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A novel method for measuring the residence time distribution in short time scale particulate systems

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Abstract

A novel, non-intrusive method is described for measuring the particle residence time distribution (RTD) in a system with a short mean residence time. The method uses phosphorescent tracer particles activated by a high intensity pulse of light at the inlet. Tracer is detected using a light sensitive photomultiplier unit. Appropriate boundary conditions are maintained by using an annular feeder fluidised bed at the entrance boundary and an inline jet mixer installed at the exit boundary. This well defined arrangement of experimental boundary conditions represents a significant advance in the measurement of unbiased particle RTDs in these systems. The method was developed for measuring the particle RTD in a circulating fluidised bed (CFB) riser, but is applicable to other particle–fluid systems where a fast response measurement is required.

Keywords: Particle residence time distribution; RTD; Experimental method; Short time scale system; Circulating fluidised bed

1. Introduction

The particle residence time distribution (RTD) in a short time scale system such as a circulating fluidised bed (CFB) can provide vital information for system designers and operators. Here, we define a short time scale system as one in which the typical mean residence time lies in the range microseconds to seconds. Some examples of short time scale particulate systems are listed in Table 1.

Compared with liquid or gas systems, stimulus response RTD experiments with solids are particularly difficult to perform without introducing bias. The numerous methods described in the literature for measuring particle RTDs all exhibit some limitation. The choice of tracer particles, the method of introducing and detecting the tracer, characterisation of the boundary conditions and the flow conditions inside the system all potentially contribute to the experimental bias.A CFB riser has been chosen as an example system to illustrate the application of the method reported here because,

(i) CFB riser RTDs have been widely studied and continue to attract research interest;
(ii) the typical mean residence time in a laboratory scale CFB riser is of the order of a few seconds, consistent with our definition of a short time scale system;
(iii) the gas–solid hydrodynamics in a CFB riser are highly complex, requiring a fast response technique to capture sufficient detail;
(iv) previous studies of the particle RTD in CFB riser's have failed to adequately characterise the experimental boundary conditions; and
(v) some previous studies have failed to capture key features of the gas–solids flow due to the limitations in the RTD method. This enables a comparison between the method reported here and techniques used previously to measure particle RTDs in short time scale systems.

To date, over a dozen different techniques have been used for fluidised bed particle residence time experiments. Examples include various stimulus response studies, radioactive, coloured, ferromagnetic, chemically doped, different temperature or different sized tracer particles and single particle studies, where a single tracer particle is tracked continuously. The method reported here is a partial adaptation of the methods of Brewster and Seader [1], Roques et al. [27] and Wei et al. [2]; it was developed to overcome the limitations of these and other techniques. It is novel and has been developed to produce well defined RTDs in systems with short mean residence times and complicated internal hydrodynamics.

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Nomenclature

CED	Cambridge Electronic Design data acquisition system
E(t)	E curve or normalised RTD curve (s^{-1})
U_{mf}	Minimum fluidisation velocity (Fig. 4) (m/s)
PMT	PMT
D	Riser diameter (Fig. 9) (m)
H	Riser height (Fig. 9) (m)
U_t	Single particle terminal velocity (m/s)
G_s	Solids circulation flux (Figs. 8 and 9) (kg/m^2 s)
U_g	Superficial gas velocity (Figs. 8 and 9) (m/s)
ΔP_t	Total pressure drop over bubbling fluidised bed (Fig. 4) (mmH_2O).

Table 1
Some examples of fast response gas–solid systems

System	Range of typical mean residence times
Riser reactor (CFB)	1–30 s
Downer reactor	0.5–0 s
FCC riser	1–30 s
Gas–solid and liquid–solid cyclones	1–10 s
Pneumatic conveying	1 s–1 min
Micro- and nano-scale reactors	1 ms–1 s

2. Reported experimental RTD techniques

The range of experimental techniques available to measure the particle RTD in a fast response particulate system is extensive. Any method able to differentiate a sample of tracer particles via a characteristic that does not alter its overall flow behaviour is potentially suitable. This section reviews and summarises published the experimental techniques for measuring mixing phenomena in particulate systems, particularly bubbling and CFBs. To date more than a dozen different methods have been reported.

The experimental techniques reviewed here are classified according to whether they are disruptive or non-disruptive of the flow conditions within the system being studied. They are then classified according to whether or not they permanently contaminate the solids inventory. For any experimental programme, the second series of methods (i.e. non-disruptive and non-contaminating) is obviously preferred, given that this minimises the experimental bias and the costs involved in replacing the solids inventory.

2.1. Disruptive techniques

2.1.1. Chemically different tracers

Rhodes et al. [3] studied the effect of operating conditions on longitudinal solids mixing in a CFB riser. They used a sodium chloride tracer, ground to a size distribution similar to the bed solids, for impulse injection experiments in a bed of non-porous alumina powder with a density similar to that of sodium chloride. The technique was based on the method of Bader et al. [4]. Sampling was performed simultaneously at three locations in the riser (just below the riser exit and at the wall and riser centreline 3 m below the exit), using a 7.5 mm probe connected to a sampling cyclone with dip leg. Solids from the cyclone dip leg were discharged into a series of containers at the end of a run using a slight pressure to minimise axial mixing. The containers were placed on a motor driven carousel. Individual samples were washed and the salt concentration determined by the conductivity of the wash solution. The time constant of the sampling method implied the technique was unable to characterise the RTD curve as fully as a fast response technique. Variations in the measured tracer concentration were observed depending upon radial location of the sampling probe which suggests that under the conditions of the experiments, the riser was operating in a core-annulus flow regime.

Bader et al.' [4] original version of the technique used a multi-injection system with four injection ports located 90° apart, designed to inject the tracer over a cross section, parallel to the solids flow. Each injection tube was positioned 0.108 m from the centre of the riser ($D = 0.31$ m). The tracer was sampled at three vertical locations in the riser using a probe positioned with its end perpendicular to the solids flow.

Zheng et al. [5], as part of a larger study of the characteristics of fast fluidised beds with internals, performed a series of experiments to determine the solids RTD using FCC catalyst saturated with sodium chloride. Tracer solids were pulse injected into the bed using high pressure air and the riser sampled at various locations (both upstream and downstream of the injection point) using a cyclone separator and slender storage tube capable of collecting a series of samples in succession. Analysis of the data was by determining a relationship between the concentration of tracer particles and the corresponding conductivity of their aqueous solution in the same manner as Rhodes et al. [3] and Bader et al. [4].

The sampling time constant of the methods of Rhodes et al. [3], Bader et al. [4] and Zheng et al. [5], a measure of the maximum rate at which samples can be taken, is dependent upon the speed of the motor driving the sampling cup holder at the bottom of the cyclone dipleg. In these studies, this resolution was about 0.5 s. Any features of the RTD (resulting e.g. from the hydrodynamics of the system) that occur within this time scale cannot be measured by this technique. This will be discussed in greater detail later.

As suggested by the section heading, these methods are also disruptive to the system hydrodynamics. Sampling probes and injection devices that are intrusive to the system disrupt the internal (solid and fluid) flow patterns and hence will have some (usually immeasurable) effect on the RTD. This criticism is common to the majority of particle RTD methods used in all types of systems (slow and fast

response), but it is particularly detrimental in short time scale systems where the relative error associated with any disturbance is increased.

Talmor and Benenati [6] performed mixing experiments on a fluidised bed using an oven dried, strongly acidic, cation exchange resin, in the hydrogen and sodium forms. The sodium form was used as a tracer in a bed of the hydrogen form. Both forms were reported to exhibit the same density and particle size distribution. Bed samples were chemically analysed to determine concentration. The bed solids were elutriuated to remove any remaining tracer before each experimental run, suggesting the tracer and bed material were of a different size and/or density. The method was reported as being time consuming and labour intensive, in addition to being disruptive of the flow conditions inside the bed.

Bai et al. [7] used bed material soaked with an organic substance as a solid tracer for RTD experiments in a CFB riser. Tracer particles were treated in a bubbling fluidised bed until the desorption of the organic substance from the particles was nearly constant. This tracer was then injected into the riser using a pulse of air. Gas samples were taken at the riser exit and analysed by chromatography to determine the tracer concentration (i.e. the concentration of solids was inferred from the gas phase measurements). The bed material was a silica gel with a mean diameter of 99.6 μm and density of 710 kg/m^3. There is considerable uncertainty in inferring solid tracer information from gas phase measurements for the following two reasons:

(i) gas and solids may travel significantly different paths through the bed, and have quite different mean residence times and RTDs;
(ii) it is possible that desorbed gas may be re-adsorbed by the tracer particles as they travel through the bed, giving an ambiguous measure of the residence time.

