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SYNTHESIS AND STUDY OF LAYERED DOUBLE HYDROXIDE AS A ECOFRIENDLY CATALYST

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Abstract:

This chapter describes the preparation, characterization and reasoning of choosing LDH material for further study of research work. This chapter describes detailed information about inorganic semiconductor (i.e. Layered Double Hydroxide). It explains the experimental techniques used for the synthesis of LDH particles and the characterization of synthesized particles. The various results of experimentation are presented along with the scientific explanation of the behavior of various properties of LDH particles.

Introduction:

LDH Particles

LDH have two-dimensional structures such as clay minerals [1]. Such materials may by represent by the following formula:

$$[M^{2+}(1\text{-}x)M^{3+}\ x(OH)_2](An\text{-})x/n\cdot zH_2O$$

Where, M represents metallic ions and A*n*- an interlamellar anion. In order to understand LDH structure, it should start by observing brucite mineral. This mineral has the formula $Mg(OH)_2$, and Mg cations occupy the center of the octahedron, which have hydroxyls in the edges. These octahedra share their edges forming flat and neutral layers, which are connected by H-bonds. In this type of structure, when divalent cations are isomorphically replaced by trivalent cations, the lamellae show a positive residual charge. For the electro neutrality of the system, the presence of anions between the lamellae is required; together with water molecules these anions promote stacking of layers of LDH with an interlayer field poorly ordered. In this case, the lamellae are not only linked by hydrogen bonds, as in the case of the brucite, but also by electrostatics between

the positively charged plates and interlayer anions. The LDH layers can be stacked in two symmetries, resulting in rhombohedric or hexagonal unit cells. Most of the synthetic LDH display hexagonal unit cell; solely LDH with M^{2+}/M^{3+} ratio equals to 1 exhibit orthorhombic unit cell [2].

LDH compounds have been synthesized by direct methods, which include coprecipitation [3-6], chimie douce [7], salt-oxide reaction [8], hydrothermal growth [9-12]. Indirect methods include all syntheses that use an LDH as a precursor. Examples of these are all anion exchange based methods such as direct anion exchange [13], anion exchange by acid attack with elimination of the guest species in the interlayer region [14] and anion exchange by surfactant salt formation [15]. The non-anion exchange methods include the delamination-restacking method and LDH reconstruction method [16-21]. With this review and the results obtained from these preparative routes, Classical Condensation method is found to be easy and suitable method for synthesis of LDH particles, and hence this method is adopted in the present study. We have synthesized and exploded LDH containing 3;1 ratio of $Al(NO_3)$:$9H_2O$ and $Mg(NO_3)$:$6H_20$ [22].

Materials and Methods:

The starting materials $AI(NO_3)_3.9H_2O$, $Mg(NO_3)_2.6H_2O$), 50% aq. NaOH were used of A.R. grade and obtained from commercial sources and used as received, without further purification. Deionized water was used as a solvent.

Synthesis Technique:

