



**HYDROTALCITE-BASED MATERIALS:**  
SYNTHESIS, CHARACTERIZATION AND  
APPLICATION

Editor:  
**Ravi Tomar**

**Bentham Books**

# **Hydrotalcite-Based Materials: Synthesis, Characterization and Application**

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## PREFACE

Hydrotalcite-based materials are widely used in heterogeneous catalysis and other applications. The representation of hydrotalcite (HT) and hydrotalcite-like compounds in the general formula is  $[M(II)_{1-c}M(III)_c(OH)_2]^{c+}(A^{d-})_{c/d} \cdot eH_2O$ . These compounds are well-known in heterogeneous catalysis due to their special properties and tremendous applications like base catalysts for the organic reaction, support for metal catalysts (mono-, bi-, tri-metallic), one-pot synthesis of organic molecules, water treatment, organic transformations, *etc.* A series of hydrotalcite-like compounds have been prepared by the exchange of metals with different ( $M^{2+} = Mg^{2+}, Ca^{2+}, Co^{2+}, Ni^{2+}, Fe^{2+}, Zn^{2+}, Mn^{2+}, Cu^{2+}, Pd^{2+}, Pb^{2+}, Cd^{2+}$ ;  $M^{3+} = Al^{3+}, Fe^{3+}, Cr^{3+}, Ga^{3+}, Rh^{3+}, Ru^{3+}, Y^{3+}, La^{3+}$ ) ion ratios in the brucite layer and anions ( $An^- = Cl^-, F^-, NO_3^-, CO_3^{2-}$ ) in the interlayer gallery. Due to basicity (the presence of hydroxide and hydrogen carbonate and insolubility in water), absorption capacity, and insoluble nature of HT, it has various applications as heterogeneous catalysis as a base: epoxidation (olefins,  $\alpha,\beta$ -unsaturated ketones), aldol condensation of aldehydes and ketones, condensation of the carbonyl group with methylene group-containing compounds (Knoevenagel and Claisen-Schmidt reactions), as metal supported catalyst in aerobic oxidation of alcohols and polyols, synthesis of 2,5-Diformylfuran, glycerol carbonate, 5-hydroxymethylfurfural, 5-Formyl-2-furancarboxylic acid, methyl isobutyl ketone, CO chemisorption and phenol hydrogenation, reduction of nitrates, hydrogenation of nitrobenzene, oxidation and reduction of xylose and glucose, reduction of unsaturated aldehydes/ketones, isomerization, biodiesel synthesis, as raw material for refractories, and many more. Hydrotalcite and Hydrotalcite-like compounds have been synthesized using co-precipitation, sol-gel, microwaves-assisted, and ultrasound-assisted methods. Hence, in this book, hydrotalcite-based materials are summarised along with their applications in various fields such as industrial, catalytic, biomedical, and so on.. We sincerely hope that this book will be a great help to the researchers.

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# Hydrotalcite and Hydrotalcite-Based Materials

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**Abstract:** This chapter deals with the history of hydrotalcite and hydrotalcite-based materials. A rare mineral known as hydrotalcite was found in Sweden sometimes in the 1840s. Magnesium aluminum hydroxycarbonate,  $\text{Mg}_6\text{Al}_2(\text{OH})_{16}\text{CO}_{34}\text{H}_2\text{O}$ , is its chemical name, and Taylor and Allmann independently determined its layered structure. For a long time, hydrotalcite and other isomorphous minerals (such as piroaurite, sjogrenite, and takovite) were the focus of most mineralogical studies. However, beginning in the 1970s, it was discovered that these rare minerals, also known as anionic clays, could be prepared quickly and affordably in a laboratory and have a variety of intriguing chemical properties. The different arrangements of the stacking of the layers, the ordering of the metal cations, as well as the arrangement of anions and water molecules in the interlayer galleries, result in a variety of stoichiometry in hydrotalcite, which are layered double hydroxides. Due to their unique characteristics, including their enormous surface area, ion exchangeability, insolubility in water, and most organic sorbents, among others, the compounds of the hydrotalcite group demonstrate a wide variety of potential uses.

**Keywords:** Acid neutralization, Brucite, Building materials, Carbon dioxide absorbers, Co-precipitation, Corrosion inhibitors, Hydrotalcite, Hydrotalcite-like compounds (HTlc), Layered double hydroxide, Photocatalyst, Polymer composites.

## 1. INTRODUCTION

Hydrotalcites, which exist in both natural and synthetic forms, exhibit a structural relationship with the mineral brucite, denoted as  $\text{Mg}(\text{OH})_2$ . Hydrotalcites typically consist of three distinct layers: an inner intermediate layer that accommodates water molecules and replacement anions, as well as two outer layers characterized by a positive charge and one outer layer characterized by a negative charge. The mineral hydrotalcite, with the chemical formula  $\text{Mg}_6\text{Al}_2(\text{OH})_{16}\text{CO}_{34}\text{H}_2\text{O}$ , is widely recognized as the most prominent and ancient example of layered double

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hydroxides (LDHs) [1]. In this study, we aim to investigate the impact of social media usage on mental health. For a considerable period, extensive mineralogical investigations primarily centered around hydrotalcite and other isomorphous minerals, including piroaurite, sjogrenite, and takovite. However, a significant shift occurred in the 1970s when it was revealed that these uncommon minerals, commonly referred to as anionic clays, could be readily and inexpensively synthesized within a laboratory setting. Moreover, these synthetic anionic clays exhibited a diverse range of captivating chemical characteristics.

The compounds produced were known as layered double hydroxides (LDH) or hydrotalcite-like compounds (HTlc), and they are usually denoted with the empirical formula  $[M(II)_{1-x}M(III)_x(OH)_2]^{x+} [A^{n-}_{x/n}]^x \cdot mH_2O$ , here M(II) and M(III) are appropriately sized bivalent and trivalent metal cations, respectively. The molar ratio of trivalent cations in the hydroxide layers,  $M^{3+}/(M^{2+}+M^{3+})$ , is used as the value of x in the calculation above, and it typically ranges between 0.20 to 0.33 [2]. These substances possess a wide range of applications, including their utilization as agents for decolorization, stabilizers, filters, adsorbents, catalyst supports, and ion exchange materials [3]. In recent studies, researchers have explored the potential of utilizing these materials for CO<sub>2</sub> adsorption due to their notable characteristics, including their strong adsorption behaviour [4], stable anion exchange properties, efficient mobility of anions and water molecules facilitated by their expansive surface areas, and robust frame structure.

Hydrotalcites have been widely employed in the production and processing of polymers, primarily in the capacity of flame retardants, neutralizing additives, and thermal stabilizing compounds for PVC processing [5 - 7]. Hydrotalcites that are extensively employed in many applications within the field of heterogeneous catalysis, primarily serving as precursors for the synthesis of catalysts that rely on mixed oxides. Furthermore, they can be employed in sorption and cleaning methodologies, as well as for the intercalation of various molecules, including pharmaceutical substances.

In the interlayer region, the presence of anions and water molecules balances out the extra positive charges that come from the substitution of trivalent cations. This creates an appropriately electrically balanced structure [8]. The chemical structures of hydrotalcites can be modified to enhance their adsorption activity and postpone particle sintering by including dopants such as alkali metals or altering the metal composition of the framework. To make it easier for carbon dioxide molecules to be absorbed, HTlcs with the carbonate ion have more empty space and wider gaps between the layers than those with OH (0.765 nm vs. 0.755 nm) [8, 9].

## 2. HISTORY AND MAIN FACTS ON HYDROTALCITE BASED MATERIALS

A study by Yong *et al.* found that the best CO<sub>2</sub> adsorption happened when the composition had an equal amount of divalent (Mg<sup>2+</sup>) and trivalent (Al<sup>3+</sup>) cations. The addition of aluminium in HTLCs leads to an enhancement of layer charges, resulting in a contraction of the interlayer gap and a reduction in the number of adsorption sites.

The alteration of the anion from OH<sup>-</sup> to CO<sub>3</sub><sup>2-</sup> in the HTLCs resulted in an increase in adsorption capacities from 0.25 to 0.5 mmol/g at a temperature of 300 °C and a pressure of 1 bar. This can be attributed to the larger interlayer spacing and higher layer charge observed in the HTLCs with CO<sub>3</sub><sup>2-</sup> anions.

In their study on Mg-AlCO<sub>3</sub> layered double oxide (LDO), Reddy *et al.* observed both physisorption and chemisorption happening at the same time during the adsorption process. Additionally, they observed both reversible (physical) and irreversible (chemical) adsorption of CO<sub>2</sub>. As the increase in temperatures occurred, there was a noticeable decrease in the adsorption capacity. Nevertheless, it was observed that the optimal capture capacity was achieved at a temperature of 200 °C instead of 100 °C, indicating that physical adsorption plays a more significant role at a higher temperature. This shows that CO<sub>2</sub> adsorption on Mg-Al-CO<sub>3</sub> layered double oxide (LDO) is a process that involves both chemical and physical processes that happen in many places. A significant proportion of the adsorption seen within the examined temperature range, specifically over 78%, was found to be reversible. Increased kinetic energy or elevated temperatures enhance the favourability of desorption from the surface of the sorbent.