2.1.2. Radioisotope tracers

Short half-life radioisotope and low-ion radiation tracer methods have been used extensively for fluidised bed and CFB studies. The application of these methods has given the most comprehensive understanding of CFB RTDs to date. They provide a fast time response and are able to capture key features of the RTD. Their main drawback however is significant. The potential health risks associated with radioactive tracers can be considerable and the subsequent requirement to find and dispose of the tracer particles at the end of an experiment can severely limit their applicability.

Patience et al. [8] completed a series of RTD experiments in a CFB unit using radioactive sand as the tracer particle. Si^{28} was irradiated in a fast neutron flux reactor to produce Al^{28} which has a half-life of 2.24 min. The Al^{28} emitted high energy gamma rays that were detected using a sodium iodide scintillation counter. Ten gram of tracer was injected using a chamber consisting of two ball valves, one isolating the mains air and the other isolating the CFB riser; the chamber contained a photodiode which activated the recording system at the start of an experiment. The original pulse air pressure used for experiments (790 kPa) caused serious perturbations in the operation of the unit and so a much lower pressure was used (40 kPa). At this lower pressure, the delta pulse assumption was not valid and a correction for the injection time was required.

Ambler et al. [9] also conducted a series of solids RTD experiments using a radioactive pulse tracer technique. The tracer, Ga^{68}, was input via a pneumatic injector. Ga^{68} has a half-life of 68 min, a stable daughter isotope (Zn^{68}) and is easily doped onto the bed material by evaporating a milking solution after mixing the solution with the tracer solids. Scintillation detectors were used to detect the tracer as it passed by the exit. Unlike Patience et al. [8], Ambler et al. were able to discern a bimodal RTD in their experiment, although this might have been due to measurement of still active tracer in the solids recycled around the CFB loop. This hypothesis cannot be checked because no information was given about the solids inventory in the loop.

May [10] performed a series of experiments using radioactive solid tracers to determine the emulsion mixing coefficient (i.e. diffusion coefficient) in a large scale bubbling fluidised bed. Tagged solids were injected steadily near the top of the bed and the appearance of radioactivity was monitored at various locations below the injection point using scintillation counters. Tests continued until an equilibrium concentration value was reached. Radioactive I^{132} was used to tag FCC catalyst particles by an unspecified method. The isotope has a half-life of 2.4 h.

Hull and Von Rosenberg [11] also used radiochemical tracing of solids to study the mixing patterns in fluidised bed units. They conducted experiments in a pilot scale FCC unit, using Zr-Nb^{95} adsorbed on catalyst as the tracer material. The tracer particles were prepared by tagging FCC catalyst using a radioactive solution provided by an external supplier (no further details presented). The specific activity of the tagged catalyst was 1–2 μCi/g. Approximately 1–2 g of tracer was used for each test, in a total solids inventory of approximately 90 kg. Zr-Nb^{95} has a 65-day half-life.

Lin et al. [43] used tracer particles containing radioactive Mn^{56} to investigate the hydrodynamics of a commercial scale CFB boiler. The tracers had a half-life of about 2.6 h and were manufactured to give a similar size and bulk density to that of the bed material. This study highlighted a further advantage of radioactive tracers; applicability to measurements in large scale equipment. The authors focussed their measurements on the standpipe and loop seal region of the CFB and were able to measure the concentration of tracer with time, as it passed by a series of sodium iodide detectors. Tracer was added to the bed in batches using a high pressure pulse of air.

In all of the radioactive methods reported, disturbances to the bed hydrodynamics were caused by the tracer injection (i.e. air pulse) and occasionally by the bed sampling techniques. In addition, contamination of the bed material by the tracer often required long lag times between experiments for

the tracer to decompose (or for the solids inventory to be replaced).

2.1.3. Magnetic tracers

Avidan [12] and Avidan and Yerushalmi [13] investigated solids mixing of group A powders with a ferromagnetic tracer in an expanded top fluidised bed (i.e. a fluidised bed with an expanded disengagement section). The tracer detection system was based on a very sensitive bridge circuit developed by the Fitzgerald et al. [14]. Avidan [12] also reports ferromagnetic tracers used by other workers (Razumov [15] and Cranfield [16]).

The method of Avidan and Yerushalmi [12] utilised an injection port in the centre of the bed and two inductance probes located up to 1.5 m above and below the injector. Tracer was typically injected within 0.5–1.0 s using an injection air volume flow rate that was typically 1.5–6.0% of the fluidising air rate. The authors did not observe any change in local pressure drop at the time of injection. A 0.15 kg of tracer (0.5% of bed inventory) was used in each pulse experiment.

Tracer was prepared from finely crushed (<37 mm) manganese–zinc transformer parts. The ferromagnetic powder was incorporated into polyurethane foam to match the bulk density of the bed catalyst, then crushed and sieved to give the same particle size distribution. The authors observed that the tracer particles were free flowing like the bed material and did not segregate at any time; even upon defluidisation.

An assessment of the detection system showed that the force acting on the particles from the detection system was negligible and that the lower detection limit of the probes was 5 g of tracer. The detection system gave a signal proportional to the amount of tracer within the detection volume. The authors also noted that the detection probes gave more weight to tracer particles travelling near the wall. This error coupled with the high value for the lower detection limit of tracer, introduce uncertainty into the RTD measurement and reduce the effective resolution of the method.

Sutherland [17] outlined a study of solids mixing in a fluidised bed of copper shot. The technique employed the use of nickel as the tracer material, with separation of the tracer after sampling using a roll magnetic separator. He also described two further methods, the first utilising coloured glass ballotini in a bed of the same material and the second using a single irradiated nickel particle in a recirculating bed of copper shot. The three methods discussed by Sutherland are not suitable for short time scale RTD measurements because the time constant for each technique is typically greater than the mean residence time for a fast response system.

2.1.4. Subliming tracer particles

Bellgardt and Werther [18] developed a novel experimental technique for studying particle mixing in gas–solid systems using subliming tracer particles. Their technique involved the use of carbon dioxide ‘pellets’ as the tracer material in a bed of quartz sand. The pellets were cylinders of solid CO_2, with a uniform diameter of 10 mm and a mean length of 10 mm.

At the sublimation temperature of carbon dioxide (194 K at 1 bar), it is possible to detect the solid tracer using two different methods. The first involves the use of local temperature measurement in the bed after injection of a pulse of tracer or during continuous tracer feeding. The second is by measuring the concentration of the gaseous sublimation product. Both methods give a measure of the tracer concentration and hence can be used to determine the bed residence times. Tracer was fed to the bed using a screw feeder for continuous addition or for batch addition via a box containing the pellets emptied at some location within the bed. This method, whilst novel, has some serious limitations for measuring the particle RTD. These include:

(i) tracer injection is non-ideal, i.e. not a delta pulse and this was not considered in calculating the RTD;
(ii) the motion of gas and solids in a fluidised bed are significantly different and hence the inferred solids RTD contains uncertainties, similar to those arising from the method of Bai et al. [7];
(iii) the tracer particles are significantly different from the bed material;
(iv) the rate at which the CO_2 pellets sublime was not included in the analysis.

2.1.5. Particle sampling

Bai et al. [19] studied the hydrodynamic behaviour of a binary solids CFB system using a non-isokinetic sampling technique. Samples were drawn from taps at the riser wall. Separate experiments determined the sampling error to lie within ±20%. They described the isokinetic technique as being “tedious” and subsequently developed a non-isokinetic method.

Chesonis et al. [20] measured solids mixing rates using a petroleum coke tracer in a CFB of alumina solids. A 2.4 kg charge of coke was injected into the system which had a total solids inventory of between 19 and 22 kg. Sampling was conducted at five locations along the riser using a single sampling probe, facing downwards, at the centre of the riser. In order to move the probe to sample from different ports, the fluidisation air had to be shut off and the bed allowed to slump. It was then restarted when the probe had been moved. Subsequent experiments were performed using the same solids inventory without recharging with uncontaminated alumina. Results were presented as a plot of local solids composition (wt.% carbon) versus time. This method does not produce RTD curves but rather the time for equilibrium mixing to occur. The key disadvantages are the accumulation of tracer solids in the bed and the requirement to allow the bed to defluidise to move the sampling probe. The errors introduced by these problems are not quantifiable but are believed to be significant.

