



	Experiment title: DRIFT/XAS analysis of single-site Pt supported on UiO-66(Ce) for H ₂ production	Experiment number: CH-6664
Beamline: Bm23	Date of experiment: from: 30/03/2023 to: 03/04/2023	Date of report: <i>Received at ESRF:</i>
Shifts: 12	Local contact(s): Davide Salusso	
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Report:

In the panorama of H₂ production strategies we are currently exploring the water gas shift (WGS) reaction over metal single sites supported on cerium-based UiO-66(Ce). We have recently prepared a Ce³⁺ rich UiO-66(Ce) catalyst with anchored Pt single sites. The catalyst presented similar catalytic activity than the conventional Pt/CeO₂ catalyst at lower temperature (150 vs 50°C). We then proposed to study the prepared Pt/UiO-66(Ce) under WGS reaction conditions by DRIFT/XAFS. Indeed, the combination of the two techniques is the only available solution to track surface and bulk properties of the catalyst under reaction condition.

During the beamtime we successfully collected operando DRIFT/XAS spectra for Pt/CeUiO-66 sample and for two Pt/CeO₂ samples with different Pt contents. The Pt loading was <1% hence fluorescence detector was installed within the DRIFT cell.

The spectra were collected during the protocol: I) RT, He, II) heat to 100C under He and steady state for 1h, III) exposure to CO saturated H₂O for 2h, IV) flush with He and cool to 100C. The Pt L₃-edge XAS spectra indicated the presence of PtO₂ in the as prepared catalyst with low Pt-O coordination number. Despite the low Pt content we successfully achieved to record high quality EXAFS spectra ($k \approx 11 \text{ \AA}$) in 1h. During reaction we clearly observed a reduction of Pt accompanied with a decrease of Pt-O shell. However, XAS could not follow the reaction time scale due to long collection time implied by the low Pt content. Nevertheless, having coupled DRIFT allowed to monitor Pt surface oxidation state as expected. Indeed, the presence of CO induced the formation of Pt-CO species which stretching vibration can be used as a fingerprint of Pt surface oxidation state. The results analysis is still in progress however, it clearly indicated the high versatility of the employed set-up. At the same time the Mass Spectrometer recorded signals evolution comparable with laboratory catalytic

results confirming the reliability of the measurement.

Overall, the beamtime was extremely successful providing high quality XAFS and DRIFT data which will allow to disclose Pt reconstruction under reaction conditions.

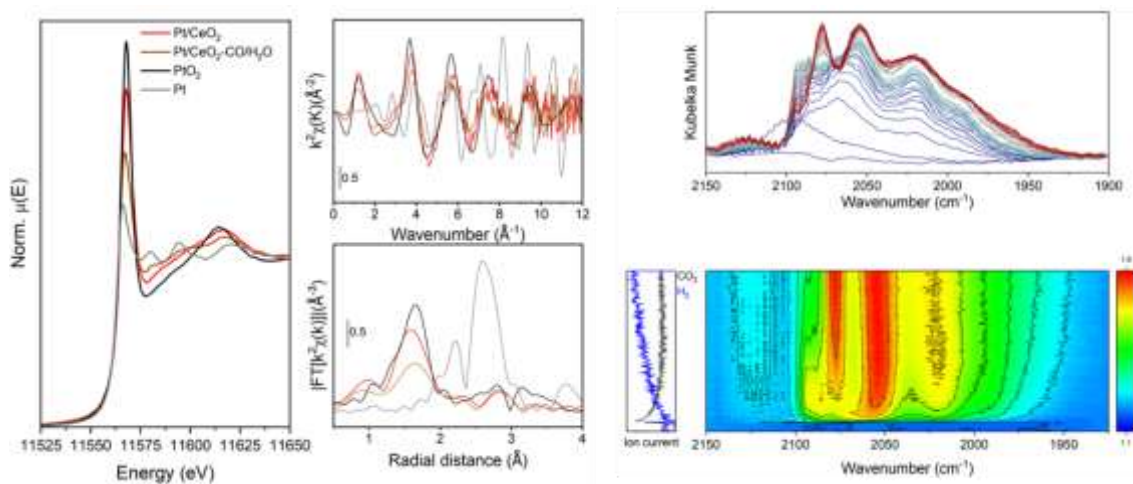


Figure 1 Pt L₃-edge left) XANES and center) EXAFS and FT-EXAFS (magnitude) spectr. Right) DRIFT spectra collected during WGS reaction. Time increases from blue to red line.