RECONSTRUCTION PECULIARITY IN CO-PRECIPITATED Mg/Al AND Mg/Al/Ce LAYERED DOUBLE HYDROXIDES

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Abstract

The layered double hydroxides (LDHs) are anionic compounds made up of positively charged brucite-like layers with an interlayer gallery containing charge compensating anions and water molecules. The metal cations occupy the centers of shared octahedral whose vertices contain hydroxide ions that connect to form infinite two-dimensional sheets [1]. After calcination at temperatures from 300 to 600 °C, LDH is converted to mixed metal oxides (MMO) [2], which have high adsorption capacity. Their high adsorption capacity and high anion exchange capacity are comparable to those of anion exchange resins. This facilitates LDH application as adsorption materials, catalyst precursors and catalyst supports [3]. MMO are able to recover the original layered structure, a property known as „memory effect" [4]. If MMO are put into aqueous solution, in the presence of anions, the layered structure is recovered with anions incorporated in the interlayer. A more irregular structure of agglomerated flake-like platelets has been observed after reconstruction [5] (Figure 1).

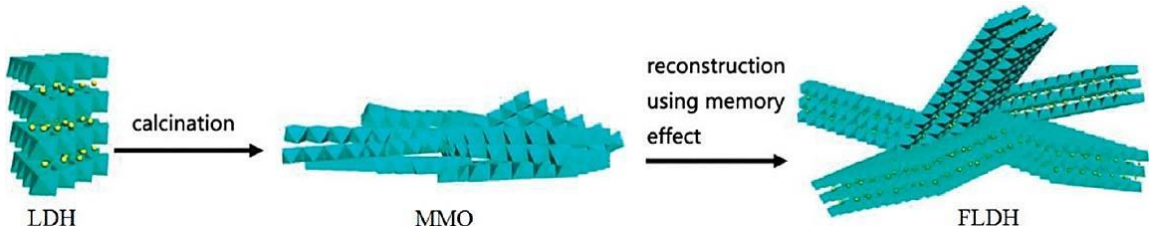

Figure 1. Schematic representation of flake-like LDH formation process

Cerium based inhibitor creates a passive insoluble oxide layer that stops the oxygen diffusion from the aggressive environment to the surface [6]. In this study, the intercalation of cerium in the Mg/Al layered double hydroxide was investigated, for the first time to our knowledge. The simple co-precipitation method was used for the fabrication of Mg/Al and Mg/Al/Ce specimens. LDH samples were synthesized by adding a mixture of $Mg(NO_3)_2 \cdot 6\ H_2O$ and $Al(NO_3)_3 \cdot 9\ H_2O$ with molar ratio (3:1) drop by drop to the solution of $NaHCO_3$ and NaOH (1:2) under vigorous stirring. The pH of the solution was kept between 7 and 9 during the synthesis. The formation of the MMO was achieved by heating LDH structure at 650 °C for 4 h. The MMO powders were reconstructed in water at 80 °C for 6 h under vigorous stirring at pH ≈ 8.5 (2 g of mixed oxide in 40 mL of water). Synthesis of Mg/Al/Ce compounds were performed in the same way as Mg/Al LDH, but the pH of the solution during the synthesis was 10. The effect of Ce^{3+} ion concentration on phase structure of $Mg_3Al_{1-x}Ce_x$ system was studied. The Ce^{3+} concentration in crystal lattice was changed from 0.05 to 2 mol%. The influence of Ce^{3+} ions content and synthesis conditions on phase composition, crystal size and morphology of $Mg_3Al_{1-x}Ce_x$ will be discussed. All synthesized samples were analysed and characterized using X-ray diffraction (XRD) analysis, scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX).

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