

# Physical Characterization of Solid-Liquid Slurries at High Weight Fractions Using Optical and Ultrasonic Methods

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## Research Objective

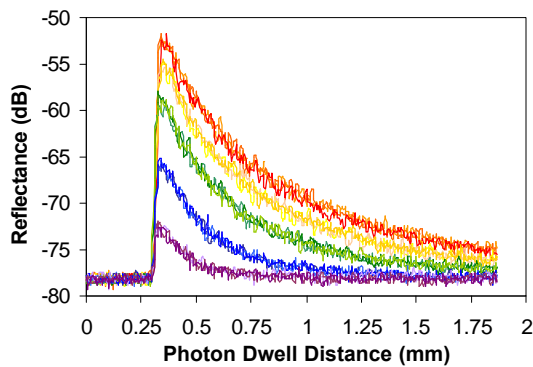
The goal of this proposed work is to directly address the need for rapid on-line characterization of the physical properties of HLW slurries during all phases of the remediation process, from in-tank characterization of sediments to monitoring of the concentration, particle size, and degree of agglomeration and gelation of slurries during transport. This will be done with both optical and ultrasonic methods. There are three tasks: 1) develop optical and acoustic measurements to provide the fundamental science needed for successful device development and implementation, 2) develop theories that describe the interrelationship between wave propagation and the physical properties of the slurry, and 3) solve, in the framework of these theories, the inversion problem and compare them with the experimental measurements to non-intrusively characterize slurries.

## Research Progress and Implications

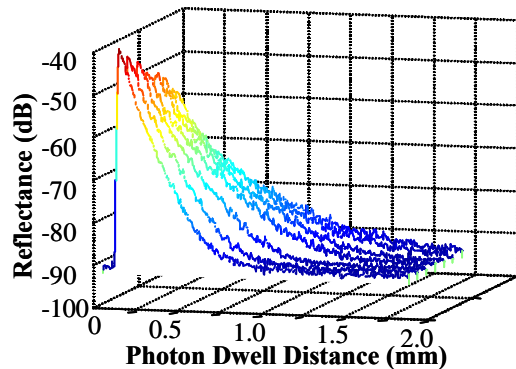
The optical measurements are based on the investigation of slurries with Optical Low-Coherence Reflectometry (OLCR). OLCR is a white-light interferometric technique that incorporates a broadband light source with a classical Michelson

interferometer. In previous works<sup>1</sup> we have demonstrated that OLCR is an effective tool for nondestructive analysis of multiscattering systems.

During this FY, the optical work focused upon using standard, monodispersed polystyrene nanospheres purchased from Duke Scientific Corporation to act as a model for the slurries. Standards are available in the smaller size range only at 1% by weight, while larger spheres can be obtained at higher concentrations. The 180° configuration of the OLCR system determines that only backscattered light is detected, and thus the signal intensity increases with increasing concentration. The OLCR signal is on a logarithmic scale and we have worked previously with scattering systems up to 70 wt % solids. Tailing decay profiles are formed which contain information about the properties and distribution of the material making up the scattering matrix. Figure 1 shows the profiles for nanospheres ranging in size from 20 nm (the bottom profiles) to 100 nm (the uppermost profiles).



**Figure 1. OLCR decay profiles from 1 wt % monodispersed polystyrene nanospheres, increasing in size by 20 nm increments from 20 nm to 100 nm.**



**Figure 2. OLCR decay profiles from 308 nm monodispersed polystyrene nanospheres, ranging in concentration from 0.25 % to 10 wt %.**

The decay pattern can be split into three sections for modeling purposes, based upon correlation length and density of fluctuations of particle coordinates. We believe that the OLCR signal in the first section is due to surface characteristics of the material. The second portion can be interpreted as a linear decay, which is related to photons scattered within the material (weak localization). We have interpreted the last portion of the decay profile, a double exponential, to be related to strong localization, or photons which are trapped within the matrix and so are delayed from returning, still in phase, to the detector.