This chapter will primarily focus on the synthesis of hydrotalcite and its various applications in construction materials, radioactive waste repositories, corrosion prevention in reinforced concrete, cements and mortars, polymer composites, and acid neutralization. Hydrotalcite has garnered significant attention from the scientific community due to its diverse applications. Consequently, there has been a growing body of literature exploring the utilization of hydrotalcite-based products and their possible environmental advantages.

### 2.1. Hydrotalcites Synthesis

The most common way to make hydrotalcite is for two metal salts to react with each other in an alkaline solution that stays at a pH level of about 10. Usually, the hydrothermal method is used to make hydrotalcite, which means that the best reaction conditions need to be set up to get consistent product quality [10 - 14].

## CHAPTER 2

# Synthesis and Characterization of Hydrotalcite-Based materials

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**Abstract:** Hydrotalcite (HT) has the chemical formula  $Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$ , with a stacked crystal structure comprising layers of positively charged hydroxides that are interlayer anion neutralization as carbonate and contain  $H_2O$  particles. These double hydroxides in layers (LDH) are a class of highly fascinating materials for the industry due to the simplicity of their production and the ability to add additional layer cations and interlayer anions. Hydrotalcite-based materials such as magnesium aluminide (MgAl) hydrotalcite with a range of magnesium to aluminium (Mg/Al) molar ratios are used to prepare catalysts for effective changes of organic molecules. X-ray diffraction and scanning electron microscopy (SEM) characterization measure the catalyst's crystallinity, surface area, and shape. The HT has been employed as a scaffold for immobilizing numerous metals, enabling highly selective organic reactions, *i.e.*, the dehydrogenation of alcohols and the deoxygenation of epoxides. It may also work with other metal catalysts to speed up subsequent responses in a single pot. It offers a vital method for the environmentally responsible synthesis of valuable compounds. This chapter overviews the hydrotalcite synthesis, classification, and application research.

**Keywords:** Characterization, Hydrotalcite, Hydrotalcite synthesis, Layered double hydroxide.

## 1. INTRODUCTION

In 1842, the mineral  $Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$ , known as hydrotalcite, was discovered in Sweden. It has a well-organized crystal structure comprising layers of positively charged hydroxide balanced by carbonate-containing intermediate anions. A set of synthetic materials have a configuration resembling a layer. It has divalent cations in  $(Mg(OH)_2)$  in which some of the divalent cations have been swapped out for trivalent ones referred to as "hydrotalcite". Fig. (1) illustrates the result of this substitution, which created positively charged sheets piled on top of one another and kept electrically neutral by intercalating anions [1, 2]. They may be written as  $[M_xII My_{III} (OH) (2_x+2_y)]$  in their general formula. Layered double

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hydroxides (LDHs) or anionic clays are isostructural materials formed when the water content ( $m$ ) and interlayer anions (for example,  $A^{n-}$ ,  $Cl^-$ ,  $(CO_3)^{2-}$ ,  $OH^-$ ,  $(NO_3)^-$  and  $(SO_4)^{2-}$ ) vary widely. In contrast to typical clays, which have positive interlayer charges [3 - 5].

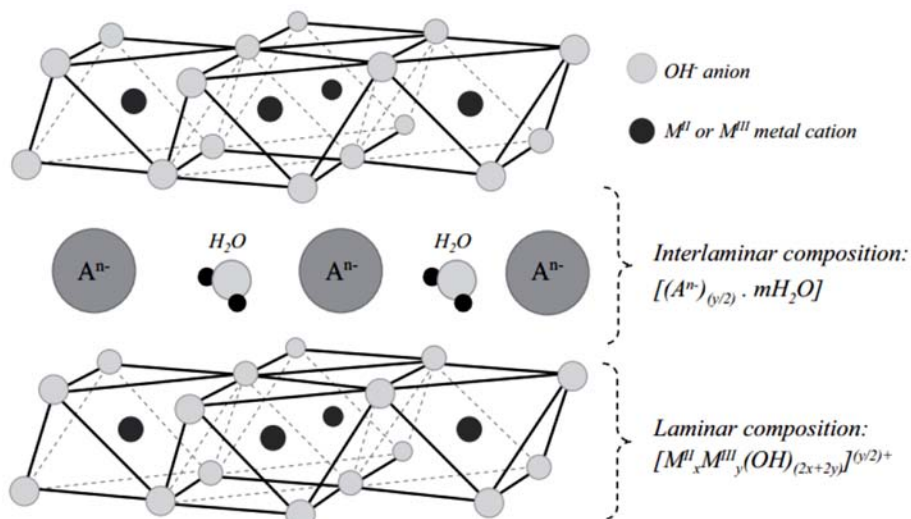


Fig. (1). Hydrotalcite structure [1].

In the past ten years, magnesium-aluminium hydrotalcite (MAH,  $Mg_6Al_2(OH)_{16}(CO_3) \cdot 4H_2O$ ) has garnered the most interest among the different types of hydrotalcite due to its numerous technological uses, including its use as a polymer composites' acid scavenger and clinic anti-acid [6, 7].

The HT has received much interest because of its adsorption capacity, cation-exchange ability of the Brucite layer, anion-ion exchangeability of the intermediate space, and variable basicity of the surface. These characteristics of the HT have been handled using a variety of synthetic approaches, using one-pot synthesis with materials obtained from biomass for green and sustainable chemistry. These methods employ the HT as a metal support and heterogeneous base catalyst for powerful liquid-phase organic reactions [8 - 10].

Compared to the standard HT with a 2D structure like a plate, the HT with a 3D design offers more wider application potential in electrochemistry, biomedicine, catalysis, and adsorption. This results from their outstanding chemical stability and adaptability, attracting several scientists to conduct an in-depth study [11 - 14]. Self-assembling lamellae with uniform size and good crystallinity are often responsible for producing hydrotalcite-like compounds (HTLCs) with 3D layered structures. Nevertheless, hydrotalcite made using traditional approaches usually

has a morphology that resembles platelets and stone blocks [15]. Numerous researchers have controlled the formation of hydrotalcite lamellae and then forced the lamellae to self-assemble to create 3D lamellar structures by adding a third component (template and surfactant) or changing the solvent. LDHs with a 3D multilayer structure that can be entirely hydrolyzed by alkali dissolution were created [16]. Induction was carried out using  $\text{SiO}_2$  as a sacrifice template. However, the results demonstrated that post-processing (the template removal approach) impacted the crystallinity and crystal shape of the output [17 - 19]. Similar studies showed the limits of peaceful testing environments and excessively complex post-processing methods. The three-dimensional (3D) structure of HTLCs must thus be produced in a single step without further processing.

## 2. SYNTHESIS OF MAGNESIUM-ALUMINIUM HYDROTALCITE (MAH)

MAH may be produced in various methods, each of which has a reactant and the finished product, distinct time-energy profiles, and purity requirements exist [20 - 24]. The most widely used technique makes use of creating an aqueous solution of aluminium and magnesium salts ( $\text{AlNO}_3$  and  $\text{MgCl}_2$ , respectively) as the first step in the co-precipitation process (Fig. 2) for instance, Fig. (2a) while maintaining a close to 3:1 or 2:1 (this ratio specifies the amount of substitution of the trivalent cations in the  $\text{Mg}(\text{OH})_2$  layers) molar ratio between them. The  $\text{Mg}^{2+}$  and  $\text{Al}^{3+}$  cations still dissolve in the steady-state solution at neutral-acid pH (Fig. 2b). The pH level of the solution is then progressively elevated to get the double hydroxide to precipitate typically,  $\text{NaHCO}_3$  or  $\text{NH}_4\text{HCO}_3$  are usually used for this since they also function as sources of  $(\text{CO}_3)^{2-}$  anions, as illustrated in Figs. (2c and d) [23, 24]. Adjusting the stirring force, pH, temperature, or nucleation-precipitation process may be regulated to provide the appropriate particle dimensions and form (Fig. 2e) [24]. The counter-cation used to elevate the pH (in this case,  $\text{Na}^+$  or  $\text{NH}_4^+$ ) remains in the hydrotalcite solution after the reaction. It must be removed typically using ultra-filtration, dialysis, or centrifugation. (Fig. 2f). The particle size distribution may be changed, and the particle crystallinity can be enhanced (Figs. 2g and h) using additional chemical methods [25]. To stop particle aggregation, use a dried powder, spray-drying, or lyophilization (Fig. 2i) [7].

