



**UNIVERSITI PUTRA MALAYSIA**

**PHYSICO-CHEMICAL PROPERTIES OF HEAT-  
TREATED LAYERED DOUBLE HYDROXIDE  
NANOCOMPOSITES**

**SIVAJOTHI NADARAJAH**

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**MASTER OF SCIENCE  
UNIVERSITI PUTRA MALAYSIA**

**1999**



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DOUBLE HYDROXIDE NANOCOMPOSITES**

**By**

**SIVAJOTHI NADARAJAH**

**Thesis Submitted in Fulfilment of the Requirements for the Degree of Master of  
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Universiti Putra Malaysia**

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## LIST OF ABBREVIATIONS

LDH: Layered Double Hydroxide

HTlc: Hydrotalcite-like compound

SDS: Sodium Dodecyl Sulphate

ZACORN: Zn/Al Layered Double Hydroxide-congo red nanocomposite

CACORN: Co/Al Layered Double Hydroxide-congo red nanocomposite

ZASDSN: Zn/Al Layered Double Hydroxide-Sodium Dodecyl Sulphate nanocomposite

TZACORN: Heat-treated Zn/Al Layered Double Hydroxide-congo red nanocomposite

TCACORN: Heat-treated Co/Al Layered Double Hydroxide-congo red nanocomposite

TZASDSN: Heat-treated Zn/Al Layered Double Hydroxide-Sodium Dodecyl Sulphate  
nanocomposite

Abstract of thesis to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

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May 1999

**Chairman: Associate Professor Mohd Zobir Hussein, Ph. D.**

**Faculty: Science and Environmental Studies**

Nanocomposite material of Zn/Al LDH-congo red (ZACORN), Co/Al LDH-congo red (CACORN) and Zn/Al LDH-SDS (ZASDSN), were synthesised by spontaneous self-assembly of the inorganic and organic phase from homogenous aqueous solutions technique. The X-ray diffraction patterns of heat-treated samples showed the formation of new phases ZnO and ZnAl<sub>2</sub>O<sub>4</sub> at 900°C and 1000°C for ZACORN, Co<sub>3</sub>O<sub>4</sub>, Co<sub>2</sub>AlO<sub>4</sub> and CoAl<sub>2</sub>O<sub>4</sub> for CACORN and only ZnO for ZASDSN. ZACORNs and CACORNs prepared at various concentrations of congo red showed that the concentration of congo red does not effect the basal spacing. Quantitative analysis of colour difference,  $\Delta E^*_{ab}$ , for TZACORNs and TCACORNs showed that they change exponentially with the heating temperature. For TZASDSN,  $\Delta E^*_{ab}$  increased rapidly at low temperature up to 300°C and then decreased as the temperature increased. FTIR spectra showed the presence of four main peaks, 3700-3200 cm<sup>-1</sup> is due to the hydroxyl groups, 1650-1500 cm<sup>-1</sup> is due to the presence of water molecules, 1130 cm<sup>-1</sup> and 620



$\text{cm}^{-1}$  is due to the presence of sulphate anion whereas peak at  $800\text{-}400\text{ cm}^{-1}$  is Co-Al-O stretching and bending and metal-oxygen stretching. Scanning electron microscopy showed a mixture of amorphous and needle-like crystals for ZACORN, cubicle-like crystals for CACORN and spherical-like crystals for ZASDSN when heated at  $900^\circ\text{C}$ .



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains.

