

Ultrasonic Techniques for the *In Situ* Characterization of Nuclear Waste Sludges – 11275

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ABSTRACT

Ongoing research at the University of Leeds, being undertaken as part of the DIAMOND university consortium, is exploring the effectiveness of various ultrasonic technologies as *in situ* probes to characterize and monitor simulant nuclear wastes. Through use of a commercial Ultrasonic Velocity Profiler (UVP) and an Acoustic Backscatter Sensor (ABS) both with 1 – 5 MHz transducers, various properties of free-settling oxide simulant sludges were determined. Work was focused upon characterizing essentially ‘static’ sludges for prospective use within U.K “Legacy” waste sites; although, the sensors have potential as monitors in many storage or thickener/de-watering systems across a wide range of industrial processing operations. The UVP, which correlates particle velocities by measuring the associated frequency Doppler shifts, was able to determine the free-settling rates of sludge particles for a 1-dimensional profile down a settling column, with results highlighting considerable hindered settling effects within certain zones. By focusing on velocity changes in the near bed region, the formation and compaction of the sediment bed over time was also tracked, as well as the movement of the sludge cloud-front. The ABS, which measures the raw attenuation of an ultrasonic pulse through dispersions, was calibrated to measure changes in particulate concentration within a settling suspension, and showed that observed hindered settling also led to an increase in particulate concentration over the sludge zone. Lastly, a quartz crystal microbalance (QCM) probe developed by RheokinisisTM was used to track yield stress changes in magnesium hydroxide sludges. Here, changes to the vibrational frequency and electric resistance of a piezoelectric crystal inserted into sludges of various concentrations and at a fixed concentration undergoing consolidation (aging) were measured. Increases to both vibrational frequency and resistance were associated with increases in the sludge matrix yield-stress.

INTRODUCTION

The problem of so called ‘Legacy’ nuclear waste deposits from first generation British nuclear sites is a major economic, logistical and technological barrier to advancement of the U.K nuclear power industry. The Legacy nuclear wastes mainly consist of dissolved magnesium alloy (‘Magnox’) canisters from spent reactor fuel rods. However, the specific nature of the resultant oxide sludges are highly complex, with properties varying extensively between sites, due to different storage conditions and other low and intermediate level wastes stored within the same deposits [1].

Characterization of this waste is therefore an extremely difficult proposal and is made all the more complex due to the potential radiation risks (which also vary considerably between sites) making sampling analysis time-consuming and logistically challenging. While a great deal of information on sludge properties has been gathered over time [2-4] (especially in respects to sludge composition) the heterogeneity of the sludges in various locations has led to major problems in acquiring sufficient data across the legacy sites. Particularly, information on sludge behavior is required that will allow operators to better plan for waste retrieval and any future storage and processing; such as sludge particulate concentrations, settling and sedimentation behavior and material yield stresses. One possible answer to the challenge of characterization is to use an *in situ* measurement system, where equipment can be installed and analysis conducted on site without the need for sampling. An *in situ* system would allow for much higher rates of data collection; however, attainable system information is often more qualitative than available from a full laboratory analysis [5].

The basis for many multiphase *in situ* characterization systems is to use a robust source signal, such as ultrasonic sound, electric capacitance or radiation, and measure the change in the signal through the testing medium [6-8]. The technical complexity arises from the de-convolution of the returned source signal to gain information on specific parameters; such as particulate or fluid flow rate, settling rates, particle concentration, aggregation state and size. The major advantage of ultrasonic devices is that the transducer can be separated from the control hardware, and hence intrusion into potentially toxic waste environments kept at a minimum. Work completed within the University of Leeds, as part of the DIAMOND university consortium, looked to ascertain the effectiveness of various ultrasonic technologies as *in situ* probes to characterize and monitor simulant nuclear wastes. The main focus of the research has been to integrate and port ultrasonic sensors that have been used extensively in hydraulic engineering and marine sedimentology applications, to systems and environments encountered within the nuclear industry. Ultrasonic techniques have proven effective in measuring various dispersion properties within marine environments, such as tracking changes to consolidated beds, measuring particle shift velocities and concentration profiling [9]. Through the current research initiative, it is hoped that ported technologies may not only have uses in characterizing current Legacy waste deposits, but also as monitors during transport and future storage of these wastes; a critical concern, as sludge behavior may change dramatically upon retrieval and pumping.