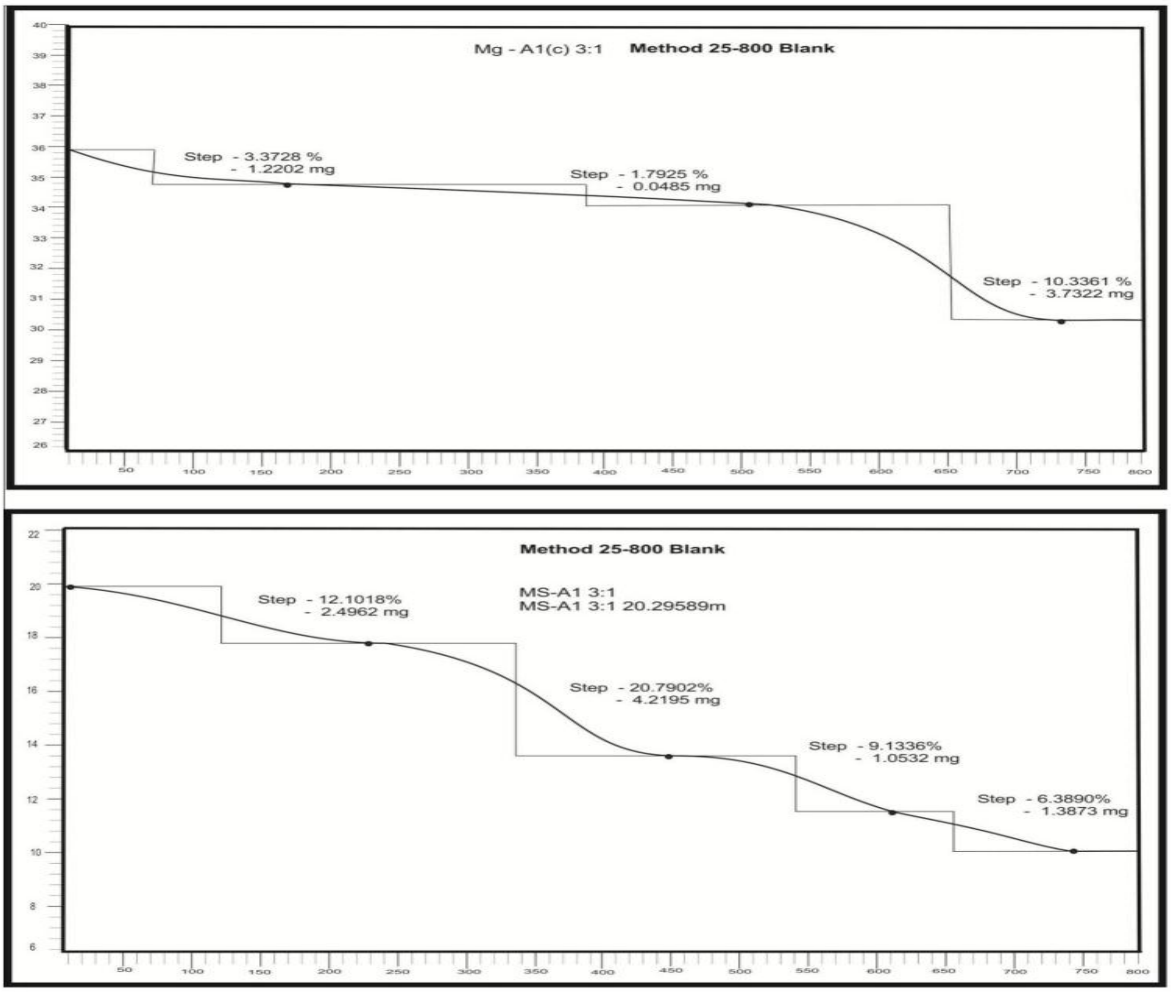
In a given research work classical condensation techniques is used for the synthesis of LDH.

In a typical synthesis, a solution was prepared by dissolving $Mg(NO_3)_2.6H_2O$(9.6g, 0.75M) and $Al(NO_3)_3.9H_2O$(9.25g, 0.25M) in a double distilled water (50 ml) was added drop wise with continuous stirring to 25ml 50% NaOH and pH was maintained close to 10 by simultaneous addition of 2M NaOH solution. The mixture was stirred for 24 hrs at room temperature. The resulting gel like slurry was aged overnight at room temperature, filtered and washed several times with double distilled water and kept in oven for drying.

Results and Discussion:

Thermogravimetric Analysis:

TGA is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes. This measurement provides information about physical phenomena, such as phase transitions, absorption and desorption; as well as chemical phenomena including chemisorptions, thermal decomposition, and solid-gas reactions (e.g., oxidation or reduction). Thermogravemetry curves of synthesized LDH are shown in above figure, it shows two step decomposition, the first one corresponding to removal of water and second one is due to dehydroxalation of layers and subsequent collapse of layered structure. The curve of Mg-Al shows metal oxide formation at 750^0C, without loss of any other as in calcinations they are removed already.

