

DEVELOPMENT OF A CONTINUOUS MICROWAVE REACTOR FOR THE
PRODUCTION OF ZINC OXIDE

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ABSTRACT

This study involves experimental design and development of continuous microwave reactor for the production of zinc oxide (ZnO) nanoparticles. The reactor consisted of a modified household microwave oven which was operated with a frequency of 2.45 GHz and a maximum output power of 800 W. The modified microwave reactor was equipped with two peristaltic pumps and connected to glass columns via T-junction to allow for mixing of the reactants, zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and sodium hydroxide (NaOH), in the microwave system. The effect of process parameters such as retention time, microwave power and reactant concentration on the phase composition, particle morphology and optical properties of ZnO was investigated. At a fixed concentration of the reactants, crystalline phase of ZnO was formed as observed from the X-ray Diffraction (XRD) patterns. Also, the phase crystallinity of ZnO was found to improve when the retention time of the reactants in the reactor was increased up to 20 min and increasing of the microwave power until 600 W. Besides, increasing the concentration of reactants has successfully produced crystalline phase pure ZnO nanoparticles which were obtained using the mole ratio of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}:\text{NaOH}$ of 1:2. Transmission Electron Microscopy (TEM) image revealed spherical-shape ZnO nanoparticles with sizes ranging from 6 to 12 nm and the result matched well with the crystallite sizes determined from the XRD data using the Scherrer equation. The ZnO nanoparticles exhibited a strong absorption in the 390 nm region of the Ultraviolet-Visible (UV-Vis) spectra which is red-shifted from bulk ZnO (370 nm) with the band-gap value of 3.15 eV. The peak intensity was increasingly decreased along with increasing of retention time, microwave power and reactant concentration while the band-gap energies were found to decrease upon increasing of the retention time, microwave power and reactant concentration. The green band emission observed in the region of 350-470 nm in the Photoluminescence (PL) spectra suggests the presence of high oxygen vacancies in the ZnO lattice which then further reduced with increasing of retention time, microwave power and reactant concentration.

ABSTRAK

Kajian ini melibatkan rekabentuk eksperimen dan pembangunan reaktor gelombang mikro berterusan untuk penghasilan zarah nano zink oksida (ZnO). Reaktor tersebut adalah terdiri daripada gelombang mikro isi rumah terubahsuai yang beroperasi pada frekuensi 2.45 GHz dan kuasa pengeluaran maksimum 800 W. Reaktor gelombang mikro terubahsuai tersebut dilengkapi dengan dua pam peristaltis dan dihubungkan dengan turus kaca melalui persimpangan-T untuk mencampurkan reaktan, zink nitrat heksahidrat ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) dan natrium hidroksida (NaOH) dalam sistem gelombang mikro. Kesan parameter proses seperti masa penahanan, kuasa gelombang mikro dan kepekatan reaktan ke atas komposisi fasa, morfologi zarah dan sifat optik ZnO yang terbentuk telah diselidiki. Pada kepekatan reaktan yang tetap, fasa habluran ZnO terbentuk seperti yang diperhatikan pada corak pembelauan sinar-X (XRD). Juga, fasa kehabluran ZnO didapati bertambah apabila masa penahanan reaktan di dalam reaktor dilanjutkan sehingga 20 min dan meningkatkan kuasa gelombang mikro sehingga 600 W. Di samping itu, meningkatkan kepekatan reaktan telah berjaya menghasilkan fasa habluran tulen zarah nano ZnO yang diperoleh menggunakan nisbah mol 1:2 untuk $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}:\text{NaOH}$. Imej Mikroskopi Penghantaran Elektron (TEM) mendedahkan zarah nano ZnO berbentuk sfera dengan saiz dalam julat dari 6 hingga 12 nm dan keputusan ini sepadan dengan saiz kristalit yang ditentukan daripada data XRD menggunakan persamaan Scherrer. Zarah nano ZnO mempamirkan penyerapan kuat di kawasan 390 nm pada spektrum Ultra-lembayung-Nampak (UV-Vis) yang teranjak merah berbanding ZnO pukal (370 nm) dengan nilai ruang jalur 3.15 eV. Keamatan puncak tersebut semakin menurun dengan pertambahan masa penahanan, kuasa gelombang mikro dan kepekatan reaktan manakala nilai ruang jalur didapati semakin menurun dengan pertambahan masa penahanan, kuasa gelombang mikro dan kepekatan reaktan. Pancaran jalur hijau yang diperhatikan dalam kawasan 350-470 nm pada spektrum fotoperdarcahaya (PL) mencadangkan terdapat banyak kekosongan oksigen dalam kekisi ZnO yang semakin berkurangan dengan pertambahan masa penahanan, kuasa gelombang mikro dan kepekatan reaktan.

