

## Insights into the structural modification of electrochemically synthesized layered double hydroxides by X-ray photoelectron spectroscopy

Reinhard Denecke<sup>1</sup>, Elisa Musella<sup>2</sup>, Thomas Ruf<sup>1</sup>, Isacco Gualandi<sup>2</sup>, Domenica Tonelli<sup>2</sup>, Marco Giorgetti<sup>2</sup>

<sup>1</sup> Wilhelm-Ostwald-Institut für Physikalische und Theoretische Chemie, Universität Leipzig, Linnéstr. 2, 04103 Leipzig, German

<sup>2</sup> Department of Industrial Chemistry "Toso Montanari", University of Bologna, Viale Risorgimento 4, 40136 Bologna, Italy  
denecke@uni-leipzig.de

Layered double hydroxides (LDHs) are important and versatile two-dimensional nanomaterials with a variety of application fields such as energy storage, environmental protection and remediation, medical treatment, drug delivery, electronics and catalysis [1]. Ex situ XPS experiments have been carried out with the aim to better understand the modifications of Co based LDHs in alkaline media which seem to appear during the first anodic segment of a cyclic voltammogram. The Co LDHs have been prepared with Al and Fe in order to investigate the role of trivalent cation on the redox behaviour. XP spectra of the different core levels recorded after stopping the first anodic potential sweep at different potential values display drastic and irreversible changes starting at a particular voltage for each system studied (Co/Al and Co/Fe). These changes are related to changes in oxidation state which in turn signal structural changes.

Fig. 1 displays Co 2p spectra for the Co/Al system obtained after a certain potential has been applied during the first voltammetry cycle. The spectra start with a clearly Co(II) state for the pristine situation which switches to Co(III) above 0.525 V. This is in line with a change from a  $\text{Co}^{\text{II}}(\text{OH})_2$  to a  $\text{Co}^{\text{III}}\text{O}(\text{OH})$  configuration [2]. Similar changes are also found in the other core levels. The situation for Co/Fe is similar but not as clearly observable. Subsequent cathodic potentials and repeated cycles do not cause significant changes.

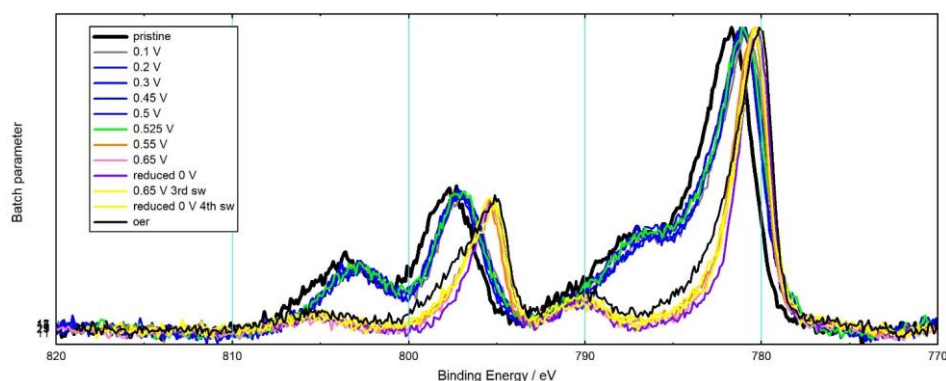


Fig. 1: Co 2p spectra recorded after the first anodic potential sweep was stopped at the marked voltages or preparation steps.

- [1] C. Hobbs, S. Jaskaniec, E. K. McCarthy, C. Downing, K. Opelt, K. Güth, A. Shmeliov, M. C. D. Mourad, K. Mandel and V. Nicolosi, *npj 2D Mater. Appl.*, 2018, **2**, 4.
- [2] E. Scavetta, B. Ballarin, C. Corticelli, I. Gualandi, D. Tonelli, V. Prevot, C. Forano and C. Mousty, *J. Power Sources*, 2012, **201**, 360.