Solution A was created by dissolving magnesium and aluminium nitrates in 150 mL of deionized water at a stoichiometric ratio (30.7688 g and 0.12 mol, respectively). In 150 mL of deionized water, 12.0 g (0.3 mol) of sodium hydroxide or 1.590 g (0.015 mol) of sodium carbonates were dissolved to produce Solution B. At around two drops per second, solutions A and B were simultaneously injected into a flask using a burette. The mixture was swirled to



## Divergent Applications of Hydrotalcite-Based Materials

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**Abstract:** Hydrotalcites (HDTL) are layered double hydroxides of the anionic clay family. They possess a large surface area, ability to accommodate divalent and trivalent metallic ions, anion exchange capacity and intercalation ability. HDTL play a vital role in nanotechnology, specifically in various nanomaterial production, functionalization, and applications. HDTL nanohybrids with unique properties are created through intercalation with various compounds like inorganic anions, organic anions, biomolecules, active pharmaceutical ingredients, and dyes. Their adaptive layered charge density and chemical combination constitute HDTL as resourceful materials befitting for a broad spectrum of applications. There are a variety of methods for preparing HDTL based nanomaterials, including co-precipitation, sol gel method, ion exchange method, intercalation method and microwave assisted methods. The morphologies of HDTL materials are characterised using technologies like X-ray powder diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Thermogravimetry coupled (TGA) with Differential Scanning Calorimetry (DSC), Scanning Electron Microscopy (SEM), and Transmission Electron Microscopy (TEM). The nanocomposites of HDTL are widely used in the field of fine chemical synthesis, pharmaceutical field, water purification, and agriculture. Biocompatible HDTL nanostructures enticed remarkable attention in therapeutic and diagnostic functions. HDTL nanohybrids are prominent bio reservoirs for drug and delivery systems and used in cancer therapy. These materials have been utilised by bioimaging techniques such as MRI and CT. The HDTL-based nanomaterials are effective adsorbents and find widespread application in the water treatment industry. These are used for the amelioration of polluted water by removing heavy metals, dyes, and other impurities. These materials are also used as flame retardants, in porous ceramics, carbon dioxide adsorption and deodorants. This chapter describes in detail about the preparation methods, properties, structural characterisation, and wide applications of HDTL based nanohybrids.

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**Keywords:** Antibacterial, Biocompatibility, Bioimaging, Biosensors, Brucite, Co-precipitation, Cytotoxicity, Diagnostic, Drug delivery, Flame-retardants, Gene delivery, Intercalation, Ion-exchange, Layered Double Hydroxides, Nanocomposite, Photodynamic therapy, Porous ceramics, Theranostics, Therapeutic, Tissue engineering.

## 1. INTRODUCTION

Layered double hydroxides (LDH), also known as hydrotalcites (HDTL), are a wide class of naturally occurring and man-made minerals whose characteristics are strikingly similar to those of clay minerals, notably cationic clay [1]. HDTL is a particularly significant class of compounds in industry due to their specific morphology with vast surface area, capacity to accommodate divalent and trivalent cations, diversity of their chemical composition, and special anion exchange and intercalation capabilities [2].

The topic of nanotechnology, which focuses on the manipulation and control of matter at the nanoscale level (about 1 to 100 nm), has been gaining prominence recently due to the potential applications and revolutionary effects it has in a number of different areas. Reducing pollution and cleaning up the environment are two areas where nanotechnology might make a significant difference [3, 4]. Hydrotalcite (HDTL)-based nanomaterials have gained significant attention in various fields, including catalysis, adsorption, drug delivery, environmental remediation, and energy storage owing to their unique structural dimensions. These nanomaterials are derived from HDTL, which are layered double hydroxides (LDHs) with a unique structure and a wide range of applications. HDTL have a layered crystal structure composed of positively charged metal hydroxide layers and interlayer anions. The metal hydroxide layers consist of divalent (M(II)) and trivalent (M(III)) metal cations coordinated to hydroxide ions, forming a brucite-like structure. The general formula for HDTL is  $M(II)_{1-x}M(III)_x(OH)_2 \cdot nH_2O$ , where M(II) and M(III) represent divalent and trivalent metal cations, respectively, x represents exchangeable anions, and n represents the number of water molecules in the interlayer region. These layers are positively charged due to the presence of metal cations with a +2 charge. The interlayer region contains exchangeable anions and water molecules, providing opportunities for the intercalation of various species including inorganic anions like chloride, carbonate, nitrate, and phosphate, organic anions like carboxylates and sulphates, biomolecules like DNA, RNA, proteins and enzymes, active pharmaceutical ingredients, and dyes [5, 6]. The ability to intercalate different ions, molecules, or nanoparticles into the interlayer region is one of the defining characteristics of HDTL-based nanomaterials. This intercalation process allows for the modification and functionalization of the material, tailoring its properties

and extending its applications. The intercalated species can impart catalytic, adsorption, or other specific functionalities to the nanomaterials [7]. HDTL offer a wide range of possibilities for various applications due to their unique structure, tailorable properties, and intercalation capabilities. Nanomaterials made of HDTL have enormous potential as catalysts and catalytic supports. The excellent adsorption properties of HDTL nanoparticles make them helpful for removing contaminants, heavy metals, and organic molecules from water and other aqueous solutions [8]. Due to this, these materials have been employed in environmental remediation processes [9, 10]. Drug delivery methods based on HDTL offer better drug stability, increased bioavailability, and tailored distribution, resulting in more efficient and individualised therapies. HDTL nanomaterials can be incorporated into polymer matrices to form nanocomposites with improved mechanical, thermal, and barrier properties and they are used as flame retardants in the textile industry [11 - 13]. Their versatility and multifunctionality make them a promising platform for advancing technologies in diverse fields [14 - 16].

## **2. STRUCTURE AND PROPERTIES OF HYDROTALCITE-BASED NANOMATERIALS**

HDTL nanoparticles are extremely adaptable and suited for a wide range of applications due to their distinctive structure and combination of characteristics.

### **2.1. Structure**

HDTL nanoparticles differ from their bulk counterparts due to particular structural characteristics. Key features of the structure of HDTL nanocomposites are summarised as follows [17, 18].

#### **2.1.1. Particle Size**

The sizes of HDTL nanoparticles typically range from a few nm to about 100 nm. Due to their small size and high surface area to volume ratio, they are more reactive and interact with other substances on a larger scale.

#### **2.1.2. Layered Structure**

Metal hydroxide sheets are layered in layers to form HDTL-based nanomaterials. These layers are made up of hexagonally close-packed layers of positively charged metal cations, usually divalent (M(II)) and trivalent (M(III)) metals.

#### **2.1.3. Interlayer Region**

An interlayer area separates the metal hydroxide layers. This interlayer area in HDTL comprises water molecules and exchangeable anions. The interlayer

## Hydrotalcite-Based Materials In Nanotechnology

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**Abstract:** This book chapter focuses on the use of hydrotalcite-based materials in nanotechnology. Hydrotalcites are layered double hydroxides with unique structures and properties that make them attractive for various applications in nanotechnology. The chapter provides an overview of the synthesis, characterization, and applications of hydrotalcite-based materials in various fields of nanotechnology, including catalysis, drug delivery, sensing, and environmental remediation. The chapter also discusses the potential challenges and future directions in the field of hydrotalcite-based nanotechnology. Overall, this chapter highlights the importance of hydrotalcite-based materials as promising candidates for developing advanced nanotechnological applications.

**Keywords:** Hydrotalcites, Hydrotalcite synthesis, Material Science, Nanotechnology, Nanocatalyst.

### 1. INTRODUCTION

#### 1.1. A Brief Overview of Hydrotalcites and their Structure

Hydrotalcites, also known as layered double hydroxides (LDHs), are synthetic materials that possess a unique layered structure [1]. They have garnered significant attention in various scientific fields due to their versatile properties and wide range of applications. In this chapter, the author has provided a brief overview of hydrotalcite, explored their structure in detail, and highlighted some notable examples of their applications.

Hydrotalcites are classified as anionic clays, which are a subclass of layered double hydroxides. These materials consist of positively charged metal hydroxide layers interleaved with anions in the interlayer region. The metal cations within the layers are typically divalent, such as magnesium ( $Mg^{2+}$ ) or zinc ( $Zn^{2+}$ ), while

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the anions can be carbonate ( $\text{CO}_3^{2-}$ ), hydroxide ( $\text{OH}^-$ ), or other species. The layers are held together by electrostatic forces, allowing for the exfoliation and restacking of the material [2].

One notable feature of hydrotalcite is its ability to undergo anion exchange. The interlayer anions can be easily replaced by different anions through ion exchange reactions. This property makes hydrotalcite highly versatile, as its composition can be tailored to suit specific applications [3]. For example, by selecting suitable interlayer anions, hydrotalcite can be used as an effective catalyst in various chemical reactions. They can also act as host materials for the controlled release of drugs and other active compounds.

