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# Ultrasonic velocimetry for the *in situ* characterisation of particulate settling and sedimentation

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# ABSTRACT

This paper reports on the development of an *in situ* ultrasonic velocimetry technique, to study the settling and sedimentation behaviour of particle dispersions. Specifically, the technique utilises a commercial ultrasonic velocity profiler (UVP) equipped with a 1 MHz transducer–receiver, to measure both particle velocities in the dispersion and the evolution of the sediment bed interface with time. It was found in systems of bi-modal non-coagulated glass particles (with a major size-peak of ~10  $\mu$ m) that measured velocities suggested dispersion segregation, although generally values were not reliable as particle settling velocities were below the instrument's threshold. For particle systems coagulated in 1 M KCl, measured dispersion velocities were within the machine's resolution and a high level of system detail could be extracted from the velocity profile maps, such as the development of hindered settling above the bed and movement of the cloud-front. For both coagulated and non-coagulated dispersions, the evolution of the sediment bed height with time could be measured, by analysing particle velocities in the near-bed region. Bed profiles indicated the non-coagulated particles settled slowly into a compact bed, while the coagulated particle-aggregates initially settled faster into a loosely packed bed that compressed over-time.

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## 1. Introduction

The measurement of particle settling and sedimentation in multiphase dispersions is important to many engineering operations. Being able to accurately describe the tendency for particles to settle under gravity in liquid systems is important for areas such as hydraulic conveying, fluidized beds and flotation cells, as well as being critical in the development of efficient thickener or clarifier systems (Shukla et al., 2007) (i.e. free-settling systems). Pointedly, many mineral processing operations will involve the transport and final disposal of multiphase particle-in-liquid wastes, where thickeners are used to induce settling and allow dewatering of the dispersion (Bedell et al., 2006; Dronste, 1997). Although most characterisation in such industrial operations is done through sampling-analysis of concentration and cloud-front propagation (Binnie et al., 2002), there is increasing desire to use in situ based techniques as a way to increase the efficiency of data collection, and to reduce problems from intrusion that may result in disturbance of the natural system (Scott et al., 1998; Williams et al., 1990). For example, the possibility of faster data collection afforded from *in situ* characterisation would be advantageous in water treatment systems to monitor the efficiency of clarifiers and thickeners susceptible to seasonal variation in water quality (Dronste, 1997). Another application for *in situ* monitoring devices in free-settling multiphase systems, is in the analysis of toxic sludges and waste deposits, where sampling may pose major health risks, such as found in many nuclear waste environments (Hastings et al., 2007).

Most automated systems used to study particle sedimentation rely on correlating the concentration of particles to changes in a source signal transmitted through the dispersion, such as laser light (e.g., turbidity meters),  $\gamma$ -ray radiation (Kaushal and Tomita, 2007; Wheeler and Chatterji, 1972), electrical capacitance, impedance or conductance (Guerin and Seaman, 2004; Holdich and Butt, 1996; Vergouw et al., 1997) and ultrasonic sound (Challis et al., 2005; Guerin and Seaman, 2004; Shukla et al., 2007). Other techniques rely on physical parameters such as pressure balances and vibration dampening (Mahgerefteh and Kamugasha, 2000; Mantovanelli and Ridd, 2008) or even automated CCD video analysis (Hubner et al., 2001; Zhu et al., 2000). Ultrasonic (US) techniques in particular hold great promise for industrial deployment, due to their relative robustness, low cost (compared to radiation transmitters) and instrument design flexibility, which



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can afford true in situ deployment. In principle, US systems work similarly to laser based measurements, where a burst of US sound (normally 1-10 MHz) is sent through a dispersion and the scattering or absorption of the signal describes particle properties such as size and concentration (Challis et al., 2005; Dukhin and Goetz, 2001; Guerin and Seaman, 2004). The advantage of US systems, in terms of *in situ* deployment, is that they can be operated with a single transducer-receiver, which both produces the US pulse and can measure the return 'echo' signal from the dispersion. Hence, they do not require the signal to be passed through a specific sample unit, such as most turbidity and conductivity meters. In addition, ultrasonic systems have been used for a number of years in marine environments to study long term shifts of sediment beds and particle transport (Hosseini et al., 2006; Thorne and Hanes, 2002), and are often preferred over laser systems, due to their applicability in opaque environments (Williams et al., 1990).