Hirschberg et al. [21] conducted a series of experiments using a binary mixture of quartz sand and iron ore to

determine mixing parameters in a large, pilot scale CFB unit in a similar manner to Chesonis et al. [20]. A 48 kg charge of iron ore particles was used as the tracer in a system with a total solids inventory of approximately 1 tonne. The local solids composition was determined by continuous sampling at a given location within the riser. No information was given on how the samples were analysed. Experimentation concluded when steady state mixing conditions were achieved. The experimental results were presented in an identical manner to that of Chesonis et al. [20], giving similar experimental uncertainties.

The limitations of RTD methods which rely on bed sampling to determine tracer concentration were discussed by Avidan [12]. They are as follows:

(i) the measurement of solid particle velocity is not always direct; and
(ii) the probe disrupts local flow patterns.

It is the second problem which limits the technique to dilute pneumatic conveying systems only and prohibits it's use for fast fluidisation conditions such as exist in a CFB riser.

2.1.6. Particle sizing techniques

Wang et al. [22] completed a series of riser residence time experiments in a 200 mm square cross section riser, using a pulse tracer input which consisted of particles of a narrow size interval separated from the main solids inventory. Experiments were performed using tracer in the size ranges 0.4–0.6, 0.6–0.8 and 0.8–1.0 mm. The bed solids had a mean particle size of 471 mm. Sampling was performed with an adjustable, stepwise, motor driven sample collector located at the cyclone underflow. The minimum motor time step was 0.1 s. Samples were collected into sample cups located on a plastic plate and then sieved to determine tracer concentration. The overall solids inventory was kept constant during the experiments.

2.1.7. Coloured tracer techniques

Lim et al. [23] very briefly summarised a number of techniques suitable for both the local investigation of the motion of individual particles and the gross behaviour of solids mixing using tracer techniques. These included radioactive, coloured, magnetic, chemically different, subliming and heating methods. The main limitations were; the first four are only suitable for single short transient experiments, after which the bed must be sectioned for analysis (or in the case of radioactive tracers, allowed to decay for sufficient time). Subliming or heated tracer techniques require many detailed assumptions to complete the analysis of an experiment. The authors' choice for a tracer system however was to develop an image analysis method utilising a CCD video camera and computer recording system. They argued that the limitations of the above techniques required a new approach.

They performed mixing experiments in a two-dimensional bubbling fluidised bed using a prussian blue dyed glass ballotini tracer. Analysis of the data was by developing a calibrated relationship between grey scale intensity and dyed glass concentration, which they are determined to be unique and monotonic.

The main limitation of the technique however is that image analysis systems are not readily able to penetrate into dense particulate systems, such as a three-dimensional fluidised bed and hence the method is really only suitable for measurements close to the walls or in specially built two-dimensional units with lighting to highlight any mixing effects. This limits applicability.

Bi et al. [24] performed a series of experiments to study the lateral mixing of coarse particles in fluidised beds of fine particles. They used orange coloured lignite pellets as tracer particles and time recording equipment to record the residence time of individual particles as they progressed through the bed.

2.2. Non-disruptive techniques

2.2.1. Optical methods

Brewster and Seader [1] developed a tagging method for measuring particle residence times in industrial pneumatic transport systems. The method was specifically developed as an alternative to radioactive tracer methods and was tested in a cocurrent downward flow pneumatic conveying system containing pulverised coal and air. Particles were coated with powdered phosphorescent pigment using glue, illuminated with light over an extended period, and subsequently injected using a high pressure pulse of air. The tracer particles were detected using a series of photomultiplier tubes (PMTs) located downstream. The method was effective in measuring coal particle velocities in systems where the gas velocity ranged from 14 to 41 m/s. The key advantage of the method according to the authors was its cost effectiveness when compared with radioisotope methods. The major disadvantage was the need for a 'see-through' detector window in the pipe which limited the method to use in relatively low pressure systems. The high velocity pulse of air used for tracer injection also introduced uncertainty into the measurement.

Roques et al. [27] used phosphorescent pigment particles to measure the solids RTD in a gas–solid downflow transport reactor. The typical mean residence time in this system ranged from 140 ms to 1.5 s. The technique used phosphorescent pigments circulating through the system acting as both bed material and tracer. Tracer was activated using a high intensity pulse of visible light (of typical duration 4 ms). The concentration of activated tracer was measured using five light detectors positioned along the length of the apparatus. Tracer decay (i.e. the decay of light intensity with time) was fitted to a hyperbolic decay function to correct the measured response curves to the RTD. The detector response was also taken into account when calculating the RTD.

Researchers in the Department of Chemical Engineering at Tsinghua University, Beijing, developed a phosphor tracer technique to measure lateral and axial solids dispersion and solids mixing in a variety of CFB units (Wei et al. [2], Wei

Table 2
Summary of single particle methods for fluidisation mixing studies

Method	Author	System	Details of Method	Geometry
Radioactive tracer	Weinell et al. [30]	CFB riser	Local particle velocities of a variety of single radioactive tracer particles are monitored using scintillation detectors placed along the length of the riser	$D = 0.14\,\text{m} \times 0.18$ m, $H = 3.0\,\text{m}$
Radioactive tracer	Weinell et al. [31]	CFB riser	Single radioactive tracer particle monitored using scintillation detectors	$D = 0.14\,\text{m} \times 0.18\,\text{m}$, $H = 3.0\,\text{m}$
Radioactive tracer	Weinell et al. [32]	CFB riser	Single radioactive tracer particle monitored using scintillation detectors	$D = 0.14\,\text{m} \times 0.18\,\text{m}$, $H = 3.0\,\text{m}$
Radioactive tracer	Stellema et al. [33]	Interconnected fluidised bed	Single radioactive particle measured with a positron emission particle tracking system (PEPT)	$D =$ unknown, $H =$ unknown
Radioactive tracer	Abellon et al. [34]	Interconnected fluidised bed	Single radioactive particle measured with two scintillation detectors.	$D =$ unknown, $H =$ unknown
Radioactive tracer	Godfroy et al. [35]	CFB riser	Single radioactive particle measured with eight scintillation detectors.	$D = 0.082\,\text{m}$, $H = 7.0\,\text{m}$
High speed video	Matsuda et al. [36]	CFB riser	High speed video system recording at 2000 or 3000 frames s^{-1} of particle swarms.	$D = 0.060\,\text{m}$, $H = 4.5\,\text{m}$

and Zhou [25]) similar to that developed by Brewster and Seader [1] and Roques et al. [27]. The technique used a phosphorescent material, typically alumina powder coated with a phosphorescing compound which, when excited by a light source, emits light immediately and continues to emit with decreasing intensity for up to a few minutes. Both point source and planar excitation of the tracers were used. Calibration of the decay process allowed it to be fitted to a power law function which could then be included in the analysis to correlate the tracer concentration with the emissive light strength. The technique was pioneered in a cocurrent downflow CFB, with a typical residence time of less than 1 s. The technique has since been applied in binary solids systems (Wei and Zhou [25]) and CFB risers for both axial and lateral solids mixing studies.

Kojima et al. [26] at the University of Tokyo used fluorescent dye-tagged FCC catalyst particles to examine axial dispersion in CFB units. They used a pulse of tracer particles (1.0–2.0 g) introduced into the riser and detected at various heights using optical fibre probes, located at the central axis of the riser. Ultraviolet light was transmitted into the bed through the optical fibres and the visible light reflected from the tracer particles was transmitted back through parallel fibres to filtered photomultipliers. Kojima et al. [26] believed the technique to be robust to the location of the detection system when using it to determine local particle velocities. They also determined an axial dispersion coefficient from the difference between the second central moments generated from two intensity curves, one for each of two detectors, spaced at 0.10 m. As with Brewster and Seader [1], the high velocity pulse of air used for tracer injection introduced uncertainty into the measurement.

2.2.2. Thermal methods

Thiel and Potter [28] studied the axial mixing of solids in slugging gas-fluidised beds using bed temperature measurements. The bed was simultaneously heated and cooled at different locations until steady state conditions were achieved and then bed temperatures were recorded at different heights and radial positions in the experimental section between the positions of heating and cooling. The cooling water flow rate and inlet and outlet water temperatures were also recorded to allow calculation of the quantity of heat being axially transferred through the bed section. Temperatures were recorded using thermocouples.

