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Ultrasonic techniques for the *in situ* characterisation of legacy waste sludge

T.N. Hunter[1], S.R. Biggs[1], J. Peakall[2]*

[1]Institute for Particle Science & Engineering, University of Leeds
[2]School of Earth & Environment, University of Leeds
(* correspondence: j.peakall@see.leeds.ac.uk)

Abstract
Owing to the relatively large size and diverse nature of Legacy waste sites, in addition to obvious issues with contamination, traditional sampling techniques for the characterisation of the various sludges and slurries is severely limited. The ability to use an *in situ* device to quickly and safely gain a large amount of data, whether qualitative or quantitative is very attractive. This report details some initial investigations into the applicability of ultrasonic techniques to characterise the settling behaviour of oxide sludges. The type of single transducer-receiver system trialled has been used for many years in the study of fast moving flowing slurries in pipe loops; however, it also has potential to analyse the free settling sludge systems encountered in Legacy waste ponds, where high particle concentrations render them opaque and unsuitable to light based characterisation. Specifically here, a 1 MHz Met-Flow UVP-DUO system was used to quantitatively characterise the settling rates and bed formation of Spheriglass 5000 glass powder, by studying the particle flow velocities. Attenuation of the particle beds was also analysed, and it is hoped that this technique will also enable some qualitative analysis of particle bed structure.

Introduction

Interest in designing *in situ* characterization techniques for mineral slurries, sludges and sediment dispersions has increased considerably within the last 20 years, as enhancements in modern computer and data processing have overcome many logistical difficulties [1]. *In situ* analysis is attractive to industry, as it has a number of potential benefits over traditional sampling techniques; such as the ability for constant monitoring and decreased problems from system intrusion [1, 2]. *In situ* techniques have been developed from two different perspectives; firstly, a number of smaller scale 'in-line' techniques have been developed to monitor dispersion properties such as concentration, size and rheology in flowing pipe loops [1]. Also, environmental sedimentologists have used various *in situ* techniques to study large scale estuarine and river bed flows and dispersed sediment properties [3].

Ultrasonic (US) techniques have been particularly useful in much of this latter field of work, where the received eco of an ultrasonic sound wave can be studied to determine particle properties such as concentration and flow gradients or bed morphology [4]. Generally, the loss of signal from particle scattering and adsorption (attenuation) is indicative of particle properties such as size, structure and concentration, (although specific interactions are difficult to determine [5]) and can be qualitatively analysed similar to light based turbidity systems. The significant advantage of US systems is that they can be used in particle dispersions of much higher concentration, which are optically opaque. Also, a single probe can be used to both transit and receive the audio signal, which leads to very simple in-situ probe systems. Further, the type of system used in these studies (ultrasonic velocity profiler, or UVP) can measure the Doppler shift of the transmitted audio frequency, gauging relative particle velocity [6].

Although there are potentially a number of particle and sediment properties that may be characterised using US techniques (depending on the complexity of both the instrument and data analysis) this investigation sought to simplify the system to focus on one key slurry parameter for Legacy waste characterisation, namely particle setting rates. Being able to directly measure particle setting rates will give vital information as to the behaviour of the Legacy waste once pumped to interim storage facilities. Indeed, it is hoped that techniques used here may lead to real time analysis of sludge bed formation and compression overtime and in addition give ability to monitor the success or failure of any sludge re-suspension processes. It is hoped further investigations using this technique will also enable some qualitative assessment of particle concentration and settled bed structure.

Experimental

'Spheriglass 5000' glass powder from Omya Industries was used as a model oxide particle base. Previous sizing by a Malvern Mastersizer showed that it has a bi-modal distribution, with a major peak at ~10 μm and a minor peak at 1-2 μm [7]. The instrument used for analysis was a Met-Flow UVP-DUO. It consists of a 1 Mhz Transducer-receiver attached by cable to a pulse generator and data logger, which is controlled by computer.