The synthesis of hydrotalcite involves various methods, including co-precipitation, hydrothermal synthesis, and ion exchange. Co-precipitation is a commonly employed method, where aqueous solutions containing metal cations and anions are mixed together under controlled conditions. The resulting precipitate is then washed and dried to obtain hydrotalcite-like materials [4]. Hydrothermal synthesis involves the reaction of metal salts and alkali hydroxides under elevated temperatures and pressures, leading to the formation of hydrotalcites. Ion exchange methods involve the exchange of interlayer anions in pre-existing hydrotalcites with desired anions.

To further understand the structure of hydrotalcite, various analytical techniques are utilized. X-ray diffraction (XRD) is widely employed to determine the layer spacing and crystalline nature of hydrotalcite. XRD patterns exhibit characteristic peaks corresponding to the periodic arrangement of layers within the material. Fourier-transform infrared spectroscopy (FTIR) is used to identify the vibrational modes of functional groups in hydrotalcite, providing information about the nature of interlayer anions and the presence of water molecules. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are employed to examine the morphology and particle size of hydrotalcite samples [5, 6]. Hydrotalcites have been employed as catalysts in biodiesel production, organic synthesis, and environmental remediation processes [7]. In addition to these techniques, other characterization techniques such as BET surface area analysis, thermogravimetric analysis (TGA), and photoelectron spectroscopy (XPS) can also be used to further understand the physical and chemical properties of hydrotalcite-based materials. BET surface area analysis can be used to determine the surface area of the material and its pore size distribution. TGA is used to determine the thermal stability of the material, while XPS can provide information about the elemental composition and chemical state of the material.

In addition to catalysis, hydrotalcite finds applications in adsorption and ion exchange processes. The anion exchange capability of hydrotalcite allows them to selectively remove certain contaminants from water or other solutions. For example, hydrotalcite has been utilized for the removal of heavy metal ions, organic dyes, and phosphates from wastewater. They can also be used as ion exchangers in the purification of drinking water and the removal of pollutants from industrial effluents.

Hydrotalcites have shown promise in the field of drug delivery as well. Their layered structure and the ability to accommodate different anions make them suitable for the encapsulation and controlled release of drugs. By incorporating drugs into the interlayer space of hydrotalcite, it is possible to enhance their stability, control their release kinetics, and protect them from degradation. This application has potential implications in the pharmaceutical industry for the development of improved drug formulations with enhanced therapeutic efficacy [8, 9].

Furthermore, hydrotalcite has been explored for its use in environmental remediation processes. Due to their anion exchange capability, they can effectively adsorb and remove pollutants from soil and water systems. For instance, hydrotalcite has been investigated for the removal of heavy metals, arsenic, and organic contaminants from contaminated environments. Their use in water treatment processes holds promise for the purification of drinking water and the remediation of polluted water bodies.

## **1.2. Importance of Hydrotalcite in Nanotechnology**

Hydrotalcites, or layered double hydroxides (LDHs), play a crucial role in nanotechnology due to their unique properties and versatile applications. Their significance lies in their tuneable structure, high surface area, and anion exchange capability. In this section, we will explore 10 real-time examples that highlight the importance of hydrotalcite in nanotechnology.

### **1.2.1. Nanocatalysis**

Hydrotalcites serve as effective catalysts in various nano-catalytic reactions. For instance, Wang *et al.* utilized hydrotalcite-supported metal nanoparticles as catalysts for hydrogenation reactions, demonstrating their superior catalytic performance and stability [10].

# Industrial Application of Hydrotalcite-Based Materials

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**Abstract:** Hydrotalcite is a layered double hydroxide (LDH) with a wide range of applications in catalysis, flame retardancy, PVC stabilization, fillers and reinforcements, additives in coatings and paints, pharmaceuticals and personal care, environmental remediation, and other industrial applications. This paper provides an overview of the properties and characteristics of hydrotalcite, as well as its potential applications in various industries.

The paper begins with an introduction to hydrotalcite, including its definition, structure, synthesis methods, and properties. The next section discusses the role of hydrotalcite as a catalyst, as well as its applications in the petrochemical industry and environmental remediation. The following section focuses on the flame retardant and smoke suppression properties of hydrotalcite, and its applications in plastics and polymers. The subsequent section discusses the use of hydrotalcite as a PVC stabilizer and its benefits and applications in the PVC industry.

The following sections discuss the use of hydrotalcite as a filler and reinforcement in composite materials, as an additive in coatings and paints, and in pharmaceutical and personal care formulations. The next section discusses the applications of hydrotalcite in water treatment, soil remediation, and battery and energy storage. The final section summarizes the future prospects and challenges of hydrotalcite, including emerging research areas, challenges and limitations, and potential for further industrial applications.

This paper provides a comprehensive overview of the properties and applications of hydrotalcite. It is intended to be a valuable resource for researchers, engineers, and other professionals who are interested in this versatile material.

**Keywords:** Additives in coatings and paints, Batteries, and energy storage, Catalysis, Environmental remediation, Flame retardancy, Fillers and reinforcements, Hydrotalcite, Layered double hydroxide, LDH, PVC stabilization, Pharmaceuticals and personal care.

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## 1. INTRODUCTION TO HYDROTALCITE

### 1.1. Definition and Structure of Hydrotalcite

Hydrotalcite is a naturally occurring or synthetic compound that belongs to a family of layered double hydroxides (LDH) [1]. It is also known for its chemical formula  $Mg_6 Al_2 (CO)_3(OH)_{16} \cdot 4H_2O$  [1 - 5]; the term 'hydrotalcite' [1] is often used as a generic name for a class of materials that have a similar layered structure and can incorporate various metal ions [2] in their structure. These metal ions can include other divalent cations like zinc ( $Zn^{2+}$ ), nickel ( $Ni^{2+}$ ) [2], and iron ( $Fe^{2+}$ ) in addition to magnesium ( $Mg^{2+}$ ) and aluminum ( $Al^{3+}$ ).

Hydrotalcite is also known as a type of anionic clay due to its layered crystal structure [3]. The structure of hydrotalcite [3] can be described as a series of positively charged brucite-like layers. Between the positively charged layers [3, 4], there are interlayer regions containing anions and water molecules. These layers consist of octahedrally coordinated metal hydroxide sheets, typically composed of magnesium (Mg) and aluminum (Al) ions [4]. The layers are separated by interlayer anions, which are usually carbonate ( $CO_3^{2-}$ ) ions. The chemical formula for hydrotalcite represents the stoichiometry of the compound [5]. Carbonate ions, chloride ions ( $Cl^-$ ), and nitrate ( $NO_3^-$ ) ions balance [6] the positive charge of the hydroxide layers. The  $H_2O$  molecules in the interlayer [6] region are weakly bonded to the hydroxide and carbonate layers [5, 6].

Hence the layers of hydrotalcite can be described as a hexagonal arrangement of hydroxide ions [7, 8], with the divalent and trivalent [7] metal cations occupying the octahedral sites in an alternating manner [6, 7]. The water molecules are weakly bound and can be easily removed through heating, resulting in a dehydration process [5 - 7].

Hydrotalcite is known for its excellent anion exchange properties [8, 9], which arise from the ability of the intercalated anions to be exchanged with other anions [8, 9] in solution. This property makes hydrotalcite a valuable material for various applications, including catalysis, adsorption [9], wastewater treatment, and as a host for the controlled release of pharmaceuticals or other functional compounds [9]. Overall hydrotalcite possesses a layered structure with alternating hydroxide layers and interlayer regions as shown in Fig. (1), making it a versatile material with applications in distinguished fields.



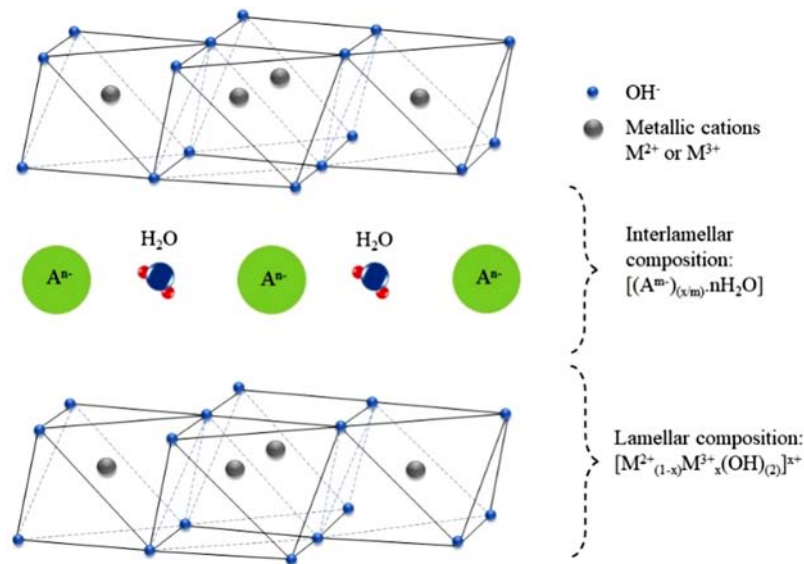


Fig. (1). The typical composition (general structure) of Hydrotalcite [8].

