Industrial Research on Catalysis using XAS at Diamond

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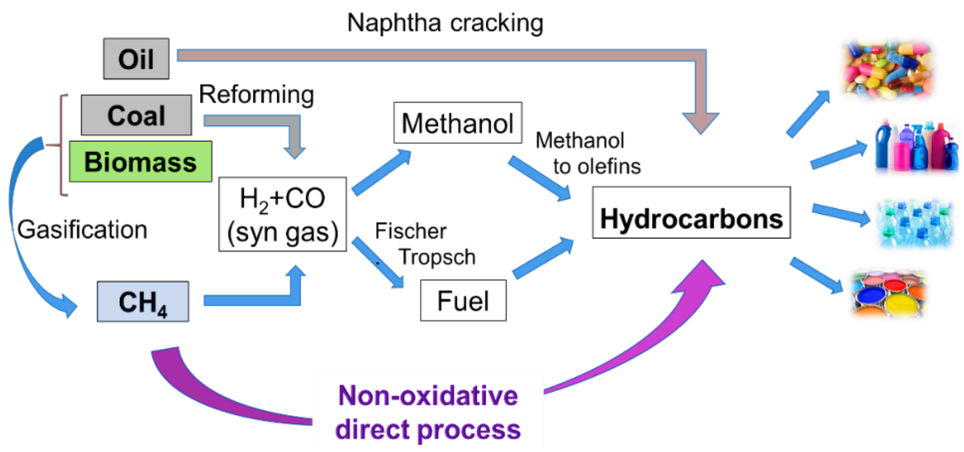
A wealth of catalysis research is carried out at Diamond and the techniques available include X-ray absorption spectroscopy (XAS), small-angle X-ray scattering (SAXS), powder diffraction, and X-ray imaging. The requirements for high selectivity and activity of catalysts are among the most crucial demands for a successful commercial application. Therefore, catalyst characterisation provides a unique opportunity for industry to develop new challenging materials for energy, chemistry and environmental technologies. Over the past decades, great efforts have been devoted to developing methods for catalyst characterisation under real operating conditions. Thus, a range of sample environments have been implemented at Diamond to accommodate the combined techniques suitable for *operando* studies.

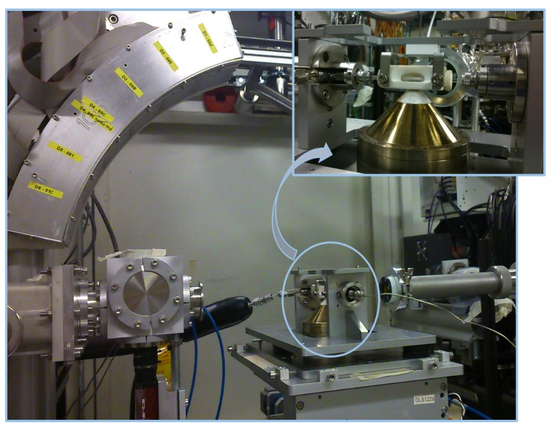
Methane upgrade to higher value chemicals

Methane Dehydroaromatisation (MDHA) is a non-oxidative reaction that converts CH_4 directly into hydrocarbons, aromatics and hydrogen.