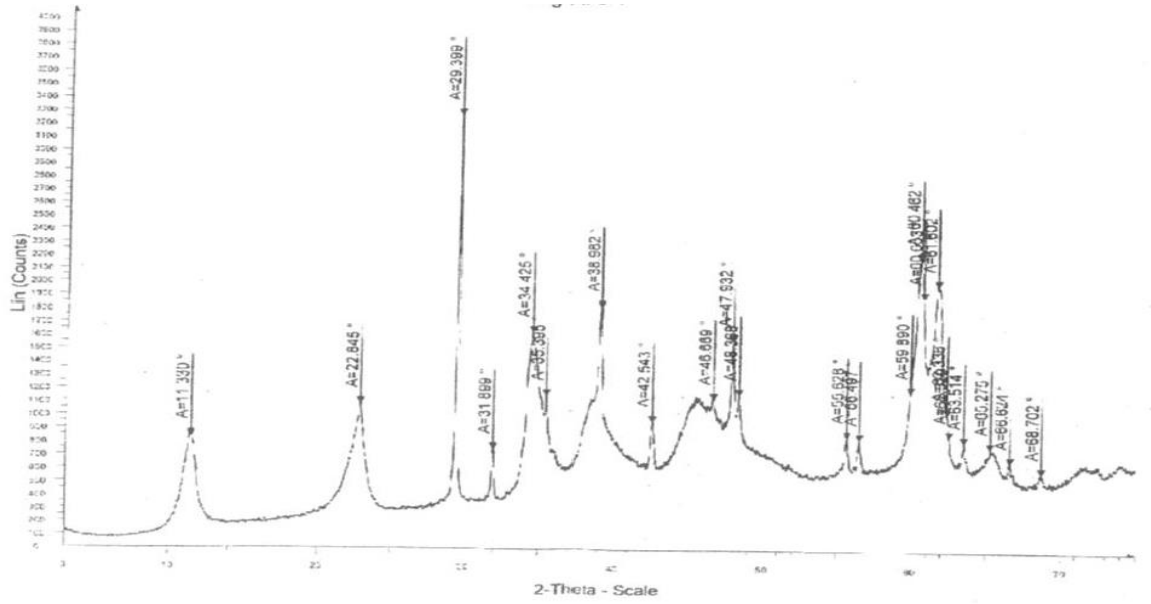


X-ray Diffractogram (XRD):

To confirm the nature of sample i.e. whether they are amorphous or crystalline, the XRD study of sample was carried out on a PHILLIPS HOLLAND PW 1710 X-ray diffractometer using CuKά radiation (λ=0.154056nm), at NEERI Nagpur. It shows the XRD pattern of LDH particles prepared by classical condensation techniques. It shows diffraction peaks at (2θ value) 11.50 and 22.50correspond to two crystal planes (003) and (006) respectively. The broadening of the diffraction peaks indicates the nanocrystalline nature of the samples. This broadness is found to be increased for the capped particles due to decrease of the particles size.

$$D = k\lambda/\beta\cos\theta;$$

Where k is a geometric factor (= 0.9), λ is the X-ray wavelength (1.546 Å), β is the FWHM of diffraction peak and θ is the diffraction angle. The XRD pattern of (Mg-Al) layered double hydroxide and (Mg-Al) calcinated are shown in figure. This (Mg-Al) layered double hydroxide shows well define crystal peak indicating that this compound is crystalline in phase. The appearance of crystallinity in (Mg-Al) LDH is due to inherent crystalline nature of that compound. XRD pattern of (Mg-Al) calcinated LDH shows more crystalline peak than normal due to removal of water from LDH.



Fourier Transform Infrared Spectroscopy (FTIR):

This is one of the most widely used tools for the detection of functional groups in unfilled compounds, mixtures of compounds and as well as compound comparisons. The FTIR technique is very important in this work since it allows one to study the specific interactions between the polymer and inorganic compound. In infrared spectroscopy the main preoccupation of an organic chemist is the region 4000–400 $cm^{-1.}$ This is the most readily examined region which covers the

Absorptions due to the fundamental vibrations of all the common functional groups

Vibration assignments of LDH particles with positions and intensities of absorption shown by IR spectra.

A small Nitrate vibration peak remained at 1384 cm^{-1}, a broad H-bonded OH stretching vibration absorbance at 3381 cm^{-1} within the interlayer as the anion species. The M-O bending vibration was present at 684.75 cm^{-1}. The bending mode band of water molecules observed close to 1680.7 cm^{-1}. These results indicate that the LDH structure and it confirms the presence of carbonate anions and cyanate anions in addition to water molecules inside the interlayer space.

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Conclusion:

After study of XRD, TGA and IR results it confirmed that prepared product is Layered double hydroxide. In this we have prepared Layered Double Hydroxide (LDH) catalyst by using easier method and using inexpensive and easily available non toxic chemicals. The adopted procedure is convenient, involves simple experimental procedure. Due to non toxic nature this Catalyst it will be useful as a ecofriendly catalyst in different reactions.

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