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LIST OF ABBREVIATIONS

ZnO	Zinc Oxide
Zn(OH) ₂	Zinc Hydroxide
Zn(NO ₃) ₂ ·6H ₂ O	Zinc Nitrate Hexahydrate
NaOH	Sodium Hydroxide
CVD	Chemical Vapor Deposition
XRD	X-Ray Diffraction
FTIR	Fourier Transform Infrared
TEM	Transmission Electron Microscope
UV-Vis	Ultraviolet Visible
PL	Photoluminescence
DNA	Deoxyribonucleic Acid
RNA	Ribonucleic Acid
GOD	Glucose Oxidase (Microorganism)
MEA	Monoethanolamine
DEA	Diethanolamine
TEA	Triethanolamine
HDA	Hexadecylamine

CHAPTER 1

INTRODUCTION

1.1 Introduction of Zinc Oxide as Nanomaterial

Zinc Oxide (ZnO) has wide applications such as biomedical material, electronic applications, solar cell and lubricants etc. It has a wide bandgap of 3.37 eV with large exciton energy of 60 meV, which makes it suitable for short wavelength optoelectronic applications. This strong exciton energy binding provides an efficient exciton emission at room temperature and makes this material a promising photonic material in the blue-UV region (Wang, 2004). ZnO has been widely used since 2000 BC as an ointment (Moezzi *et.al*, 2012) and recently has been heavily used in light emitting devices, laser diodes, photodetectors, solar cells, catalysts, pigments, cosmetics, gas sensor and industrial additives (Dumbrava *et.al*, 2013).

In rubber industry ZnO has been used to accelerate the cure rate of rubber. ZnO is also used for the protection of latex plantation due to its reactivity with the enzyme responsible for latex decomposition, thus, it is able to improve as well as maintain physical properties of rubber. In ceramic industry ZnO is used to maintain semiconductor ceramic elements at elevated temperature or high voltages during processing. Other electronic devices like varistors are composed of modified ZnO or doped with other materials and it can also be used for other electrical devices. In pharmaceutical/cosmetic industry ZnO is used in soap, ointment, dental inlays, food powders etc. It is also known for its use in hair and skin care products. On the other hand, ZnO is known for its use in food packaging industry as well as various packaging in animal and vegetable product as antibacterial material to keep product safe from bacteria and fungi.

Generally, ZnO exists in three different types of crystal structures, which are hexagonal wurtzite, cubic zinc-blend and cubic rock salt (NaCl type) (Moezzi *et. al*, 2007). Among these three structures, hexagonal wurtzite is the most thermodynamically stable structure and exists under ambient conditions. ZnO is categorized as non-toxic material as it usually does not cause skin or eye irritation, however it can be hazardous through inhalation or ingestion, while soluble zinc compounds are ecotoxic to aquatic organism. Due to its unique wide application, ZnO is capable to form diverse group of growth morphologies, such as nanocombs, nanorings, nanosprings, nanobelts, nanowires, nanocages and etc. (Wang, 2004).

ZnO structure can be described as alternate number of planes which are composed of tetrahedrally coordinated O^{2-} and Zn^{2+} ions. ZnO has three types of growth directions, which are (2110), (0110) and (0001). Bulk ZnO is normally produced using French and American Process, but there are several other processes available to produce ZnO as nanomaterial, such as solvothermal, sol-gel, hydrothermal, wet-chemical and microwave method (Fadeel and Garcia-Bennet, 2010)

1.2 Synthesis of ZnO

1.2.1 Chemical Vapour Deposition Method (CVD)

This method involves volatile precursors where the active gaseous species are generated and transported into the reaction chamber. Here, the precursors undergo gas phase reaction to form an intermediate phase. When the temperature inside the reactor gets higher, homogeneous gas phase reactions occur whereby the intermediate species undergo decomposition and chemical reaction to form powders and volatile byproducts. Powders are collected on the substrate surface, which act as crystallization centers; eventually they are transported away from the deposition chamber. The

intermediate are diffused across the boundary layer and are absorbed onto the heated substrate and heterogeneous reactions occur at the gas-solid interface that produces the deposit and byproducts.

