PREPARATION OF ZINC OXIDE NANO- AND MICRO-STRUCTURES USING HYDROTHERMAL-TEMPLATE METHOD AND THEIR APPLICATIONS

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By

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To My Beloved Husband

Dariush Damavandi

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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February 2014

Chair: Professor Mohd Zobir Bin Hussein, PhD

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Pure zinc oxide (ZnO) was successfully synthesized using various non bio- and bio-templates namely, sodium dodecyl sulfate (SDS), cetyl trimethylammonium bromide (CTAB), palm olein (PO), uncooked- and cooked rice. ZnO nano- and microstructures were synthesized through hydrothermal method. The physico-chemical properties of the resulting samples were characterized for samples synthesized at various amount of templates to zinc precursor. Different morphologies such as flower-, rod-, flake-, sphere-rose-, triangular- and star-like shapes were obtained.

Moreover, an enhancement in BET surface area and modification in pore texture were also observed. This modification resulted from either increasing the pore size and volume or the uniformity of the pores. However, optical properties like UV-Vis absorption and band gap energy are generally quite similar to that of ZnO synthesized without templating agents. Last but not least, we also investigated the effect of the addition of the as synthesized ZnO nanoparticles into polysulfone/zinc oxide mixed matrix membranes (PSf-MMMs) for the application of gas separation. The results indicated an improvement in CO_2/CH_4 separation and permeance properties of PSf/ZnO MMMs.

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

PENYEDIAAN ZINK OKSIDA STRUKTUR NANO DAN MIKRO MENGGUNAKAN KAEDAH TEMPLAT-HIDROTERMA DAN APLIKASINYA

Oleh

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Zink oksida (ZnO) tulen telah disintesis menggunakan pelbagai templat bio-dan bukan-bio seperti sodium dodesil sulfat (SDS), setil trimetilammonium bromida (CTAB), olein kelapa sawit, beras yang dimasak dan tidak dimasak. Struktur nano- dan mikro-ZnO telah disintesis melalui kaedah hidrotermal. Sifat fisiko-kimia sampel telah dicirikan bagi sampel yang telah di sintesis pada pelbagai nisbah templat terhadap zink prekursor. Pelbagai morfologi seperti bunga-, rod-, emping-, sfera-, ros-, segitiga- dan bentuk bebintang telah diperolehi.

Selain daripada itu, penambahbaikan luas permukaan BET dan modifikasi dalam tekstur liang juga telah diperhatikan. Modifikasi ini adalah hasil daripada samada peningkatan saiz liang dan isipadu atau keseragaman pada liang. Bagaimanapun, sifat optik seperti penyerapan UV-Vis dan tenaga jurang jalur adalah secara umumnya agak serupa dengan ZnO yang disintesis tanpa agen templat. Kesan penambahan zarah nano ZnO yang telah disintesis kepada matrik polisulfon/membran campuran zink oksida (PSf-MMMs) untuk diaplikasikan sebagai membran pemisahan gas telah juga dikaji. Hasil yang didapati telah menunjukkan peningkatan dalam pemisahan CO_2/CH_4 dan telapan kepada sifat-sifat PSf/ZnO MMMs.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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This is to confirm that:

- the research conducted and writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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LIST OF ABBREVIATIONS

AACVD	Aerosol-assisted Chemical Vapor Deposition
AFM	Atomic Force Microscopy
BAW	Bulk Acoustic Wave
BET	Brunauer-Emmett-Teller
BJH	Barret-Joyner-Halenda
CMC	Critical Micelle Concentration
CR	Cooked Rice
CTA^+	Cetyltrimethylammonium Cation
CTAB	Cetyltrimethylammonium Bromide
CVD	Chemical Vapor Deposition
DBE	Deep Band Emission
DC	Direct Current
DSSCs	Dye-Synthesized Solar Cells
DTG	Differential Thermogravimetric Analysis
EDS	Energy Dispersive X-ray spectroscopy
EDTA	Ethylenediaminetetraacetic acid
E_g	Band gap energy
FESEM	Field Emission Scanning Electron Microscopy
FET	Field Effect Transistor
FS	Fumed Silica
FTIR	Fourier Transform Infrared
GaN	Gallium Nitride
HMTA	Hexamethylentetramine
IPA	Isopropyl alcohol
ITQ-29	Zeolite A with higher Si/Al ratio
IUPAC	International Union of Pure and Applied Chemistry
JCPDS	Joint Committee on Powder Diffraction Standards
KBr	Potassium Bromide

