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Sensitivity of EXAFS to Crystallite Size

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Introduction: With the recent focus on nanoparticle synthesis, the problem of determining the size distribution of a nanoparticulate sample is becoming increasingly important. While many methods exist, including electron microscopy, Scherrer broadening of x-ray diffractograms, dynamic light scattering, and Brunauer-Emmet-Teller analysis, each requires different sample characteristics and each reacts differently to samples that comprise nanoparticles with a range of sizes.

Extended x-ray absorption fine structure (EXAFS) spectroscopy is also sensitive to the size (and, to some extent, the shape) of nanoparticles [1]. We are investigating this dependence for two purposes. First, EXAFS shows promise as a complementary tool for determining particle size. Second, EXAFS is currently used to analyze the structure of nanoparticles; readily implemented corrections for the sizes of the particle will improve these analyses.

Methods and Materials: Transmission EXAFS spectroscopy has been conducted on a series of palladium nanoparticles with different size distributions synthesized by nonaqueous precipitation, as well as on an iron-nickel composite in which the nickel crystallites possessed a wide distribution of sizes. In both cases the smallest crystallites were under three nanometers in diameter.

Results: For small particles, the Fourier transform of the EXAFS spectrum is damped in a predictable way. Because this reduction is dependent on particle size in a nonlinear way, EXAFS analyses that assume a single particle size tend to pick out particles at the low end of the size distribution. Based on our study of palladium nanoparticles, it appears that EXAFS can correctly rank the average sizes of two distributions even when the mean of the distributions differ by less than their standard deviations.

Conclusions: EXAFS may prove to be a useful tool in determining the sizes of nanoparticles, but more investigation remains to be done, particularly on sample comprising nanoparticles with a wide range of sizes. In particular, EXAFS shows sensitivity to the smaller particles in the sample, while most other techniques for determining particle size tend to favor the larger particles. EXAFS may thus be useful for detecting the presence of small crystallites that would be overlooked by other techniques.

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References: [1] A. I. Frenkel, C. W. Hills and R. G. Nuzzo, "A View from the Inside: Complexity in the Atomic Scale Ordering of Supported Metal Nanoparticles," J. Phys. Chem. *B* **105**, *12689-12703*.