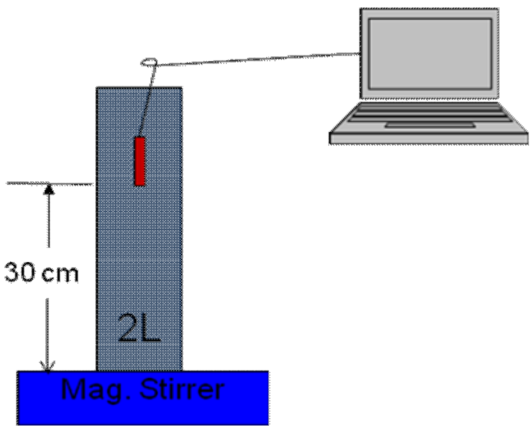

Figure 1 Schematic of experimental setup

The experiments were run as follows. A 2 litre measuring cylinder was used as the test vessel. 5 wt% particle dispersions in distilled water were made with 10^{-2}M KCl as background salt, adjusted to pH 7. The dispersion was well mixed in the cylinder and then left to settle over 90 minutes (with the UVP transducer fully submerged in the dispersion). The UVP is a rapid analyser, and measures approximately 260 distinct profiles per minute. The software automatically calculates particle velocity from the Doppler shift of the audio eco signal. By assuming a set velocity of sound (which was calibrated in a 5 wt% Spheriglass dispersion to be ~1500 m/s) the data logger discretises the eco signal into distance lengths from the transducer (i.e. giving a 1 dimensional velocity profile). Here, the transducer was set 300 mm away from the base of the measuring vessel (see Figure 1), and the UVP was set to receive echo signals (and translated velocity profiles) between distances of 50 to 350 mm from the transducer. It is noted that there is a minimum distance of 1.31 mm between each discretised velocity reading.

Results

<u>Selected Velocity Profiles</u>

Individual velocity profiles were averaged over 30 second intervals (incorporating 130 profiles) to give the mean velocity and standard error over 30 second time steps. It is noted that settling should be slow enough, for there to be no significant change in bed height or particle behaviour within each time step. Given in Figure 2 (a-c) are the average velocity results for 30 second time steps starting at 5 minutes, 60 minutes and 90 minutes.

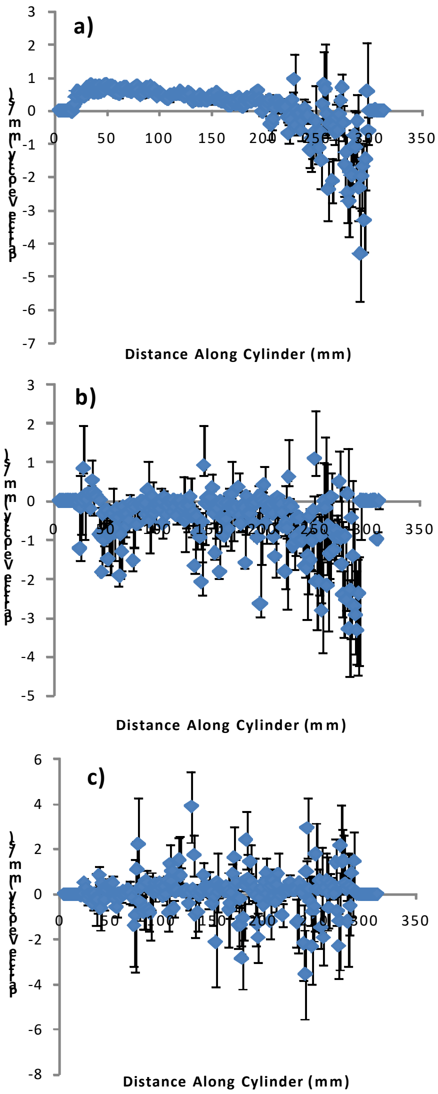

Figure 2 Velocity profile averages for 30 second time steps starting at a) 5, b) 60 and c) 90 minutes

Firstly, the profile at 5 minutes is discussed (Figure 2 a). It is observed that the profiles along most of the cylinder show particle velocities less than 1 mm/s. This would be expected for free settling particles of this size (simply estimated by calculating the non-hindered Stokes settling velocity for a 10 to 20 μm particle [8]. However, near the base of the column, the velocities seem to decrease relatively, but also the error significantly increases. It is presumed this is due to segregation in the column. As described in the experimental section, the particles are bi-modal; hence the larger particles will settle out quicker and in the lower portion of the column interact with the slower settling fines. Interactions between particle flow fields may lead to significant disruption to particle trajectories, resulting possibly even in the smaller fines being lifted by displacement. It is quite likely that the interaction between the segregating particles near the base is the cause for the increased error and velocity changes. It is noted that the given velocities are 'relative' in comparison to the UVP transducer and the complexities of the system mean that it is impossible to judge specifically the reason for the measured changes in average velocity.