Later, the deposit is diffused along the heated substrate surface forming the crystallization center for subsequent growth of the film. Finally, the gaseous byproducts are diffused away from the boundary layer and simultaneously, the unreacted gaseous precursor and byproduct are transported away from the deposition chamber (**Figure 1.1**) (Liu *et. al*, 2008).

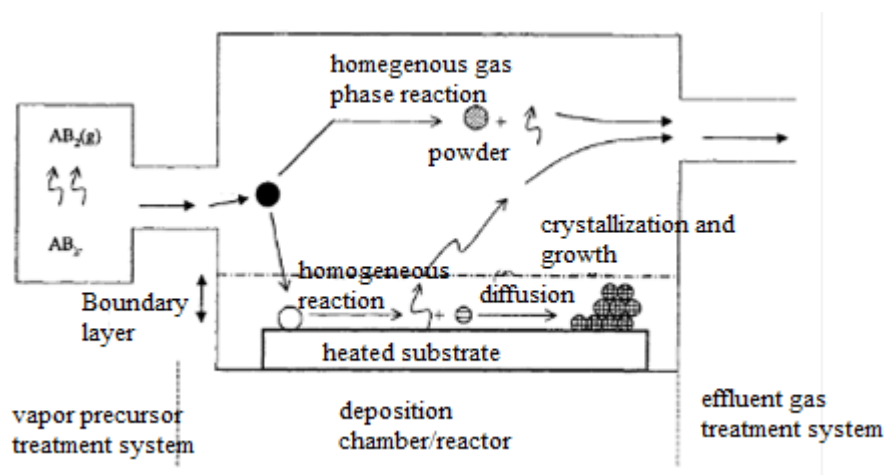


Figure 1.1: Main steps occurring in the CVD process

ZnO crystal fibers have been developed using CVD process, by using quartz tube at 1100 °C with one side opened to the air. Polycrystalline hexagonal wurtzite-phase ZnO with diameter ranging from 300 nm to 1.5 μm was prepared through this method (Chen *et. al*, 2004). Meanwhile, a dual plasma-enhanced with metal-organic chemical vapour deposition (DPEMOCVD) has been used to study the ZnO grown on polyestersulfone (PES) surface. They researchers used direct voltage and radio frequency plasma system inside DPEMOCVD to produce ZnO (Lei *et.al*, 2012).

1.2.2 Sol-gel method

The ‘Sol’ refers to a stable dispersion of colloidal particles or polymer in the solvent. These particles may be amorphous or crystalline. Meanwhile, the ‘gel’ consists of three dimension of continuous network, where it is built from agglomeration of colloidal particles. Generally, the sol may interact by Van der Waals forces or hydrogen bonds. It may also be formed by linking polymer chains and gelation process may be reversible due to the involvement of other interactions.

Simple method on sol-gel process to produce ZnO thin film was discussed detailed by Znaidi (2010), where typically ZnO thin film involves several critical parameters, which is nature of precursor and its concentration, solvent type, additive species type and its concentration, aging time on the early mixture of precursor, coating method of substrate and pre-heat, post-heat treatment of materials. **Figure 1.2** shows a detailed process mapping conducted by Znaidi (2010).