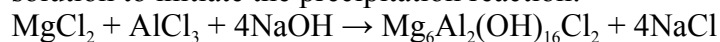
## 1.2. Synthesis Methods

Hydrotalcite can be synthesized through several methods, both in the laboratory and industrially [10]. The synthesis of hydrotalcite involves the precipitation of metal hydroxides followed by subsequent hydrothermal treatment or anion exchange [11]. Here are some common synthesis methods for hydrotalcite along with reactions.

### 1.2.1. Co-precipitation Method

It involves [12] the simultaneous precipitation of divalent metal ions (typically magnesium) and trivalent metal ions (typically aluminum) in a basic solution. The metal salts, such as magnesium nitrate [ $Mg(NO_3)_2$ ], magnesium chloride ( $MgCl_2$ ) [13], aluminum nitrate [ $Al(NO_3)_3$ ], and aluminum chloride ( $AlCl_3$ ) [13] are dissolved in water, and a base, such as sodium hydroxide, is added to raise the pH. This results in the formation of hydrotalcite precipitates, which are then separated and washed. Step-by-step description of synthesis [14] is shown in Fig. (2):

- i. Prepare a solution containing magnesium and aluminum salts [13] in the desired stoichiometric ratio.
- ii. Add a powerful base, such as sodium hydroxide [11] ( $NaOH$ ), to the metal salt solution to initiate the precipitation reaction:



## Catalytic Application of Hydrotalcite-Based Materials in Organic Transformation

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**Abstract:** Hydrotalcite-based materials or Hydrotalcites (HTs) have gained significant attention in recent years as efficient and versatile catalysts for various organic transformation reactions. The tunable surface properties, high surface area, and excellent thermal stability make them ideal catalysts for a range of chemical reactions. This book chapter provides an overview of the recent advances in the application of hydrotalcite-based materials as catalysts in organic transformations. The chapter highlights the various catalytic reactions where hydrotalcite-based materials have shown significant potential in the synthesis of a range of heterocycles such as chromenes, pyrans, pyrazoles, and triazoles, as well as oxidation, reduction, and C-C bond formation reactions. The chapter also discusses the various modifications that can be made to hydrotalcite-based materials to enhance their catalytic activity, selectivity, and stability by tailoring the electronic structure of the catalysts and supports. Additionally, the chapter covers the use of hydrotalcite-based materials in the synthesis of fine chemicals, pharmaceuticals, and polymers. Overall, this book chapter provides a comprehensive overview of the catalytic application of hydrotalcite-based materials in organic transformations. It will be of great interest to researchers and professionals in the fields of catalysis, organic chemistry, and materials science, as well as to those interested in developing more sustainable and efficient catalytic processes.

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**Keywords:** Heterocycles, Heterogeneous catalysis, Hydrotalcites (HTs), Multicomponent reactions, Organic transformations, Pyran, Pyrazole, Synthesis.

## 1. INTRODUCTION

Modern organic synthesis has advanced significantly from traditional methods to environmentally friendly ones. Green chemistry principles are used in these eco-friendly strategies [1]. Among these, the creation of organic transformation reactions has greatly advanced the understanding of the atom economy. These reactions have drawn the attention of pharmacologists and biologists due to their ability to synthesize physiologically active substances in one place. Organic transformation reactions are mainly of two types: (a) two-component reactions and (b) multicomponent reactions (MCR). In these types of reactions, one-pot synthesis not only saves time and energy by lowering the number of reaction steps but also costs less in terms of atoms. The use of benign solvents in procedures is one method for making one-pot synthesis even greener. Several factors play an important role when selecting a green solvent for a given reaction, in a Kamlet-Taft plot, the solvent and energy required to make it, its effects on human health and the environment, and its ease of removal from products without distillation. In a few instances, using water as a solvent to conduct reactions resulted in 300-fold acceleration [2]. In addition to solvent-related environmental concerns, traditional organic transformation reactions also involve harsh mineral acids and alkalis. The development of heterogeneous catalytic systems, where catalysts act as solid acids and bases, can help address this. One of the reasons why solid heterogeneous catalysts are preferred in the chemical industries is because they are thermally stable, reusable, and easily removable from the reaction fluid [3]. The number of surface-active sites and their proximity to the reactants determine the catalyst's activity in heterogeneous catalysis [4]. Numerous kinds of inorganic compounds, including zeolites and clays, have been considered in the search for the ultimate solid catalyst. Moreover, modified clays, and hetero-poly acids are also used, which have large specific surface areas, chemical and thermal stability, and the presence of active groups that can activate the adsorption reagents for acid-base or redox reactions on their surfaces. In this context, other solid heterogeneous catalysts like Hydrotalcites (HT) or related molecules act as an excellent substitute in comparison to homogenous organic catalysts due to their easy removal from the reaction system and thus represent the excellent applicability with respect to economic and environmental prospects in both smaller and larger-scale industrial applications.

## 2. DEFINITION OF HYDROTALCITE

Hydrotalcites (HT) are 2-D (two-dimensional) sheets of mixed hydroxides of water, and exchangeable charge-compensating anions [5 - 7] that are positively charged, either naturally or artificially. One of the groups of minerals with a layered structure that resembles talc is hydrotalcite (HT). HTs are primarily anionic clays, which are made up of layered double hydroxides (LDHs), have a general molecular formula of  $[M1^{2+}\text{-}xMx^{3+}(\text{OH})_2(\text{An})_{x/n}\cdot y\text{H}_2\text{O}]$  [8], and are expressed as  $[M1\text{-}x^{2+}Mx^{3+}(\text{OH})_2] [A_{x/n}^{n-}]$ , where  $A^{n-}$  represents an anion that is available in between the layers. The range of 'x' is generally between 0.15 to 0.40. They are structurally similar to  $\text{Mg}(\text{OH})_2$  in the brucite family. Six hydroxide ions ( $6 \text{OH}^-$ ) occupy the corners of the octahedron in the brucite structure, and  $M^{2+}$  or  $M^{3+}$  ions are located in the middle. The edges of each octahedral unit are joined together to produce a multilayer structure. In case of Mg-Al HT, the Trivalent Al-species replace divalent Mg ions in Mg-Al HT, or  $\text{Mg}_6\text{Al}_2(\text{OH})_{16}\text{CO}_3\cdot 4\text{H}_2\text{O}$ , causing the development of positively charged layers [5, 6]. Between the cationic layer, where carbonate species ( $\text{CO}_3^{2-}$ ) aid in charge balancing, are  $\text{H}_2\text{O}$  molecules and  $\text{CO}_3^{2-}$  ions. The layer configurations regulate the final structure of HT; for instance, a unit cell composed of three layers showed rhombohedral symmetry, whereas two layers resulted in a hexagonal symmetry [9]. In the memory of E. Manasse, who discovered the first precise formula for hydrotalcite (HT), hexagonal symmetry, which is more predominant in natural HTs, is known as manasseite [10]. Due to the development of a more enhanced catalytic surface oxide material with essential properties after calcination, hydrotalcite (HT) demonstrated interesting and excellent practical applications [11].  $\text{Mg}_6\text{Al}_2(\text{OH})_{16}\text{CO}_3\cdot 4\text{H}_2\text{O}$ , trivalent Al-species replace divalent Mg ions, resulting in the formation of positively charged HT.

$\text{Mg}^{2+}$  and  $\text{Al}^{3+}$  are two decent examples of divalent and trivalent cations having six-fold coordination with octahedral arrangements that make up infinite layers. The counter anions, like  $\text{CO}_3^{2-}$  ions, are depicted as tiny spheres in the internal layer area. The HTs and hydrotalcite-like compounds (HTlcs) have been explored as prospective bifunctional heterogeneous catalysts for various specific chemical transformations [12, 13] due to their simultaneous base and acid characteristics [14, 15]. Hydrotalcite-type molecules having different unit cell characteristics found in several minerals such as Meixnerite, Honessite, and Coalignite *etc* [11]. When the HTs are heated, then the intercalated carbonate ions ( $\text{CO}_3^{2-}$ ) and  $\text{H}_2\text{O}$  molecules are eliminated and layered structures vanish. The layered structure of HTs can be again recovered by the rehydration process and this process is known as the memory effect. In this regaining process, the catalytic efficacy and selectivity may be changed due to morphology change and also due to the introduction of new ions in their layers [16].

# Synthesis of Heterocyclic Compounds Using Hydrotalcite

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**Abstract:** Today, a promising technique for creating various biologically active organic compounds is the one-pot synthesis employing materials based on hydrotalcite. One-pot synthesis is an effective method for creating complex organic compounds because it includes the simultaneous production of many bonds and functional groups in a single reaction vessel. Layered double hydroxides (LDHs) called hydrotalcites have a special structural makeup that makes them suitable for usage as catalyst supports. The one-pot synthesis method using HT-based materials has a number of benefits, including shorter reaction times, increased product yields, and simpler reaction procedures. Utilising HT-based materials is also economical and friendly to the environment. The chapter describes a variety of one-pot methods for the creation of different heterocycles using hydrotalcites as effective catalysts.

