

• Facility for *operando* XAS studies on catalytic systems (2017)

Structure-activity correlation is vital to understand and improvise catalysis processes which are regularly used in various important experiments such as for converting harmful exhausts from automotive and industrial plants to benign products. Recently in-situ XAS has also become very important to probe the structural changes in catalysts during a reaction. These experiments not only help to understand the effect of structural changes on the catalytic activity, but also give insight into the problem of deactivation of the catalysts. A set-up has been developed (left panel top) for *in-situ* monitoring of heterogeneous catalytic reactions, which consists of a S.S cell with Be windows for X-ray transmission and heating facility of the sample upto 400°C. The inlet of the reaction cell is connected to a gas manifold with mass flow controllers to allow the flow of reactant gases in desired proportions while the outlet of the reaction cell is connected to a gas chromatograph for detection of the reaction products. Fischer Tropsch (FT) reaction which involves hydrogenation of CO to produce CH<sub>4</sub> with SBA-15 supported Co<sub>3</sub>O<sub>4</sub> nanoparticles as catalysts has been monitored using the above facility. The oxide catalyst was first reduced by heating to 400°C with a ramp rate of 10°C/min under 20ml/min H<sub>2</sub> flow and the reduction process was monitored by *in-situ* XANES measurements (bottom panel left). However, at 320°C the Co catalyst shows deactivation with time (bottom panel centre). The catalyst has been studied by *in-situ* XAS for 10 hours at 320°C and it has been observed that there is no signature of formation of CoO phase during the reaction at 320°C (bottom panel centre) which might be responsible for the deactivation of the catalyst. Another cell (right panel at top) has also been fabricated which has facility for illumination by visible and UV radiation through glass/quartz windows for photo-catalytic reactions. Operando EXAFS studies have been carried out using this cell on Cu doped TiO<sub>2</sub> catalysts during photocatalytic reduction of CO<sub>2</sub> and splitting of water and subsequent generation of methane (CH<sub>4</sub>).