In this paper we report on the development of suite of complimentary ultrasonic sensors designed to probe the behavior of Legacy nuclear wastes. Firstly, an ultrasonic velocity profiler (UVP) was used to measure the settling rates of aggregated glass dispersions as well as the build up and compression of the sediment bed. Secondly, an acoustic backscatter sensor (ABS) was used to monitor dispersion concentration changes during sedimentation. Lastly, a quartz crystal microbalance (QCM) technique was used to characterize the yield stress properties of consolidated magnesium hydroxide sludges. Although this work was performed on simple non-active simulants, there should be no reason why such techniques could not be taken to active environments, as all tested sensor types were robust and protected in design. Indeed, it is hoped in future that these sensors may be able to be fitted to robotic handlers that have been installed onsite (and previously used for sampling), allowing fully automatic *in situ* sludge analysis.

MATERIALS & METHODS

Particulate Sedimentation Measured with an Ultrasonic Velocity Profiler (UVP)

To prove the measurement capabilities of the UVP for analysis of settling aggregated suspensions, a simple oxide powder was used. Dispersions of 'Spherglass 5000' glass particles from Omya Industries were made at 5 wt% (to test similar concentration levels to the bulk Legacy wastes [2]) and aggregated via coagulation in 1M KCl. Light sizing using a Malvern Mastersizer indicated the particles formed aggregates in a broad size distribution, with a D^{50} of $\sim 35 \mu\text{m}$. The instrument used for analysis was a Met-Flow UVP-DUO. It consists of an ultrasonic 1 MHz transducer-receiver attached by cable to a pulse generator and data logger, which is controlled by computer.

To conduct sedimentation analysis with the UVP, Spherglass dispersions were well mixed in a 2 L measuring cylinder, before being left to settle over 30 minutes (with the UVP transducer fully submerged). On board software calculates particle velocity values from the Doppler shift of the audio echo signal. By assuming a set velocity of sound (which was calibrated in a 5 wt% Spherglass dispersion to be $\sim 1500 \text{ m/s}$) the data logger discretises the echo signal into distance lengths from the transducer (i.e. giving a 1-dimensional velocity profile). The transducer was set 300 mm away from the base of the measuring vessel, and the UVP was set to receive echo signals (and translated velocity profiles) between distances of 50 – 350 mm from the transducer. It is noted that there is a minimum distance of 1.31 mm between each discretised velocity reading.

To compare the settling and sedimentation profiles with a standard laboratory technique, behavior was also analyzed using a Turbiscan Lab Expert profiler (Fullbrook), which utilizes a vertical scanning laser set at 800 nm and 20 mL sample vials. Here, both settling of the top dispersion interface (cloud-front) was correlated by measurement of light transmission and the buildup of the sediment bed was correlated by measuring changes to the 45° back scattered reflectance.

Particulate Settling and Concentration Changes Measured with an Acoustic Backscatter Sensor (ABS) system

A similar experimental system was studied with the ABS, to probe how these two types of ultrasonic sensors could elucidate complimentary information on the settling behavior of dispersions. Again, Spherglass oxide particles were used, although in the ABS tests they were flocculated with anionic polymer PolyDADMAC at 0.5 ppm. The multi-frequency Acoustic Backscatter System (ABS) employed was an Aquascat 1000 from Aquatek acoustics. It consists of 1, 2, 4 & 5 MHz transducers connected via cables to a controller box and computer. As opposed to the UVP, which converts the Doppler shift of echo signals to produce quantitative settling rates, the ABS more qualitatively measures the amplitude (or backscatter strength) of an ultrasonic pulse sent through a dispersion. Again, the ABS discretises the return echo (using the speed of sound in liquid) to produce a 1-dimensional profile through the suspension. The ABS was set to scan in 2.5 mm 'bins' and a distance profile of 40 – 220 mm from the transducer.

For the ABS studies, a 5 L beaker was used as a small settling tank with the ultrasonic transducers positioned $\sim 180 \text{ mm}$ above the beaker base. The experimental programme consisted

of two series of tests. Firstly, concentration correlations were made using dispersions from 2 – 10 wt%. The ABS was set to take 5 second averages through the homogenous dispersions to compare the backscattered acoustic signal as a function of concentration. Preliminary results indicated 5 s averages were statistically sufficient, but the time-step was small enough to ensure there would be no bulk changes within the suspension during measurement. Secondly, the settling of particle dispersions was measured using the ABS. Here, dispersions at 5 wt% were left to settle for 10 minutes and the acoustic response was recorded throughout the sedimentation. 5 s averages were again used.