Valenzuela and Glicksman [29] also studied solids mixing in a fluidised system using heated particles as a tracer. They studied vertical and horizontal particle mixing in a freely bubbling two-dimensional bed of large glass particles. The steady state temperature patterns around a heated wire and the transient response to an injected pulse of heated particles were recorded with an array of thermocouples. The major drawback associated with thermal methods is the need for complicated assumptions to infer an RTD from experimental measurements.

2.3. Single particle tracers

A number of authors have utilised single particle tracer techniques in CFBs. All of these studies measure the trajectory of a single tracer particle (usually radioactive) around the CFB loop. Other types of tracers used include coloured particles and tracers of made of specific materials. The trajectories of the particles measured over many circuits are used to calculate average particle velocities and residence times. These methods are summarised in Table 2.

3. Experimental

3.1. Method development

The review of experimental methods provides an insight into the advantages and disadvantages of published

techniques used to measure particle RTDs in particulate systems, with short and long time scales. The aim of the review was to identify a safe, accurate and robust method that minimises any experimental bias. Radioactive tracer methods were not considered because of safety concerns. Of the remaining techniques the most appropriate was a modified version of the phosphorescent tracer methods reported in Section 2.2.1. The advantages of this technique, identified by Brewster and Seader [1], Wei et al. [2] and Roques et al. [27] are:

(i) the immediate activation of tracer by a light pulse with no disturbance to the bed hydrodynamics;
(ii) simple online detection of the tracer by a light detector, again with no disturbance to bed hydrodynamics;
(iii) the tracer is identical to the rest of the bed material;
(iv) no extra particles or gas are added to disturb the flow;
(v) there is no tracer accumulation in the bed; the bed particles become inactive after a sufficient period;
(vi) comparatively low cost compared with radioactive tracer studies.

The method reported here improves upon those described in Section 2.2.1 by removing their major limitations, namely:

(i) the need to combine individual RTD curves measured at different lateral positions to generate the "overall" RTD; and
(ii) ill defined boundary conditions at the inlet and outlet which give uncertainty about whether the experiment gives the true RTD curves.

It is the second of these that is the most critical limitation of methods using phosphorescent or fluorescent tracer particles to measure particle RTDs. In previous studies, the experimental methods failed to characterise adequately the experimental boundary conditions and consequently the measured concentration–time curves are not the true RTD but rather some other distribution of particle travel times. This error applies to the majority of published results regardless of the method used to measure the RTD. When measurements are made under open boundary conditions (as in the case of a straight section of riser), the concentration–time curve produced is of a transient response function. The values of the mean and variance of this distribution are only equal to the true RTD under closed boundary conditions (Nauman and Buffham [37], White [44], Nauman [45]). This is because particles which enter the system through an open boundary are then able to pass back through the same boundary. The period of time spent outside the system contributes to the total retention time but does not contribute to the residence time. There is no way of distinguishing the time spent outside the system from that spent inside, and hence distinguish the two (Nauman [45]).

Experimental studies of RTDs in CFBs have sampled from different locations along the riser axis (for a review see Harris et al. [38]), generally corresponding to the different postulated mixing zones within a riser (i.e. bottom zone, transition zone, core/annulus dilute phase, exit zone). Because the boundaries separating these region are open, the measurements have not yielded a true RTD. The studies can give useful information regarding local solids mixing within each of the hydrodynamic regions of the riser, but are of uncertain value when trying to determine the RTD.

3.2. Measuring the RTD

In the method described in this paper, particle RTDs are measured using phosphorescent tracer particles activated by a high intensity light pulse at a specific position at the base of the riser. This region is well mixed supplied by a feed which is closed (i.e. there is no back-flow into the feeder). When the tracer particles reach the riser exit, they pass through an inline jet mixing unit; at the downstream end of this unit, the tracer is detected by a single light sensitive PMT. The inline mixing unit is designed such that flow is very likely to be in one direction only (see Fig. 1). The method was developed in an academic scale CFB, 5.8 m in height, with a square cross section riser (0.14 m × 0.14 m). All the measurements were made at ambient temperature and pressure with air as the fluidising medium. Tracer particles and bed solids were identical. The method is based on the work of Brewster and Seader [1], Wei et al. [2] and Roques et al. [27]. The major limitations of these previous phosphorescent tracer methods are overcome by using novel tracer injection and detection modules, as described earlier.

A schematic of the RTD method is presented in Fig. 1. It has four key components.

(i) Tracer particles.
(ii) Tracer injection to the riser.
(iii) Tracer detection in the riser.
(iv) Data acquisition system.

Each of these is discussed in turn.

3.2.1. Tracer particles

A commercial phosphorescent pigment (Honeywell "Lumilux Effect Green-N") was used in all the experiments as both bed material and tracer. The particles emit radiation following exposure to light (as opposed to fluorescent particles which only emit whilst being irradiated). The detectable emission can last up to several minutes following a single microsecond pulse of energy. The properties of the particles used are presented in Table 3. The particle size distribution and the typical phosphorescent emission spectrum are presented in Figs. 2 and 3. The particle size distribution was measured using a Malvern laser sizer. The particle fluidisation characteristics, presented as a plot of bed pressure drop versus superficial air fluidising velocity are illustrated in Fig. 4. This curve was generated from measurements made in a 0.15 m diameter bubbling fluidised bed. The particles appear to fluidise without cohesion effects despite

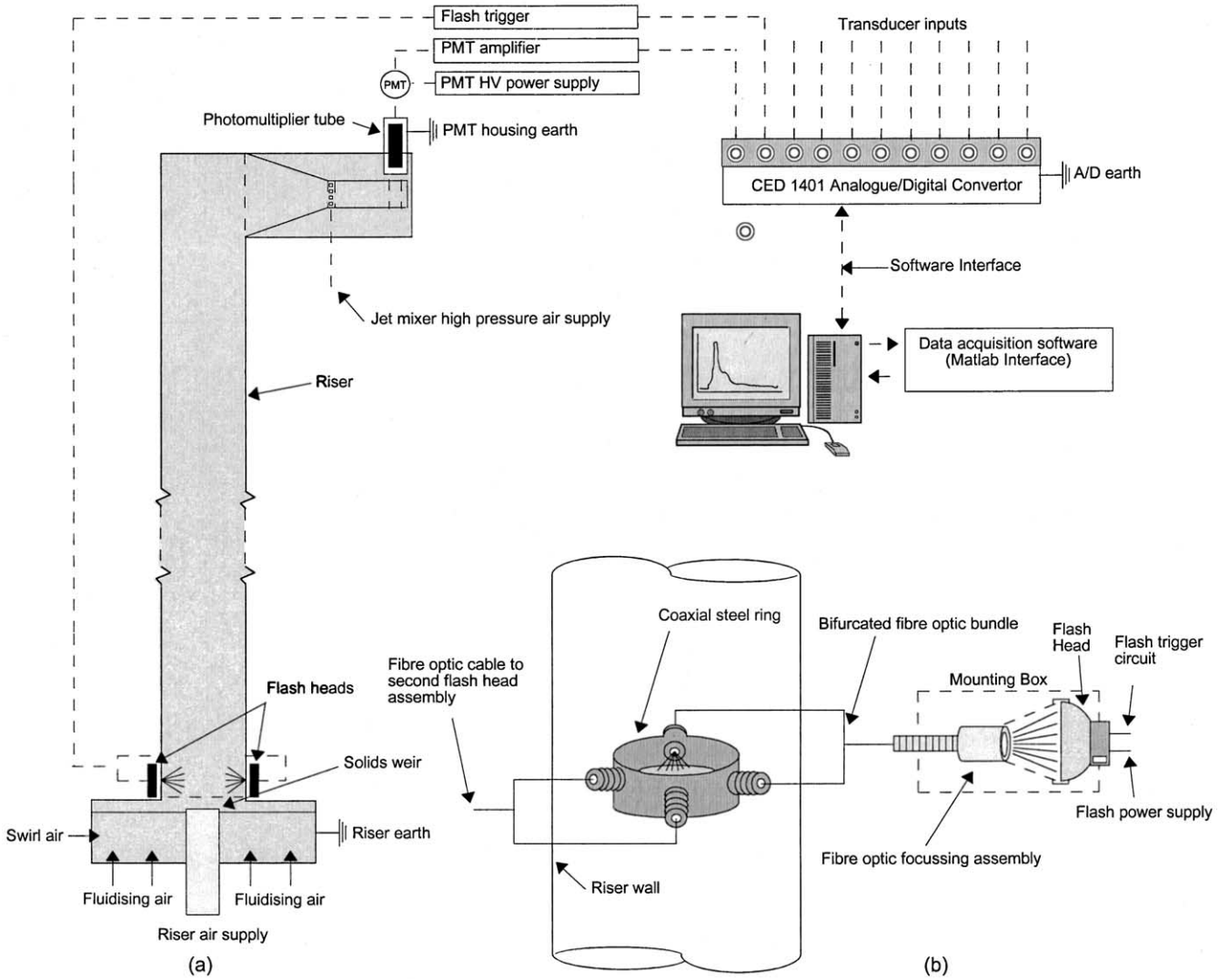

Fig. 1. Schematic presentation of the RTD method showing the two alternative method of (a) planar tracer activation and (b) point source tracer injection.

their Geldart classification. The decay characteristics of the tracer particles (i.e. decrease in emission strength with time) following a single microsecond light pulse are presented in Fig. 5.

