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ACCEPTED MANUSCRIPT

**SYNTHESIS AND CHARACTERIZATION OF LAYERED DOUBLE  
HYDROXIDES CONTAINING NICKEL IN UNSTABLE OXIDATION  
STATE +3 IN CATIONIC SITES**

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A series of layered double hydroxides (LDH) with molar ratio  $M^{2+}/M^{3+} = 3:1$  and different degrees of aluminum substitution in the cationic sites by  $Ni^{3+}$  were prepared by precipitation from solution in the presence of sodium hypochlorite as an oxidizing agent. The synthesized samples were characterized by various techniques including powder X-ray Diffraction (XRD), Thermogravimetric and Differential Thermal Analysis (TG-DTA), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy and inverse Temperature-programmed Reduction (iTPR). The results indicate that single-phase LDHs were formed during synthesis and they were of a high degree of crystallinity. The thermal stability of LDHs did not decrease essentially with increasing  $Ni^{3+}$  content. The calcination at 600°C led to the complete destruction of the layered structure and formation of mixed oxide phases. In iTPR experiments for as-synthesized LDHs the reduction of  $Ni^{3+}$  to  $Ni^{2+}$  took place above 400°C, and the reduction of  $Ni^{2+}$  to  $Ni^0$  occurred above 600°C.

### **Graphical Abstract**

Layered double hydroxides containing Ni(III) in cationic sites of brucite-like layers have been successfully prepared by precipitation from salts solution in an oxidizing environment. The synthesized samples have been characterized by various

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