# Synthesis of Pseudoionone by Aldol Condensation of Citral and Acetone on MgAlLa Catalysts.

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## Introduction

The requirement of the fine chemical industry to implement processes that besides correcting the demand of products of commercial and scientific significance are friendly to the environment has fomented the development of different catalytic solid systems [1]. In reference to the acid-base processes, the synthesis of pseudoionone (PS) is a clear example of how heterogeneous catalysis fulfills the expectations of this industry [2]. Many investigations have shown that in the route of synthesis of PS, catalytic solids, such as hydrotalcites, can eliminate the use of inorganic bases such as NaOH commonly used as a catalyst.

The catalytic inhibition for the strong adsorption of organic molecules in the catalytic surface, at low temperatures, and the great quantity of catalyst used to obtain high yields, together with the specific conditions needed for the preparation and handling of the hydrotalcites, have increased the doubts on the industrial application of these materials.

In this work we report the use of MgAl hydrotalcites, the synthesis and characterization of these materials and the catalytic activity evaluated in the aldolic condensation reaction between citral and acetone to obtain PS.

## Materials and Methods

MgAl and MgAlLa, hydrotalcites type structures, were synthesized by the coprecipitation method at low saturation [3, 4]. The solids were subjected to a calcination-rehydration process, before putting them in contact with acetone and citral [3]. Reaction products were identified and quantified by gas chromatography coupled to mass spectrometry (G.C. Agilent-6890N, capillary Column HP-5MS, 30 m X 0.250mm X 0.25 microm, Agilent 5973 Mass Detecting Selective). X-RD, TGA-DTA, NMR ($^{27}$Al), $N_2$ adsorption, SEM and $CO_2$-TPD were used for characterization.

## Results and Discussion

Under the same reaction conditions, the ternary solid present the highest catalytic activity, table 1. The difference in activity is increased at low reaction temperatures. The characterization results point to that in the ternary system (MgAlLa), during the restoration of the structure (memory effect) in the calcination-rehydration processes, the presence of lanthanum on the surface of the crystalline structure inhibits the formation of tetrahedral sites in the structure of hydrotalcite. This phenomenon hinders the strong adsorption of citral on the catalytic surface of the solid, figure 1.

On the other hand, according to this results and Angelescu [4] it could be suggested that lanthanum acts as a shield of the catalytic sites when $CO_2$ molecules from the atmosphere are present.

**Table 1. Results of citral conversion and selectivity.**

| Catalyst | Rehydration (% weight) | Reaction temperature (°C) | Reaction time (hours) | Citral conversion (%) | PS selectivity (%) |
|---|---|---|---|---|---|
| MgAlLa (3%La) | 30 | 48 | 4.0 | 95 | 95 |
| | 30 | 48 | 1.5 | 84 | 81 |
| | 60 | 48 | 1.5 | ~100 | 86 |
| | 100 | 48 | 1.5 | ~100 | 81 |
| | 130 | 48 | 1.5 | 93 | 86 |
| | **100** | **25** | **1.5** | **98** | **87** |
| MgAl | 30 | 48 | 4.0 | 60 | 59 |
| | 100 | 48 | 4.0 | 82 | 89 |
| | 130 | 48 | 4.0 | 84 | 89 |
| | **100** | **25** | **1.5** | **30** | **89** |

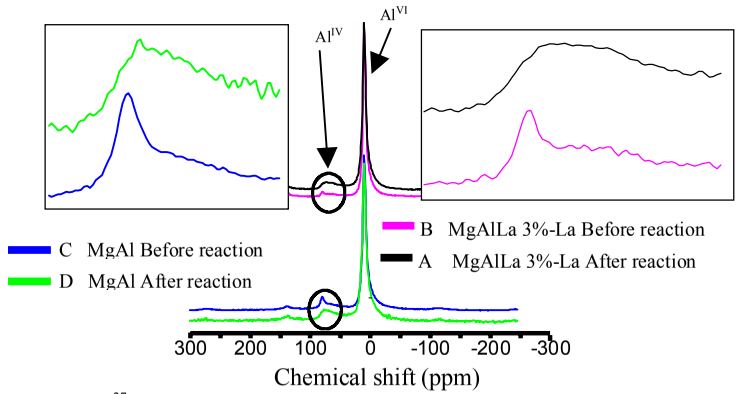


**Figure 1.** NMR ($^{27}$Al) Solids MgAlLa (3% La) y MgAl, before and after reaction.

## Significance

The good yields at room temperature together with the fact that the solid does not require special conditions of handling, makes this material a good candidate to be tested in a higher scale to produce PS.

## References

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