearly with an increase in superficial liquid velocity. With an increase in superficial liquid velocity, the movement of solid particles increases and thus the liquid is subjected to a vigorous turbulence. The similar observation has been put forth by previous researchers while investigating the liquid phase mixing characteristics of SLFB. Most of these studies report that liquid phase axial dispersion coefficient increases with an increase in superficial liquid velocity. A maximum value of axial dispersion coefficient was reported at a superficial liquid velocity corresponding to bed voidage of about 0.70 (Cairns and Prausnitz 1960; Mehta and Shemilt 1976; Kim and Kim 1983; Tan and Krishnaswamy 1989). On the contrary, in the present investigation, it has been observed that the axial dispersion coefficient monotonically increases with an increase in superficial liquid velocity. Similar results have been obtained by Asif, Kalogerakis, and Behie (1992) and Kalaga et al. (2012).

Figure 4. Effect of superficial liquid velocity on RTD of liquid phase for 0.85mm particle size in SLFB. Experimental: ($\square$) 2.02 mm.s⁻¹; (Δ) 3.50 mm.s⁻¹; ($\diamond$) 5.50 mm.s⁻¹; ($\circ$) 6.52 mm.s⁻¹; ADM: (—) 2.02 mm.s⁻¹; (...) 3.50 mm.s⁻¹; (—) 5.50 mm.s⁻¹; (—) 6.52 mm.s⁻¹.

Figure 5. Effect of the superficial liquid velocity on the dispersion coefficient in SLFB. Experimental: ($\diamond$) 0.60 mm; ($\square$) 0.85 mm; (Δ) 1.20 mm; ADM (—) 0.60 mm; (- - -) 0.85 mm; (—) 1.20 mm.

The effect of particle size on the axial dispersion coefficient is also depicted in Figure 5. It is clear that axial dispersion coefficient increases with an increase in solid particle size. For example, the values of dispersion coefficient were found to be 5.21, 6.60, and $7.68 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$

for 0.60, 0.85, and 1.20mm solid particle sizes, respectively at superficial liquid velocity of 3.50 mm.s^{-1} . All previous researchers have reported the similar finding with respect to the effect of solid particle size on the axial dispersion coefficient (Cairns and Prausnitz 1960; Chung and Wen 1968; Tang and Fan 1990; Asif, Kalogerakis, and Behie 1992; Kalaga et al. 2012). The terminal settling velocity of solid particle increases with an increase in solid particle size. Therefore, to maintain a desired voidage for particles of higher terminal settling velocity, higher relative liquid velocity is required. Consequently, the power consumption per unit volume increases which results into higher intensity of turbulence, leading to higher extent of mixing.

5.2. Novel multistage solid–liquid circulating fluidized bed

Before we embark on liquid phase characteristics of the proposed model, it is essential to establish the stable operating window of the operation for the proposed SLCFB. The operating window determines the limiting values of superficial liquid velocity and solid circulation rate for stable and smooth operation of the SLCFBs. Therefore, the present section is divided into two parts: (1) establishment of operating window and (2) study of liquid phase mixing characteristics of proposed SLCFB.

5.2.1. Operating window

In SLCFB, solid circulation rate is an important operating parameter which determines the operating window of operation together with superficial liquid velocity. It has been well established that the smooth and stable operation of the column is not solely dependent on the superficial liquid velocity but equivalently dependent on solid circulation rate. The ratio of solid circulation rate to superficial liquid velocity implicitly sets the criterion of stable and smooth operation of SLCFBs (Singh et al. 2008; Chavan, Kalaga and Joshi 2009; Chavan et al. 2018). Therefore, the operating window has been established for the smooth and stable operation of the multistage column. The superficial liquid velocity was varied for a given solid circulation rate to determine two extremes conditions, namely loading and flooding. Although the multistage column operates in conventional fluidization regime wherein superficial liquid velocity ranges between minimum fluidization velocity and terminal settling velocity, it is essential to determine flooding and loading states for stable operation of the system for given physical properties of liquid and solid phases. There is a minimum solid flow rate for a given superficial liquid velocity below which the column is considered to be flooded. At the flooding state, the fluidizing medium (water in the present study) prevents the flow of solid particles from one stage to another through the downcomer. There is another extreme state, called as loading state, wherein solid particles get excessively loaded on the stage for a given superficial liquid velocity at a certain solid flow rate. At the loading condition, the solid particles chock the downcomer, preventing the flow from one stage to another.

