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In Situ Imaging and Spectroscopy of Particles in Liquid

Xiao-Ying Yu[1], Bruce Arey[2], Zihua Zhu[3], Juan Yao[1], Fei Zhang[1], Sayandev Chatterjee[2], and Jaehun Chun[2]

[1.] Earth and Biological Sciences Directorate, Pacific Northwest National Laboratory (PNNL), Richland, WA 99354, USA.
[2.] Energy and Environment Directorate, PNNL, WA 99354, USA.
[3.] W. R & Wiley Environmental Molecular Science Laboratory, PNNL, Richland, WA 99354, USA.

This work presents an example of in situ imaging of particles, suspended in liquid, in a vacuum compatible microfluidic sample holder using a suite of tools including scanning electron microscopy (SEM) and time-of-flight secondary ion mass spectrometry (ToF-SIMS), highlighting the advantage of multiscale analysis in material sciences. Nanometer-sized boehmite (AlOOH) particles synthesized at our laboratory are used as a model system in this work [1]. Such particles exist in high-level radioactive wastes at the Hanford site. It is known that they are difficult to dissolve and cause rheological problems for processing in the nuclear waste treatment plant. Therefore, it is important to build the capability to characterize boehmite particles suspended in liquid.

A transferrable and vacuum compatible microfluidic interface, System for Analysis at the Liquid Vacuum Interface (SALVI), was used in this study. SALVI enabled surface analysis of liquids and liquid-solid interactions using ToF-SIMS and SEM [2, 3]. Its detection window is an aperture of 2 μm in diameter open to vacuum, permitting direct detection of the liquid surface. Liquid is withheld by surface tension within the aperture. The interface is composed of a silicon nitride membrane and polydimethylsiloxane microchannel [2, 3]. A variety of samples including complex liquid mixtures, ionic liquids, single mammalian cells, live biofilms, and solid-electrolyte interface (SEI) have been analyzed using in situ imaging [4, 5]. The feasibility of using SALVI for in situ characterization of nanoparticles in liquid was demonstrated in our previous work [6]. This paper shows the recent results of multimodal analysis of synthesized polydisperse boehmite particles under natural and caustic alkaline pH conditions in deionized (DI) water.

High vacuum conditions were employed in liquid SEM (FEI Quanta). Figure 1 depicts SEM imaging results of boehmite particles under natural and alkaline pH conditions in the microchannel. Optimized conditions for secondary electron (SE) images are obtained using 8 kV. The SE images can be used to study particle size and morphology. The synthesized particle size ranges from 30 nm – 100 nm. In situ liquid SEM provides descriptions of particle size, shape, morphology enhanced with elemental mapping. In situ liquid SIMS is used to study particle molecular structure and composition. The solvent microenvironment can also be captured using liquid SIMS. Figure 2 depicts a comparison of negative ion mass spectra acquired using in situ liquid ToF-SIMS (IONTOF GmbH, TOF-SIMS V) under natural and alkaline conditions. The latter is used to simulate caustic conditions found in the nuclear tank wastes. There is a difference in the m/z spectra in natural and alkaline pH conditions. It is known that the solubility of boehmite depends on pH [1]. The mass spectra provide direct evidence of this phenomenon. Both water clusters labelled in red marks and representative peaks vary as a result of pH change. SIMS provides detailed submicron molecular mapping of the particle and its surrounding water cluster environments as well as molecular identification of small molecules in liquid. These new results demonstrate advancements of in situ correlative imaging of liquid surfaces and solid-liquid interfaces using a universal microfluidic interface, SALVI [2, 3].

