



UNIVERSITI PUTRA MALAYSIA

**LAMELLA STRUCTURE OF ZINC ALUMINUM LAYERED DOUBLE
HYDROXIDE AS MOLECULAR CONTAINERS FOR THE PREPARATION
OF MESOPOROUS CARBON**

LIM SHEAU WEN

ITMA 2006 10



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OF MESOPOROUS CARBON**

By

LIM SHEAU WEN

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirement for the Degree of Master of Science**

July 2006



DEDICATION

Dedicated to my family for their love, support and encouragement.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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LIM SHEAU WEN

July 2006

Chairman : Mohd. Zobir Bin Hussein, PhD

Faculty : Institute of Advanced Technology

Open lamella systems such as layered double hydroxides (LDHs) can be used to generate new nanostructured materials of layered organic-inorganic nanohybrid type. The inorganic Zn-Al-layered double hydroxide (ZAL) was used as a matrix, hosting an active agent or a guest, toluene-4-sulphonate (TSA), 2,4-dichlorophenoxyacetic acids (24D), naphthaleneacetic acid (NAA) anthraquinone-2,6-disulphonate (AQDS) and dodecylsulfate sodium salt (SDS).

They were prepared by spontaneous self-assembly method from an aqueous solution for the formation of a new layered organic-inorganic hybrid nanocomposite material. The Zn to Al ratio at R=4 and the various concentrations of anion organics at pH 10 was found suitable to give well-ordered nanolayered organic-inorganic hybrid structure.



PXRD and FTIR analyses show that the inorganic-organic structure of LDH expanded from 8.8 Å to accommodate the anion organics for the formation of the nanocomposite. Nanocomposites were then calcined under N₂ gas at different temperatures, 500 °C, 700 °C, and 1000 °C for the formation of the carbon products. In order to remove the carbonaceous products from the template matrix, the carbonized nanocomposite was treated with 2 M nitric acid.

Powder X-ray diffraction pattern of the carbons showed that they are of amorphous type. The surface area and porosity studies show that the resulting materials are of mesoporous carbon with high BET surface area and high percentage of micropore content. No significant difference in the surface morphology of ZAL and its nanocomposites was observed under a scanning electron microscope. Both of them afforded non-uniform irregular agglomerates of compact and non-porous structure of plate-like morphology. The morphology of carbons showed agglomerates of compact and porous granular structure.



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

**STRUCTURE LAMELA ZINK ALUMINIUM BERLAPIS GANDA SEBAGAI
TAKUNGAN MOLEKUL BAGI PENYEDIAAN KARBON BERIANG MESO**

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Sistem lamela terbuka seperti hidroksida berlapis ganda (LDHs) boleh menghasilkan satu siri bahan baru yang mempunyai nanostruktur bagi nanohibrid organik inorganik berlapis. Zink aluminium hidroksida berlapis ganda (ZAL) digunakan sebagai matriks dan lima bahan organik yang aktif dipilih iaitu (TSA) metil benzena-4-sulfonat, (24D) 2,4 diklorofenoasetik, (NAA) asid naftalenaasetik, (AQDS) anthrakuinon-2,6 disulfonat dan (SDS) natrium dodesilsulfat.

Mereka disediakan dengan kaedah sintesis terus melalui satu larutan untuk menyediakan nanokomposit organik-inorganik yang baru. Ratio bagi zink dan aluminium telah ditetapkan pada $R=4$ dan kepekatan yang berlainan bahan organik digunakan untuk menyediakan nanokomposit masing-masing. Didapati bahawa nanokomposit-nanokomposit yang disediakan pada pH 10 memberi struktur nanokomposit yang baik.



Analisis PXRD dan FTIR menunjukkan jarak basal bagi struktur organik-inorganik hidroksida berlapis ganda dikembang daripada 8.8 Å untuk memuatkan bahan organik yang aktif menjadikan nanokomposit. Kemudian, nanokomposit-nanokomposit yang telah siap sedia diberi rawatan suhu dalam keadaan gas nitrogen atmosfera pada tiga suhu berlainan iaitu 500, 700 dan 1000 °C untuk menjadikan produk karbon. Asid nitrik 2 M diperlukan untuk mengeluarkan produk-produk karbon daripada templat.