Figure 6 shows the operating window for 0.60, 0.80, and 1.20 mm particle sizes. The filled symbols represent the maximum solid flow rate at a given superficial liquid velocity, which may be used for stable and smooth operation of the column without loading of the stages with excess solids. Similarly, the hollows symbols represent the solid flow rate at a given superficial liquid velocity, which may be used without flooding of the stage with water. Therefore, the difference between the two values of superficial liquid velocity defines the operating range of fluidization without loading and flooding of the stages at a given superficial liquid velocity. For example, for 0.85mm particle size, at a superficial liquid velocity equal to 3.66 mm.s^{-1} the operating range of solid circulation rate is between 1.45 and 2.40 g.s^{-1}

corresponding to flooding and loading in the multistage column. The solid circulation rate smaller than 1.45 g.s^{-1} gradually leads to the stage flooded, whereas that in excess of 2.40 g.s^{-1} causes loading of the stages. In Figure 6, the operating range is marked with vertical double-headed arrows for clarity for a given size of solid particle.

Figure 6. Operating window for multistage SLCFB. Filled symbols: loading limit. Hollow symbols: flooding limit. (□) 0.60 mm; (△) 0.85 mm; (◇) 1.20 mm.

5.2.2. Liquid phase mixing

RTD experiments were carried out in the multistage column within an operating window by varying superficial liquid velocity and solid circulation rate to ascertain the extent of liquid phase mixing for a given solid particle size. For example, superficial liquid velocity was varied in the range of 0.50 – 3.0 mm.s^{-1} and solid circulation rate was kept constant at 1.40 g.s^{-1} for 0.85 mm solid particle size to study the effect of superficial liquid velocity on liquid phase axial mixing. Similarly, the effect of solid circulation rate was investigated by varying solid circulation rate in the range of 1.0 – 2.0 g.s^{-1} at superficial liquid velocity equal to 2.17 mm.s^{-1} . Correspondingly, the effect of superficial liquid velocity and solid circulation rate on liquid phase axial mixing was studied for other solid particle sizes under consideration. It may be noted that tracer concentration was measured at the outlet of the multistage column which comprises five stages. The multistage column was operated in conventional fluidization regime wherein superficial liquid velocity varies from minimum fluidization velocity to terminal settling velocity of the solid particle. Consequently, each stage comprises SLFB at the bottom and solid particle free liquid at the top under steady state operation of multistage column. The measured RTD at the outlet is overall effect of such five stages.

Figure 7 shows typical dimensionless RTD curves measured experimentally and those derived from ADM as a function of superficial liquid velocity within an operating window for 0.85 mm particle size. All the symbols represent the experimental values while solid lines represent curves predicted from ADM. As mentioned earlier, on each stage (sieve plate) of the multistage column a conventional fluidization regime was attained. Figure 8 shows the

comparison between experimental and ADM predicted values of dispersion coefficients as a function superficial liquid velocity for a given solid particle size within a standard deviation of 10%. A similar trend has been observed in multistage column as in the conventional SLFB. However, the values of dispersion coefficient are lower for a given solid particle size when compared to the corresponding dispersion coefficient values obtained in conventional SLFB. The presence of the sieve trays decreases the axial mixing in the multistage column of the SLCFB as compared to that in the conventional SLFB. The presence of the sieve trays tends to render the velocity profile more uniform and physically reduces the backmixing in the liquid phase by eliminating any cross-flow across the sieve plates. This, in turn, reduces the axial liquid mixing, which is very desirable for any separation technique.

Figure 7. Effect of superficial liquid velocity on RTD of liquid phase for 0.85mm particle size in multistage SLCFB. Experimental: ($\square$) 0.50 mm.s⁻¹; (Δ) 1.50 mm.s⁻¹; ($\diamond$) 2.17 mm.s⁻¹; ($\circ$) 3.0 mm.s⁻¹; ADM: (—) 0.50 mm.s⁻¹; (...) 1.50 mm.s⁻¹; (- - -) 2.17 mm.s⁻¹; (-_-) 3.0 mm.s⁻¹.