**SIFAT KIMIA DAN FIZIK BAGI NANOKOMPOSIT HIDROKSIDA BERLAPIS  
GANDA TERAWAT HABA**

**Oleh**

**SIVAJOTHI NADARAJAH**

**May 1999**

**Pengerusi: Profesor Madya Mohd Zobir Hussein, Ph. D.**

**Fakulti: Sains dan Pengajian Alam Sekitar**

Bahan nanokomposit Zn/Al HBG-congo red (ZACORN), Co/Al HBG-congo red (CACORN) dan Zn/Al HBG-SDS (ZASDSN), masing-masing telah disediakan dengan kaedah pencampuran spontan dalam fasa tak organik dan organik dengan menggunakan teknik larutan akueos homogen. Kaedah pembelauan sinar-X (XRD) menunjukkan pembentukan fasa baru iaitu ZnO dan ZnAl<sub>2</sub>O<sub>4</sub> untuk ZACORN, Co<sub>3</sub>O<sub>4</sub>, Co<sub>2</sub>AlO<sub>4</sub> dan CoAl<sub>2</sub>O<sub>4</sub> untuk CACORN dan hanya ZnO untuk ZASDSN. Pembentukan ZACORN dan CACORN dengan menggunakan kepekatan congo red yang berlainan telah menunjukkan bahawa kepekatan congo red tidak mempengaruhi jarak antara lapisan. Analisis kuantitatif iaitu perbezaan warna,  $\Delta E^*_{ab}$ , bagi TZACORN dan TCACORN menunjukkan bahawa mereka berubah secara eksponen apabila dipanaskan dan TZASDSN menunjukkan bahawa  $\Delta E^*_{ab}$  bertukar secara mendadak pada suhu rendah dan kemudian menurun apabila suhu terus dinaikkan. Spektrum FTIR menunjukkan kehadiran empat puncak utama, iaitu puncak pada 3700-3200 cm<sup>-1</sup> dan 1650-1500 cm<sup>-1</sup> yang masing-masing





berkaitan dengan kumpulan -OH dan molekul air. Puncak pada  $1130\text{ cm}^{-1}$  dan  $620\text{ cm}^{-1}$  adalah berkaitan dengan kehadiran ion sulfat sementara puncak pada  $800\text{-}400\text{ cm}^{-1}$  adalah berkaitan dengan pembentukan Co-Al-O dan M-O. Kajian menggunakan mikroskop pengimbasan elektron juga menunjukkan bahawa rawatan suhu pada  $900^\circ\text{C}$  menghasilkan campuran amorfus dan hablur berbentuk jarum untuk ZACORN, berbentuk kiub untuk CACORN dan berbentuk sfera untuk ZASDSN.

## CHAPTER I

### INTRODUCTION

#### Layered Double Hydroxides

Layered double hydroxides (LDHs) has been known for very long, when these materials were first synthesised by Feithnecht (1942) through reaction between dilute solutions of metal with bases, leading upon ageing to hydrotalcite-like (HTI) materials.

Layered double hydroxide (LDH) is a layered material of anionic clay family. This type of material is characterised by structures of sheets held together by strong covalent bonds in the xy plane to form two-dimensional polymeric mixed metal hydroxide layers and a considerable weaker bond in z direction containing anions and water molecules. The expandable interlayer z direction can be exploited to introduce ions or molecules, thereby generating the so-called nanocomposite materials (Hussein *et al.*, 1993), a diverse group of material with various applications in technologies such as photochromic, organoceramics, semiconductors and control release materials.



The hydroxalcalite-like compounds are composed of a positively charged layer of di- and trivalent metal hydroxides. This charge being compensated by interstitial anions and water, hence the compensation is not stoichiometric. Figure 1 shows the basic structure of layered double hydroxides. The basic structure of the layered compound is magnesium hydroxides (brucite) in which the divalent cation occupies the octahedral position. The positive charge is created by the substitution of the divalent cation by a trivalent cation. Water molecules and anion are intercalated and are located between the layers, and so maintain the electroneutrality of the crystal. The general formula of the solid solution is  $M(II)_{1-x}M(III)_x(OH)_2A^{n-}_{x/n} \cdot mH_2O$  where  $M(II) = Mg^{2+}, Zn^{2+}, Ni^{2+}, \dots$ ,  $M(III) = Al^{3+}, Fe^{3+}, \dots$ ,  $A^{n-} = OH^-, Cl^-, NO_3^-, SO_4^{2-}, \dots$ , and  $0.2 < x < 0.33$ . In certain cases,  $x$  can be higher than 0.33 in the Zn/Al system. The above formula could be extended to include a monovalent cation in the octahedral position (Kooli *et al.*, 1993).

