

# ZnAlFe Mixed Oxides Obtained from LDH Type Materials as Basic Catalyst for the Gas Phase Acetone Condensation

Angeles Mantilla<sup>1,a</sup>, Francisco Tzompantzi<sup>2,b</sup>, María Manríquez<sup>2,c</sup>,  
 Guadalupe Mendoza<sup>2,d</sup> Jose L. Fernández<sup>1,e</sup> and Ricardo Gómez<sup>1,f\*</sup>

<sup>1</sup>CICATA-IPN, Av. Legaria 694, 11500, D.F. México

<sup>2</sup>Universidad Autónoma Metropolitana-Iztapalapa, San Rafael Atlixco 186, 09340 D.F. México

<sup>a</sup>email: [angelesmantilla@yahoo.com.mx](mailto:angelesmantilla@yahoo.com.mx); <sup>b</sup>email: [fjtz@xanum.uam.mx](mailto:fjtz@xanum.uam.mx);

<sup>c</sup>email: [marymanriquez@yahoo.com.mx](mailto:marymanriquez@yahoo.com.mx); <sup>d</sup>email: [gallinaja@hotmail.com](mailto:gallinaja@hotmail.com);

<sup>e</sup>email: [jlfernandez@ipn.mx](mailto:jlfernandez@ipn.mx); <sup>f</sup>email: [gomr@xanum.uam.mx](mailto:gomr@xanum.uam.mx)

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**Abstract:** ZnAlFe mixed oxides with high surface area were obtained by the calcination (723 K) of ZnAlFe layered double hydroxides (LDHs). The calcined materials proved as basic catalysts in the gas phase acetone condensation exhibited high activity and high selectivity towards the formation of mesityl oxide.

## 1. Introduction

Nowadays, the interest in aldol condensation reactions and their products has been increased in the fine chemical industry. Aldol condensation reactions are currently carried out with homogeneous basic catalysts and their replacement by solid basic is a goal to reach. The use of solid basic catalysts offers the advantage of decreasing corrosion and environmental problems, allowing an easier separation and recovery of the catalysts. In this way, aldol condensation reactions have been successfully carried out using MgAl hydrotalcites [1-3], zeolites [4] and alkaline doped MgO and TiO<sub>2</sub> catalysts [5,6]. However, the catalysts obtained by the calcination of layered double hydroxides LDHs (with general formula  $[M_{1-x}^{2+}M_x^{3+}(\text{OH})_2]^{x+} \cdot A_{x/n}^{n-} m\text{H}_2\text{O}$ ) have become an important alternative to obtain basic catalysts, since the composition of the mixed oxides and hence their basic properties can be tailored from the LDHs formulation [7-9].

With the purpose to develop new solid basic catalysts, in the present work, ZnAlFe LDHs materials were prepared and calcined to obtain ZnAlFe mixed oxides. In order to study their basic properties, the materials were evaluated in the acetone gas-phase self condensation. X-ray diffraction, nitrogen adsorption and Transmission Electron Microscopy (TEM) were used for the characterization of the mixed oxides.

## 2. Experimental

### 2.1 Catalysts preparation

ZnAlFe layered double materials were prepared by the co-precipitation method by using aqueous solutions of Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O:Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O and Fe(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O (J.K. Baker AR) in the appropriate amounts to obtain different ZnAlFe molar ratios. The solutions containing the precursors were added drop wise under vigorous stirring in a glass reactor vessel containing bidistilled water. Afterwards, the pH of the solutions was adjusted to 9 by adding NH<sub>2</sub>CONH<sub>2</sub> as the precipitant agent. The resulting suspensions were vigorously stirred for 4 h at 363 K and maintained under reflux for 36 h. Then, the obtained materials were filtered and washed with deionized water. Before characterization, the solids were dried at 393K (dried samples) for 12 h and finally annealed at 723 K for 12 h in N<sub>2</sub> flux (calcined samples). A reference ZnAl sample was prepared in the same way as described but without using iron precursor.