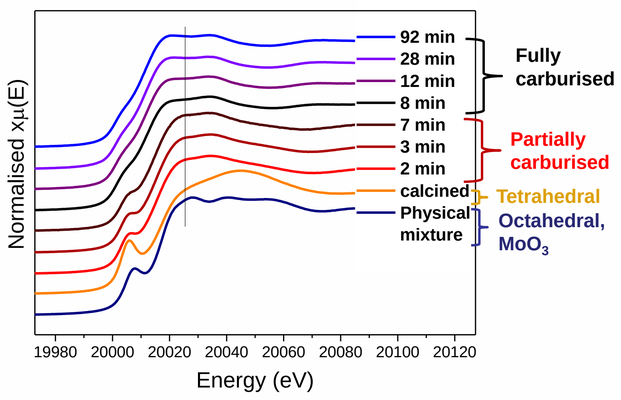


Operando Mo K-edge XAS/XRD/MS studies on B18 at Diamond

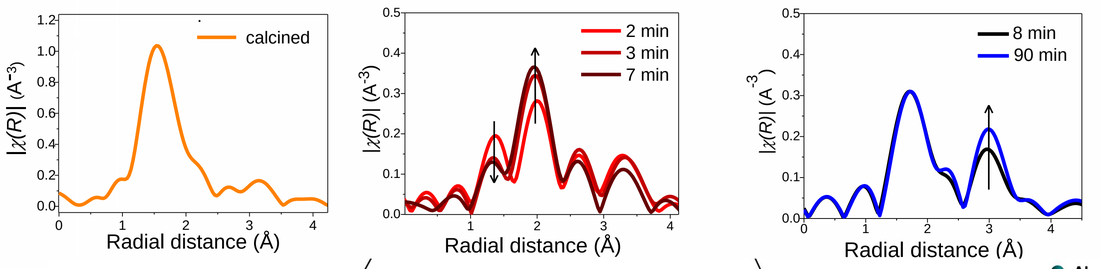
Capillary-based reaction cell set up for *in situ* XAS/XRD/MS studies

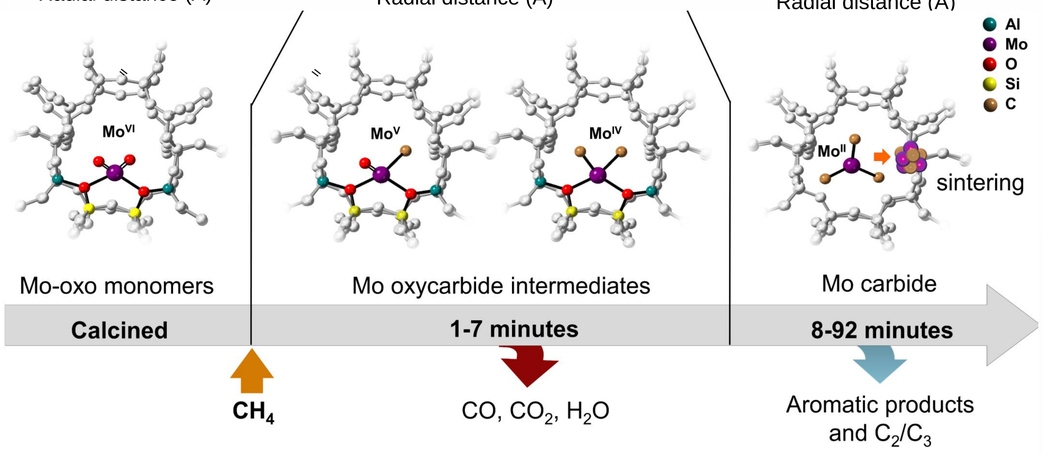


Mo K-edge XANES



Mo K-edge EXAFS





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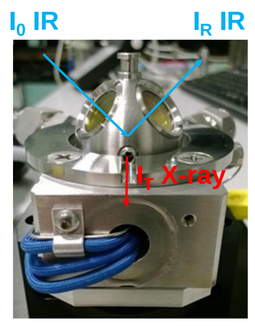
Combined set up for effective catalyst design

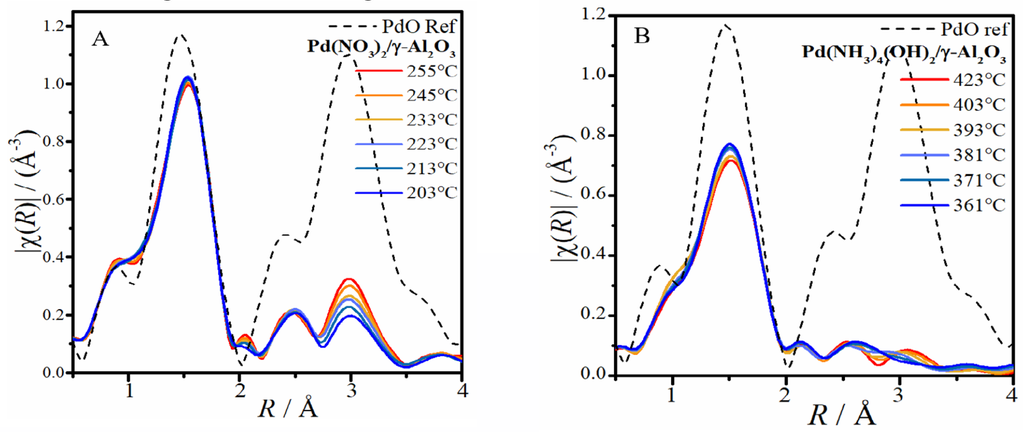
Evolution of PdO/γ-Al_2O_3 made from two different Pd precursors

