

A new versatile *operando* photocatalytic cell for quick-EXAFS: the example of CO₂ photoreduction

Anthony Beauvois^a, Sébastien Roth^b, Audrey Bonduelle-Skrzypczak^b,
Christèle Legens^b, Julie Marin^a, Laurent Barthe^a, Pascal Raybaud^b and
Valérie Briois^{a,c}

^aSynchrotron SOLEIL, L'Orme des Merisiers, 91190 Saint-Aubin, France

^bIFP Energies Nouvelles, Rond-Point de l'échangeur, 69360 Solaize, France

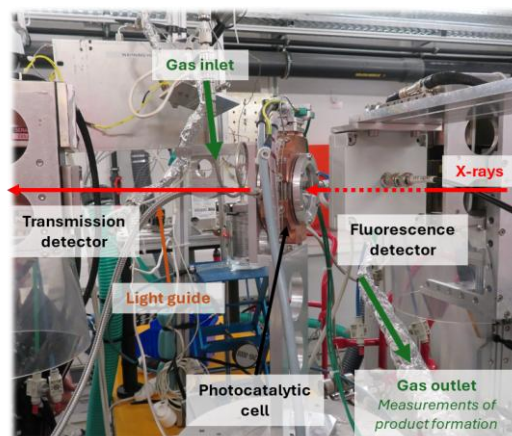
^cCentre National de la Recherche Scientifique, UR1, France

ABSTRACT

Over the past decade, photocatalysis has emerged as promising process for numerous applications including pollutant removal, water splitting or CO₂ conversion, through the harnessing of solar energy. However, achieving sufficient efficiency for industrial applications requires addressing several challenges. Most of these challenges relate to the design of the photocatalyst itself including light penetration and absorption, charges recombination, selectivity of the product formation, nature of active sites and photocatalyst deactivation¹. *Operando* quick-EXAFS measurements is a powerful technique to unravel the structure-activity of the photocatalyst and to address these challenges by monitoring the electronic structure and local environment of a photocatalyst under working conditions.

In this work², we describe a versatile *operando* cell suitable for monitoring gas-phase photocatalytic reaction by quick-EXAFS in transmission or fluorescence in the energy range covered by the ROCK beamline³, along with an in-line analysis of product formation by analytical methods such as mass spectrometry or gas chromatography. This development was carried out in the framework of the French 'PEPR LUMA'⁴.

The capabilities of the *operando* cell will be demonstrated by studying Mo oxysulfides supported on TiO₂ used for the CO₂ photoreduction. After validating the cell by comparing the performances obtained at ROCK with that of a well-tested lab-cell, the behavior of these catalysts will be monitored by XAS at the Mo K-edge, using a Xe lamp (250-650 nm) as UV-visible source. To overcome the volume sensitivity of XAS for studying photocatalytic reactions (occurring at the sample surface where light interacts with the adsorbed species), a modulation-excitation⁵ XAS was implemented. This technique has proven relevant and powerful to highlight the contribution of active species compared to silent spectator species during the photocatalytic process.



REFERENCES

1. Gong *et al.*, *Energy Environ. Sci.*, 2022, **15**, 880-937, DOI: [10.1039/D1EE02714J](https://doi.org/10.1039/D1EE02714J)
2. Roth *et al.*, *J. Synchrotron Rad.*, 2026, **33**, DOI: [10.1107/S1600577525008768](https://doi.org/10.1107/S1600577525008768)
3. Briois *et al.*, *J. Phys.: Conf. Ser.*, 2016, **712**, 012449, DOI: [10.1088/1742-6596/712/1/012449](https://doi.org/10.1088/1742-6596/712/1/012449)
4. <https://www.pepr-luma.fr/en/homepage/>
5. Urakawa *et al.*, in *Springer Handbook of Advanced Catalyst Characterization*, 2023, DOI: [10.1007/978-3-031-07125-6_42](https://doi.org/10.1007/978-3-031-07125-6_42)