Exploration into the Ionic Exchanges in the Ni/Fe Lamellar Double Hydroxide by in situ Techniques Under Polarization

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Lamellar Double Hydroxides (LDH) are solids with a structure similar to that of brucite Mg(OH)\textsubscript{2} in which some divalent cations are replaced by trivalent cations. This substitution creates an excess of positive charges in the octahedral layers, which is compensated by the presence of exchangeable anions in the interlayer space.\textsuperscript{1} The choice of electroactive cations for the synthesis of LDH-coated electrodes enables their reversible electrochemical oxidation and reduction that are observed using the cyclic voltammetry method. Coupling this technique with a crystal quartz microbalance permits the measure of the variations in mass of the coated film and witnesses the reversible transfer of anions\textsuperscript{1} during a series of oxidation and reduction steps of cations constituting the layers. Reversibility of anionic exchanges (like nitrate, nitrite or arsenate) is of fundamental and industrial interest as it paves the way to applications in water treatment against numerous anionic pollutants.\textsuperscript{2}

In this presentation the nature of the transferred species under the polarization of a thin film of LDH are further investigated with \textit{in situ} techniques. For this study, the Ni/Fe LDH was chosen for its high electrical conductivity and the facile reduction and oxidation of Ni. It was chemically synthesized by the coprecipitation method with variable pH to form nano-platelets that increase the LDH/aqueous medium interface and enhance the anionic exchanges. Its physical and chemical properties have been characterized by XRD, SEM, infrared spectroscopy and Energy Dispersive X-ray Spectroscopy. For the study of the ionic exchanges, we employ the \textit{ac}-electrogravimetry\textsuperscript{3} technique. It is a laboratory-made nondestructive \textit{in situ} technique that measures the transfer function $\Delta m/\Delta E(\omega)$ of the polarized film to distinguish the contribution of several species (cations, anions, solvent) occurring in the exchange, as well as their molar mass, their concentration and kinetics. We also follow the intercalation/deintercalation of species by \textit{in situ} XRD measurements by observing the (003) peak which is a marker of the interlamellar distance in the LDH. The use of \textit{in situ} techniques under polarization (\textit{ac}-electrogravimetry and XRD), with the contribution of other common physical analyses, illuminate the ionic exchanges in the Ni/Fe LDH.

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References