**Keywords:** Catalyst, Heterocycles, Hydrotalcites, Layered Double Hydroxides and Organic Compounds.

## INTRODUCTION

Heterocyclic compounds are a class of organic compounds characterized by the presence of one or more heteroatoms in their ring structure. The inclusion of heteroatoms imparts unique chemical and physical properties to heterocyclic compounds, making them highly significant in the field of organic chemistry [1, 2]. These compounds are found extensively in nature and are also synthesized in laboratories for various applications. They play vital roles in biological systems, being integral components of important molecules such as DNA, RNA, vitamins, hormones, and pharmaceutical drugs [3]. Due to their diverse reactivity and biolo-

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gical activities, heterocyclic compounds are often targeted in drug discovery and development processes. Several common types of heterocyclic rings exist, including:

Pyridine, Pyrimidine, Pyrazine, Furan, Thiophene, *etc.* These examples represent only a fraction of the heterocyclic compounds known and explored in chemistry. The field of heterocyclic chemistry encompasses a wide range of applications, including drug discovery, materials science, and agrochemical development. By modifying the structure of heterocyclic compounds, chemists can tailor their properties and create new compounds with specific desired characteristics [4].

One-pot synthesis, also known as multicomponent reactions or domino reactions, is a highly efficient strategy in synthetic chemistry. It involves combining multiple reactants in a single reaction vessel to produce the desired product or a series of interconnected reactions [5]. This approach simplifies the synthesis process by reducing the number of reaction steps, isolations, and purifications required, resulting in time, resource, and energy savings. Traditional organic synthesis often involves a sequential approach, with each reaction requiring isolation and purification before proceeding to the next step [6]. This can be time-consuming, labor-intensive, and prone to side reactions or product loss. One of the major advantages of one-pot synthesis is its ability to utilize the reactivity of different starting materials, which may have different functional groups, to create complex molecular structures. By carefully selecting compatible reactants and optimizing reaction conditions, chemists can orchestrate a cascade of reactions where intermediates from one step become starting materials for subsequent transformations. This cascade effect allows for the rapid construction of complex molecules in an efficient manner [7].

Furthermore, one-pot synthesis promotes atom economy by minimizing the formation of unwanted byproducts or waste. By directly combining multiple reactants in a single vessel, it reduces the consumption of solvents and reagents, enhancing the overall sustainability of the synthesis process. One-pot synthesis has found extensive applications in various areas of chemistry, including medicinal chemistry, natural product synthesis, polymer chemistry, and materials science [8]. It has revolutionized the way chemists approach the synthesis of complex molecules, enabling the rapid discovery and development of new compounds with diverse biological, pharmaceutical, and functional properties. Overall, one-pot synthesis is a powerful and versatile strategy that combines efficiency, selectivity, and sustainability, making it an invaluable tool in modern synthetic chemistry.

Hydrotalcite is a type of catalyst that has garnered considerable attention for its use in various chemical reactions. It belongs to the class of compounds known as layered double hydroxides (LDHs) and is characterized by its structure, which consists of positively charged metal hydroxide sheets separated by interlayer anions. {The unique properties of hydrotalcite make it a versatile catalyst in a range of chemical processes.} One notable characteristic is its strong basicity, derived from the hydroxide ions present in its structure. This basic nature enables its application in acid-base catalysis, such as transesterification reactions or Knoevenagel condensations. In addition to its basicity, hydrotalcite demonstrates excellent thermal stability even at high temperatures [9, 10]. {This stability ensures that it can maintain its catalytic activity over extended reaction times and under harsh conditions.} Hydrotalcite also serves as a suitable support for various active species, including metal nanoparticles or complexes, which further enhances its catalytic performance. This attribute is particularly advantageous in reactions such as hydrogenation, oxidation, and carbon-carbon bond formation. {Moreover, the composition, structure, and textural properties of hydrotalcite catalysts can be modified to control their selectivity [11, 12]. This level of control is highly desirable in the synthesis of fine chemicals and pharmaceutical intermediates.} Hydrotalcite-based catalysts have demonstrated significant potential in environmental applications. They have been extensively studied for their ability to remove pollutants from aqueous solutions, facilitating reactions such as oxidative degradation of organic compounds, removal of heavy metals, and conversion of harmful substances into less toxic forms. Due to its insolubility in many solvents, hydrotalcite can be easily recovered and reused as a heterogeneous catalyst. This characteristic reduces waste generation and enhances its viability for industrial applications [13].

### **CHEMISTRY OF HYDROTALCITE BASED MATERIALS FOR THE ONE POT SYNTHESIS OF ORGANIC MOLECULES**

**Ken Motokura *et al.*** introduced a new method in which Mg-Al HT can facilitate the one pot synthesis of quinoline derivatives (**3a-o**) through a hydrogen transfer process. By carrying out the Meerwein-Ponndorf-Verely (MPV) reaction, in which hydrogen transfer between alcohols (**1**) and carbonyl compounds (**2**) is base-catalyzed. (**Scheme 1**) [14]

**Keshav *et al.*** used aromatic aldehyde (**4**), malonitrile (**5**), phenylhydrazine (**6**) and Mg-Fe hydrotalcite as heterogeneous catalysts to develop 5-amino--H-pyrazole-4-carbonitrile and its derivatives(**7a-g**).

# Synthesis of Organic Molecules Using Hydrotalcite Based Materials

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**Abstract:** Hydrotalcites (HTs) belong to layer double hydroxide (LDH) structure and are the anionic clays that contain a double-layered structure similar to brucite [Mg(OH)<sub>2</sub>]. The layering structure allows the intercalation with a variety of anionic species, oxometallates, and palmitoleates including absorbed water in the interlayer as well as on the surface. Hydrotalcites (HTs) are the high-performance solid catalysts for the one-pot synthesis of organic compounds in the industry as well as the laboratory by the various chemical transformations such as Claisen-Schmid, aldol, Knoevenagel condensations, and Michael addition, isomerization, and Friedel-Craft alkylation owing to their surface Lewis base catalyst. This chapter offers an exposition on the diverse strategies employed for crafting various organic scaffolds by using Hydrotalcites as a catalyst and their applications within the spheres of biomedical and pharmaceutical industries.

**Keywords:** Aldol condensations, Alkylation, Claisen–Schmidt condensations, Coupling reactions, Catalyst, Double layer, Deoxygenation, Epoxidation, Hydrogenolysis, Hydrotalcites and Knoevenagel Michael additions.

## 1. INTRODUCTION

Hydrotalcite-like materials are composed of layered double hydroxide (LDH) clays with the general formula  $[M(II)_{1-x}M(III)_x(OH)_2]^{x+}[(A^n)_{x/n} \cdot mH_2O]^{x-}$ , where M(II) and M(III) are divalent (such as Mg, Ni, Co, Zn) and trivalent (such as Al, Cr, Fe, In) metal ions, respectively (Fig. 1). (A<sup>n</sup>) in this formula represents an anionic species such as CO<sub>3</sub><sup>2-</sup>, SO<sub>4</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, Cl<sup>-</sup> etc. The typical structure of an HT encompasses layers composed of positively charged metal hydroxide sheets, interweaved with water molecules and anionic radicals between the two-layer regions. Within the hydroxide layers, the metal cations adopt a Brucite-like confi-

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guration, and hydroxide ions (-OH) are coordinated in an octahedral manner surrounding metal ions *via* metal-oxygen bonds. Anions were attached to this metal hydroxide layer through electrostatic interactions and sometimes with hydrogen bonding, and neutralize the positive charge of the metal hydroxide layers. The lack of covalent bonding renders these anions readily exchangeable. Whereas the presence of water molecules contributes to the stability of the structure through hydrogen bonding with anions as well as ionic interactions with metal ions.

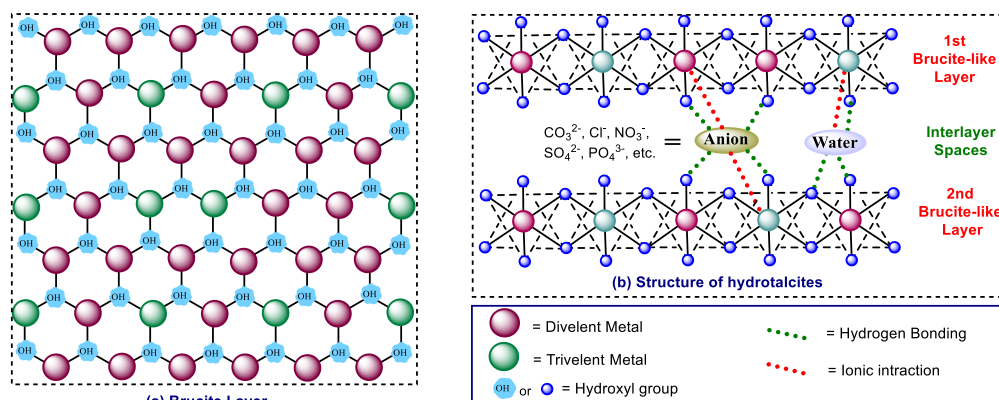


Fig. (1). Schematic diagram of hydrotalcite (A) Brucite Layer (top view) (B) Basic structure (side view).

