Accelerating Nanoparticle Synthetic Development Using In Situ Small Angle X-ray Scattering

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The fine control of monodisperse nanoparticles (NPs) over their size, shape, and structure is essential for understanding their properties, especially catalytic properties. In the past two decades, significant developments have been achieved in the colloidal synthesis of monodisperse NPs. However, synthesizing well-defined NPs in a predictable way is still challenging. Here, we use synchrotron-based in situ small-angle X-ray scattering (SAXS) to experimentally monitor the formation of NPs during solution phase synthesis. We choose Pd NPs as an example. Through quantitative analysis of the SAXS data, the number, size, and size distribution of Pd NPs during the synthesis are determined. By real-time probing the evolution of Pd NPs, we systematically study the effects of different surfactants (e.g., oleylamine, trioctylphosphine) on the kinetics of NP nucleation and growth. Taking advantage of this in-situ SAXS technique, we are able to better understand the growth mechanism of Pd NPs and as a result optimize the synthetic parameters to precisely control the Pd NP size from 2 to 10 nm, and these well-controlled Pd NPs serve as a model system for studying their size-dependent catalysis for methane combustion reaction.