It is noted that although results were recorded for all transducers with frequencies 1 – 5 MHz, only the 5 MHz results are shown for concision, as it was found this frequency best highlighted the dispersion changes.

Sludge Yield Stress Measurements with a Quartz Crystal Microbalance (QCM) and Rheokinisis™ Dip Probe

To measure *in situ* changes to sludge yield stresses, a method and dip probe developed by the University of Leeds and patented by Rheokinisis™ [10] was employed. It consists of a 5 MHz piezoelectric QCM crystal system embedded flush into a Perspex holder designed to be inserted vertically into sludges. QCM crystals are similar to the piezoelectric crystals used in ultrasonic transducers. Whereas ultrasonic transducers apply a voltage over the crystals to produce a compression (audio) wave, the QCM applies a voltage laterally to cause the crystal to vibrate at a certain frequency. The Rheokinisis system used a 5 MHz crystal, attached by the probe to a ‘QCM-200’ frequency generator and logger from SRS Instruments. Yield stress behavior is essentially correlated from changes to the vibrational frequency and resistance of the crystal, as it is inserted into sludges of varying properties.

Two experimental systems were investigated. Dispersions of Versamag fine magnesium hydroxide powder (from Rohm & Haas) were made at various concentrations between 30 and 50 wt%. QCM frequency and resistance changes were tracked between the different dispersions (using the crystal response in air as a standard comparison). The overall behavior trend was compared to Versamag yield stress measurements in the same concentration regime, using the standard ‘Vane’ blade method [11] with a Brookfield DV-II Pro viscometer. Secondly, the aging of magnesium hydroxide Legacy simulant sludges at 45 wt% concentration was measured with the Rheokinisis probe. The simulant sludges were supplied by National Nuclear Labs (NNL) Workington, U.K, and consist of a defined mixture of fine graded magnesium hydroxide. Here, probe frequency and resistance changes were measured for a non-disturbed sludge undergoing aging over the course of a week.

RESULTS & DISCUSSION

Particulate Sedimentation Measured with the UVP

Fig. 1 shows a ‘color map’ of a particular settling experiment recorded with the UVP. The color plot is an internally calibrated 2-dimensional velocity profile map, which displays tracked changes to instantaneous velocity values down the settling column with time. Different velocity regimes are distinguished with different color profiles, where ‘green’ shades indicate small

negative values (i.e. particles settling down the column), yellow shades small positive values (i.e. particle or fluid 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. Given in the insert of Fig. 1 is the Turbiscan data for comparison, showing the change in the cloud-front interface (upper graph) and bed thickness from a zero basis (lower graph).