Figure 8. Effect of the superficial liquid velocity on the dispersion coefficient in SLCFB. Experimental: ($\diamond$) 0.60 mm; ($\square$) 0.85 mm; (Δ) 1.20 mm; ADM: (—) 0.60 mm; (- - -) 0.85 mm; (- - -) 1.20 mm.

Figure 9 shows the RTD curves as a function of solid circulation rate for a given superficial liquid velocity for 0.85mm solid particle size. Figure 10 demonstrates the effect of solid circulation rate on axial dispersion coefficient for solid particle sizes under consideration. It can be seen that axial dispersion coefficient increases with an increase in solid circulation rate. Although solid and liquid phases contact counter-currently in multistage column, solid particles meet cross-currently with liquid phase on each stage contact. An increase in solid circulation rate tends to enhance cross-current flow solid particles which in turn intensify radial movement of liquid phase. Consequently, RTD curve spread increases which result into increase in axial dispersion coefficient with solid circulation rate. Similar result has been demonstrated by Singh et al. (2008) while investigating the effect of solid circulation rate on axial dispersion coefficient. It is also clear from Figures 9 and 10 that the dispersion coefficient values predicted by ADM are in good agreement with experimental values.

Figure 9. Effect of solid circulation rates on RTD of liquid phase for 0.85mm particle size in SLCFB. Experimental: ($\diamond$) 1.0 g.s⁻¹; ($\square$) 1.40 g.s⁻¹; (Δ) 1.75 g.s⁻¹; (o) 2.0 g.s⁻¹ ADM: (—) 1.0 g.s⁻¹; (- - -) 1.40 g.s⁻¹; (—) 1.75 g.s⁻¹; (...) 2.0 g.s⁻¹.

Figure 10. Effect of the solid circulation rate on the dispersion coefficient in SLCFB. Experimental: ($\diamond$) 0.60 mm; ($\square$) 0.85 mm; (Δ) 1.20 mm; ADM: (—) 0.60 mm; (- - -) 0.85 mm; (—) 1.20 mm.

5.3. Correlation for dispersion coefficient in multistage SLCFB

Voluminous reports are available in the literature wherein empirical correlation has been proposed to estimate axial dispersion coefficient as a function of physical properties of solid and liquid phases, and superficial liquid velocity for a given geometrical parameters (Chung and Wen 1968; Krishnaswamy and Shemilt 1972; Krishnaswamy, Ganapathy, and Shemilt 1978; Kikuchi et al. 1984; Tan and Krishnaswamy 1989; Tang and Fan 1990; Kalaga et al. 2012). However, there is no correlation available in the literature to predict axial dispersion coefficient in multistage column with due consideration of solid circulation rate. Kalaga et al. (2012) have made an attempt to propose a correlation in multistage column. However, their correlation does not elucidate the effect of solid circulation rate on axial dispersion coefficient since RTD measurements were carried out as a function of superficial liquid velocity by keeping the solid circulation rate constant for all solid particle sizes under investigation. Therefore, there is need to propose a unified correlation in order to account the effect of solid circulation rate on axial dispersion coefficient.

Literature indicates that the liquid phase dispersion coefficient is a strong function of physical properties of solid and liquid phases and superficial liquid velocity. Therefore, following equations can be written:

$$D_L = f[V_L, d_p, \mu_L, \rho_L, (\rho_S - \rho_L), g] \quad (7)$$

$$D_L = K(V_L)^a (d_p)^b (\mu_L)^c (\rho_L)^d (\rho_S - \rho_L)^e (g)^f \quad (8)$$