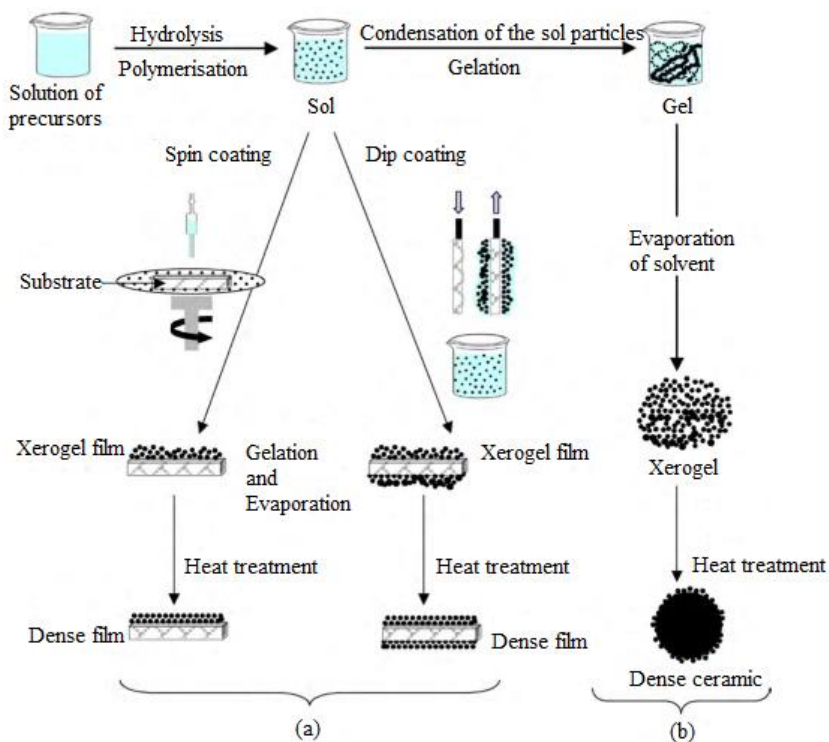


Figure 1.2: Process mapping of sol-gel method to produce ZnO thin film based on Znaidi (2010) study.

Based on figure above, a solution will undergo a transformation in (a), which is hydrolysis of the molecular precursor and transformation (b), which is polymerization via successive bimolecular additions of ions, to form a group of oxo-, hydroxyl or aquabridges. Also, based on the figure, the main steps of preparation by sol-gel process can be summarized in three parts, where the first part is the preparation of precursor solution, then followed by deposited of the sol on the substrate and finally, the heat treatment process of the xerogel film. Later this xerogel film will be dried gel at the ambient pressure.

1.2.3 Hydrothermal Process

Hydrothermal process involves heterogeneous reaction in the presence of aqueous solvents under high pressure and temperature in a closed system. Continuous hydrothermal process has also gained its popularity due to its quick reaction times, and it is able to achieve high purity of nanoparticles without any post treatment (Shin *et.al*, 2009). Makishima *et.al* (2009) have successfully developed a continuous hydrothermal method, where its reactor structure consisted of material feeder, tube shape hydrothermal reactor and product discharge unit (**Figure 1.3**).

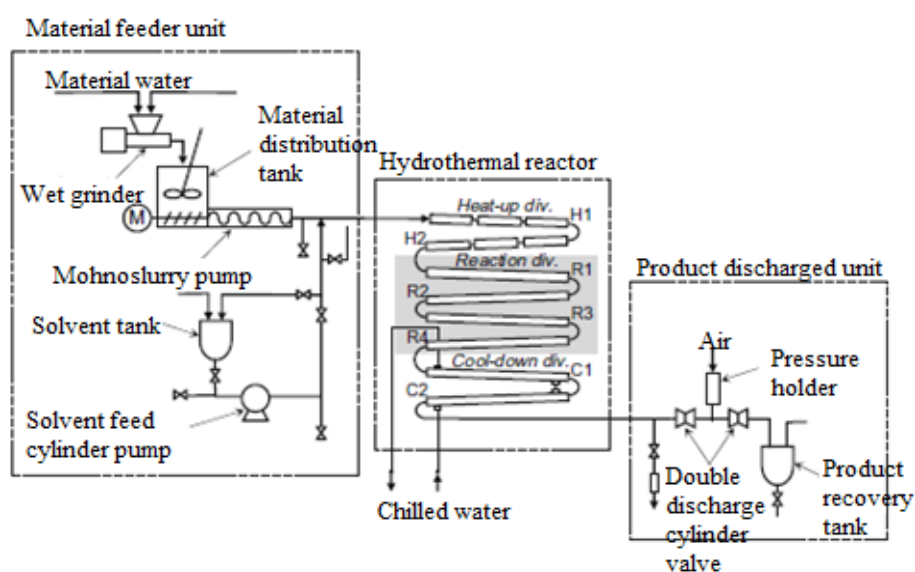


Figure 1.3: Continuous hydrothermal process from Makishima *et.al*, 2009

However, a drawback of this process is that it requires high temperature and pressure in order to furnish products with high purity and crystallinity. The reactor works most best at the critical temperature of water (374 °C) at this temperature the reactor presents serious safety concerns and long term operation of the reactor is fairly costly. Furthermore, continuous flow hydrothermal flow system suffers from poor heat transfer control, limited crystal growth due to the short dwell time inside the heating zone and particles are highly agglomerated (Aimable *et.al*, 2009). The reactor also works best at low concentrations of the reactants to avoid blockage of stainless steel pipes and pressure valves, furthermore the corrosion of pipes and valves are also major drawbacks of the system.