Table 3
Tracer and bed particle properties

Property	Value
Sauter mean particle size (d_{32})	25 μm
Particle composition	Cu:ZnS
Bed inventory	200 kg
Particle density (reported by manufacturer)	4060 kg/m^3
Bulk particle density (reported by manufacturer)	1800 kg/m^3
U_t (calculated for a single particle)	0.071 m/s
U_{mf} (measured)	0.013 m/s
Geldart classification	AC Boundary
Colour	Yellow-green
Emission colour	Green

3.2.2. Tracer activation

Tracers are activated at the base of the riser just above the solids feed. The feeder consists of an annular bubbling fluidised bed that distributes the incoming solids and spills them over a weir into the upflowing riser air supply. This activation point represents a closed boundary (Fig. 6).

Because the tracer material is identical to the remainder of the bed solids, no actual physical pulse (of gas or solids) is required, avoiding any hydrodynamic flow disturbances. The tracer activation and flash setup are illustrated in Fig. 1. Two types of tracer activation were developed; planar (Fig. 1a) and point source (Fig. 1b). Both methods use a dual head commercial photographer's flash unit attached to the riser: the unit produces a horizontal collimated beam. Point source activation utilises wide bore fibre optic cables attached to an open ended, 30 mm diameter circular chamber located in the centre of the riser, just above the riser feeder, see Fig. 1b. The particles situated within the chamber at the time of the flash

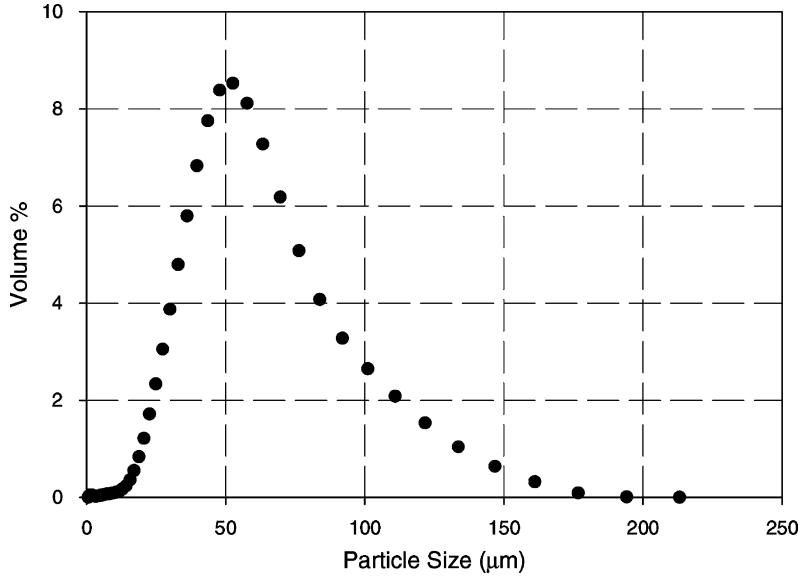

Fig. 2. Typical particle size distribution (Malvern laser size).

are activated and begin to glow. The volume of the chamber is $7.9 \times 10^{-5}\,m^3$ with the quantity of activated solids (i.e. tracer) dependent upon the local solids concentration (inferred from pressure measurements made with pressure transducers located in the measurement plane). Planar tracer activation uses opposing transparent square slits in the riser wall to allow light to reach the particles in the riser, Fig. 1a. It is assumed that all particles in the flash plane are activated. Tests using a light meter positioned to measure the transmission of light through the solids suspension in the riser at typical operating conditions show this is a valid assumption. The slits are located on the same plane as the point source injection ring (which is removed when not being used). The properties of the flash unit are presented in Table 4. The typical time duration of the flash means a delta pulse assumption is valid for the input boundary.

Table 4
Tracer activation properties

Property	Value
Flash output energy (reported by manufacturer)	125 J
Flash duration (reported by manufacturer)	2 μs (typical)
Flash intensity (calculated)	42.5×10^6 lumens

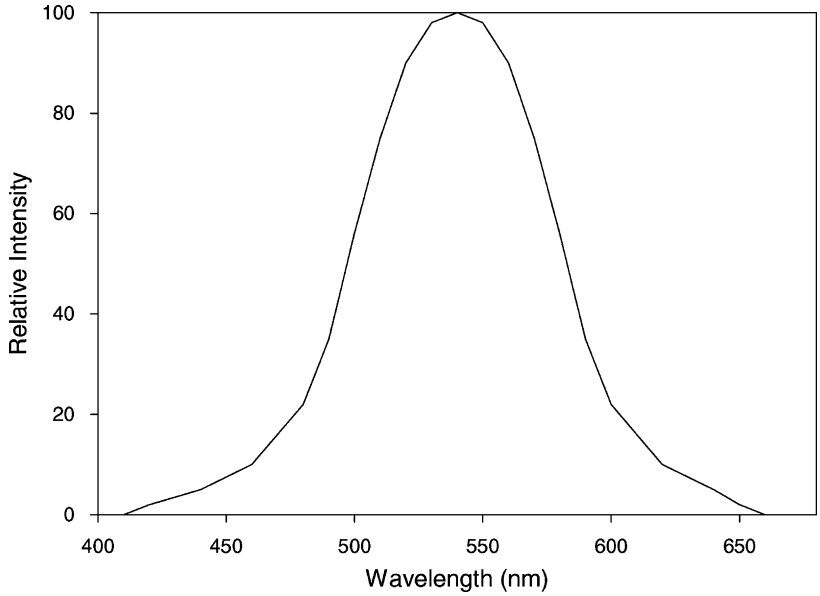

Fig. 3. Typical particle phosphorescence emission spectrum.

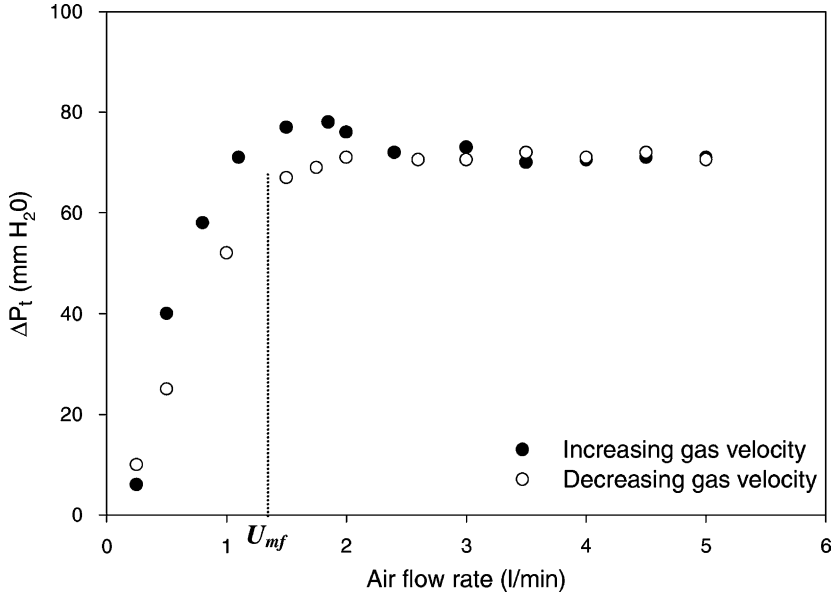

Fig. 4. Particle fluidisation characteristics, measured in a 0.15 m bubbling fluidised bed with high pressure drop porous distributor.