Probably the most common type of ultrasonic device for sedimentology applications are Ultrasonic-Doppler Velocity Profilers (UVPs) (e.g., Kostaschuk et al., 2005; Sime et al., 2007), where the Doppler-shift of a transmitted US pulse scattered through dispersion is converted to a relative velocity of the dispersion particles. These systems are also often employed to measure the velocity profiles of multiphase systems in hydraulic conveying (Birkhofer et al., 2008; Wiklund and Stading, 2008); yet, as with their use in moving estuary environments, there is a question as to whether such UVP devices have the resolution to track the free-settling of particles.

In fact, ultrasonic devices have been used previously to study particle settling behaviour. Shukla et al. (2007) used a dual transducer and separate receiver system fitted at a particular height up a settling column. Results highlighted changes to both the US attenuation signal and calculated speed of sound as the sediment bed rose above the transducer. The limitation to this technique was that it could only track changes around the transducer plane, and not the progressive sedimentation with time. Razavian et al. (1991) used a single transducer-receiver system fitted to the base of an in-house designed sample unit, and by measuring the dissipation of the US pulse, were able to accurately gauge sedimentation rate with time, although they did not directly measure particle velocities. More recently velocity rates of settling dispersions were measured by Chinaud et al. (2010) with ultrasonic speckle velocimetry, again using an in-house designed profiler and sample unit. This previous research has highlighted the potential of ultrasonic transmission to gauge settling behaviour, but work is still required to establish whether a single-probe system could be employed as a viable in situ solution for an industrial situation. The focus of our current research was to ascertain whether a commercial acoustic Doppler velocimeter, as used in the study of multiphase flows in pipes and channels (e.g., Felix et al., 2005; Wiklund and Stading, 2008; Baas et al., 2009), could be also used to study free-settling suspensions. By measuring US Doppler profiles along a dispersion column, it was hoped that both freesettling particle velocities along a column depth, as well as the evolution of the sediment bed and cloud-front with time could be measured.

#### 2. Materials and methods

## 2.1. Materials

All tests were conducted using 'Spheriglass 5000' glass powder from Potters industries Inc. to simulate a simple industrial oxide. Spheriglass particle dispersions were made using distilled water and adjusted to pH 7 with HCl and KOH. Potassium Chloride (KCl) salt was also mixed into the dispersions in two different concentration regimes to induce particle-coagulation. A background 'low-salt' regime at  $10^{-2}$  M KCl and a 'high-salt' regime at 1 M KCl were tested. Sizing of the particles with a Malvern Mastersizer 2000, indicated the low-salt dispersion had a bi-modal distribution, with a minor (~20 vol.%) peak at 1–2 µm and a major peak at 8–10 µm, while the high-salt coagulated dispersion had a broad distribution with a mean peak around 35 µm. A representative sizing distribution average is shown for both dispersion types in Fig. 1. All trials were carried out with Spheriglass concentration set at 5 wt.%.

## 2.2. Methods

To gauge the settling and sedimentation profiles for the Spheriglass dispersions, behaviour was firstly followed using a Turbiscan Lab Expert profiler (from Fullbrook Systems), which utilises a vertical scanning laser set at 800 nm. Here, both settling of the top dispersion interface (cloud-front) was correlated by measurement of light transmission and the build up of the sediment bed was correlated by measuring changes to the 45° backscattered reflectance. For the Turbiscan studies, 20 mL sample vials (giving a 40 mm total liquid height) were used.