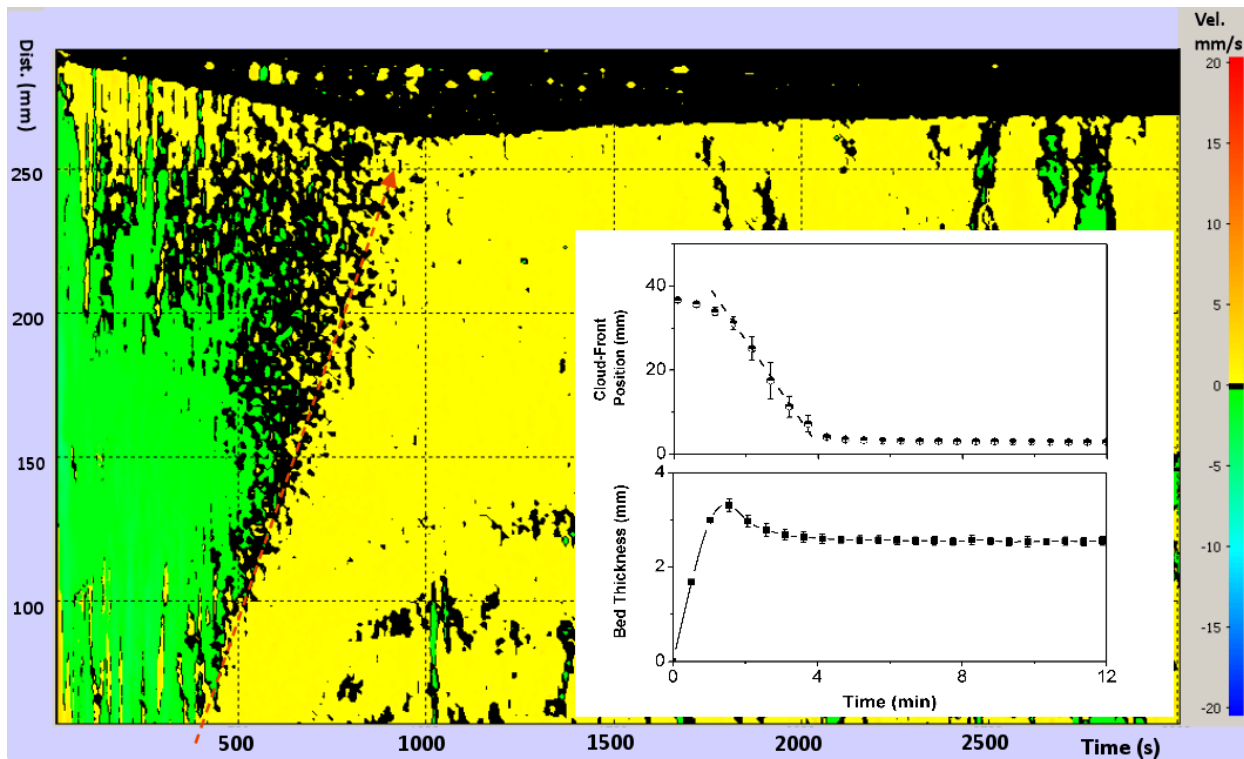


Fig. 1. Velocity ‘color map’ showing changing instantaneous velocity profiles down a settling column with time for a 5 wt% coagulated Spherglass dispersion, as measured by the UVP. The chart bar is expressed in mm/s. Orange dotted arrow indicates settling cloud-front. Insert: Turbiscan analysis of the same system showing the consolidated sediment bed (lower graph) and cloud-front (upper graph).

The color plot shown in Fig. 1 actually contains a considerable amount of detail. Firstly, average settling velocities were analyzed from the middle of the column (~150 – 200 mm from the transducer) and measured as ~ 1 – 3 mm/s, which is in line with theoretical Stokes estimates based on the likely size of the aggregates. It is noted that velocity values were analyzed within the first 500 s, as the particle dispersion had completely settled out within 800 – 1000 s. Also in the image, the evolution of the sediment bed is clearly seen being tracked from the black (zero) velocity region at the top of the image. Interestingly, it seems initially there are a number of non-zero velocity readings from inside the bed as it is formed, signifying there is rearrangement of the sediment aggregates. These areas of non-zero velocity inside the bed are reduced as time increases, suggesting perhaps some longer-term bed compaction. Indeed, the ‘black’ region at the top of the image representing the stationary bed clearly reduces in thickness after 1000 s, consistent with bed compression.

Further information on sedimentation can also be gained from the color map. Because of the aggregated nature of the particle dispersions, they tended to settle with a clear upper supernatant region separated by a dispersion cloud-front. This is observed by a distinct change in measured velocity values, between the supernatant water, which is essentially in up-flow, and the dispersion interface that is moving down. The color map shows this behavior in the yellow/black front that forms in the dispersion diagonally down the column image over time, highlighted by the arrow (again within the first 1000 seconds). The linear slope of this line (which is ~ 0.35 mm/s) correlates to the settling flux rate of the cloud-front. In addition to the cloud-front, some insight into the nature of the settling behavior within the dispersion can be gained from the color map. There is a region on top of the sedimented bed where measured particle 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 1000 s settling period). This change in the near-bed velocities is consistent with hindered-settling effects within the dispersion, likely 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 [12]. 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. It is lastly noted that as all the particles had settled out after 1000 s, the calculated velocity measurements from within the bulk dispersion after this time are only related to system noise.