3.2.3. Tracer detection

In order to ensure the boundary condition is properly characterised at the exit measurement plane (i.e. a closed boundary exists), a novel inline jet mixer unit (Fig. 7) was designed and installed downstream of the riser exit bend. The purpose of this unit was four-fold:

(i) to provide a well defined measurement zone for RTD measurements;
(ii) to provide a fully mixed sample at the measurement plane;
(iii) to ensure steady flow of solids at constant velocity past the measurement plane;
(iv) to ensure no deposition of tracer at the measurement plane.

The inline mixer consists of a constricting entrance, slot and jet mixing nozzles to lift and mix particles near the floor and wall of the unit, followed by a flow developing region. The ratio of jet mixer air to riser air is about 1%. At the exit from the mixer, a PMT (Electron Tubes model 9124B) is incorporated to detect light emissions from the tracer particles

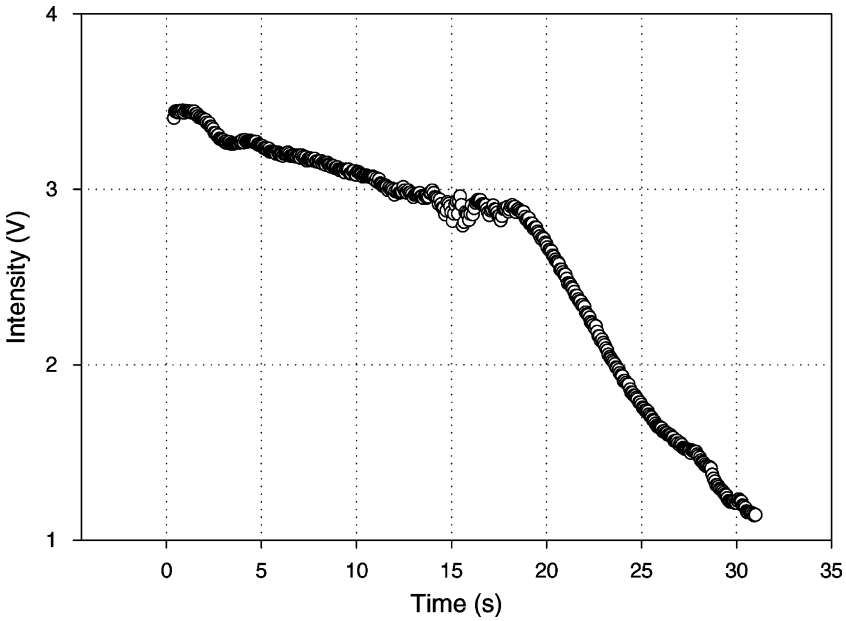

Fig. 5. Typical particle emission decay characteristics (PMT feed voltage = 750 V, signal amplification × 10).

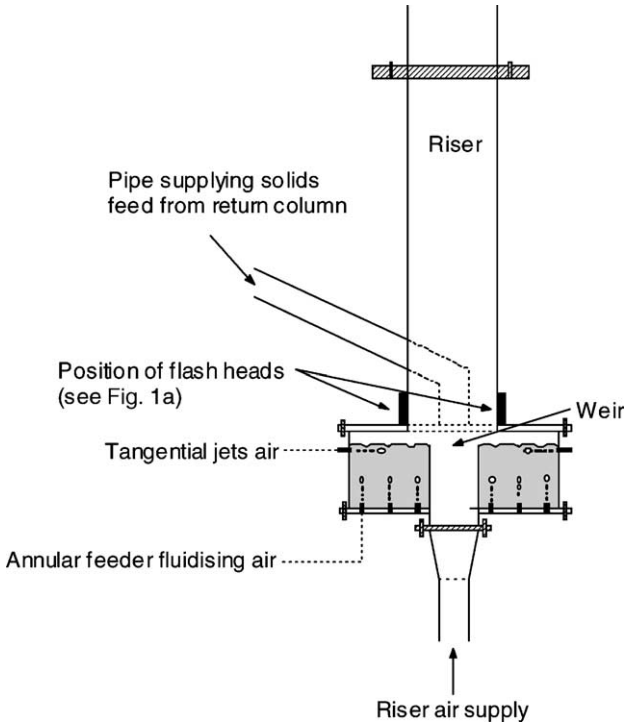

Fig. 6. Solids feeder annular fluidised bed and lower riser section (circular cross section).

that are travelling in steady flow past the measurement plane. The measurement of light emissions replaces the need to take many samples in some other techniques. At the point of measurement, particles are estimated to be travelling in excess of 18 m/s. The photomultiplier exhibits peak sensitivity at 550 nm and was selected to maximise detection of green wavelength light corresponding with the emission spectrum in Fig. 3. Details of the inline mixing unit are presented in Fig. 7. Observation of the solids flow within the mixing unit confirms:

(i) the riser exit boundary is closed, i.e. there is no reverse flow;
(ii) solids flow past the measurement plane is steady;
(iii) no solids accumulate in the measurement section; and
(iv) a substantial dune is formed at the entrance to the mixer unit.

The presence of a solids dune at the riser exit was described by van der Meer [39] who suggested that it contributed to riser back mixing and the 'exit effect' described by many workers (Harris et al. [40]). These observations are typical for the inline mixing unit over the range of riser superficial gas velocities and solids fluxes used in the experiments.

3.2.4. Data acquisition system

The PMT is connected via a transimpedance amplifier to the data acquisition system. Signals are typically amplified by a factor of 10 before being processed by the data acquisition software. The glass measurement window of the PMT is 30 mm across and is protected by a 10 mm section of Perspex. At the emission wavelengths of the tracer particles, Perspex is completely transparent and very limited absorption of the tracer emissions occurs. The PMT is powered by a high voltage unit supplying up to 2000 V. The maximum operating voltage of the tube is 1673 V and the typical operating voltage is 750 V. The light sensitivity of the PMT may be increased by increasing the operating voltage of the tube (to the detriment of its operating lifetime). PMTs produce random signals with zero input (dark counts). A 5-point median signal filter was used to delete this background noise from the RTD measurements.

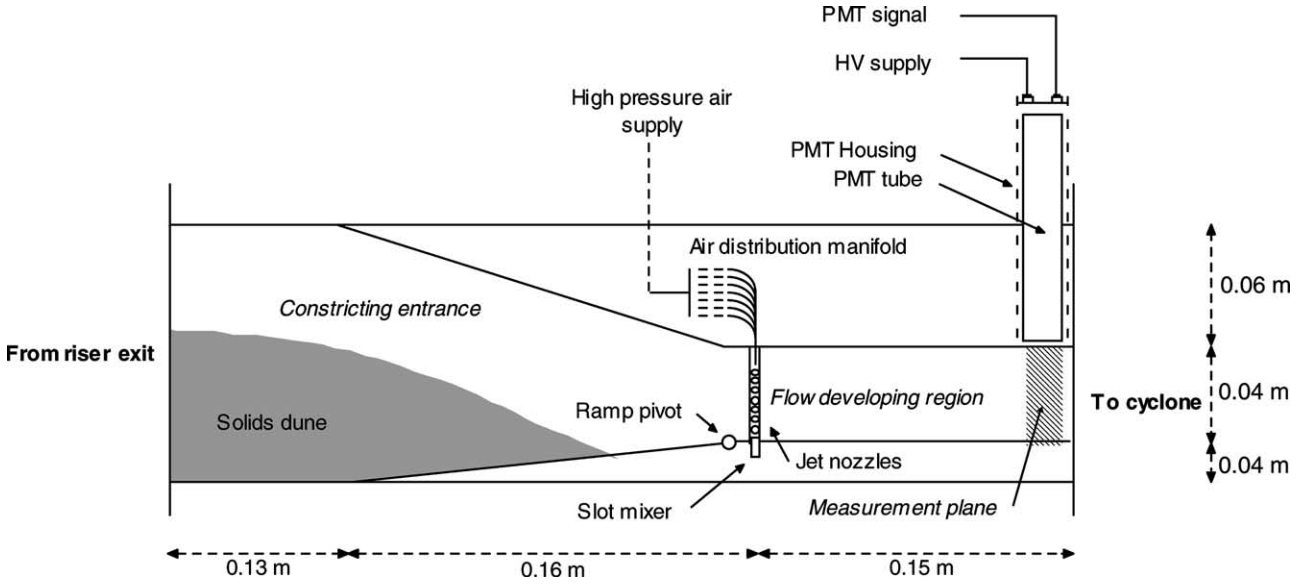

Fig. 7. Schematic presentation of the inline jet mixer unit positioned downstream of the riser exit (not to scale). The photomultiplier measures light emissions from the phosphorescent particles in the particle exit stream.