For the ultrasonic studies, a 'UVP-DUO' ultrasonic velocity profiler (UVP) was used from Met-Flow, Switzerland (see Takeda, 1991; Best et al., 2001). The UVP-DUO can both calculate particle velocity using the Doppler-shift method and can track solid walls or interfaces using an internally calibrated attenuation reading (Met-Flow, 2009). For the settling studies, a single 1 MHz transducer-receiver probe was used, attached by cable to the control box and computer data logger. A 1 MHz transducer was chosen, as this probe frequency has adequate resolution to measure the free-settling dispersions, and attenuation from the particulate fluid is lower than for higher frequencies (the UVP can use up to 8 MHz probes). Experiments were conducted in a 2 L measuring cylinder (8 cm diameter) filled with the testing dispersions. The 1 MHz probe, which has a diameter of 15 mm and a length of 60 mm (and spreading half angle of  $\sim 2^{\circ}$  from the focus point), was positioned ~300 mm from the base of the cylinder and completely submerged in the fluid. The cylinder was placed on top of a magnetic stirrer, to facilitate initial mixing of the dispersions (the stirrer bar was removed immediately prior to measurement so as not to interfere with results).

The experimental protocol was as follows. Firstly, each testdispersion was mixed for 15 min in the measuring cylinder to



Fig. 1. Average particle size distribution for the Spheriglass dispersion in  $10^{-2}$  M ('low-salt') and 1 M ('high-salt') KCl.

ensure a homogenous dispersion and that any aggregation had reached equilibrium. The sample was then left to settle for 90 min. The UVP-DUO was set to measure the 1-dimensional particle velocity profiles for distances between 50 mm and 350 mm from the transducer, at a scan rate of 260 scans per minute. To try to reduce any initial effects from mixing turbulence in the column, the system was left for 1 min before the UVP measurement was commenced, and, depending on the noise associated with any random turbulence fluctuations, the first 30-60 s of data was also generally ignored (which was considered reasonable, due to the relatively long time frame of the settling). It is noted that for this measurement distance and transducer frequency, the UVP discretises its distance measurements into 1.31 mm 'bins', which in-effect equates to the maximum resolution of the device. Because individual profiles generally contain a substantial amount of noise and scatter, the settling velocity profiles were averaged over 50 scans  $(\sim 12 \text{ s})$ . These 'averaged' settling profiles were then tracked over the 90 min time frame. Of course, any time averaging may result in errors, as the settling dispersions essentially represent a continually evolving system, but initial tests indicated that the  $\sim 12$  s time-step period was small enough, so that there should not be substantial change in the system, even for the high-salt dispersions.

#### 3. Results and discussion

# 3.1. Turbiscan sedimentation profile

The particulate settling and sedimentation profiles of the Spheriglass dispersions were firstly monitored with the Turbiscan, to assess any general differences in behaviour. To gauge settling rates, the cloud-front was monitored by laser transmission. Normally, for dispersions resulting in formation of a clear supernatant, a distinct peak in the transmission signal is observed. The growth in this peak is a correlation of the settling profile of the cloud-front. Likewise, the formation of a solid bed in the bottom of the sample unit creates a peak in the backscattered signal, and tracked changes represent the growth of the sediment bed. Fig. 2 shows the transmission and backscatter Turbiscan results for the 1 M salt dispersion and the backscatter results for the  $10^{-2}$  M salt dispersion. It is noted that because there was a high number of colloidal sized



**Fig. 2.** Turbiscan analysis of transmission peak thickness for 5 wt.% Spheriglass in 1 M KCl (upper graph) and backscatter peak thickness for 5 wt.% Spheriglass in  $10^{-2}$  M & 1 M KCl (lower graph). Dashed line in transmission graph represents the linear approximation of the settling flux.

particles in the low-salt dispersions, no clear supernatant formed due to the influence of Brownian diffusion on particle settling profiles (Mirza and Richardson, 1979) and hence no transmission data was gained.