## 1.1. Preparation of Hydrotalcite

It was initially detected in Hungary in 1842, and thereafter it was found in other places all over the world. Since it resembles the mineral talc and can hydrate (absorb water), the name “hydrotalcite” was coined. HTs naturally form over prolonged times in particular geological conditions. It is generally formed *via* natural processes involving the alteration of magnesium- and aluminum-rich minerals or prolonged weathering effects. In addition to these natural occurrences, various laboratory techniques have been firmly established for LDH synthesis that are discussed below.

### 1.1.1. Co-precipitation

In this approach, an alkaline solution of inorganic salts is evaporated or its pH is altered to obtain a supersaturated solution which precipitates out in LAH [1]. The pH fluctuation approach is more frequently employed for HT synthesis. The degree of the supersaturation of the solution influenced the structural morphology and particle size of the crystal. The selection of the saturation pH necessitates careful consideration and selection and is significantly influenced by the nature of the cation types employed. Metallic ions may undergo dissolution at very high pH

levels of solution, whereas, complete precipitation of all ions from the solution may not occur at low pH levels [2]. Co-precipitation of these solutions can manifest at varying degrees of supersaturation, but a notably higher crystallinity is achieved under conditions of relatively lower supersaturation. This technique involves the gradual mixing of aqueous solutions containing divalent and trivalent metal salts, with careful control of the desired pH throughout the process. Simultaneously the aqueous solution of the salt having desired interlayer anions was added and stirred continuously to enable the complete precipitation of LAH. After that, it was isolated by filtration or centrifuge and washed to remove impurities [3]. The ratio of trivalent and divalent metal ions in the Brucite layer could be regulated by adjusting the ratio of metal salts during mixing.

### **1.1.2. Sol-gel Method**

This technology enables the cost-effective synthesis of highly pure HTs, which could be used in biomedical applications [4] and as a drug carrier [5]. This process involves the hydrolysis of metallic precursor salts or their organic counterparts at room temperature in water or an organic solvent. However, for insoluble salts, higher temperatures are required, or sometimes alternative solvents are used to make them soluble. The structural characteristics of such HTs are directly influenced by temperature, precursor composition, and the ageing period [6]. In this method, the liquid phase (sol) colloidal suspension of two metal alkoxides (salts) was slowly transformed into a solid (gel) in the presence of acid [7].

#### **1.1.2.1. Urea Hydrolysis**

Urea slowly hydrolyses to ammonium cyanate *i.e.*,  $\text{NH}_4^+\text{NCO}^-$  in water. It is a mild Bronsted base that is utilised as a precipitating agent to alkali during hydrotalcite synthesis. The slow hydrolysis of urea has an advantage over  $\text{Na}_2\text{CO}_3$ , resulting in gradual supersaturation during precipitating. Additionally, LDH synthesised from the urea hydrolysis method is easy to purify through washing. When an alkali solution was employed in the process, multiple washing of precipitates may be necessary to eliminate alkali metal ions along with their counterions [8]. The urea hydrolysis technique, pioneered by Costantino *et al.*, was initially utilized for the synthesis of Mg-Al hydrotalcite [9]. In this method, the particle size determination of hydrotalcite could be modulated through temperature control. At lower temperatures, the nucleation rate is lower, and hence larger particles were generated, whereas smaller particles were observed at high temperatures.

## Recent Trends in the Application of Hydrotalcite-based Materials in Organic Synthesis

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**Abstract:** Hydrotalcites (HT) and hydrotalcite-based materials are viewed as attractive and feasible choices among heterogeneous catalysts, particularly in organic catalytic transformations. These catalysts have been widely researched as promising candidates in one-pot organic synthesis. They are synthesised *via* the standard co-precipitation method and can be used as support for transition metals and nanomaterials. Compared to the corrosive, hard to reutilise and, detrimental to the environment homogenous catalysts, HTs are high-functioning alternatives that deliver several benefits like profitable yields, recyclability and excellent selectivity. They are being studied today as precursors for a variety of scientific purposes such as in the production of renewable fuels, polymers, olefins and pharmaceuticals. Such processes involve essential organic reactions like isomerisation, oxidation, hydrogenation, methanation, nucleophilic additions, transesterification, and many more. In this chapter, examples of such organic reactions where hydrotalcite-based materials posed as an optimal catalyst are discussed.

**Keywords:** Hydrogenation, Methanation, Magnesium-aluminium hydrotalcite, Nucleophilic addition, Nanomaterials, Oxidation, Transition metals.

### 1. INTRODUCTION

Hydrotalcites are structured materials comprising double hydroxide layers with the chemical composition  $[M^{2+}_{1-x}M^{3+}_x(OH)_2]^{x+}A^{n-}_{x/n} \cdot mH_2O$ , where  $M^{2+}$  is a divalent cation,  $M^{3+}$  is a trivalent cation and  $A^{n-}$  refers to the interlayer anions [1]. The large surface area provides them with the ability to accommodate a variety of di- and trivalent ions, on its surface giving rise to numerous possible hydrotalcite-based catalysts (Fig. 1).

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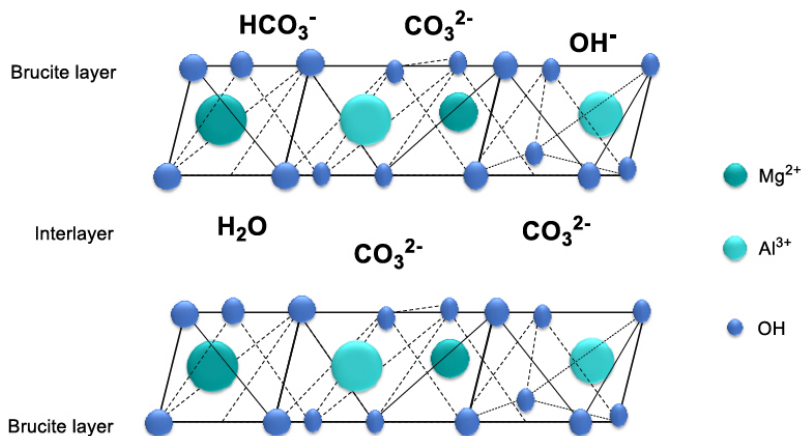


Fig. (1). Hydrotalcite.

The most extensively used hydrotalcite type is magnesium-aluminium hydrotalcite (Mg-Al HT), containing Mg<sup>2+</sup> and Al<sup>3+</sup> ions in the brucite layer. The anions and water molecules are present in the interlayer and on the surface. The anions present on the surface are responsible for the strong basicity. This is a pivotal characteristic of hydrotalcite since it enables it to function as a proton abstractor, making it an attractive choice as a base catalyst in base-catalysed organic reactions [2].

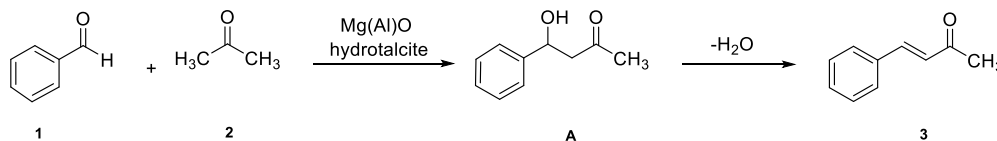
They are synthesised *via* the co-precipitation method at certain reaction conditions. To avoid impurities (the precipitation of aluminium and magnesium hydroxides), a pH range between 7-10 is considered optimum [3]. Hydrotalcites, also serve as promising supports for various metal species. Such species are immobilised on their surface owing to their large surface area. And these catalysts have been researched to promote organic transformations in a one-pot fashion. One-pot synthesis reduces the number of reaction steps, thereby optimising the reaction [4 - 6].

Over the last decade, a remarkable effort has gone into designing environmentally benign synthetic protocols using such catalysts. They offer unique properties like profitable yields, recyclability and, excellent selectivity in organic reactions, over the corrosive, hard to reutilise and, detrimental to the environment homogenous base catalysts like sodium or potassium hydroxide. The scope of this catalyst has been mostly in, but not limited to, the production of renewable fuels [7, 8], precursors for value-added chemicals [9], and pharmaceuticals [10]. This chapter is divided into sub-sections according to the different metal-based hydrotalcites and their application as catalysts in different organic reactions.

