Development of *in-situ* EXAFS facilities to probe structural changes during growth of nanoparticles and hetergenous catalysis at Indus-2 SRS, INDIA

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In-situ studies on the synthesis of nanoparticles from a solution phase are extremely important since the information can be used to fabricate tailor made nanoparticles. Synchrotron radiation based XAS, comprising of both XANES and EXAFS, can simultaneously give insight into the structural changes in the species during growth of nanoparticles as well as in the oxidation states of the metal cations or in other words into the step-by-step reduction process of the precursors. Similarly structure-activity correlation is vital to understand and improvise any catalysis process. 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. In order to study growth of nanoparticles and heterogeneous catalytic reactions on-line, several *in-situ* setups have been designed and installed at the EXAFS beamlines at Indus-2 SRS at RRCAT, Indore, INDIA which are presented in this paper.

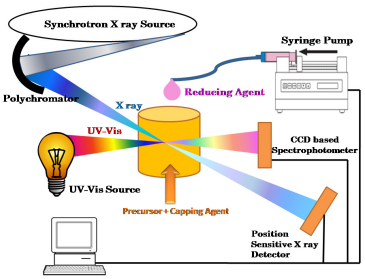

Fig.1

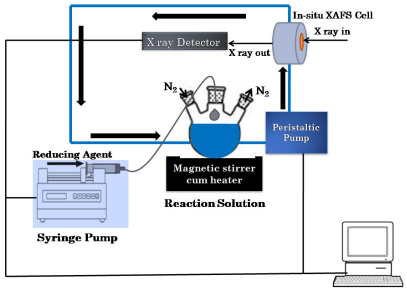

Fig.2

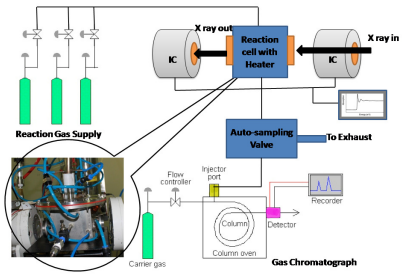

Fig.3

Fig.1 shows the schematic of the reaction cell designed for *on-line* probing of growth of nanoparticles by simultaneous *in-situ* measurements of XAS and UV-Vis spectroscopy. The reaction cell is made up of teflon and have paths for both X-rays and UV–Vis radiation in mutually perpendicular directions. The cell has been used in monitoring *in-situ* growth of Au, Pt monometallic and Au@Pt core shell nanoparticlers. Such an experimental set-up is however useful only for one-pot synthesis and has very less flexibility where the growth needs special ambience like inert atmosphere. For this a flow set-up, as shown schematically in fig.2, has been designed and installed where the synthesis takes place in a reaction vessel under continuous nitrogen bubbling and the measurement is carried out in a separate measurement cell. The reaction product is pumped to the *in-situ* XAS measurement cell from the reaction vessel through viton tubes using a peristaltic pump. *In-situ* study on growth of Cu nanoparticles has been successfully carried out using this set-up. Fig.3 schematically shows the experimental setup to monitor catalytic reactions, which consists of a S.S cell with Be windows for X-ray transmission and heating facility of the sample upto 400^{o}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. The outlet of the reaction cell is connected to a gas chromatograph for detection of the reaction product. This setup has been used to study Fischer-Tropsch reaction for production of methane from reactant gases of CO_2 and H_2.

References: (i) C. Nayak et.al., *J. Synchrotron Rad.*, 23 (2016) 293-303, (ii) C. Nayak et.al.,*J. Synchrotron Radiat.*, 24 (2017) 825-835, (iii) C. Nayak et.al., *AIP Conf. Proc.*, 1832 (2017) 060003.