Firstly, the 1 M KCl transmission data is discussed (Fig. 2, upper graph). The slow initial change in peak thickness is interpreted as being due to the effects of sample mixing leading to a small time lag until the cloud-front is clearly resolved. Then there is a period of constant change, with the linear gradient giving a settling rate of ~0.2 mm/s. This approximately represents the free-settling velocity of a 15 µm particle; using the unhindered Stokes' equation in laminar flow (Lu et al., 2005). Using the size distribution in Fig. 1, the weighted mean size of the particles is  $30-35 \mu m$ , giving corresponding theoretical unhindered mean settling rates of 0.75-1 mm/s. Although the measured cloud-front settling rate is below this mean, it is also noted from the size distribution in Fig. 1 that there is still a relatively large proportion of particles within a lower 10–15 µm range, corresponding to this rate. For such a broad particle distribution, there will be considerable internal separation of the dispersion, due to differences in relative particle settling rates, and thus the cloud-front will quickly segregate to represent the settling flux of essentially the smallest particles undergoing freesettling. The measured settling rate is consistent with this assumption.

The backscattered peak data for the 1 M KCl system (Fig. 2, lower graph), representing the growth of the sediment bed, is consistent with that expected for a highly aggregated system (Gohel et al., 2010). A relatively high sediment bed initially forms rapidly (here, within the first 2 min), which then compresses over-time. The initial bed peak is due to large inter-aggregate spacing. The weight of the upper particulate layers pressing down in the bed, will lead to internal-rearrangement of the aggregate structure, expelling the excess inter-particle liquid and effectively compacting the bed with time to an equilibrium configuration. Results for the 10<sup>-2</sup> M KCl dispersion (also Fig. 2, lower graph) suggest the low-salt bed forms slowly into a compact non-compressible arrangement, in a non-linear fashion. This profile is consistent with a non-aggregated bi-modal system (Kondrat'ey and Naumoya, 2006; Spannenberg et al., 1996), where segregation due to differences in settling rate will be extremely pronounced. Initially, the larger  $\sim 10 \,\mu m$  fraction will dominate the bed sedimentation, before slowly the colloidal fines fraction settle.

#### 3.2. UVP measurement of the speed of sound

Since the UVP uses a single probe acting as both a transducer and receiver, distance calculations require an accurate estimation of the speed of sound (SoS) through the dispersion. As the distance from the transducer to the base of the cylinder was set at 300 mm, calculation of the SoS was completed by tracking the measured distance to the cylinder base for different input SoS values, in the lowsalt and high-salt mixtures. To check the accuracy of the machine, SoS measurements were also completed in distilled water. An initial SoS estimate was taken near the known value for water (~1484 m/s, Benedetto et al., 2005) and values increased until the measured distance to the cylinder base matched the set 300 mm value. It is noted that the cylinder base signal was shown by both a strong attenuation peak and a clear point where the calculated particle velocities dropped to zero. Even with distilled water (where all velocity calculations were effectively noise, due to lack of particulate matter to give a Doppler-shift signal) a clear zero was seen in values at the cylinder base. Speed of sound measurements were made over a 30 s run. Fig. 3 shows the measured distances to the cylinder base for different input SoS from 1480 to 1560 m/s, with water and 5 wt.% particle dispersions in low and high salt conditions.



**Fig. 3.** Change in the distance to the column base as measured by the UVP, for water and 5 wt.% Spheriglass in  $10^{-2}$  M & 1 M KCl, given different input speeds of ultrasound. Dashed line represents correct distance value.