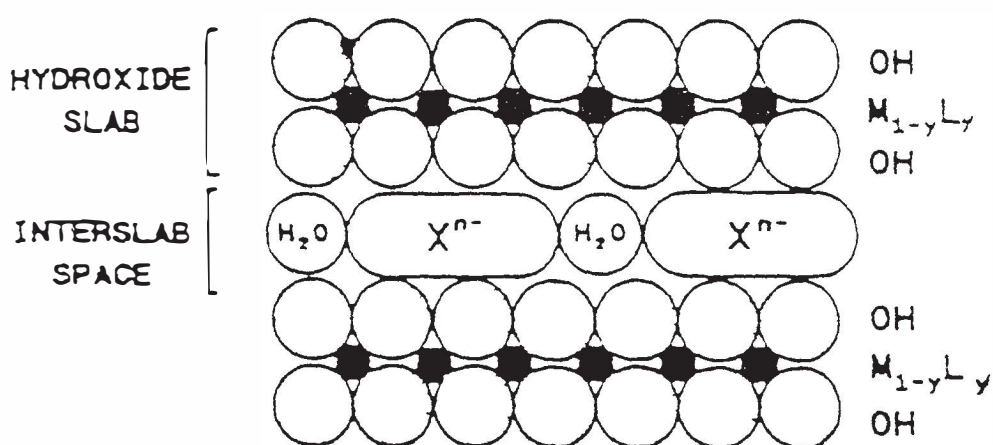


Figure 1: LDHs structure.

The main features of HTlc structures are determined by the nature of the brucite-like sheet, by the position of anions and water in the interlayer region and by the type of the brucite-like sheets. Figure 2 shows the structure of brucite lattice  $[\text{Mg}(\text{OH})_2]$ .

The sheets containing cations are built in a brucite layer, where the cations randomly occupy the octahedral holes in the close-packed configuration of the  $\text{OH}^-$  ions. The anions and water are randomly located in the interlayer region, being free to move by breaking their bonds and forming new ones (as in liquid water). The oxygen atoms of the water molecules and the anions are distributed closely around the symmetry axes that pass through the hydroxyl groups of the adjacent brucite-like sheet.

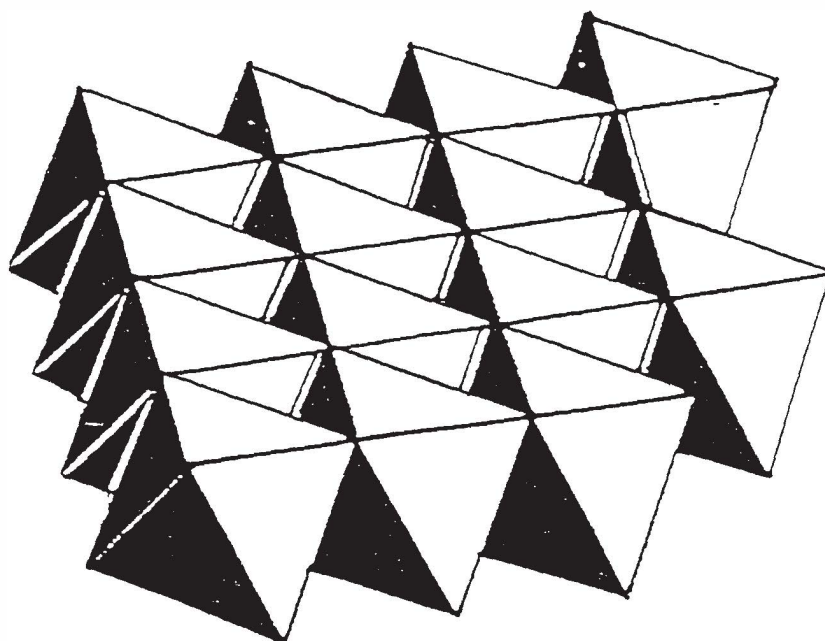


Figure 2: Brucite lattice  $[\text{Mg}(\text{OH})_2]$  top view.