We have demonstrated that information regarding particle size and concentration can be extracted from the decay profiles. The maximum amplitude of the leading edge, caused by the initial interaction between the light and the matrix, is proportional to the mean size and concentration of heterogeneities present in the system. Figure 2 shows

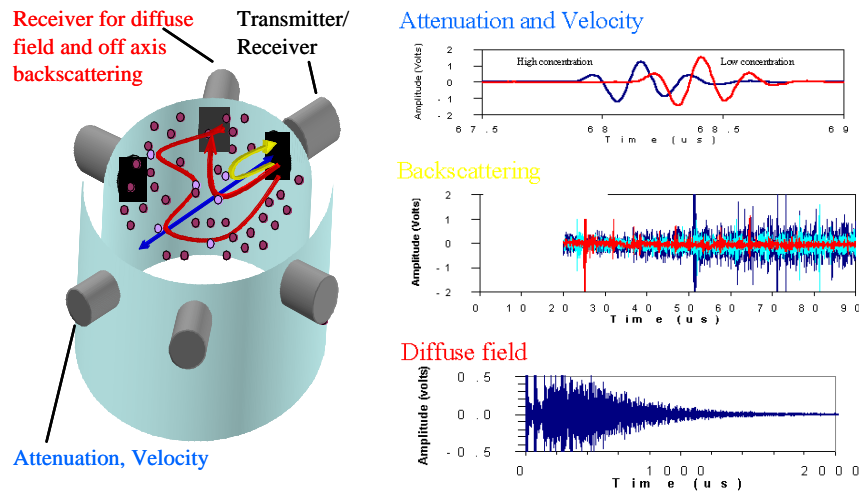
<sup>1</sup> Thurber, S.R., A.M. Brodsky, and L.W. Burgess. 2000. "Characterization of Random Media by Low Coherence Interferometry." *Applied Spectroscopy*, 54(10), 1506-1514.

Thurber, S.R., L.W. Burgess, A. Brodsky, and P.H. Shelley. 2000. "Low-Coherence Interferometry in Random Media II. Experiment." *Journal of the Optical Society of America A*, 17(11), 2034-2039.

Brodsky, A., Thurber, S.R., and L.W. Burgess. 2000. "Low-Coherence Interferometry in Random Media I. Theory." *Journal of the Optical Society of America A*, 17(11), 2024-2033.

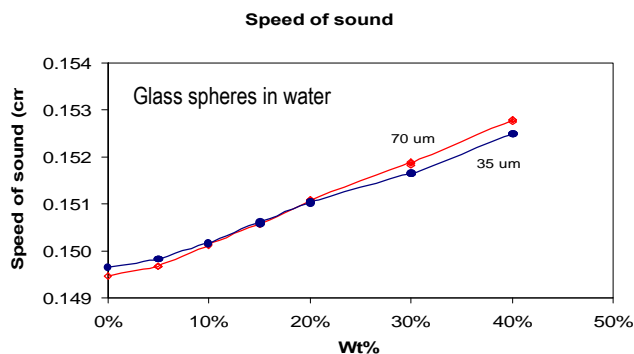
that the shape, as well as the maximum intensity, of the profiles changes with concentration. The profile can also be split into two pieces for further analysis. The derivative of the beginning section of the profile is proportional to  $1/\text{particle radius squared}$ . The derivative of the second half is directly proportional to particle size. Additional information about system clustering characteristics can be gained from investigating the fluctuation of the signal from the mean throughout the profile. Current and future work will apply the same data analysis tools to model polydispersed systems.

During this FY we have also been developing ultrasonic measurement methodologies to characterize solid liquid suspensions at concentrations up to 40 wt%. Ultrasonic measurements have the beneficial feature that the waves penetrate deep into materials and produce an average property. We have focused ultrasonic backscattering measurements that have the best opportunity to provide quantitative results at the high concentration expected to occur in the waste remediation process. A schematic of these measurements is provided in Figure 3. The ultrasonic measurements included the speed

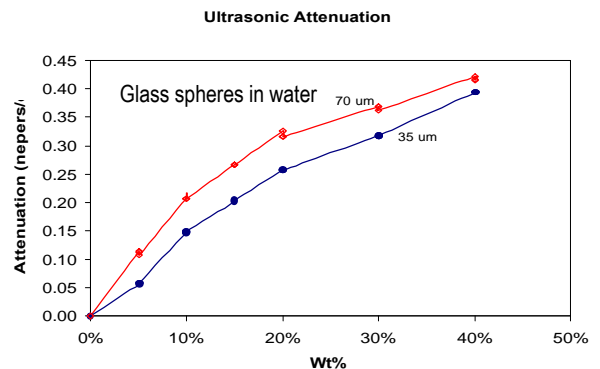


**Figure 3. Schematic illustration of ultrasonic measurements and the resulting signals for each.**

of sound (Figure 4), the attenuation (Figure 5), the backscattering (Figure 6) and the diffuse field (Figures 7a and 7b). Notice the similarity in signal shape between the diffuse field acoustic measurement and the OLCR signal. The speed of sound and the attenuation were used to characterize the slurries and as inputs for analysis of the



**Figure 4. Speed of Sound vs. Concentration.**



**Figure 5. Ultrasonic Attenuation vs. Concentration.**

backscattering data. Current results for glass spheres in water indicate that the ultrasonic measurements are very sensitive to both particle size and concentration.