The distilled water results shown in Fig. 3, firstly confirm the accuracy of the distance discretisation, with the estimated distance value passing through the set-distance of 300 mm as the input SoS was between 1480 and 1490 m/s. It is also shown with the distilled water that if input SoS is increased above the real value, the measured distance to the cylinder base increases, as the sound is actually travelling slower than estimated. For the low-salt Spheriglass dispersion (where all particles should be non-aggregated) the input SoS needs to be increased to  $\sim$ 1500 m/s for accurate distance

calculations. This is consistent with propagation theory (Challis et al., 2005; Shukla et al., 2007; Xue et al., 2010). Simply, owing to the fact that sound travels faster through solid systems, due to the increased efficiency of compression propagation, the introduction of the Spheriglass particles act similarly to enhance the transmission properties of the fluid. Interestingly, for the high-salt system, where particles are aggregated, the real SoS through the dispersion increased dramatically up to  $\sim$ 1550 m/s. This shows the effects of increased structure in the fluid on sound propagation, but considering that both salt systems contain the same bulk concentration of particles (5 wt.%) this increase is quite important. Indeed, similar velocity increases have previously been found in coagulated food dispersions, such as milk (Bakkali et al., 2001), and highlight how such techniques can be used to monitor the extent of aggregation (Kaatze et al., 2008). From these results, the input SoS was held at 1500 m/s for the low-salt studies and initially at 1550 m/s for the high-salt studies.

3.3. UVP measurements of dispersion sedimentation in the 'low-salt' regime

#### 3.3.1. Selected velocity profiles

The evolution of the settling velocity profiles for Spheriglass dispersions with  $10^{-2}$  M KCl with time, during a 90 min test run is shown in Fig. 4. Here, 12 s 'averaged' time-step profiles along the distance of the column are shown with their relative errors after 5 min (A), 30 min (B), 60 min (C) and 90 min (D).

Firstly, the profile after 5 min is discussed (Fig. 4A). It is observed that the absolute values of the particle velocities along the first two-thirds of the column are less than 1 mm/s. Considering the unhindered settling velocities of sub 10  $\mu$ m particles



**Fig. 4.** Velocity profiles averaged over  $\sim 12$  s time-steps measured by the UVP along the distance of the settling column, for a 5 wt.% Spheriglass dispersion in  $10^{-2}$  M KCl ('low-salt' regime). Profiles are shown after 5 min (A), 30 min (B), 60 min (C) and 90 min (D).

should be only in the region of  $\sim$ 0.1–0.2 mm/s (again, simply estimated from Stokes' free-settling equation) it may be presumed these small values are within expectations. However, in the lower portion of the column it is noted that the particles velocities are much greater (in absolute terms, momentarily ignoring the relevance of the negativity), although the error also increases markedly. As it is known the un-aggregated particles are bi-modal in nature and are prone to segregate with time, it may be presumed this distinct change in velocity profile highlights the separation of the large particle fraction in the bottom third of the column. However, the magnitudes of the absolute values in the lower portion of the column are far beyond those expected even for particles up to 30 µm (Spannenberg et al., 1996). It also has to be noted that the relative particle velocities given are in reference to the transducer, with negative values indicating the particles are falling away from the transducer (settling) and positive values indicating the particles are coming towards the transducer (rising). Hence, the fact that positive velocity values are shown in the upper portions of the column would suggest either some positive particle or fluid displacement, as perhaps the larger particles move down through the slower settling fines, or more probably, the system as a whole settles too slowly to be accurately measured by the UVP-DUO. As the instrument is designed to measure flowing slurries (Felix and Peakall, 2006; Birkhofer et al., 2008; Kantoush et al., 2008; Wiklund and Stading, 2008), this is perhaps not surprising, especially considering the interference of Brownian diffusion on the flow profiles of the fines, which will lead to the random migration of colloidal material throughout the dispersion. As, the UVP effectively assumes 1-dimensional movement either towards or away from the transducer, any random movements (especially from very slow moving fines) will be picked up as noise fluctuations by the instrument. In fact, the Brownian diffusion of colloidal fines was significant enough to produce no clear supernatant during sedimentation (as with the Turbiscan studies).