What is evident however is that once at the base of the column (around 300 mm) the particles abruptly stop as they form into a settled bed. Indeed, this abrupt change in velocity is also evidenced at the longer time lengths, and indicates that the evolution of the settled sediment bed can be correlated to the sudden measured halt in particle movement. Hence, by following this incipient point of bed formation, we are able to measure bed settling rates.

The general velocity behaviour at 60 and 90 minutes is also discussed. It is perceived that at these longer time scales there seems to be a less significant difference between particle velocities in the upper and lower portions of the column, while average error increases in the upper portions of the column. As the larger 10 μm particles settle out more quickly than the fines, it would be unsurprising that by 60 or 90 minutes most would have settling into the bed, and hence the dispersion segregation observed at 5 minutes would be reduced. The reason for the increased velocity error is due to the way in which the software computes particle movement. As the instrument uses the Doppler shift from the eco signal off dispersion particles, a higher dispersion concentration (within the limits of the machine) gives the software a larger average to calculate instantaneous velocity. At 90 minutes, the dispersed particle concentration is probably so low as to effectively render the velocity calculations meaningless.

<u>Measurement of Particle Settling Rates</u>

Trying to track the bed formation by observing the velocities of the particles is a difficult proposal, as essentially one has to take a set of time averaged profiles, which are then tracked over larger time ranges. Again, 30 second (130 profile) averages were taken over a period of 90 minutes. The point of bed formation was taken by comparing both the average particle velocities and relative errors, in the vicinity of the particle bed (from 250-350 mm). A simple set of Boolean expressions were set up in Excel to track the moving bed. As noted in Figure 2, although measured particle velocities were extremely variable in the vicinity of the bed, the relative error was also high. However, once lodged in the particle bed, the velocities were given as a consistent zero reading with no error. The insipient point of bed formation was defined as being the first distance point where the absolute value of the average velocity was < 0.001 AND the absolute error was < 0.01. By treating the raw data in such a way, a plot of the increasing bed build up in the column was calculated, as displayed in Figure 3. Here, the initial column length (from the base of the transducer) was divided by the calculated bed distance, to show the bed increase from an initial zero height. Also shown in Figure 3 is an average of 4 separate runs in the same system, where the settled bed height was manually measured by eye.

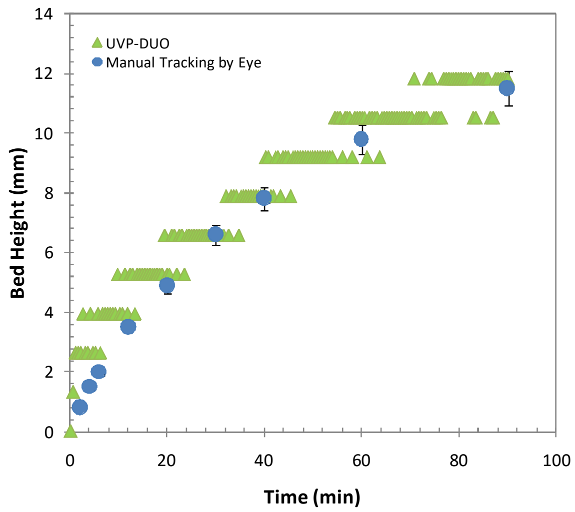

Figure 3 Tracked sediment bed height over time from the UVP-DUO and from manual measurements

It is clearly observed that the UVP system deployed successfully tracks the formation of the Spheriglass bed, and the results compare very favourably with the manual measurements. It is noted that the calculated bed heights jump between discrete distances over time, a result of the fact that distance is discretised into 1.31 mm sections. This in effect highlights the error within this system of measurement; however for larger beds this error would not be as significant. Nevertheless, the average change in height over time for agrees extremely closely to the manual measurements.