The results from the UVP trials also compared favorably to the settling behavior as measured from the Turbiscan analysis (Fig. 1 insert). The Turbiscan uses 20 mL sample vials, hence the settling volumes do not compare, meaning that the time scale for sedimentation was much smaller. Nevertheless, the general settling trends shown by the cloud-front and bed analysis are in agreement with the UVP results. The Turbiscan analysis for the bed profile (Fig. 1 insert, lower graph) highlights the same behavior, of an initially fast settling larger bed (here displayed from a zero basis) that compresses over time to an equilibrium thickness. By analysis of the linear portion of the cloud-front movement (Fig. 1 insert, upper graph) the settling flux was calculated as ~ 0.25 mm/s. This is slightly lower than the cloud-front settling flux measured from the UVP; however, it only equates to a shift in theoretical particle settling sizes of around $5 \mu\text{m}$. Due to the large size distribution of the coagulated particles, such differences are well within those expected from experimental variation, noting the color plot shows the analysis of one particular settling run, whereas the Turbiscan data is averaged over a number of runs. 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.

Particulate Settling and Concentration Changes Measured with the ABS

The main focus of the ABS studies was to ascertain additional characterization information on settling dispersions not available from the UVP; namely, concentration correlations. Being able to measure concentration changes in suspensions would be extremely advantageous for operators monitoring sludge retrieval, as bulk conditions must be closely controlled due to the potential for sludge build-up in pumps and pipe work. Also, it was hoped that measuring concentration changes would confirm any hindered settling effects in aggregated Spheriglass dispersions indicated by the UVP analysis.

To measure the concentration within a settling dispersion, firstly the attenuation of the ABS through different equilibrium suspensions from 2 – 10 wt% was analyzed to create a correlation plot. It was found that at each concentration in this range, the backscatter strength was attenuated in a linear fashion with dispersion depth, where the gradient slope increased with concentration. This type of linear regression profile is common for multi-phase dispersions under certain concentration thresholds [13, 14]. As discussed in more detail in a recent paper for the DIAMOND nuclear consortium [15], a plot was generated to allow the *in situ* calculation of Spheriglass concentration, based on the slope of acoustic attenuation through the dispersion (assuming homogenous mixing).

The acoustic response of the ABS through a 5 wt% settling flocculated Spheriglass dispersion was analyzed at various time periods within a 10 minute settling run, and resultant 5 second average attenuation profiles are shown in Fig. 2 a). It is noted firstly that the 1 – 5 s signal demonstrates a similar response to the equilibrium concentration profiles as discussed (and reported previously [15]) with the signal strength being attenuated in a linear fashion with depth, until a peak is observed at 180 mm, which represents the scattering from the beaker base. As particle sedimentation commences, a clear second peak is seen in the signal nearer the transducer, moving down the settling cylinder with time (highlighted by the black diamonds, for the time averages between 41 – 45 and 141 – 145 s). These peaks represent the movement of the cloud-front, above which a clear supernatant formed. This was correlated by manual observations of the cloud-front movement with time. A peak is generated, as the clear water in the supernatant zone will produce a very weak signal, while the cloud-front will act as a strong ‘surface’ to scatter the ultrasonic pulse.