These hydroxyls are tied to the anions directly or via intermediate  $H_2O$  through hydrogen bridges:  $OH-A-HO$  or  $OH-H_2O-A-HO$ . The anions are situated flat in the interlayer and  $H_2O$  is loosely bound; they can be eliminated without destroying the structure.

The brucite-like sheets stack one on the other with two different symmetries, rhombohedral or hexagonal. If we call ABC the three-fold axis of the OH groups in the brucite-like sheet, the stack may have the sequence BC-CA-AB-BC, thus having three sheets in the unit cell, or BC-CB-BC with two sheets in the unit cell with hexagonal symmetry. Figure 3 shows the stacking sequences in HTlcs with different symmetries.

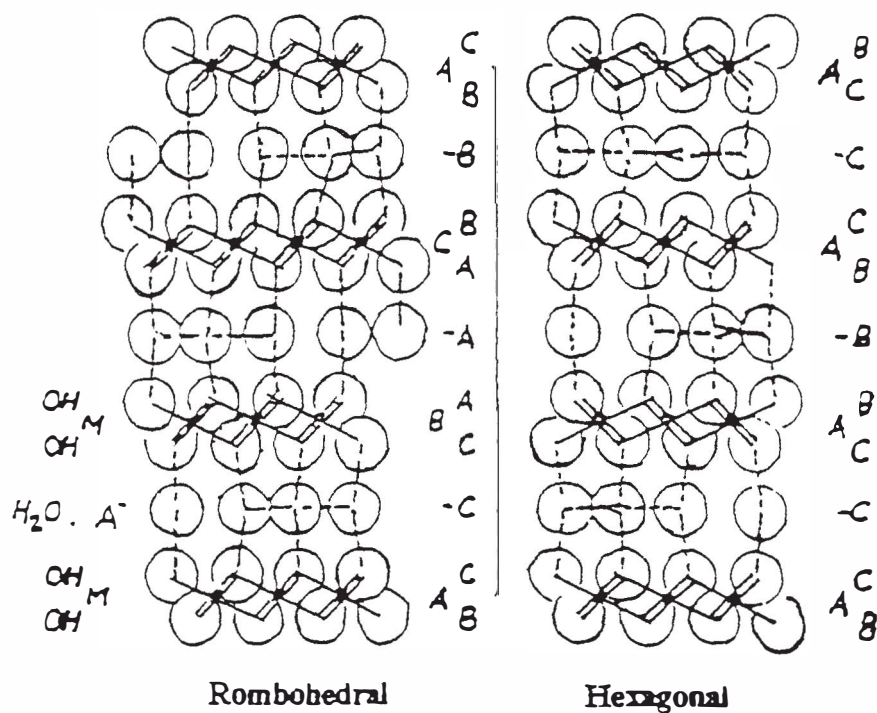


Figure 3: Stacking sequences in HTlcs with different symmetries.

## Historical Background

Clays break down into two broad groups, namely cationic clays, which nature prefers and anionic clays which is less common. Clays can be synthesised and chemical modification sometimes needed in order to enhance certain desirable properties.

Hydrotalcite is a mineral that can be easily crushed into a white powder similar to talc, discovered in 1842. It is a hydroxycarbonate of magnesium and aluminium and occurs in nature in foliated and contorted plates and/or fibrous masses.

