TUTORIAL

Chemometric analysis of large XAS dataset Practical: From quick-XAS to hyperspectral imaging

Organizers : A. Beauvois, V. Briois (ROCK beamline) and A. Tougerti (S2UO)

Number of participants : 20

The development of sub-second time resolution in X-ray Absorption Spectroscopy (XAS), also refers as quick-EXAFS, allows the monitoring of chemical kinetics such as (themo)-(photo)-(electro)-catalysis reactions [1-6], battery cycling [7], nanoparticles nucleation-growth [8-9]. However, such *in situ/operando* experiments generate large volume dataset (often hundreds of spectra collected during one experiments) which poses a significant challenge for traditional analysis workflows. To overcome this bottleneck, chemometric techniques combining **Principal Component Analysis (PCA)** and **Multivariate Curve Resolution with Alternating Least Squares (MCR-ALS)** have emerged as a powerful method to quantify and extract spectra of intermediate species from XAS spectra of evolving mixtures upon reaction [1-9].
At the ROCK beamline, a user-friendly interface has been recently developed to handle the huge amount of data collected upon reaction monitoring, from data extraction to X-ray absorption spectra alignment in energy and normalization [10]. This data processing quickly creates normalized matrices containing several hundreds of X-ray absorption spectra, serving as direct inputs for MCR-ALS analysis, which is performed using free toolbox developed by the Tauler's group available on the commercial Matlab® platform [11]. To enhance accessibility, the Fastosh software, a free user-friendly platform built on MATLAB for XAS data processing and integrating the Tauler group's toolbox, has been adopted for interactive data processing [12].

This tutorial will introduce the core principles of chemometrics for XAS (PCA + MCR-ALS), followed by a hands-on session using Fastosh to analyze a Quick-XAS dataset collected during a reaction. Attendees will learn to apply MCR-ALS to extract chemically meaningful components and interpret their evolution. The tutorial will also demonstrate how powerful is the chemometric approach to handle the very huge dataset (~million of spectra) generated by the recently developed hyperspectral full-field quick-EXAFS imaging methodology at the ROCK beamline [13].

The practical requires to bring his laptop and to install the Fastosh software.
(Download : https://www.synchrotron-soleil.fr/en/beamlines/samba)

[1] W. Cassinelli, L. Martins, A. R. Passos, S. H. Pulcinelli, C. V. Santilli, A. Rochet, V. Briois, Cat. Today 229, 114 (2014).
[2] J. Hong, E. Marceau, A. Khodakov, L. Gaberova, A. Griboval-Constant, Jean-Sebastien Girardon, C. La Fontaine, V. Briois, ACS Catal. 5, 1273 (2014).
[3] H.W. P. Carvalho, S. H. Pulcinelli, C. V. Santilli, F. Leroux, F. Meneau, V. Briois, Chem. Mater. 25, 2855 (2013).
[4] A. Rochet, B. Baubet, V. Moizan, C. Pichon, V. Briois, Comptes Rendus Chimie (2016) http://dx.doi.org/10.1016/j.crci.2016.01.009.
[5] A. Voronov, A. Urakawa, W. van Beek, N. E. Tsakoumis, H. Emercih, M. Ronning, Analytica Chimica Acta, 840, 20, (2014).

[6] M. Staniuk, O. Hirsch, N. Kränzlin, R. Bölhen, W. van Beek, P. M. Abdala, D. Koziej, Chem. Mater. 26, 2086 (20134).
[7] F. Eveillard, C. Gervillié, C. Taviot-Guéhot, F. Leroux, K. Guérin, M. T. Sougrati, S. Belin, D. Delbègue, New J. Chem., 44, 10153 (2020)
[8] D. Vantelon, M. Davranche, R. Marsac, C. La Fontaine, H. Guénet, J. Jestin, G. Campaore, A. Beauvois, V. Briois, Env. Sci.: Nano, 6, 2641 (2019)
[9] B. Azeredo, T. B. Ghzaiel, N. Huang, S. Nowak, J. Perron, M. Giraud, J. Balachandran, O. Taché, L. Barthe, J.-Y. Piquemal, V. Briois, L. Sicard, Phys. Chem. Chem. Phys., 25, 22523 (2023)
[10] C. Lesage, E. Devers, C. Legens, G. Fernandes, O. Roudenko, V. Briois, Cat. Today, 336, 63, (2019)
[11] J. Jaumot, A. de Juan, R ; Tauler, Chemom. Intell. Lab. Syst.140, 1, (2015).
[12] G. Landrot, E. Fonda, J. Synchrotron Rad., 32, 1085, (2025)
[13] V. Briois, J.-P. Itié, A. Polian, A. King, A. S. Traore, E. Marceau, O. Ersen, C. La Fontaine, L. Barthe, A. Beauvois, O. Roudenko, S. Belin, J. Synchrotron Rad., 31, 1084, (2024)