K-M	Kubelka Munk
LED	Light Emitting Diodes
LPCVD	Low-pressure Chemical Vapor Deposition
MDMO-PPV	2-methoxy-5-(3′,7′-dimethyloctyloxy)-1,4-phenylenevinylene
MEMS	Micro-Electromechanical Systems
MMMs	Mixed Matrix Membranes
MOCVD	Metalorganic Chemical Vapor Deposition
NEMS	Nano- Electromechanical Systems
NMP	1-methyl-2-pyrrolidone
OGM	Octaethylene Glycol Monododecyl ether
P3HT	Poly (3-hexyl thiophene)
PECVD	Plasma-enhanced Chemical Vapor Deposition
PEG	Polyethylene Glycol
PEI	Polyethyleneimine
PFO	Polyfluorene
PL	Photoluminescence
PLD	Pulsed Laser Deposition
PO	Palm Olein
PSD	Particle Size Distribution
PSf	Polysulfone
PVD	Physical Vapor Deposition
PVP	Poly vinylpyrrolidone
PXRD	Powder X-ray Diffraction
RF	Radio-Frequency
SAW	Surface Acoustic Wave
SDA	Structure Directing Agent
SDS	Sodium Dodecyl Sulfate
SEM	Scanning Electron Microscopy
SiO_2	Silicon Dioxide

TEM	Transmission Electron Microscopy
TGA	Thermogravimetric Analysis
THF	Tetrahydrofuran
UR	Uncooked Rice
UV-VIS	Ultraviolet Visible
VLS	Vapor-Liquid-Solid
VS	Vapor-Solid
XRD	X-ray Diffraction
ZIF	Zeolitic Imidazolate Frameworks
ZnO	Zinc Oxide

CHAPTER 1
INTRODUCTION

1.1 Nanoscience and Nanotechnology

1.1.1 History

Nanotechnology has been grown its roots in a pioneering talk by a prominent physicist, Richard Feynman in 1959. His tremendous speech, namely "There is plenty of room at the bottom" was pointed out the producing, manipulating and controlling things on a very small scale. He suggested the possibility of arranging atoms in a "way that we want" through the physic's principles in the future (Ozin & Arcenault, Andre C, 2005).

Thereafter, the term nanotechnology was seen for the first time in an article, named "On the Basic Concept of Nanotechnology" by Prof. Norio Taniguchi in 1974 (Klusek, 2007). He described nanotechnology as a process in which separation, consolidation and deformation of the material may occur by one atom or molecule. In fact, nanotechnology differs from conventional technologies since the "bottom-up" approach is preferred in nanotechnology while in conventional technologies usually the "top-down" approach is considered. The term of "top-down" describes processes starting from large pieces of material and producing the intended structure by mechanical or chemical methods, whereas "bottom-up" expression is used to ascribe atoms or molecules which construct the building blocks to produce nanomaterials (Vollath, 2008).

Therefore, scientists focused on exploring the fundamental nature of materials during past decades. They exploited the nanoscience principles to synthesize materials with unique structures and properties by controlling their size and composition. This made nanomaterials valuable for many different applications. It even found extensive applications in different areas e.g. electronics, agriculture, textiles, medicine and antibacterial consumables energy and environment, etc. Today, nanotechnology has become one of the most challenging, multi-disciplinary and competitive fields (Yousaf & Ali, 2008). Moreover, the ultimate goal of nanotechnology is to fabricate self-replicating nano-robots which will overrun the earth (Ozin & Arcenault, Andre C, 2005).

1.1.2 Nanomaterials

Nanomaterials can be defined as materials with at least one dimension less than 100 nm and second dimension below 1 µm. In a narrower definition, nanomaterials benefit from at least two dimensions below 100 nm (Kohlar & Fritzsche, 2007). In fact, they attract lots of attention since they found very unique properties which depend inherently on their small grain size (Vollath, 2008). In other words, the properties of nanomaterials are size-dependent. Nanomaterials can be classified into zero-dimensional (e.g. nanoparticles), one-dimensional (e.g. nanorods or nanotubes), or two-dimensional (e.g. thin film or stacks of thin films).

Nanomaterials have unique properties compared to their bulk counterparts, such as mechanical, catalytic, magnetic and optical properties which will be investigated according to the desirable target. In fact, decreasing the material scale to nanometer can promote or even lead to new property. For instance, large amount of grain boundaries in nanomaterials allows sliding which leads to plasticity. Moreover, due to the large surface, nanomaterials exhibit catalytic properties. Additionally some nanomaterials show the supermagnetism property which in combining with particles with high energy of anisotropy can lead to a new class of permanent magnetic materials. Furthermore, preparation of non-agglomerated particles with distribution in polymer can produce nanomaterials with non-linear optical properties (Vollath, 2008).

1.2 Zinc Oxide

1.2.1 Basic properties of zinc oxide

Zinc oxide is a II-VI semiconductor compound with density of about 5.6 g/cm^3. It has a hexagonal wurtzite crystal structure with lattice parameters of a = 0.325 nm and c = 0.521 nm. Zinc oxide is thermodynamically stable under ambient condition. In addition, zinc oxide is transparent in visible light due to the band gap energy of about 3.3 eV. It also has relatively high exciton binding energy of about 60 meV which increases its light emission efficiency (Jagadish & Pearton, 2011; Morkoç & Özgür, 2008). Moreover, zinc oxide benefits from wide range of properties which made it the attention centre for past decades including piezoelectricity, pyroelectricity, high transparency, room-temperature ferromagnetism, wide band gap semiconductivity, chemical sensing and huge magneto-optic effect (Schmidt-Mende & MacManus-Driscoll, 2007).