Hydrotalcite,  $[\text{Mg}_6\text{Al}_2(\text{OH})_{16}\text{CO}_3^{2-}\cdot 4\text{H}_2\text{O}]$  is an anionic clay mineral, containing  $\text{Mg}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{CO}_3^{2-}$ . Compounds with similar structure to hydrotalcite are called “Hydrotalcite-like compounds” (HTlcs).

At the same time, another mixed hydroxycarbonate of magnesium and iron was found, which was called pyroaurite (because of a likeness to gold when heated) and which was later recognised to be isostructural with hydrotalcite and other minerals containing different elements, all of which were recognised as having similar features.

The hydrotalcite formula reflects that the atomic contents for the structural element of the two polytype structures, and shows that it is possible to synthesise a number of compounds with different stoichiometries. The value of x for natural element

is generally equal to 0.25 and the most common anion is carbonate. It is possible to synthesise HTlcs with the above formula with more than two metals and two anions.

From the X-ray investigations, Aminoff and Brome (1930) (Cavani and Trifiro, 1991), recognised the existence of two polytype of hydrotalcite; hexagonal manasseite, with  $a = 3.1 \text{ \AA}$  and  $c = 15.3 \text{ \AA}$  and rhombohedral hydrotalcite; with  $a = 3.1 \text{ \AA}$  and  $c = 23.1 \text{ \AA}$ . These are respectively the 2- and 3-layer polytype of the same basic structure.

Hydrotalcite-like compounds feature well known anion exchange capabilities (Ulibarri and Hernandez, 1986). They are some of the few known inorganic anion exchangers. Their anion exchanger properties are governed by the anion size, its electric charge and the molar  $M^{2+}/M^{3+}$  ratio. A number of small anions such as  $\text{CO}_3^{2-}$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$ ,  $\text{Cl}^-$ , *etc.*, have been used in exchange reactions. The interlayer spacing of these compounds varies with the size of the intercalated anion. The intercalation of large anions are some of the interests to adsorption and catalysis.

A wide range of derivatives containing various combination of  $M^{2+}$ ,  $M^{3+}$  and  $A^{n-}$  ions can be synthesised either by direct crystallisation from aqueous solution or by anion exchange of a precrystallised LDHs clay. LDHs will form a complementary class of materials to the two dimensional clays and it swell to only one or two layers. This coupled with a high layer charge density leads to an interlayer space that is filled with anions and not accommodate penetration of organic molecules. Low surface areas and

low catalytic activity often results; however, these variables can be enhanced by employing a number of synthetic methods.

The stability of hydrotalcite-like compounds during thermal and hydrothermal treatments, as demanded by operation conditions and regeneration treatments. It has been proposed that the thermal stability of intercalated clays depends on the structure and properties of both the clay component and the pillaring agent. According to Trillo *et al.*,

hydrothermal stability of pillared clays. As pointed out by Messersmith and Stupp (1995), high temperature treatment of the intercalated clays produced oxides and non-stoichiometric spinel phases. The thermal treatment on the hydrotalcite-like compounds will increase the specific total (or micropore) surface areas, but a further increase in the calcination temperature produced a decrease in the specific surface areas. Similar behaviour was observed for the specific pore volumes.

According to Puttaswamy and Kamath (1997), thermal decomposition of various anions (X) of  $[\text{Zn}_2\text{Cr}(\text{OH})_6]\text{X}.\text{nH}_2\text{O}$  where  $\text{X} = \text{F}^-$ ,  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{CO}_3^{2-}$ , and  $\text{NO}_3^-$  can be used as ion conductors because the system uptake is unusually high of  $\text{H}_2\text{O}$ , leading to more swelling and to anion mobility. The  $\text{Zn}^{2+}$ - $\text{Cr}^{3+}$ - $\text{Cl}^-$  produced the conductivities as high as  $10^{-3} \Omega^{-1}\text{cm}^{-1}$  at  $17^\circ\text{C}$  were observed, presumably due to movement of chloride ion through channel under various humidity conditions. Other anions showed ion conductivities between  $10^{-3}$  and  $10^{-4} \Omega^{-1}\text{cm}^{-1}$ .