- The aim of this work is to understand how PdO nanoparticles are formed using a combined set up of XAS/DRIFTS/MS and find the optimal route for designing a palladium oxide based catalyst;
- **Pd0/γ-Al_2O_3** was formed using $Pd(NO_3)_2$ and $Pd(NH_3)_4(OH)_2$ precursors;
- Pd catalysts can be used in a number of catalytic applications:
  - CO and CH_4 oxidation;
  - Upgrading bio-oils;
  - Automotive three-way catalysts.

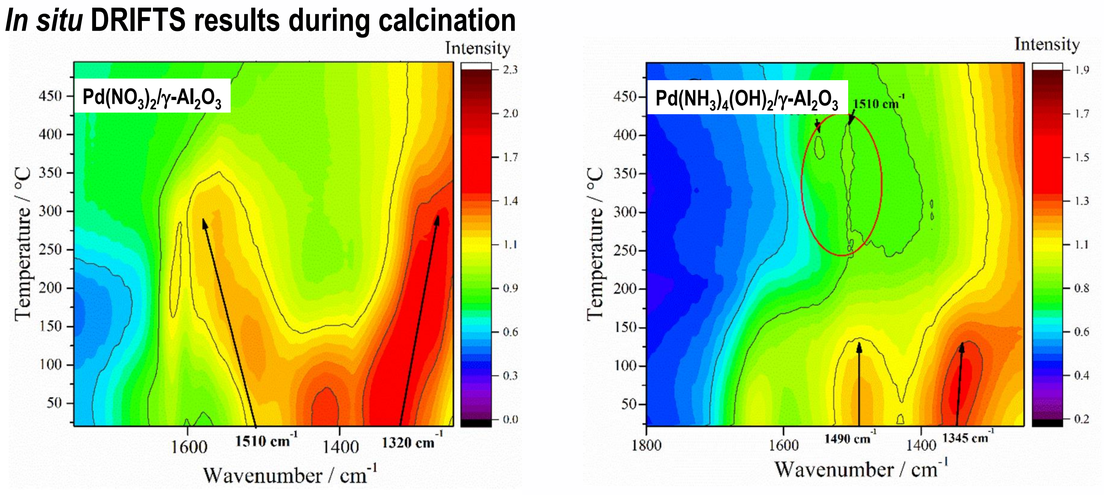


Customised **Harrick DRIFTS Cell**

***In situ* Pd K-edge EXAFS during calcination**



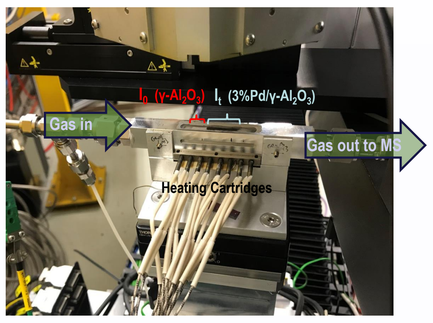
***In situ* DRIFTS results during calcination**

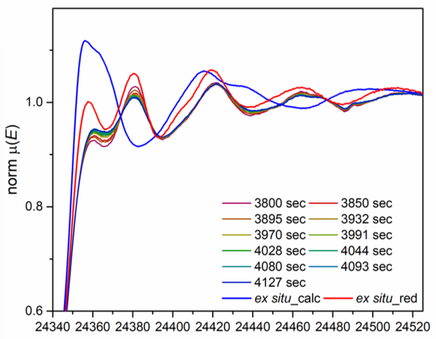


$Pd(NO_3)_2$/γ-Al_2O_3 forms pre-associated molecular assemblies upon impregnation due to bridging interactions of nitrate ligands and an increase in the Pd-Pd scattering contribution

The Pd centres from **$Pd(NH_3)_4(OH)_2$/γ-Al_2O_3** appear to adopt isolated $[Pd(NH_4)_4]^{2+}$ sites upon impregnation, evidenced by the absence of any Pd-Pd distances

Newly developed XAS/DRIFTS/MS reaction cell on I20-EDE





***In situ* Pd K-edge EDE-XANES during CO oxidation**

Ellie Dann, *et al.* Chem.Mater. 2017, **29**, 7515 − 7523

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