Because the strength of the emitted light decays with time, the concentration–time curves require correction. The correction is made using data from the decay calibration curve (Fig. 5). The measured signal is increased by the ratio [ordinate (Fig. 5) at $t = 0$]/[ordinate (Fig. 5) at time t]; this correction is made at intervals of 10 ms, giving the RTD curve. Over the length of an experiment (typically 20 s), the correction produces little effect on the measured curves.

Data from the measurement transducers (PMT signal voltage, pressure, temperature and mass flow) are input to a PC for analysis via a 32 channel analogue to digital converter (Cambridge Electronic Design model 1401). The interface between the data acquisition unit and PC is via a series of 32 bit Matlab® routines called MatCED (Ringach [41] and Coleman [42]). Separate signal filtering and data analysis software is used to generate RTD curves and pressure profiles, and determine superficial gas velocities and solids fluxes once the data are transferred to the PC. This data analysis software was custom written using the Matlab® software. All data acquisition hardware required shielding from static electric charge build-ups.

4. Results and discussion

An example of a typical RTD signal measured using the method reported here, is presented in Fig. 8a. Measured curves were corrected for the decaying light emission of the

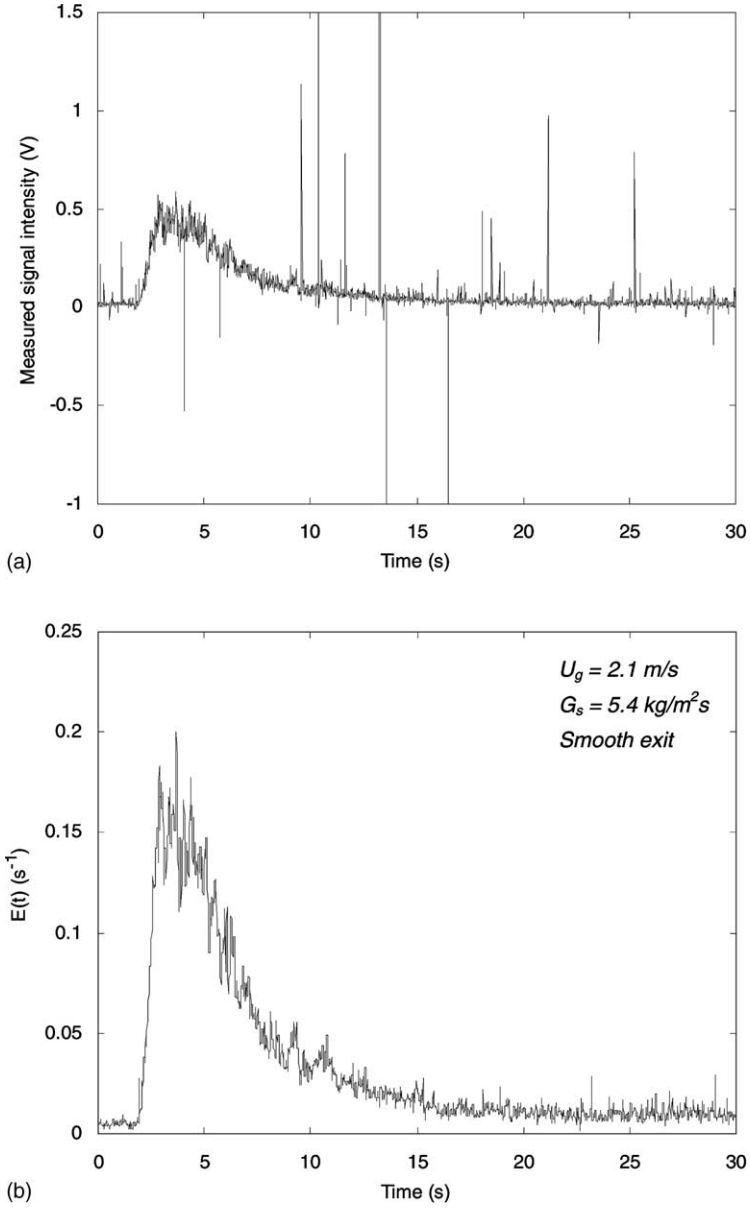

Fig. 8. Typical RTD curves deduced from the optically tagged tracer particles: (a) raw residence time data; (b) RTD filtered, normalised and corrected for tracer emission decay.

tracers and filtered using a 5-point median filter to remove outlying data points resulting from background noise (dark counts) from the photomultiplier. Fig. 8b illustrates a filtered and decay-corrected version of the data presented in Fig. 8a; the curve in Fig. 8b was normalised to give $\int_0^\infty E\,\mathrm{d}t = 1$. The method is highly sensitive to the presence of tracer particles and therefore detects low concentrations; also, it is able to sample at sufficiently high data acquisition rates to ensure all points of interest in the RTD are accurately captured.

4.1. Applicability of the method to other fast response systems

It is clear from the curve in Fig. 8b that the method presents a significant amount of detail regarding the solid suspension. The curve exhibits local irregularities that stem from the stochastic nature of gas–solid flow in a riser. This detail cannot be captured by a slow response technique (e.g. NaCl tracer and probe sampling system or single particle studies).

Thus, the phosphorescent method is applicable in any system where a fast response is required and where the experimental boundary conditions can be adequately characterised. An example is the work of Roques et al. [27], described earlier, who measured the particle RTD in a downer reactor using a phosphorescent pigment. The nature of their reactor configuration led to the experimental boundaries characterising the system being closed. Hence, the concentration time curve measured by Roques et al. [27] is a true RTD, rather than a transient response function described in Section 3.1.

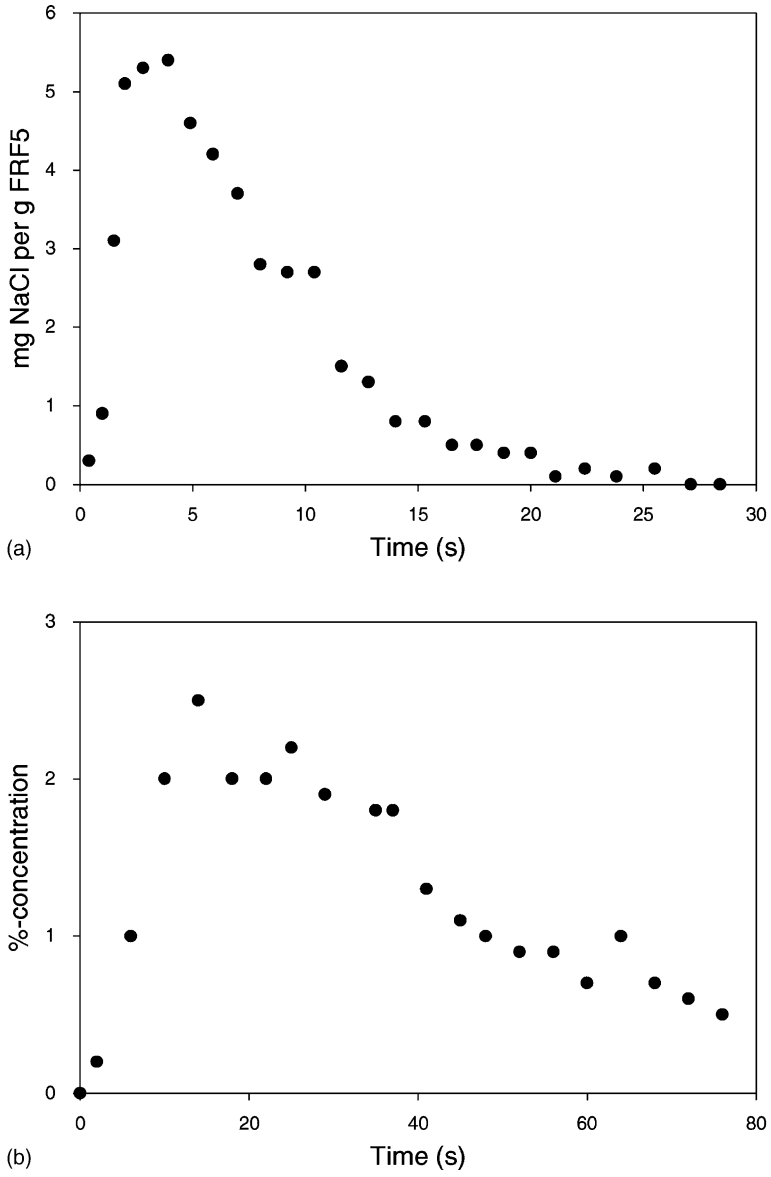

Fig. 9. Published RTD data using as tracer practice (a) NaCl particles ($U_g = 2.8$ m/s, $G_s = 30$ kg/m^2 s, $D = 0.15$ m, $H = 6.2$ m) and (b) size fraction separated from the solids inventory ($U_g = 3$ m/s, $G_s = 3.25$ kg/m^2 s, $D = 0.2$ m, $H = 4$ m).

