



4th International Conference on Process Engineering and Advanced Materials

Synthesis and Structural Analysis of Double Layered Ni-Mg-Al Hydrotalcite Like Catalyst

Umair Sikander^a, Suriati Sufian^{a,*}, Md. Abdus Salam^a

^aChemical Engineering Department, Universiti Teknologi PETRONAS, Bandar Sri Iskandar, 32610, Perak, Malaysia

Abstract

Hydrotalcites are double layered lamellar clays. It has high surface area and different types of active sites can be fused on its surface this make hydrotalcites an ideal structure for hetero generous catalytic reactions. In this article synthesis of Ni-Mg-Al hydrotalcite by coprecipitation is discussed alongwith its characterization. The hydrotalcite formation is verified by powder XRD. FTIR and BET characterization is carried out to understand the structural characteristics of the material and its possible use as a catalyst. High surface area with large porosity and a consistent atomic percentages was measured during these analysis.

© 2016 Published by Elsevier Ltd. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

Peer-review under responsibility of the organizing committee of ICPEAM 2016

Keywords: Hydrotalcite; double layered hydroxides; coprecipitation; nano-particles

1. Introduction

Several organic production methods of fine chemicals use homogeneous catalysts like minerals, organic acids and bases. Such homogeneous catalysts are difficult to recover, cause waste disposal problems and can corrode the pipes too by salt formations [1]. A greener solution to these problems is by using solid; acid or basic catalysts for heterogeneous catalytic reactions. Furthermore, a polar solvent can completely be removed by their use and such catalysts can also be used under rigorous high temperature and pressure conditions [2].

Layered double hydroxides [LDH], generally named as Hydrotalcites [HTs] is a lamellar clay and is an ideal material for solid catalyst, catalyst precursor and catalyst support [3, 4]. Their layered structure provides high surface area with the freedom to compensate various dimetallic (M^{II}) and trimetallic (M^{III}) ions on its surface [5]. HTs consists of brucite-like positively charged layers, with an anionic layer sandwiched between them compensating the positive charge, along with the water molecules trapped inside these layers[6, 7]. This layered structure of HTs provides high surface area, thermal stability and maximum concentration of active sites on the catalyst surface. HTs existed in the form of both bivalent and trivalent cationic layers, most commonly occurring HT compound is: $Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$ [8]. A variety of highly active catalysts can be made by replacing Mg and Al

* Corresponding author. Tel.: +605-368-7587.
E-mail address: suriati@petronas.com.my

cations by M^{II} and M^{III} catalytically active species respectively in the octahedral sheets during synthesis [9, 10]. It is worth mentioning that both the layered structure (as synthesized) and mixed metallic oxides (obtained after calcinations) is catalytically active in nature [11]. In this work, Nickel is used as an active site, fused within the octahedral layer of Mg-Al hydrotalcites. Its synthesis by co-precipitation method and characterization is presented in this article.

2. Experimental

2.1. Materials

Magnesium Nitrate ($Mg(NO_3)_2 \cdot 6H_2O$), Aluminum Nitrate ($Al(NO_3)_3 \cdot 9H_2O$), Nickel Nitrate ($Ni(NO_3)_2 \cdot 6H_2O$), and Sodium Carbonate (Na_2CO_3) were purchased from Sigma Aldrich® Ltd., for the synthesis of Ni-Mg-Al HTs. Deionized water was used during the synthesis.

2.2. Synthesis of Mg-Ni-Al Hydrotalcite

0.25 M solutions of $Mg(NO_3)_2$ and 0.5 M solutions of $Al(NO_3)_3$ and of $Ni(NO_3)_2$ are prepared. 1 M solution of Na_2CO_3 was used as a precipitating agent. A mixture of known quantities of Mg, Al, and Ni nitrate solution was added drop wise to a vigorously stirred 1 M solution of Na_2CO_3 in a glass reactor. Temperature was kept constant at $70^\circ C$ and pH is kept constant at 10pH by drop wise addition of NaOH solution. Precipitates formed were washed by deionized water followed by filtration. Vacuum filtration of solution was carried out 5 times until the precipitates are free from sodium ions. Precipitates thus produced were then dried in air drier for 18hrs at $80^\circ C$ and are further calcined at $700^\circ C$ for 18hrs. Catalysts of three different concentrations were synthesized by repeating the same method. Their concentrations are given in Table 1.

Table1. Percentage concentration of synthesized Samples

Catalyst	%age Concentration		
	Mg	Ni	Al
MNA-121	25	50	25
MNA-211	50	25	25
MNA-535	12.5	75	12.5

2.3. Characterization

Elemental analysis and topographical images of MNA HTs were taken by Zeiss Supra 55-VP Field-Emission Scanning Electron Microscope (FESEM). Powder X-Ray Diffraction (XRD) patterns of hydrotalcites were recorded in the 2θ range from 2° to 80° with a 0.02 per second step increase on D8 Advance Bruker X-Ray Diffractometer. FTIR spectra is recorded in the range of $400-4400\text{cm}^{-1}$ on Shimdazu 8400S-FTIR. N_2 physisorption at $-150^\circ C$ is used to characterize the BET surface area, pore volume, and pore dia using ASAP 2020 surface area analyzer.

3. Results and Discussion

3.1. Elemental Analysis

EDX analysis of the synthesized HTs was carried out to determine their chemical composition. The analysis revealed that Ni/Mg/Al ratio is in close proximity to the pre-calculated ratios. Measured atomic percentages of HTs compared with calculated values are shown in Table 2. This result confirmed effectiveness of the co-precipitation technique for the synthesis of HTs.

Table2. Measured and calculated Atomic Percentages

Catalyst	Ni:Mg:Al Atomic %age	
	Measured by EDX	Calculated
MNA-121	28:43:28	25:50:25
MNA-211	46:33:21	50:25:25
MNA-535	17:68:15	13:75:12

Download English Version:

<https://daneshyari.com/en/article/853286>

Download Persian Version:

<https://daneshyari.com/article/853286>

[Daneshyari.com](https://daneshyari.com)