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 CH₄ directly into hydrocarbons, aromatics and hydrogen.

**Combined set up for effective catalyst design**

Evolution of PdOₓ-Al₂O₃ 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;
- PdOₓ-Al₂O₃ was formed using Pd(NO₃)₂ and Pd(NH₃)₂(OH)₂ precursors;
- Pd catalysts can be used in a number of catalytic applications:
  - CO and CH₄ oxidation;
  - Upgrading bio-oils;
  - Automotive three-way catalysts.

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

- Pd(NO₃)₂+Al₂O₃ 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(NH₃)₂(OH)₂+Al₂O₃ appear to adopt isolated [Pd(NH₃)₂]²⁺ sites upon impregnation, evidenced by the absence of any Pd-Pd distances

**Newly developed XAS/DRIFTS/MS reaction cell on I20**

- Customised Harrick DRIFTS Cell
- In situ DRIFTS results during calcination

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