Typical results from slow response techniques are illustrated in Fig. 9. The results presented are of two experiments at similar conditions in CFB risers using (i) a NaCl solid tracer technique (Rhodes et al. [3]) and (ii) tracers of a size fraction separated from the bed material (Wang et al. [22]). These results can be compared with the phosphorescent tracer result presented in Fig. 8b. The experimental conditions are not identical but are sufficiently similar for comparison. The phosphorescent tracer method captures significant detail about the curve which is not present with the slow response techniques illustrated in Fig. 9. The general form of the RTD is clear but the micro-scale detail is not. This micro-scale detail is important in fast response systems predominantly because it provides a clearer picture of the flow pattern in the system (indicating regions of backmixing, bypassing or recycling) and eliminates ambiguity in assessment of the RTD by providing sufficient detail. In CFB risers the micro-scale detail may include second or consecutive peaks in the distribution, detail about the length of the tail of the distribution, and local irregularities stemming from the formation of particle clusters in the riser. This information is important for CFB designers, modellers and operators. For example, Harris et al. [46] were able to develop a new model for the particle RTD in a CFB riser by interpreting the micro-scale detail in experimental RTD curves as a stochastic component. Slow response measurements do not give sufficient detail to observe this phenomenon.

In addition to the primary aim of characterising the RTD in short time scale systems, the high level of detail detected by this new method can also be used as follows:

(i) To give a clear characterisation of the tail of the RTD. For some RTD measurements, the exact length of the tail is uncertain, leading to substantial errors (in the region of 50%) in calculating the moments of the distribution, see White [44] and Nauman [45].
(ii) To give clear characterisation of the peak of the RTD; both the time of first appearance of tracer and the maximum height of the peak (or peaks). This type of detail can be lost in slow response methods.
(iii) Cross correlation of pressure signals and fluctuations in the RTD from a CFB riser using time series analysis, can illustrate common operating frequencies useful in characterising system operation (see e.g. Johnsson et al. [47]).

It is also possible to use different types of particles for the RTD experiments; commercial phosphorescent pigments are available in a range of particle sizes. Depending upon the bed solids required, it is possible to coat the outside of other particles using the pigment and a binder or glue. In this way, any particles that can be coated may be used for fast response RTD experiments. Wei et al. [3] coated 1 mm glass ballotini for RTD studies using this technique.

4.2. Reproducibility and robustness of the method

The method enables very rapid measurement of RTD curves (a typical experiment can be repeated within about 60 s with sufficient solid inventory in the system) and

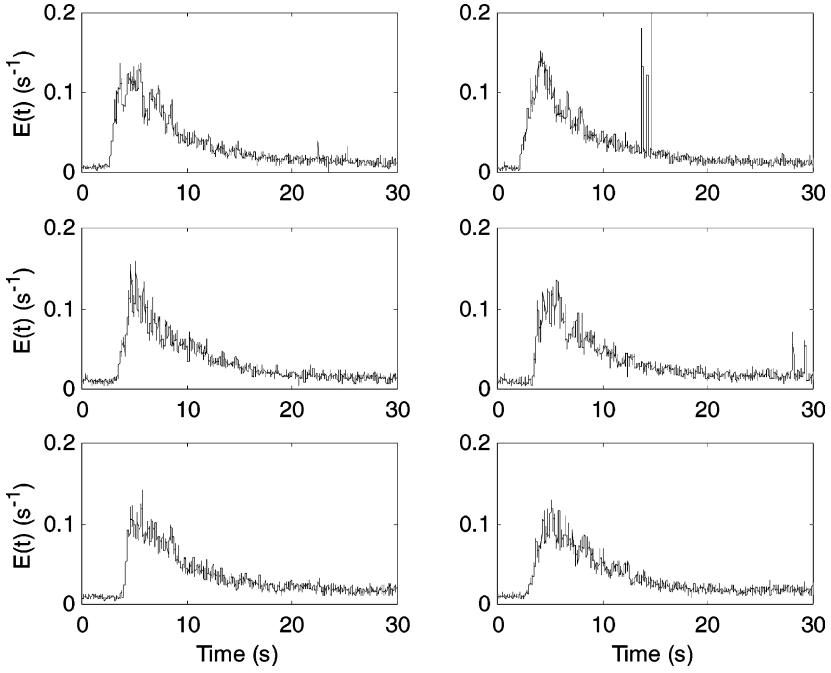

Fig. 10. Reproducibility of the RTD measurement. The six runs were performed at intervals of 60 s at the same operating conditions ($G_s = 8.6\,kg/m^2\,s$, $U_g = 1.8\,m/s$).

Table 5
Measures of the RTD for the curves presented above

Run	Mean, μ (s)	Variance, σ^2 (s^2)	Skewness, s^3
1	10.61	7.07	19.85
2	10.45	7.15	19.86
3	11.42	7.06	17.39
4	11.84	7.55	17.50
5	12.19	7.32	15.69
6	11.72	7.47	17.54

multiple repeats are possible to check reproducibility. A somewhat longer interval between experiments is needed to allow the emission from activated particles to decay to zero, see Fig. 5. Examples of RTDs measured at intervals of 60 s at nominally the same operating conditions in a CFB riser are presented in Fig. 10, together with the key statistical parameters describing the curves, namely the mean, variance and skewness of the distributions determined from the moments (Table 5). Differences between the individual measurements are probably due to local flow irregularities in the riser, hence the importance of a stimulus response with a sufficiently fast time constant.

The method presented here is cost effective and robust to operating conditions. The range of experimental conditions studied included dilute and dense phase particulate regimes, up to a solids concentration of about 45 vol.%.

4.3. Limitations of the method

In systems where the residence time experiment extends past 3 min, uncertainties due to the correction for tracer decay (Fig. 5) become significant. This may be compensated for by increasing the detection sensitivity e.g. (i) increasing the number of photomultipliers at the detection plane or (ii) using a more phosphorescent (and consequently more expensive) tracer. In addition, in systems where the solid concentration is above 45%, the assumption that all particles in the flash injection plane are activated is likely to be invalid because of the shielding effect.

It is also important that the experimental conditions defining the entrance and exit boundaries are adequately described. For the CFB riser reported here, this was achieved by using novel modules at the entrance and exit of the riser. In other systems similar injection and detection modules may be required to ensure closed boundaries so that the true RTD is measured.

5. Conclusions

A novel experimental technique is reported for the non-intrusive measurement of particle RTDs in fast response systems using phosphorescent tracer particles activated by a high intensity pulse of light and detected by a light sensitive photomultiplier. The method improves upon techniques reported in the literature by providing accurate characterisation of the experimental boundary conditions. Well defined boundary conditions are shown to be critical in measuring a well defined, unbiased RTD.

The method has been shown to be suitable in systems where the mean residence time is small (i.e. microseconds to seconds). The upper residence time limit of the method is approximately 3 min because of emission decay of the activated particles. The method is limited to systems with solids concentrations less than about 45 vol.%.

The technique was been developed for the vertical riser of a CFB, but is applicable in other solid–fluid systems requiring a fast response measurement. The advantages of the phosphorescent particle RTD method over other particle RTD methods are as follows:

(i) the immediate “injection” of tracer by a light pulse with no disturbance to the bed hydrodynamics;
(ii) simple online detection of the tracer by a light detector with no disturbance to the bed hydrodynamics;
(iii) the tracer is identical to the rest of the bed material;
(iv) no extra particles or gas are added to disturb the flow;
(v) there is no tracer accumulation in the bed;
(vi) comparatively low cost compared with radioactive tracer studies.

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