Despite the large error with the raw velocity values in the dispersion, it is observed in Fig. 4A that once the particles settle to the cylinder base (around 300 mm) a clear zero reading is shown for the velocities, also with a zero relative error. Hence, despite the fact that the instrument does not have the resolution to accurately measure such small velocities, it is able to distinguish the bottom interface very clearly. Indeed, as we look at the longer time periods (Fig. 4B–D) we see the same phenomenon, of a clear zero velocity reading with low related error at the bed interface. The longer time periods also emphasis the segregation in the bottom third of the column. The noted absolute change in velocity values in the lower third of the column is measured again at 30 min (Fig. 4B), but is far less distinct at 60 min (Fig. 4C), where most of the larger particles would be expected to have settled out. It is further noted that the system noise and error at 60 min is increased throughout the entire column. At 90 min (Fig. 4D) calculated velocities are most likely only noise signals throughout the column, as the majority of particles should have sedimented by this time, and so dispersion concentrations would not be high enough to produce meaningful estimates of particle Doppler-shifts.

Because the bed interface could be tracked, despite the large errors associated with the actual velocity measurements, the bed sedimentation behaviour was analysed in more detail. This was achieved by focusing upon the interfacial region and calculating the point at which the bed formed, from the supposition that associated velocity values (and related error) at the bed interface dropped to approximately zero (as highlighted by the profiles in Fig. 4). The resulting analysis is discussed in the next section.

## 3.3.2. Plotting the evolution of the low-salt Spheriglass bed

To plot the settled bed interface, a 100 mm section between 250 and 350 mm was analysed, which incorporated the bed interface.

Using the evidence (as discussed) that the bed interface was shown by a distinct drop in both average velocity and relative error, a simple set of Boolean expressions was set up in Excel<sup>TM</sup> to track the first point at which BOTH the absolute value of the velocity was less than 0.001 mm/s AND the related standard error was less than 0.01. By using both velocity values and related errors, any erroneous points above the bed with a close-to-zero velocity were invalidated, as it was established the errors associated with these points were large.

Fig. 5 shows an example of the resulting change in the bed interface height with time, for a low-salt system. By relating the measured distances to the sediment bed from the initial distance, the bed height is shown from a zero basis. Also shown is a manually tracked comparison, where the height was measured by eye (using a graduated ruler) over the same time period, giving a direct visual comparison of the settling behaviour. It is firstly observed in Fig. 5 that the UVP data jumps between different discretised distances. This result would be expected, because of the defined size of the distance 'bins' (at 1.31 mm), which underlines the instrumental resolution. If the actual bed height was between two discretised bins, the algorithm would show the calculated bed height jumping between the two closest measurement bins (some profiles would show the height as one distance bin while others an adjacent bin, with the real bed height lying somewhere in-between). However, it is noted that a column of this size only produces a total bed height of a little over 10 mm after settling is completed, and industrially bed volumes would be much greater, where such resolution would not be an issue. Despite the uncertainty involved with the distance discretisation, it is clearly seen in Fig. 5 that the manual measurements taken during the same period compare closely with the trend measured from the UVP, giving great confidence in the validity of the technique.

3.4. UVP measurements of dispersion sedimentation in the 'high-salt' regime

#### 3.4.1. Velocity colour map

Initial tests with the UVP-DUO to gauge the threshold limitations of the velocity measurements, suggested particles in the high-salt regime settled with a great enough velocity to be within the instrument's capabilities, due to the particle aggregation. Indeed, detailed analysis of the profiles further indicated this was the case, as evidenced from the velocity 'colour maps'. Fig. 6 shows



**Fig. 5.** Change in the sediment bed height with time for a 5 wt.% Spheriglass dispersion in  $10^{-2}$  M KCl, as measured by the UVP and manually tracked by eye.