Produk karbon yang dihasilkan kemudian dianalisis oleh PXRD dan mendapati bahawa corak belauan sinar-x bagi bahan tersintesis berstruktur armofus. Kajian ASAP pula menunjukkan bahawa bahan tersintesis adalah karbon berliang meso dan memberikan BET luas permukaan spesifik yang tinggi dengan mempunyai peratusan kandungan mikro yang tinggi. Pemerhatian mikroskopi imbasan elektron menunjukkan tiada perbezaan ketara antara zink aluminium hidroksida berlapis ganda dan nanokomposit. Pemerhatian mikroskopi imbasan elektron bagi produk-produk karbon di bawah rawatan suhu menunjukkan berliang dan mempunyai partikel yang bersaiz kecil.



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I certify that an Examination met on 6th December 2005 to conduct the final examination of Lim Sheau Wen on her Master of Science thesis entitled “Lamella Structure of Zinc Aluminium Layered Double Hydroxide as Molecular Containers for the Preparation of Mesoporous Carbon” on accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotation and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

LIM SHEAU WEN
Date: 3 January 2007



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LIST OF ABBREVIATIONS

2 θ	Two theta
Å	Amstrong
R	Ratio
JCPDS	Joint Committee on Powder Diffraction Standards
ASAP	Accelerated Surface Area and Porosity
BET	Bruneur, Emmett and Teller
BJH	Barret, Johner and Halenda
CHNS	Carbon, Hydrogen, Nitrogen and Sulfur Analysis
PXRD	Powder X-ray Diffraction
TGA-DTG	Thermogravimetry Analysis-Derivative Thermogravimetry Analysis
FTIR	Fourier Transforms Infrared
SEM	Scanning Electron microscopy
LDH	Layered Double Hydroxide
ZAL	Zn-Al-Layered Double Hydroxide
TSA	Toluene-4-Sulfonate Acid
AQDS	Anthraquinon 2,6 disulfonate
SDS	Dodecylsulfat
24D	2,4-dichlorophenoxy Acetate
NAA	Naphthalene Acetate



CHAPTER 1

INTRODUCTION

1.1 The evolution of material technology

Material technology has had such a profound impact on the evolution of human civilization. The current ‘Synthetic Materials Age’ has been precipitated by humankind’s demand for materials with superior performance characteristics. It is catalyzed by a technological revolution that will exploit several emerging technology, such as materials science, biotechnology, biomimetics, nanotechnology, molecular electronics, neural networks and artificial intelligence (Gandhi and Thompson, 1992).

In materials science, a composite implies that the material is composed of a mixture of two or more constituents that differ in composition. Accordingly, one may well classify among the composite materials nearly all substances such as wood, bones, shells, etc, and also some man-made materials, such as certain powder metallurgy products, electrical insulators, resin-bonded magnetic materials, powder-charged plastics, paper laminates, etc, (Schiller, 1978).

Revival of research on latest composite fabrication is a result of increasing interest focused on advanced technology material due to its interesting promising features. Advanced materials maybe defined as materials, which have enhanced mechanical and physical characteristics, compared to traditional materials. The characteristics



either allows for every significant, allow for new technologies that are not achievable using conventional materials (Schwartz, 1993).

To date the vast majority of advanced technology research has been focused on nanotechnology, which showed the trend to obtain control of the composite size to a molecular level. A lecture delivered in 1959, by the late Noble Laureate in Physics, Richard P. Feynman, titled “There’s Plenty of Room at the Bottom”, discussed the prospect of fabricating materials and devices at the atomic molecular level confirmed that the concept of nanotechnology is not relatively new. Nanotechnology encompasses all aspects of science and technology involved in the study, manipulation, control individual atoms and molecules, making it possible to build machines on the scale of human cells, or create materials and products with nanostructures containing highly desirable properties. It is predicted that the nanotechnology will give an enormous impact on every aspect of human life (Hamid and Yarmo, 2003).

Research performed over the last two decades has identified a variety of syntheses methods for the so-called ‘nanoparticles’ or ‘nanocomposites’. Although this term in its broadest sense refers to particles that have nanoscale dimensions (usually from 1 to 100 nm particle diameter), it now also often implies that the presence of these particles in a device, system or formulation should result in improving properties and performance that are directly derived from the nano size character of the constituent particles. The high surface-to-volume ratio of the particles is the key to attaining novel and useful properties. For such cases, smaller nanoparticles are preferred, and it is generally found that prospective applications require particle diameters of 30 nm



or less to provide significant improvements over use of ‘conventional’ nano or micron scale particles.

