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#### Research paper

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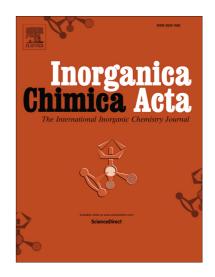
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## **ACCEPTED MANUSCRIPT**

# Examination of dehydration and dehydroxylation of synthetic layered (oxy)hydroxides through thermal analysis (TG-DSC-EGA-MS) and a discussion to the second Pauling's rule

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#### Abstract

This paper summarizes a reinvestigation of the dehydration and dehydroxylation of synthetic layered (oxy)hydroxides through thermal analysis (TG-DSC-EGA-MS). As well a discussion to the second Pauling's rule have been done. Simple metal-hydroxides i.e. Mg(OH)<sub>2</sub> and Al(OH)<sub>3</sub>, closely related aluminium oxide hydroxide, AlO(OH) and (Mg-Al-OH) type layered double hydroxides (LDHs) were prepared by different synthesis procedures and evaluated by X-ray diffraction (XRD).

According to this research, the thermal stability of hydroxyl groups in synthetic reference layered (oxy) hydroxides is  $(Al_2)$ -OH-boehmite sites  $(534^{\circ}C) > (Mg_2Al)$ -OH-hydrotalcite sites  $(432^{\circ}C) > (Mg_3)$ -OH-brucite sites  $(401^{\circ}C) > (Mg_3)$ -OH-hydrotalcite sites  $(330^{\circ}C) > (Al_2)$ -OH-gibbsite sites  $(250^{\circ}C)$  at a heating rate of  $10^{\circ}C$ -min<sup>-1</sup>. The reorganization of the hydroxyl groups within the magnesia-alumina hydrating blended pastes was established and evaluated in three aspects. First, the results from EGA-MS reveal that the  $(Mg_3)$ -OH sites in brucite lattice progressively reorganize and move continuously towards lower temperatures with curing time. Second, the optimized chemical composition of MgO-Al<sub>2</sub>O<sub>3</sub> dry mixture designed as hydrotalcite precursor in corundum-spinel castables is given in mol% as follows 50%MgO-50%Al<sub>2</sub>O<sub>3</sub>. Third, the hydrothermal activation strongly accelerates the formation of hydrotalcite Mg<sub>6</sub>Al<sub>2</sub>CO<sub>3</sub>(OH)<sub>16</sub>·4H<sub>2</sub>O phase.

**Keywords**: thermal stability of hydroxyl group; hydrotalcite; thermal analysis; structure of layered (oxy)hydroxides; second Pauling's rule

#### 1. INTRODUCTION

#### 1. 1 General overview

Simple metal-hydroxides i.e. Mg(OH)<sub>2</sub> and Al(OH)<sub>3</sub>, closely related aluminium oxide hydroxide, AlO(OH) and (Mg-Al-OH) type layered double hydroxides (LDHs), have received extensive research in recent years, as they find many potential applications, such as flame retardants, raw materials in conventional and advanced ceramic materials technology and layer structure inorganic ion exchangers, respectively [1-2]. Most synthetically produced layered hydroxides are deriving from processing of simple metal-oxides i.e. fine nano- and micro-powders of MgO and Al<sub>2</sub>O<sub>3</sub> in unshaped refractory materials (castables) technology [3-4]. As has been found so far in all cases investigated, the fineness and particle size distribution result in a size-dependent hydration mechanism and kinetics for powders of MgO and Al<sub>2</sub>O<sub>3</sub>, and also determine cementitious (hydraulic) activities [3]. Going in the forward direction there would be several additional points to address: (1) Effect of curing time and temperature on phase transformation of the MgO-Al<sub>2</sub>O<sub>3</sub>-H<sub>2</sub>O castable matrix, (2) The

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