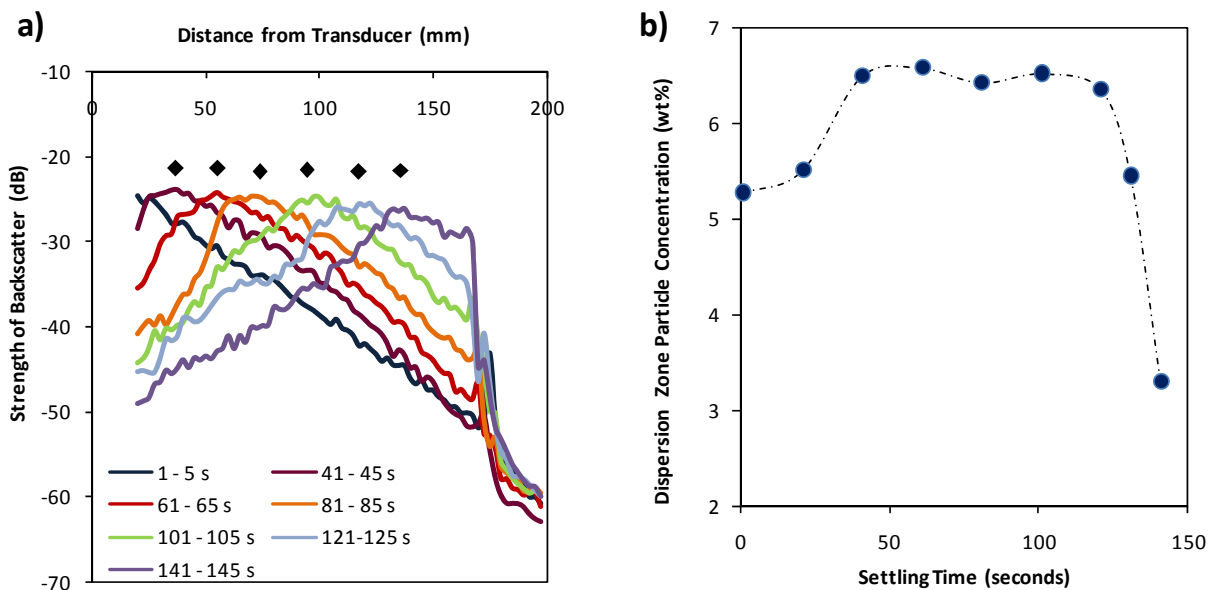


Fig. 2. Acoustic backscatter profiles at various times (5 second averages) through a settling 5 wt% flocculated Spheriglass dispersion ‘a)’, and the correlated change to particle concentration within the dispersion zone during the same time period ‘b)’. The black diamonds in ‘a)’ represent the movement of the cloud-front peak during sedimentation.

The raw attenuation profiles were then analyzed to observe any changes to concentration within the dispersion zone during sedimentation. This was completed by approximating the acoustic response through the dispersion at different times (from the cloud-front peak to the beaker base) as linear profiles and calculating the gradient slope. These values were then compared to the correlation plot and the changes to the gradient slopes converted to dispersion concentrations. The resulting behavior is shown in Fig 2 b), which displays how the particulate concentration changes within the dispersion zone during the settling run. It is emphasized that by ‘dispersion zone’, we mean the area from the cloud-front to the sediment bed. The concentration clearly increases in this zone from the initial homogeneously mixed 5 wt% to ~6.7 wt% during settling, before it reduces sharply near the completion of sedimentation. This reduction in concentration can simply be explained by the fact that at these later time periods much of the dispersion would have already sedimented. As the suspension contains quite a broad particulate size distribution, it is likely that there will be some segregation over time as the larger particulates settle faster, which will eventually lead to a reduction in dispersion zone concentration. The initial increase in concentration is more pertinent however, and again points to hindered settling effects within the dispersion zone. As falling particulates interfere with the flow-fields of their neighbors, settling rates will drop over time (as observed with the UVP in the near-bed region) and this hold up will in-turn lead to a buildup of particulates, increasing the bulk concentration.

The homogeneity of the dispersion over time was considered by looking at the linear fit of the attenuation profiles, which were used to correlate the concentration changes. If the settling dispersions contained significant segregation overtime, it was assumed that the profiles would no longer fit a linear trend. Indeed, at long time periods when segregation would be most pronounced (above 120 s) the R^2 fit values (Taken simply from an Excel linear trend-line fit) were reduced (<0.97); although for most time periods, R^2 values were $>.99$, indicating a good fit. It is noted that at 41 – 45 s and 61 – 65 s (when the dispersion zone concentration is first observed to increase) the linear R^2 values also were below levels considered a good fit (<0.98). It is thought that perhaps initially dispersion segregation increases with larger particulates settling lower into the dispersion until hindered settling interactions effectively slow down these larger particulates and reduce the segregation to some extent. Nevertheless, in general terms, the R^2 linear fit values were high enough to give confidence that the correlated concentration changes driven by hindered settling effects were a real and a significant feature of the settling system, as evidenced by both the UVP and ABS studies.

Sludge Yield Stress Measurements with the QCM and RheokinisisTM Dip Probe

Fig. 3 shows the results of the Versamag QCM correlations, comparing the frequency shift (Fig. 3 a)) and the crystal resistance (Fig. 3 b)) measured by the Rheokinisis dip probe to the same yield stress data (as measured using the vane method) across a sludge concentration range of 25 – 50 wt%. It is noted that the frequency results are displayed as the ‘frequency shift’ from the crystal oscillation frequency in air (at around 5 MHz).

