nature reviews materials https://doi.org/10.1038/s41578-025-00844-9

Supplementary information

Capturing rapid nanomaterial transformations with cross-platform operando characterization

In the format provided by the authors and unedited

Supplementary Information for

Capturing rapid nanomaterial transformations with cross-platform operando characterisation

Swaroop Chakraborty[1], Superb K. Misra[2], Jamie R. Lead[3,4], Iseult Lynch[1,5*]*

[1] School of Geography, Earth and Environmental Sciences, University of Birmingham, UK, B152TT

[2] Materials Engineering, Indian Institute of Technology, Gandhinagar, India, 382355

[3] School of Geography, Earth and Environmental Sciences, University of Plymouth, UK, PL4 8AA

[4] School of Biological and Marine Sciences, University of Plymouth, UK, PL4 8AA

[5] Centre for Environmental Research and Justice, University of Birmingham, Edgbaston, UK

*s.chakraborty@bham.ac.uk; i.lynch@bham.ac.uk

Table S1: *Comparative Overview of Advanced Cross-platform In-Operando Techniques for Capturing rapid Nanomaterial Transformations at short time scales*

Technique	Time Scale of measurement	Examples of Mechanistic Insights	Technical Constraints	Example NM Transformations
Single-Cell ICP-MS *(sc-ICP-MS)*	min–hrs	Cell associated metal quantification, dissolution kinetics at cellular scale	Cell isolation required; limited throughput, cell lysis upon sample introduction (destructive method)	Intracellular dissolution of Ag-NMs in bacteria[1]
Single-Particle ICP-MS *(sp-ICP-MS)*	min	Particle-by-particle dissolution rates, concentration profiling	No chemical speciation resolution. Multi-ion analysis possible (in parallel or sequentially)	Ag^+ ion release from metallic Ag-NMs; Distribution of NMs accumulation in cells[2]

X-ray Absorption Spectroscopy *(XAS)*	s–min	Real-time oxidation states, coordination environment at atomic scale	Synchrotron dependency; limited accessibility	Sulfidation, redox cycling ($Ce^{3+} \leftrightarrow Ce^{4+}$), phosphatidation etc.[3,4]
X-ray Free-Electron Laser *(XFEL)*	fs	Captures ultrafast bond cleavage, transient intermediate species formation	High operational costs, scarce beamtime access	Photocatalytic bond rupture in TiO_2 (UV exposure)[5]
Liquid-Cell TEM	ms	Direct visualisation of dissolution, nucleation, aggregation dynamics	Beam-induced artefacts; 2D imaging constraints	Dissolution of Ag nanoparticles[6]
Small-Angle X-ray Scattering *(SAXS)*	s	Real-time particle size evolution, aggregation kinetics	Low sensitivity for dilute suspensions	Rapid homo-/heteroaggregation[7] (e.g., CeO_2–clay systems)
Quasi-Elastic Neutron Scattering *(QENS)*	ns–μs	Molecular-level hydration dynamics, proton transport	Requires isotopic contrast (deuteration); low neutron flux	Water restructuring at NM interface[8]
In-situ Synchrotron Infrared micro spectroscopy	s	Surface functionalisation kinetics, protein corona formation mechanisms	Limited to vibrational transitions (IR-active)	Protein corona evolution on polystyrene-NMs

References:

1. Gimenez-Ingalaturre, A. C. *et al.* Performance of single-cell ICP-MS for quantitative biodistribution studies of silver interactions with bacteria. *J Anal At Spectrom* 39, 743–753 (2024).

2. Guo, Z. *et al.* Biotransformation modulates the penetration of metallic nanomaterials across an artificial blood–brain barrier model. *Proc Natl Acad Sci U S A* 118, e2105245118 (2021).

3. Thalmann, B., Voegelin, A., Sinnet, B., Morgenroth, E. & Kaegi, R. Sulfidation kinetics of silver nanoparticles reacted with metal sulfides. *Environ Sci Technol* 48, 4885–4892 (2014).

4. Larquet, C. *et al.* Synthesis of Ce2O2S and Gd2(1-y)Ce2yO2S Nanoparticles and Reactivity from in Situ X-ray Absorption Spectroscopy and X-ray Photoelectron Spectroscopy. *Inorg Chem* 56, 14227–14236 (2017).

5. Obara, Y. *et al.* Femtosecond time-resolved X-ray absorption spectroscopy of anatase TiO2 nanoparticles using XFEL. *Structural Dynamics* 4, 44033 (2017).

6. Ahn, T. Y., Hong, S. P., Kim, S. Il & Kim, Y. W. In situ liquid-cell transmission electron microscopy for direct observation of concentration-dependent growth and dissolution of silver nanoparticles. *RSC Adv* 5, 82342–82345 (2015).

7. Guénet, H. *et al.* Characterization of iron–organic matter nano-aggregate networks through a combination of SAXS/SANS and XAS analyses: impact on As binding. *Environ Sci Nano* 4, 938–954 (2017).

8. Plekhanov, M. S., Thomä, S. L. J., Magerl, A., Appel, M. & Zobel, M. Quasi-Elastic Neutron Scattering of Citrate-Capped Iron Oxide Nanoparticles: Distinguishing between Ligand, Water, and Magnetic Dynamics. *Journal of Physical Chemistry C* 128, 11661–11671 (2024).