In situ structural characterization of homogeneous metal catalysts using XAFS spectroscopy in combination with complementary techniques.

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The availability the Third Generation light sources has greatly enhanced the opportunities for investigating chemical change in real time.[1] This presentation describes studies carried out at the ESRF and Diamond Light Source investigating chemical processes in solution, using techniquies developed from those applied to nickel catalysts for alkene oligomerisation.[2]

The nature of catalysts for the selective tri- and tetramerisation of ethene to linear α-alkenes which are generated by treatment of Cr(III) complexes incorporating bi- and tri-dentate soft-donor ligands with aluminium alkyl reagents has been investigated by XAFS spectroscopy. New complexes with S-donor ligands have been synthesised and investigated with Cr(III), and analogues on scandium(III) (for NMR purposes) and molybdenum(III) (a more amenable energy of in situ XAFS spectroscopy).

The acquisition time required by conventional scanning mode methods at the Cr K-edge was longer than normal reaction times.[3] Hence a sampling technique combining stopped-flow[2,4] with freeze-quench techniques has been developed to probe these catalysts.[5] These recent results will be described, with the aim of throwing more light onto the nature of these commercial catalysts.

A demonstration experiment using a germanium microstrip detector has shown that energy dispersive EXAFS can be employed to investigate the nature of photoexcited states.[6] Plans to establish these techniques at the Diamond Light Source and apply them to study primary steps in homogeneous catalysis will be outlined.

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