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News

Fundamental Research

Catalysis and reaction kinetics | Chemical analysis

Converting CO_2 into clean energy is a key challenge for both the climate and the energy transition, and using a photocatalytic approach requires an understanding of the mechanisms at play on a very small scale. Thanks to close collaboration between IFPEN and the SOLEIL Synchrotron as part of a PhD research project[1], a pioneering operando cell now makes it possible to probe the behavior of catalysts under the influence of light on an atomic scale. This

represents a major step forward in accelerating the potential development of an industrial process.

The objective: to overcome the challenges to artificial photosynthesis

The photocatalytic conversion of CO_2, which uses **solar energy (a resource 400 times greater than global demand)**, is one of the great challenges of the century. However, **its efficiency remains inadequate** for industrial-scale use due to complex phenomena, including the deactivation of catalytic materials or the recombination of electronic charges [1]. Until now, the lack of tools for observing the catalyst in action, i.e. under *operando* conditions, had made it impossible to precisely identify the causes of these obstacles.

A synergy of technical expertise

The creation of **a new analysis cell** was the result of a close technical partnership between the teams at the two organizations. The design of the equipment was directly inspired by the photocatalytic reactors already in use in IFPEN's laboratories. This platform was then adapted and optimized by the synchrotron[2] teams to meet the specific requirements of SOLEIL's ROCK beamline, thereby enabling an innovative integration of **gas-phase photocatalysis** and **X-ray analysis** [2].

Cross validation between laboratory and synchrotron

The reliability of this new tool has been demonstrated through a rigorous validation process involving both partners:

- **Catalytic performance:** The results obtained at the synchrotron were compared with and validated against those obtained at the test facility located at the IFPEN site, thereby guaranteeing that X-rays do not alter the catalyst's behavior.
- **Analytical precision:** The X-ray absorption spectroscopy (XAS) measurements performed in the cell were confirmed by ex situ analyses conducted in capillaries, ensuring optimal signal quality despite the limitations of the *operando* setup.

Unraveling the secrets of the origin and evolution of photocatalytic activity

The new equipment was used to study **photocatalysts based on molybdenum oxysulfides (MoO_xS_y) supported on TiO_2**. The combination of cutting-edge XAS techniques with **an excitation modulation approach under transient illumination** made it possible to identify the nature of the active phase (**MoO_xS_y nanoclusters**) and to understand the evolution of its active site during the CO_2 photoreduction reaction [3]. The result marks a first for this particular reaction in the gas phase.

A shared tool for the future

This innovative and versatile set-up, now available on the SOLEIL Synchrotron's ROCK beamline, can be adapted for other reactions and combined with new analytical techniques such as Raman spectroscopy. It provides the scientific community with a cutting-edge tool, developed as a result of

collaboration between institutional research and a major research infrastructure, to design the decarbonization solutions of the future.

[1] Sébastien ROTH, ***Molecular exploration of supported Mo sulfide and oxysulfide nanostructures for the photocatalytic reduction of CO?***, PhD thesis, ENS Lyon, 2024.

[2] With the support of Agence Nationale de la Recherche (ANR – French National Research Agency) within the framework of the ANR-22-EXLU-0003 and ANR-10-EQPX-45 projects

References:

[1] Roth, S., Bonduelle-Skrzypczak, A., Legens, C., Raybaud, P. ***Meeting the Industrial Challenges of CO_2 Photocatalytic Reduction: Moving From Molybdenum Disulfides to Oxysulfides Based Materials?*** ChemSusChem 2025, 18, e202400572.
>> DOI : https://doi.org/10.1002/cssc.202400572

[2] Roth, S., Bonduelle-Skrzypczak, A., Legens, C., Marin, J., Barthe, L., Beauvois, A., Briois, V., Raybaud, P. ***Operando photocatalytic cell for time-resolved XAS/GC analysis of gas phase CO_2 photoreduction***. J. Synchrotron Rad. (2026). 33, 123–12.
>> DOI : https://doi.org/10.1107/S1600577525008768

[3] Roth, S., Bonduelle-Skrzypczak, A., Legens, C., Marin, J., Beauvois, A., Briois, V., de Zwart, F. J., Mougel, V., Copéret, C., Raybaud, P. ***TiO_2-supported Mo oxysulfide nanostructures for the CO_2 photoreduction: active states from operando X-ray absorption spectroscopy*** (under reviewing)

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CO2 photocatalytic reduction IFPEN and the SOLEIL Synchrotron are jointly developing an operando spectroscopic analysis cell
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