Synthesis and Electrochemistry of Layered Double Hydroxide Thin Films by Layer-by-Layer Assembly Method

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Two-dimensional nanosize sheets as macromolecules have received a great attraction in the last couple of years. Studies on nanosize sheets, especially those of transition metal layered oxides, which possess negative charges on the layer surface, have revealed the potential of nanosize sheets in the photoelectrochemical, electrochemical and photocatalytic applications. In this respect, exfoliation of layered double hydroxides (LDH) to positively charged nanosize sheets have been studied as an alternative in recent years. In this study, we have investigated the synthesis of several LDHs, such as Ni-Ga, Ni-Fe and Ni-Al LDHs with hydrothermal method. The reaction was alternatively carried out in the presence of hexamethylenetetramine (HMTA) as a hydrolysis agent instead of the most widely used urea in similar reactions. Total metal content and ratio of the hydrolysis agent in the reaction solution have found to be determining factors for the crystallinity and morphology of LDH products. Exfoliation of LDHs was carried out in formamide and exfoliation was confirmed by the images obtained from Atomic Force Microscope (AFM). The observed thickness of sheets was in agreement with the theoretical thickness of LDH layers. Exfoliated single nanosize sheets were deposited on a substrate by Layer-by-Layer Assembly (LBL) method, which utilizes the electrostatic attraction principles between oppositely charged units. Positively charged LDH sheets were intercalated with various anionic molecules, such as ferro- and ferric- cyanides, ferric oxalate, polyanions and negatively charged metal oxide layers by subsequent adsorption of these species on a substrate. UV-vis spectroscopy of the films showed that deposition of intercalated LDH thin films with LBL was successful and a desired number of layers can be obtained. Electrochemical properties of the films were also investigated in this study. Cyclic voltammograms of the films revealed that LDH layers and intercalated species are electrochemically active. The redox peaks observed for Ni^{2+} containing LDHs in the region of -0.45 V were reversible. Scan rate controlled experiments revealed a linear increase in the current densities of the peaks with the increasing scan rate. In conclusion, we have demonstrated in this study that various LDH nanosize sheets can be intercalated with anionic species in a Layer-by-Layer deposition manner. In addition, LDH thin films prepared by LBL method showed electrochemical response

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