**Fig. 6.** Velocity 'colour map' showing the changing instantaneous velocity profiles down the settling column length with time for a 5 wt.% Spheriglass dispersion in 1 M KCl ('high-salt' regime). The colour chart bar is expressed in mm/s. Dotted arrow indicates settling cloud-front. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

a representative colour map for the first 3000 s of a particular settling run. The colour map is an internally averaged 2-dimensional image, showing the tracked changes of the instantaneous velocity down the length of the column with time. Different velocity regimes are distinguished with different colour profiles, where 'green' shades indicate small negative values (i.e. particles settling down the column), yellow shades small positive values (i.e. particle rise) and black indicates areas of close to zero velocities. Note, the image is produced in terms of absolute distance from the transducer; hence the bottom of the cylinder is at the top of the image.

The colour plot shown in Fig. 6 gives a considerable amount of detail. Firstly, average settling velocities in the middle of the column (within the first 500 s) were measured as  $\sim$ 1–3 mm/s, which is reasonable considering the measured size of the aggregates. Also, the evolution of the sediment bed is clearly seen being tracked from the black (zero) velocity region at the top of the image. Initially, it seems there are a number of non-zero velocity readings from inside the bed as it is formed, signifying rearrangement of the sediment aggregates, as interpreted from the Turbiscan results. These areas of non-zero velocity inside the bed are reduced as time increases, as would be expected from longer-term bed compaction. In fact, it is clearly seen that the black region at the top of the image associated with the bed reduces in thickness after 1000 s, again consistent with the long term compaction of the bed evidenced in the Turbiscan. In addition, some insight into the nature of the settling dispersion can be gained from the colour map. There is a region on top of the sedimented bed where measured particle (or associated fluid) velocities decrease to near zero or even begin to rise off the bed (highlighted by the yellow and black region near the bed within the first 1000 s). This change in the near-bed velocities is consistent with hindered-settling effects within the dispersion, caused by interaction between the particle aggregates. Water up-flow from aggregates settling lower in the dispersion will disrupt the flow of nearby particulates, slowing their settling rate (Tadros, 1987). Due to the likely build up of particulates in the lower portions of the column from size segregation of the dispersion, it would not be surprising that these effects are most evident in the near-bed region.

Lastly, the evolution of the cloud-front with time can be evidenced by a distinct change in measured velocity values, between the supernatant water, which is essentially up-flowing and the dispersion interface that is down-flowing. This is shown in the colour plot by the vellow/black front that forms in the dispersion diagonally down the column image over-time, highlighted by the arrow (again within the first 1000 s). The linear slope of this line (which is  $\sim$ 0.35 mm/s) correlates to the settling flux rate of the cloud-front. This is greater than the rate measured from the Turbiscan, however it only equates to a shift in theoretical particle size from  $\sim 15 \ \mu m$  to  $\sim$ 20  $\mu$ m. Due to the large size distribution of the coagulated particles, such differences are well within those expected from experimental variation, noting this is a colour plot of one particular settling run. Potential differences in settling dynamics between the small sample cell used in the Turbiscan analysis and the large 2 L cylinder used with the UVP also cannot be discounted.

It is noted that at longer time periods (after  $\sim$ 20 min or 1000 s) the cloud-front has sedimented, leaving a nominally clear supernatant, although measurements within the dispersion supernatant (throughout the entire settling run) continue to show non-zero values (clearly seen in Fig. 6). It is assumed these values are effectively system noise from any trace particles still present. In reality, any colloidal fines, trapped dust or any trace matter in the water will be measured as a velocity value by the UVP, even when it is in concentrations far below that required to produce a statistically meaningful result. Actually, the associated error from these velocity values in the supernatant at long time periods were extremely large (although the errors are not shown by the colour plot itself), and effectively these readings are ignored. It is also pertinent to highlight that the lack of internal error analysis in the colour plot illustrates why it is limited to the analysis of settling systems with low associated noise, which is why a colour plot was not able to be produced for the low-salt systems.