### Possible Applications of LDHs

The anionic clays based on hydrotalcite-like compounds have found many practical applications due to the varied properties of metal cationics, anionics, size and particles, morphology, their physical and chemical properties. Figure 4 shows a schematic picture of the possible application of hydrotalcite-like compound (Cavani *et al.*, 1991).

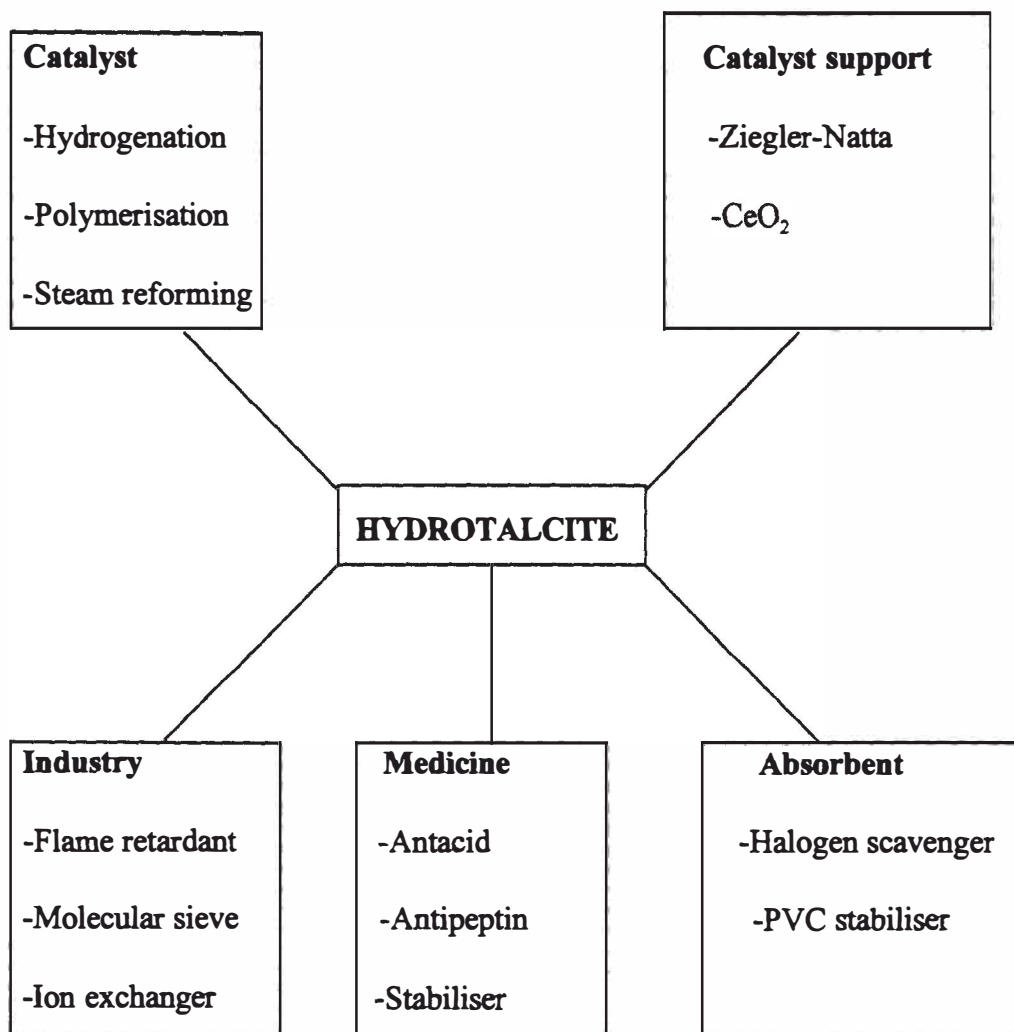


Figure 4 : Possible applications of LDHs