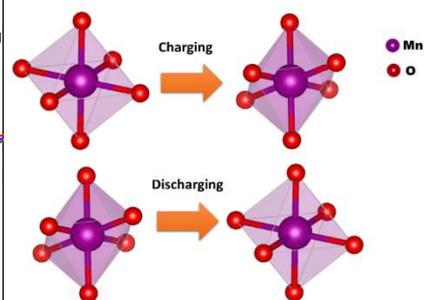
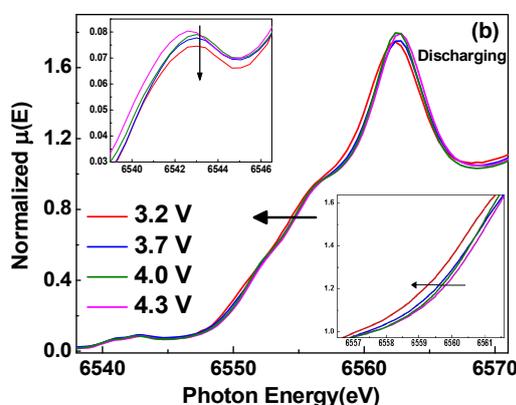
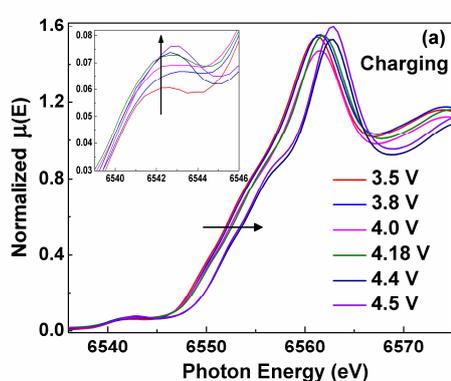
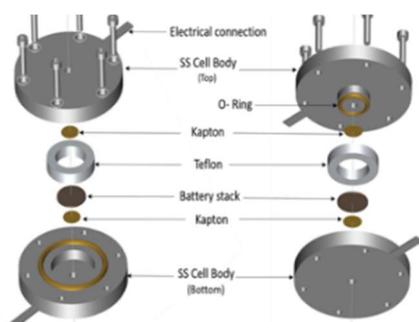


- Facility for *in-situ* XAS monitoring of charging-discharging of Li-ion batteries (2019)



A facility has been developed for *in-situ* XAS measurements and on-line monitoring of structural changes in cathode or anode materials during charging/discharging of Li & Na ion batteries. A versatile, easy to assemble and re-usable electrochemical battery cell has been designed and developed for this (top left panel) and arrangements have been made for *in-situ* XAS measurements of the cathode materials in transmission mode at the BL-09 EXAFS beamlines (top right panel). Kapton films as X-ray windows and Al foils as current collectors have been used in the cell in such a way that it ensures leak tightness so that proper electrochemical reactions of the battery can take place and at the same time X-rays can pass through it so that transmission mode XAS measurements can be done. A Li ion battery with  $\text{LiMn}_2\text{O}_4$  and Li foil as cathode and anode materials respectively has been assembled inside a glove box under argon ambience with Kapton windows on both sides for transmission of X-rays. The electrochemical performance of the cell vis-a-vis a standard coin cell made up of similar electrode and electrolytes has been ascertained by galvanostatic testing on the cell by several cycles. Subsequently *in-situ* XAS measurements have been carried out on the cell at the BL-09 beamline (Indus-2) at Mn K-edge. *In-situ* XANES measurements (bottom panel left and centre) disclosed co-existence of  $\text{Mn}^{+3}$  and  $\text{Mn}^{+4}$  in pristine  $\text{LiMn}_2\text{O}_4$  and Li de-intercalation during charging process leads to oxidation of  $\text{Mn}^{+3}$  to  $\text{Mn}^{+4}$ . *In-situ* EXAFS also corroborate that upon charging Mn environment changes from a mixed coordinations of two different Mn-O octahedra to a uniform coordination of  $\text{MnO}_2$  (bottom panel right). However, the above changes are found to be not fully reversible when the battery is discharged. Cyclic instability of Li-ion batteries during the charging/discharging processes are due to such variations in the local structure of the electrode materials around the active atoms and above *in-situ* study provides a direct technique to detect these changes in real time.