<u>Attenuation Profiles</u>

The UVP-DUO measures a relative attenuation profile, which is unlike most ultrasonic devices. It is internally calibrated to highlight walls and other solid structures by outputting a large attenuation peak. Indeed, it can be coupled with the velocity measurements as a method to verify the point of bed formation, which displays an attenuation peak. However, owing to the fact it is a relative signal that cannot be absolutely calibrated with any system, means its use in this way is limited. Nevertheless, it was hoped that attenuation peaks attributed to the particle beds may be able to give some qualitative insight into the structure or density of a particular bed.

Firstly, a 5 wt% Spheriglass dispersion was left to settle for 24 hours. Then the average of 100 attenuation profiles were collected to observe the effect of the Spheriglass bed on the attenuation signal. In addition, about 400 cm^3 of 50 μm Ballotini particles were poured onto the settled Spheriglass bed, and attenuation re-analysed. Figure 4 shows the average attenuation profiles for the Spheriglass bed and the Spheriglass bed with the addition of the Ballotini particles.

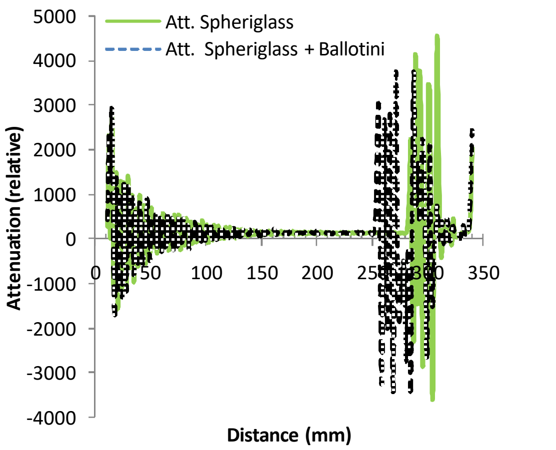

Figure 4 Average attenuation profiles for a 5 wt% Spheriglass dispersion after 24 hours with and without addition of a Ballotini bed

The initial small attenuation peaks are due to ringing inference from within the transducer, while the large peaks near the base of the cylinder are clearly correlated to the particulate beds. The initial attenuation peaks from the Ballotini (at around 3000 relative units) do seem to be smaller than the same initial signal from the Spheriglass (at around 4000 relative units) although natural variation is too high to draw any definite conclusions. There also seems be a definite shift in signal strength in the transition from the Ballotini bed to Spheriglass bed (blue dashed line), perhaps suggesting a way in which bed structure changes with depth may be able to be monitored. It is noted that these are only very cursory observations from the initial attenuation measurements, and much work is required to probe the usefulness of these attenuation profiles.

Conclusions and future work

We have shown that particle setting rates and bed formation can be tracked on a real time basis using an ultrasonic velocity profiler (UVP). This has obvious advantages to manual measurements and sample based techniques in the monitoring of particle beds in a nuclear environment, but would also be useful to track settling in many systems where direct measurements are not easily achieved (e.g. Water treatment thickeners). The Attenuation profiles from the UVP-DUO are far less useful quantitatively, although, by measuring the penetration through beds one may be able to define some differences between bed structure.

Future work will focus on canvassing a larger range of sediment types to investigate the limits of this technique. Highly aggregated particle dispersions from both salt coagulation and polymer flocculation will be tested to observe whether aggregation affects the strength and quality of both velocity and attenuation signals. This initial system was a slow setting system, where particles formed a dense bed. Questions remain as to whether the formation of the bed can be as clearly tracked in highly aggregated fast settling systems and systems where there is significant bed compression over time. Also, it is hoped that this technique can incorporate a second transducer to measure the raw (non-relative) attenuation of the dispersion, which may give more detailed information on the concentration and structure of the dispersed aggregates and bed [9, 10].

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