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Mapping magneto-chemical heterogeneity in laser-synthesized bimodal colloids via synchrotron XAS and STXM

Mohammad Sadegh Shakeri, Arkadiusz Zarzycki, Zaneta Swiatkowska-Warkocka
Institute of Nuclear Physics Polish Academy of Sciences, Krakow, PL-31342 Poland
* ms.shakeri@ifj.edu.pl

Abstract
The engineering of magnetic nanomaterials with tailored stoichiometry and bimodal size distributions is critical for applications ranging from targeted drug delivery to high-contrast MRI. This study investigates a novel sub-ablation dual-synthesis regime during the laser processing of magnetite colloids. We hypothesize that a hot-skin effect triggers simultaneous surface atom shedding and confined volume fusion, resulting in a bifurcated population of ultrafine nanoclusters and submicron spheres [1]. To resolve the complex magneto-chemical gradients inherent in this non-equilibrium process, we employed a multi-modal synchrotron-based approach. Hard X-ray absorption spectroscopy (XAS) at the Fe K- edge was utilized to quantify the phase transformation between thestarting magnetite and the resulting fragmented cloud [2]. Through linear combination fitting of the XAS data, we identified a significant drop in Fe^{2+} content (from 31% to <8%) in the fragmented population, signaling a rapid, solvent-mediated oxidation into maghemite. This allowed us to correlate the laser-induced thermal gradients with the resulting cationic vacancy density. To visualize these transitions at the single-particle level, scanning transmission X-ray microscopy (STXM) was performed. STXM mapping at the Fe L2,3-edges provided spatially resolved chemical fingerprints of the bimodal distribution. By extracting local absorption spectra from the submicron fused cores and the surrounding fragmented clusters, we directly mapped the magneto-chemical heterogeneity of the colloid. STXM results confirmed that while the fused spheres maintain a magnetite-rich core, the ejected fragments undergo a complete phase shift. This synergy of XAS and STXM provides an unprecedented view of the structural and electronic evolution in laser-driven colloids, validating our predictive multiscale models and offering a blueprint for the deterministic synthesis of graded magnetic nanomaterials.

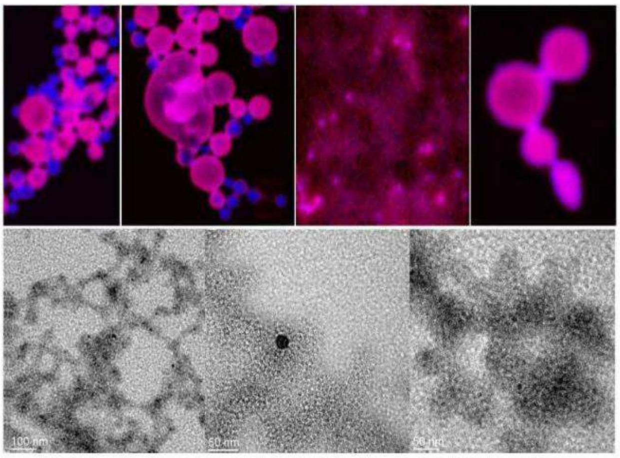

Fig. 1. STXM and HRTEM of biomodal synthesis

[1] Mohammad Sadegh Shakeri, et al. Advanced Functional Materials, 2023, 2304359.
[2] Mohammad Sadegh Shakeri, et al. Composites Part B: Engineering, 2025, 112457.