The backscattering and diffuse field measurements are especially appealing because of the relative simplicity of the theoretical description of the scattering processes.

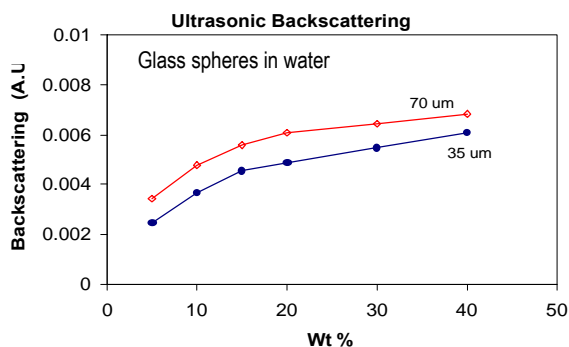


Figure 6. Ultrasonic Backscattering vs. Concentration

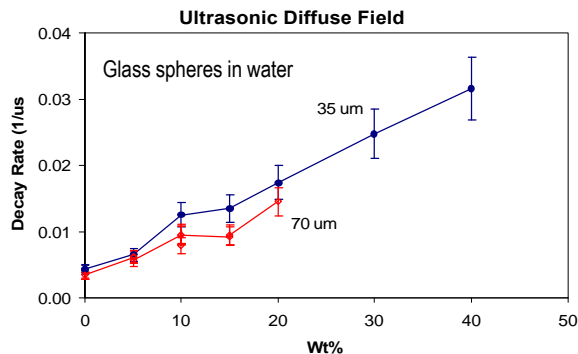


Figure 7a. Diffuse field decay rate vs. Concentration

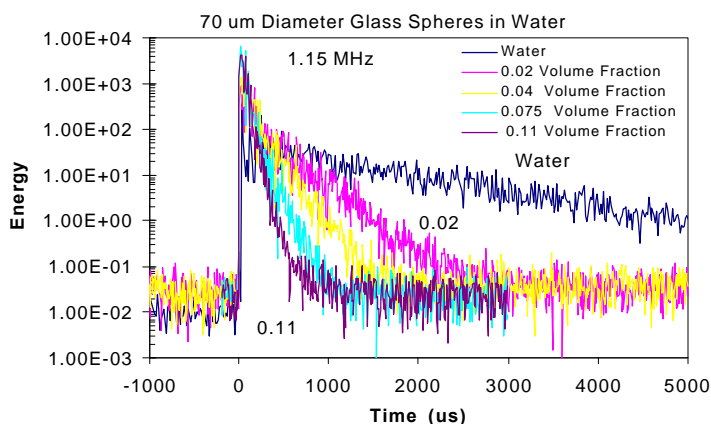


Figure 7b. Diffuse field as a function of particle concentration.

Future plans include extending existing theories to include multiple scattering and particle-particle interactions, which occur at high concentrations. It will then be possible to compare the developed acoustic and optical theory with the experimental results. Theoretical work has been started on the coherence effects in acoustic scattering by nonuniform media. The main goal of the calculations is to determine the expressions which will allow a solution to be found for the inverse problem—characterization of studied media (including dense slurries) by acoustic scattering data. The applied theoretical technique is analogous to that previously exploited by us in optics<sup>1</sup> and applied to the analysis of optical low-coherence reflectometry.

Several meetings have taken place to discuss data and future coordination between the two research areas. Progress has also been presented twice at the semiannual sponsor meetings of the Center for Process Analytical Chemistry.

**Planned Activities**

Upcoming work will include evaluation of polydispersed standard systems for both the optical and acoustic methods. The polydispersed systems will be formulated by mixing several of the previously analyzed monodispersed systems to provide average, and known, particle characteristics. Then studies will be performed upon individual slurry surrogate components. Eventually we will analyze a complex matrix composed of multiple surrogate components with both optical and acoustic techniques.