References:

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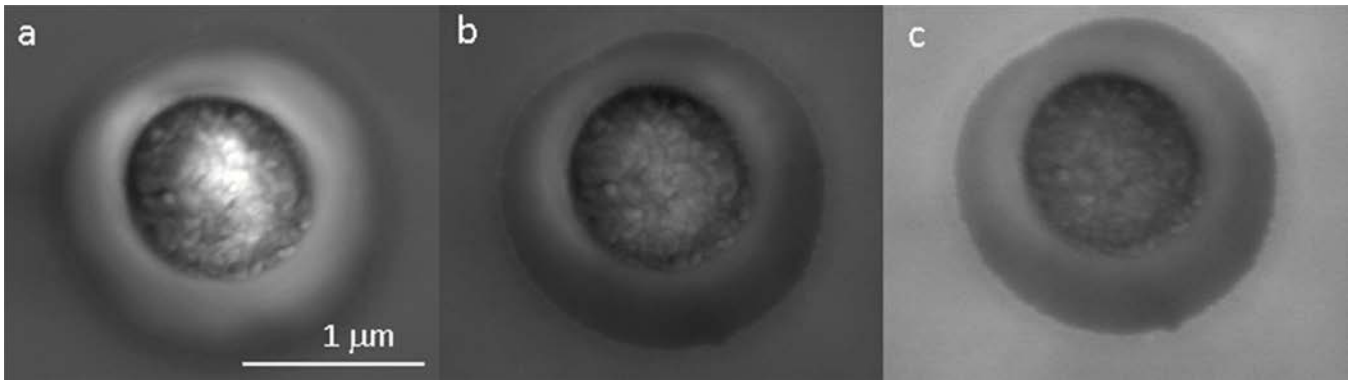

Figure 1. Liquid SEM SE imaging of boehmite particles in DI water: (a) 5 kV; (b) 8 kV; and (c) 10 kV.

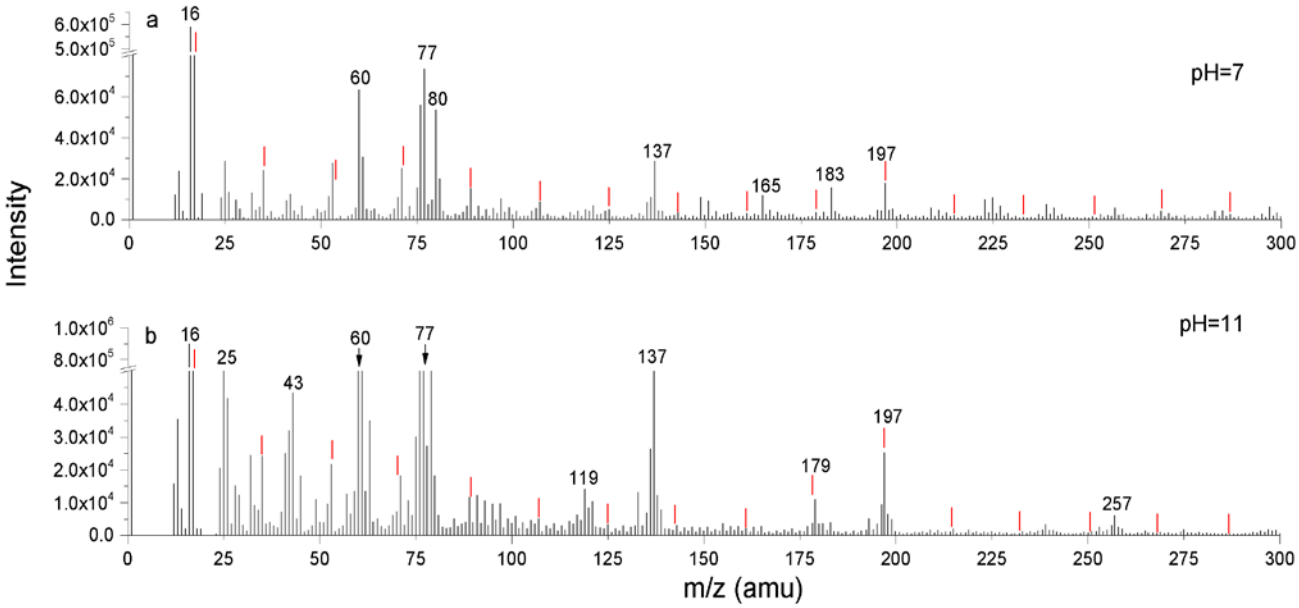

Figure 2. Negative m/z spectra of synthesized boehmite particles in DI water by ToF-SIMS: (a) natural pH and (b) alkaline pH. Red bars indicate locations of water clusters.