1.2 Nanocomposite Materials

In materials science, a ‘composite’ implies that the material is composed of a mixture of two or more constituents that differ in composition. Thus, the term “nanocomposite” implies that the physical arrangement of the different constituents is on a scale of 1 to 100 nm (1 nm= 10^{-9} m, i.e., one billionth of a meter) (Roy *et al.*, 1986).

Nanostructured materials are becoming of major significance and the technology of their production is rapidly growing into a powerful industry. These fascinating materials include nanofilms, nanocrystal, alloys, nanocomposites and semiconductors (Nalwa, 2000). The synthesis of materials of nanoscale dimension is important because the small size of these materials endows them with unusual structural and optical properties that might find application in catalysis electro-optical devices. Such materials may also be valuable precursor to strong ceramic. These kinds of materials and their base technologies have also opened up exciting new possibilities for future applications in aerospace, automobile, batteries, insulators, printing, color imaging, drug delivery, medicine and cosmetics.

The preparation of nanostructure materials depend on the following four common microstructural features:

- The grain size and size distribution (<100 nm).



- The chemical composition of the constituent phases.
- The presence of interfaces, more specifically, grain boundaries, heterophases interface, or the free surface.
- Interaction between the constituent domains.

The presence and interplay of these four features largely determine the unique properties of the nanostructured materials.

A two-dimensional layered structure consisting of thin crystalline inorganic layers with a thickness in nanometer range can be used as an ideal host of layered nanocomposite or organic-inorganic hybrid materials. One of the candidates for this type of structure is layered double hydroxide (LDH). A variety of anionic species can be inserted as guest into the interlayer spaces of the LDH, resulting in an expansion of the interlayer distance to a nanometer sized dimension to form a new nanocomposite material (Yamanaka, 1991).



CHAPTER 2
LITERATURE REVIEW

2.1 Layered Double Hydroxide

Layered double hydroxides (LDHs) are also known as anionic clays. Feitknecht discovered it about 50 years ago, but their structure was only determined in 1970 by Allman for the Mg-Fe LDH (pyroaurite and sjögrenite) and by Brown and O'Hare for the Mg-Al LDH (hydrotalcite and manasseite) (Ehisissen *et al.*, 1993). These compounds have a structure of sheet held together by strong covalent bonds in the xy plane to form a two-dimensional polyhydroxyl cation layers (Figure 2.1). These crystalline layers containing anions and water molecules are stacked considerable weaker bonds in the z direction (Hussein *et al.*, 1995).

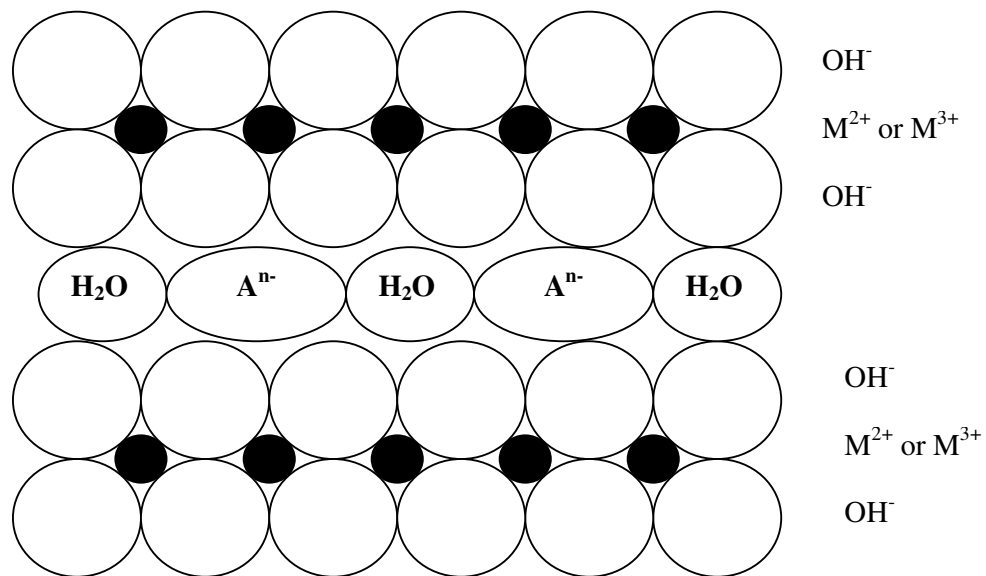


Figure 2.1 Structure of LDH