1.2.2 Zinc oxide applications

Zinc oxide has very extensive applications due to its exclusive and multiple properties. It is used widely in transducers, energy generator, photocatalysis for hydrogen production (Z. L. Wang et al., 2004), varistors, optoelectronic devices, transparent conducting electrodes (Jagadish & Pearton, 2011), UV-light emitting diodes, sensors, solar cells and etc. It is also used as catalysts in production of methanol out of CO or CO_2 and H_2. Zinc oxide combined with other compounds like Al_2O_3 and Cu is considered as a very good catalyst. Due to its antibacterial activity, lots of ointments, creams and bandages are applied with zinc oxide. In addition, zinc oxide is biocompatible and biodegradable; it is commonly applied in medical and pharmaceutical products. Moreover, it can be found in cosmetics like facial powders and sunscreens due to their UV absorption and photo stability properties. Furthermore, it is known as additives in lubricants, cement and rubber and generally used as non-poisonous white pigment in paint industry (Klingshirn, 2010).

1.3 Problem Statement

ZnO nanomaterials encounter some limitations in their applications due to their restricted behavior in different media. These limits are revealed whereas inorganic nanomaterials need to tune their properties and applications according to nanotechnology prospects. Nanotechnology is exploring its future in the multifunctional nanomaterials to fabricate multifunctional nano-devices. Thereby

ZnO nanomaterials properties require to be manipulated synchronously in order to suit to new and multifunctional applications. This requirement will be fulfilled with different types of surface modification. One of the most novel and outstanding methods of surface modification for ZnO nanostructures is biotemplating which will be addressed thoroughly in Chapter 2. In spite of the advantages of cost- and time-effective approach which applies very sophisticated biological structures, biotemplating route has some limitations. Very small size of some biotemplates is counted as disadvantage since unable them to be used in industrial large-scale applications. In addition, some fascinating biotemplates with very specific morphological structures are rare and unavailable to be studied and applied. In some other cases, the materials that applied biotemplates become so sensitive to be manipulated and applied for functional devices. In conclusion, choosing a suitable biotemplate which simultaneously enjoy benefits from unique physicochemical or morphological properties to guide the assembly of ZnO structures and assure the accurate and precise replication of that special property is a serious challenge in ZnO nanomaterials synthesis. To overcome this problem, large numbers of trial and error experiments need to be done to get some reliable and reproducible results which lead to synthesis of high quality and uniformity ZnO nanomaterials in large scale production.

1.4 Significance of the study

In this study, we put all our efforts to overcome the limitations of the templated-assisted synthesis and produce mesoporous ZnO nanomaterials. To do so, the templates applied in this work were chosen from the cheapest and most available and biocompatible existing templates. Moreover, this study not only focused on the synthesis and optimizing the morphological structure of ZnO nanomaterials to achieve the modification of properties and improved performances, but also tried to find out the functional mechanism between templates and the nanoparticles and structures of synthesized ZnO. In addition, this work put its step beyond the synthesis and extended to the application of modified final product. The potential application of the synthesized ZnO nanostructures has investigated in the Chapter 7.

1.5 Objectives of study

The objectives of this study are:

1- To synthesize and characterization of the ZnO nanostructures using various templates.

2- To investigate the effect of various concentration of templates on physico-chemical properties of ZnO nanostructures.

3- To propose the growth mechanism of ZnO crystals and the role of structure directing agent on it.

4- To investigate the potential applications of as-synthesized ZnO-SDS/CTAB in gas separation.

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LIST OF PUBLICATIONS

1. D. Ramimoghadam, M.Z. Bin Hussein and Y.H. Taufiq-Yap 2012. The effect of sodium dodecyl sulfate (SDS) and cetyltrimethylammonium bromide (CTAB) on the properties of ZnO synthesized by hydrothermal method. *Int J Mol Sci* 13(10):13275-13293.

2. D. Ramimoghadam, M.Z. Bin Hussein and Y.H. Taufiq-Yap 2013. Synthesis and characterization of ZnO nanostructures using palm olein as biotemplate. *Chem Cent J* 7:71-80.

3. D. Ramimoghadam, M.Z. Bin Hussein and Y.H. Taufiq-Yap 2013. Hydrothermal synthesis of zinc oxide nanoparticles using rice as soft biotemplate. *Chem Cent J* 7:136-145.

4. P. Moradihamedani, N.A. Ibrahim, D. Ramimoghadam, M.Z.W Yunus and N.A. Yusof 2013. Polysulfone/zinc oxide nanoparticle mixed matrix membranes for CO_2/CH_4 separation. *J. Appl. Polym. Sci.* doi: 10.1002/app.39745.