#### 3.4.2. Evolution of the high-salt Spheriglass bed

Similarly to the low-salt dispersions, the settling velocity data was analysed to produce a graph of the bed interface with time. Although, in essence, the sediment behaviour could be seen with the colour map, a quantitative assessment of the sedimentation was carried out to confirm the bed formation structure and any effects from different input speed of sound values. Again, velocity values around the bed interface were considered in detail and a set of Boolean expressions used in Excel™ to track the incipient point of bed formation. For the high-salt systems, because of the loose-aggregated nature of the beds, slightly higher threshold velocity and error values were used. Here, the first point was tracked with BOTH the absolute value of the velocity as <1 mm/s and error as <0.05. Fig. 7 shows two different sedimentation runs with the bed-height tracked in this way, where the input speed of sound was set at 1550 m/s (A) and 1500 m/s (B). Again, the runs are directly compared to manual measurements, where the bed interface was measured by eye.

Two different input speeds of sound (SoS) were tested, because it was known that although the high-salt SoS was  $\sim$ 1550 m/s at the onset of sedimentation, once settling commenced (and so bulk particle concentration dropped) so too would the SoS. This problem was not so evident in the low-salt regime, due to the relatively small difference between the SoS of the dispersion and that of



**Fig. 7.** Change in the sediment bed height with time for a 5 wt.% Spheriglass dispersion in 1 M KCl, as measured by the ultrasonic velocity profiler and manually tracked by eye, using an input speed of sound of 1550 m/s (A) and 1500 m/s (B).

water. Indeed, it is clearly seen in Fig. 7A, when using an input speed of sound of 1550 m/s, the UVP-DUO slightly overestimates the bed height throughout the majority of the settling run, suggesting a systematic error. Although, clearly, the continual change in dispersion characteristics with sedimentation would also lead to continual changes in the SoS, a value of 1500 m/s was chosen, as it was felt this would represent a mean that may better predict the system overall. In fact, it is clearly evident from Fig. 7B that using this lower speed of sound resulted in a closer bed height bed comparison throughout the sedimentation. The settling profiles correlate well with the Turbiscan data, again highlighting that the coagulated particles initially form a large loose-packed bed which compresses over-time. Indeed, the ability to track these changes to bed structure and density on a real time basis highlights the technique's potential to monitor the efficiency of industrial settling systems and sediment aging effects.

## 4. Conclusions

This report has shown that coagulated particle settling rates, sediment bed formation, and bed compaction can be tracked on a real time basis using an *in situ* ultrasonic velocity profiler. This technique is very advantageous, as it relies solely on commercial cost-effective off-the-shelf equipment, and as it uses a single transducer–receiver system, intrusion is kept at a minimum. Although tests shown in this report were completed in a 2 L cylinder, the system does not require any particular vessel size, and could easily be used in most industrial settling systems, such as clarifiers, thick-eners or multiphase waste deposits.

The work completed here was focused upon ascertaining the limits of this system, for the measurement of free-settling particle velocities. Measured velocity profiles of non-aggregated 10 µm particles produced considerable error, however the bed interface was seen clearly, and particle sedimentation was still able to be tracked quantitatively. For a coagulated high-salt system, measured settling velocities were accurate enough to produce a full colour map, illustrating not just sediment bed formation, but quantitative measurements of particle setting velocities down the column as well as the movement of the cloud-front, in addition to highlighting areas of hindered settling. The evidence for hindered particle settling within the dispersion is a particularly important result, as this level of system detail would not be possible with a traditional turbidity meter system, such as used for comparison in this work. Indeed, generally, the ability for US systems to work in opaque environments, thus giving them the ability to penetrate through dispersions allowing full settling profiles, is a major advantage for their industrial deployment. Despite the technique's limitations (in terms of measurement in the colloidal regime) the aggregated nature of most industrial dispersion systems means it is likely the UVP would be suitable for a wide range of free-settling multiphase environments.

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