Tracking the Nanostructure Change of Ordered Mesoporous Materials for Li Storage by *in Operando* Small Angle X-ray Scattering

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To monitor dynamic volume changes of electrode materials during electrochemical lithium storage and removal process is of utmost importance for developing high performance lithium storage materials. We herein report an *in operando* probing of mesoscopic structural changes in ordered mesoporous electrode materials during cycling with synchrotron-based small angle X-ray scattering (SAXS) technique. *In operando* SAXS studies combined with electrochemical and other physical characterizations straightforwardly show how porous electrode materials underwent volume changes during the whole process of charge and discharge, with respect to their own reaction mechanism with lithium. This comprehensive information on the pore dynamics as well as volume changes of the electrode materials will not only be critical in further understanding of lithium ion storage reaction mechanism of materials, but also enable the innovative design of high performance nanostructured materials for next generation batteries.