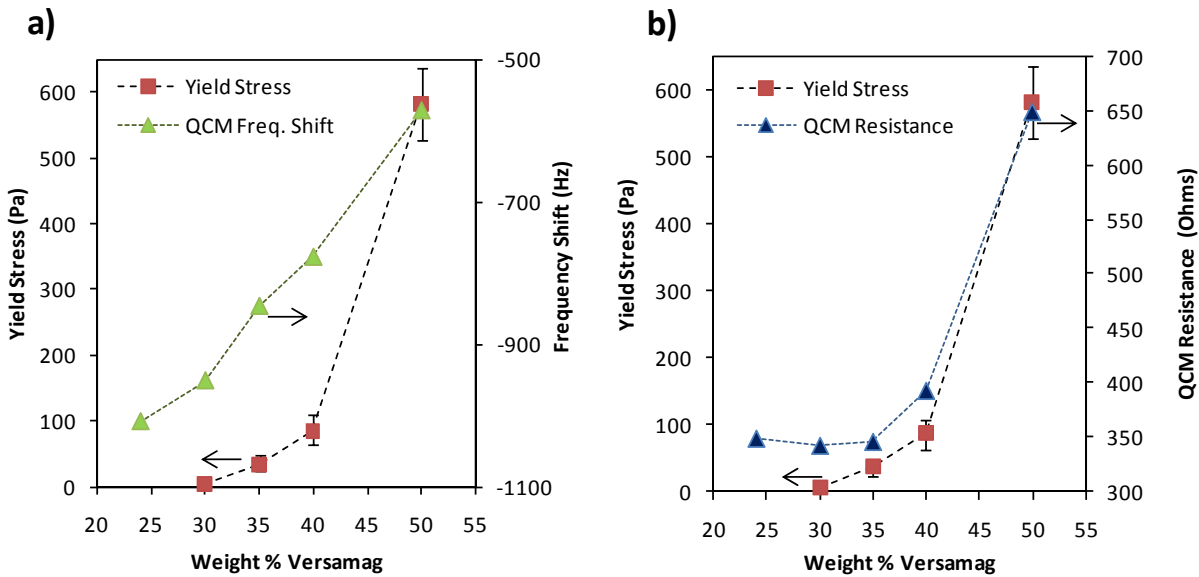


Fig. 3. Comparison between yield stress data obtained from a Brookfield viscometer for Versamag sludges between 30 and 50 wt% with QCM frequency shift ‘a’) and crystal resistance ‘b’) for a Rheokinisis dip probe in the same sludges. Note the dotted lines are to help guide the eye only.

It is observed in Fig. 3 that both the base frequency of vibration increases (i.e. frequency shift reduces) as well as the crystal resistance, correlating with the change in Versamag yield stress, although there are some differences between behavior that will be discussed. It is noted firstly that the shear stress behavior is loosely exponential in nature, with a critical point where yield increases significantly between 40 and 50 wt%. The frequency response of the QCM crystal to the changes in the sludge yield properties is however a fairly gradual and continual increase, with no distinct behavior change around this critical point of 40-50 wt%. Theoretically, the frequency behavior of the crystal in multiphase systems is linked to the extent of ordering within the system. Multiphase fluids will naturally dampen the crystal and reduce the frequency of oscillation. However, these changes are linked to the dissipation mechanics of the system. If systems are highly structured around the crystal (such as in the case of high yield dispersions) the number of dissipation pathways increases and the vibration dampening will be less pronounced (and resulting vibrating frequency will be greater). Although this trend is tracked in the Versamag studies, complications arise from dissecting the ordering effects from the increase in yield to the effects from simply increases in the bulk particulate concentration.

The changes to the crystal resistance perhaps more closely track the changes to the Versamag yield properties, with a significant increase occurring between 40 – 50 wt%, similar to the critical change in yield stress. Although very promising results, there are a number of problems with quantitatively linking changes from multiphase properties to the theoretical resistance changes. It is known that high viscosity Newtonian liquids increase crystal resistance, by not only altering the base frequency, but also dampening the resonant oscillation, a viscous loss that is manifested in an increase to the series resonance resistance [16]. How this theory is transcribed to non-Newtonian fluids and generally multiphase systems is complicated, and future work is planned to better isolate the effects of different system properties (such as concentration, yield stress and bulk viscosity). Nevertheless, qualitatively, the use of both frequency and

resistance to monitor sludge yield changes has been shown here to be potentially extremely useful, especially as crystals are relatively cheap and disposable. Additionally, as with the ultrasonic sensors, the probe and crystal can be separated from the control logger, allowing their use in a multitude of waste environments.