Equation (8) can be written in terms of dimensionless numbers by using Rayleigh's method of dimensional analysis as follows:

$$\frac{D_L}{d_p V_L} = K(Re)^{(-c-2f)} (Ga)^f \left(\frac{\rho_L}{\rho_S - \rho_L} \right)^{[(3c+2f)/3]} \quad (9)$$

where

$$Re = \frac{d_p V_L \rho_L}{\mu_L}$$

$$Ga = \frac{d_p^3 \rho_L (\rho_S - \rho_L) g}{\mu_L^2}$$

Equation (9) can be extended to multistage SLCFB with following modification to take into account the solid circulation rate:

$$\frac{D_L}{d_p V_L} = K(Re)^{(-c-2f)} (Ga)^f \left(\frac{\rho_L}{\rho_S - \rho_L} \right)^{[(3c+2f)/3]} (M_S)^h \quad (10)$$

where M_S is a normalized mass flow rate of solid particles, defined as follows:

$$M_S = \frac{G_S}{V_L A \rho_L}$$

where G_S is a solid circulation rate and A is a cross-sectional area of column.

To the best of authors knowledge, Singh et al. (2008) and Kalaga et al. (2012) have carried out RTD measurements in multistage SLCFB and reported liquid phase dispersion coefficients with due consideration of solid circulation rate. Therefore, the correlation constants in Equation (10) have been obtained by using their experimental data sets and present

experimental data by least square regression analysis. The following expression has been obtained:

$$\frac{D_L}{d_p V_L} = 6.52 \times 10^4 (Re)^{0.64} (Ga)^{-0.46} \left(\frac{\rho_L}{\rho_s - \rho_L} \right)^{-1.60} (M_s)^{0.62} \quad (11)$$

Figure 11 shows that values of DL predicted using Equation (11) match satisfactorily with experimental values with a maximum deviation of 17.21%.

Figure 11. Parity plot. (□) Singh et al. (2008); (◇) Kalaga et al. (2012); (Δ) Present work.

6. Conclusions

The liquid phase axial dispersion characteristics of novel SLCFB were investigated using the pulse response technique within an established operating window. The ADM was successfully employed to determine axial liquid phase dispersion coefficient. The experimental findings show that the liquid phase axial dispersion coefficient increases with an increase in superficial liquid velocity, particle diameter, and solid circulation rate in multistage column. The following correlation has been proposed to predict axial dispersion coefficient in multistage column with due consideration of solid circulation rate:

$$\frac{D_L}{d_p V_L} = 6.52 \times 10^4 (Re)^{0.64} (Ga)^{-0.46} \left(\frac{\rho_L}{\rho_s - \rho_L} \right)^{-1.60} (M_s)^{0.62}$$

The correlation satisfactorily predicts axial dispersion coefficient value with a maximum deviation of 17.21%. In all the cases, good agreement was observed between the experimental values of the dispersion coefficient and those predicted by ADM.

Nomenclature

- A = cross sectional area of multistage column, (m²)
C = concentration of tracer at a given time, (g.m⁻³)

D	= column diameter, (m)
D_L	= liquid dispersion coefficient, ($m^2.s^{-1}$)
d_p	= diameter of solid particle, (m)
$E(t)$	= RTD function, (s^{-1})
$E(\Theta)$	= dimensionless RTD function, (-)
g	= gravitational acceleration, ($m.s^{-2}$)
Ga	= Galileo number, (-)
G_s	= solid circulation rate, ($g.s^{-1}$)
H	= suspension height, (m)
K	= correlation coefficient defined by equation 8, (-)
m	= number of data points, (-)
M_s	= normalized solid circulation rate, (-)
n	= Richardson – Zaki parameter, (-)
Re	= Reynolds number based on superficial liquid velocity, (-)
t	= time, (s)
t_m	= mean time, (s)
U	= interstitial liquid velocity, ($m.s^{-1}$)
V_L	= superficial liquid velocity, ($m.s^{-1}$)
$V_{s\infty}$	= terminal settling velocity of solid particle, ($m.s^{-1}$)
z	= bed height, (m)

Greek letters

ε_L	= voidage of the bed, (-)
μ_L	= viscosity of liquid, ($kg\ m^{-1}\ s^{-1}$)
ρ_L	= liquid density, ($kg\ m^{-3}$)
ρ_s	= solid density, ($kg\ m^{-3}$)

Subscripts

O	= initial condition
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