## 2. MAGNESIUM-ALUMINIUM HYDROTALCITES

### 2.1. Aldol Condensation

A simple method to synthesise reconstructed hydrotalcite *in-situ* for use in aldol condensation without the safety of an inert atmosphere was found. Owing to the memory effect property of hydrotalcites, calcined hydrotalcites can be rebuilt back to its authentic structure on rehydration. During this process, Brønsted base sites are constructed by the substitution of carbonate ions by the hydroxyl ions. However, the hydrotalcites prepared this way posed several disadvantages: complicated protocol, consumes a lot of time, and requires an inert environment due to loss of activity when exposed to air. Therefore, this work achieved to overcome the drawbacks by adding water and Mg-Al mixed oxides to the solution of benzaldehyde and acetone in the absence of an inert gas. And the reconstructed Mg-Al hydrotalcite, was found to catalyse the cross-aldol condensation of benzaldehyde **1** and acetone **2** in aqueous medium more actively than the hydrotalcite prepared conventionally and had better water tolerance. Water worked as the rehydrating agent and as the solvent in the condensation reaction (Scheme 1) [11].



**Scheme (1).** Rehydrated magnesium aluminium oxide hydrotalcite catalysed aldol condensation.

Through self-aldol condensation under solvent-free conditions, Yang *et al.*, synthesised a bicyclic ten-carbon hydrocarbon **5** at an excellent yield (up to 80%) using Mg-Al hydrotalcite. The conversion of cyclopentanone **4** was tested over a series of base catalysts. To justify the activity difference between the different catalysts, they were characterised by N<sub>2</sub>-adsorption, CO<sub>2</sub>-Temperature Programmed Desorption (TPD) and NH<sub>3</sub>-TPD. From the CO<sub>2</sub>-TPD curve, it was obvious that the desorption peaks corresponding to Mg-Al HT at high temperatures (>873 K) implied stronger basicity. NH<sub>3</sub>-TPD results showed that the hydrotalcite catalyst has a higher amount of acid sites per unit mass of the catalyst. And since, aldol condensation favours the synergetic effect of both acidic and basic sites, the enhanced activity of hydrotalcites among the others was explained (Scheme 2) [12].

# One-Pot Synthesis Of Organic Molecules Using Hydrotalcite-Based Materials

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**Abstract:** The one-pot reaction has recently received a lot of attention due to its significant advantages over traditional multi-step reactions. One of the main advantages of one-pot reactions is that it can save time and resources by eliminating the need for multiple reaction steps and purification processes. It can lead to the more efficient and cost-effective synthesis of target molecules. In addition, one-pot reactions can also frequently be conducted under milder reaction conditions, such as lower temperatures and pressures, resulting in higher yields and fewer side reactions. They can also reduce the formation of hazardous waste and environmental impact. One-pot reactions also offer more opportunities for synthetic creativity, as they allow for the simultaneous manipulation of multiple functional groups in a single reaction vessel. As a result, new synthetic pathways and novel compounds might be discovered.

In recent years, the combination of one-pot reactions and hydrotalcite has gained significant importance recently due to their complementary advantages. Hydrotalcite is a mineral with the chemical formula  $Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O$  and is commonly found in primary and ultrabasic igneous rocks, serpentinites, and carbonate-rich sediments. It is made up of interlayer anions like carbonate or nitrate and positively charged layers of magnesium and aluminum hydroxides. The layers of magnesium and aluminum hydroxides, which are positively charged, have a large surface area and a net negative charge in hydrotalcite as a result of the interlayer anions. Its large surface area and net negative charge allow it to stabilize reactant molecules, enhancing the yield as well as the selectivity of the expected product. Additionally, altering the interlayer anions, the Mg/Al ratio, and doping with additional metal ions can further improve the catalytic activity of hydrotalcite.

Compared to traditional catalysts, such as metal salts or complexes, hydrotalcite offers several advantages, including high stability, low toxicity, and easy separation from the reaction mixture. As a result, combining one-pot reactions with hydrotalcite as a catalyst can lead to more efficient and sustainable synthetic processes with greater synthetic creativity. Therefore, the aim of the book chapter is to summarize recent examples of one-pot reactions in which hydrotalcites have been used as catalysts. By

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exploring these examples, readers can gain insights into the potential applications of hydrotalcite as a flexible and efficient catalyst for organic reactions.

**Keywords:** Green synthesis, Green catalyst, Hydrotalcite, One-pot reactions, Organic synthesis.

## 1. INTRODUCTION

The field of organic synthesis has made remarkable strides since Wöhler's pioneering synthesis of urea in 1828. The complicated method of producing small compounds with revolutionary features and fascinating chemical structures, however, is something that today's synthetic chemists are equally enthused about. The urge to innovate has led to the development of novel catalysts, reagents, and reactivity that enable chemists to use their imagination when planning sequential reactions to get molecules of limitless complexity. While achieving these milestones has long been considered a resounding success, chemists in the past three decades have become increasingly concerned about the process by which they reach their goals.

In the titles of synthesis publications nowadays, words like “concise,” “efficient,” and “convergent” are often used. They serve as defining principles that chemists consider while developing their synthesis techniques, in addition to serving as buzzwords. The environmental effect of the chemical industry has also been recognized by process chemists, who have actively attempted to reduce it. Because of this knowledge, the idea of “green chemistry” has emerged, allowing process and synthetic chemists to think critically about these ideas and apply them to the development of cutting-edge syntheses.

Green chemistry aims to reshape how chemists conceive synthesis. Its efforts are not just directed at creating new techniques, but also at providing viable substitutes for those already in use. Most notably, it promotes various synthetic techniques that take environmental factors into account at an early stage of the process design. MCRs—multicomponent reactions—come into play in this situation. In MCRs, at least three reactants are combined in a single vessel to produce a final product that contains most, ideally all, of the atoms from the initial materials. Their exceptional atom economy, effectiveness, gentle reaction conditions, high convergence, simultaneous decrease in reaction steps, compatibility with ecologically friendly solvents, and important place in the arsenal of environmentally friendly synthetic techniques all support this claim [1].

The significance of multicomponent reactions (MCRs) in diverse fields of chemistry cannot be overstated. In their respective fields, including drug

discovery initiatives, medicinal chemistry, combinatorial chemistry, agrochemistry, natural product synthesis, and polymer chemistry, researchers from a variety of disciplines have noted the expanding relevance of MCRs. The versatility and efficiency of MCRs have opened up new avenues for synthetic strategies and accelerated the development of novel compounds with potential applications in these fields.

***In medicinal chemistry and drug discovery programs***, MCRs have become important instruments for the quick creation of a variety of compound libraries. By combining multiple reactants in a single step, MCRs enable the efficient synthesis of structurally complex molecules with drug-like properties. This streamlined approach facilitates the exploration of chemical space, aiding the discovery and optimization of lead compounds for therapeutic applications (Fig. 1).

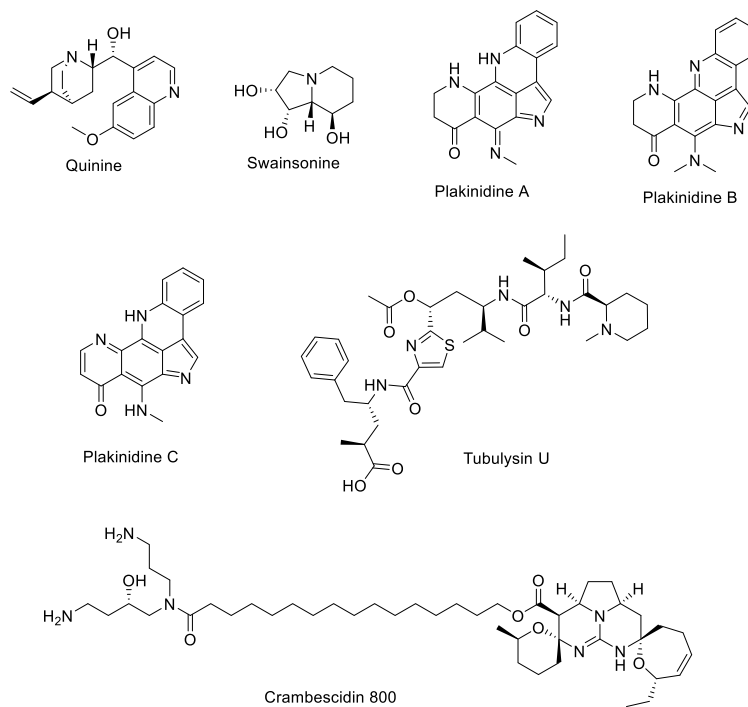


Fig. (1). Examples of lead compounds for therapeutic applications [2].

***Combinatorial chemistry***, which focuses on the quick synthesis and screening of vast chemical libraries, has also embraced MCRs as a powerful technique. By employing MCRs, chemists can assemble vast arrays of structurally diverse compounds, allowing for high-throughput screening to identify biologically active



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