To further investigate the QCM technique to correlate yield stress changes, and to highlight the probes potential use as a constant on-line monitor, the effect of bed aging on the frequency and resistance response was measured. Here, the 45 wt% magnesium hydroxide sludge was prepared and an initial QCM reading was made after 1 hour. QCM readings were again performed after 24 hours and 6 days to highlight any changes from significant bed consolidation. The results are shown in Fig.4, with frequency changes on the right hand axis and resistance changes on the left.

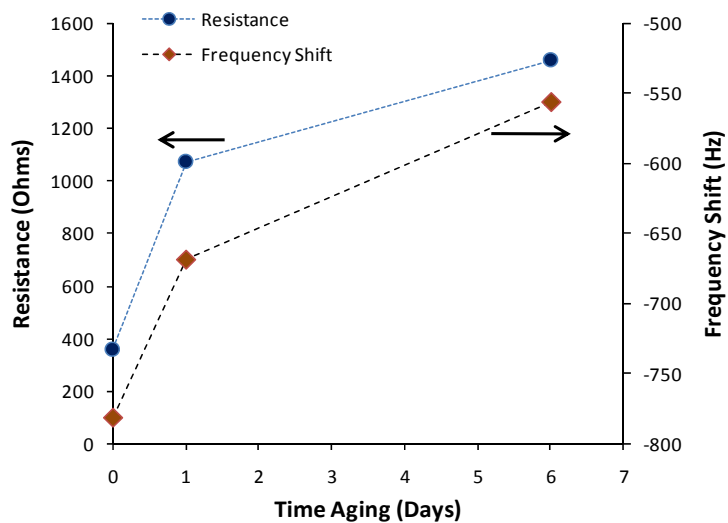


Fig. 4. Effect of 45 wt% magnesium hydroxide sludge consolidation on QCM resistance (left) and frequency shift (right) over 6 days. Note the dotted lines are to guide the eye only.

Both the oscillation frequency and crystal resistance in the sludge increase over time. Previous work from National Nuclear Labs (Workington, U.K) had established that these hydroxide sludges show significant increases in yield stress (due to bed aging) over similar time periods, and the QCM results correlate very close with these changes (although, unfortunately due to confidentiality issues, the exact yield stress data was not supplied with the hydroxide samples). Again, the relative changes in the crystal resistance were more significant than the frequency shift, with the frequency increasing in real terms by ~250 Hz (or a change of ~30%) whereas the resistance increased by 900 Ohms (or change of over 300 %). As with the Versamag tests quantitative assessment is difficult, but this evidence highlights the QCM probes potential to at least qualitatively measure differences relating to critical changes in sludge behavior.

CONCLUSIONS

Work completed within the University of Leeds as part of the DIAMOND consortium sort to ascertain the effectiveness of two different ultrasonic sensor types and a novel QCM probe to characterize simulant nuclear wastes, for potential use in 'Legacy' deposits. It was found that

the ultrasonic velocity profiler was able to measure the settling of aggregated glass powder dispersions with results comparing closely to standard turbidity analysis of the sedimentation behavior. As well as bulk settling velocities, the UVP allowed the monitoring of the growth and consolidation of the sediment bed, and also, information was gained on the movement of the cloud-front. Additionally, velocity changes within the near bed region indicated development of hindered settling in the lower portions of the column.

The acoustic backscatter sensor was able to provide complimentary information on the settling profile of aggregated dispersions, with attenuation profiles highlighting concentration changes within the dispersion zone during settling, and again significantly, showing particulate build up from development of hindered settling regimes. Lastly, a quartz crystal microbalance probe developed by Rheokinisis was used to measure changes to consolidated sludge matrix yield stresses for magnesium hydroxide slurries, similar to those encountered within the Legacy deposits. Correlations were found between an increase in yield stress (associated with both changes in magnesium hydroxide concentration and bed aging) to increases in the QCM crystal frequency and resistance.

Collectively, work here has shown the potential for *in situ* sensors to give detailed information on the behavior of multiphase dispersions that may be invaluable for future processing of Legacy wastes. The ability to correlate the sludge yield stress and ageing in the Legacy ponds (and during future transportation, interim storage and dewatering) will give operators the ability to assess the erosion and re-dispersion potential of the sludges; while the ability to measure dispersion setting rates, segregation and concentration will allow operators to assess for potential hazardous conditions upon redispersion. These techniques may also be useful in a multitude of waste environments, where sample analysis is difficult or dangerous.

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