1.2.4 Solid-state Process

This solvent less process is often referred to as ceramic method. A mixture of solid starting materials, which usually do not react together at room temperature are heated at elevated temperature between 1000 °C-1500°C. This process requires high temperature in order to provide necessary energy for the process to occur. **Figure 1.4** shows the formation of product in the solid-state process.

Reaction starts when two precursors, which is A and B is on contact, and reaction will only occurs at contact points between grains of precursor A and B. Then, at high temperature, the product of C will grow from diffusion process of A and B, and high temperatures provides the necessary energy for the reaction to occur and it usually leads to thermodynamically stable product. However, diffusion process to produce C is very slow, due to it requires breaking and forming many bonds for complete disruption of structure A and B.

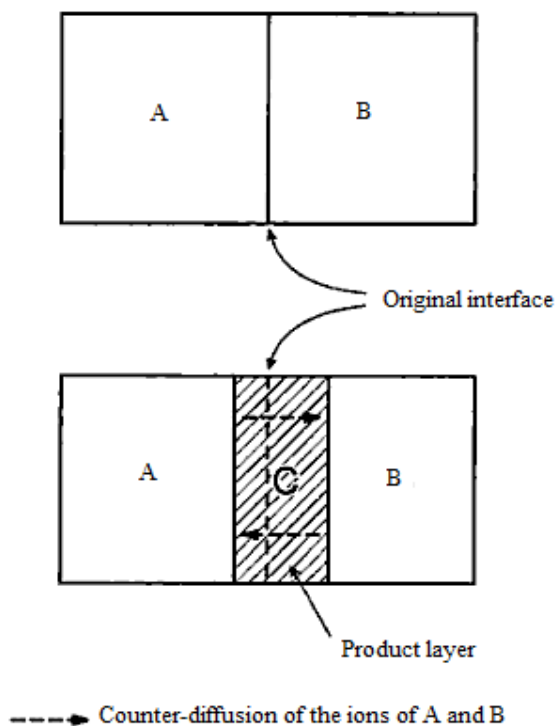


Figure 1.4: Solid-state process to produce nanopowders

1.2.5 Co-precipitation Process

In a co-precipitation process a product is precipitated from the solution containing other ions, and it occurs when a solution is supersaturated with a substance and forming a precipitate or when a melt is super cooled. Generally, there are three major processes involved in co-precipitation process. The first process is known as inclusion, where the impurity in substance occupies a lattice site in the crystal structure of the carrier. The second process is called occlusion, where an adsorbed impurity is physically trapped inside the crystal during growth. Third process is called adsorption, where the adsorbed impurity is weakly bound to the surface of the precipitate. After these three processes are completed, a powder is often washed and dried to obtain pure nanopowders.

1.3 Microwave Assisted Synthesis

Microwave assisted syntheses in the modern research have received tremendous attention due to their shorter processing time. In certain cases, it has successfully replaced the traditional method like sol-gel, hydrothermal, solid-state reaction, emulsion and microemulsion synthesis (Parhi *et.al*, 2004). Microwave heating enables thorough direct heating to the materials through molecular interaction with electromagnetic field (Thostenson and Chou, 1999). It offers advantages such as non-contact heating, efficient energy transfer, rapid volumetric heating and provides higher level of safety and automation (Menendez *et.al*, 2010).

Microwave heating also gives advantage to ceramics and polymer processing, where these materials are well known for having low thermal conductivities, and thus, it can reduce processing time significantly (Thostenson and Chou, 1999). There are several advantages of using microwave processing compared to conventional method, such as speed, where microwave heating occurs not just at the surface of the material, but it occurs within the material itself, making the heating process much faster. Besides, it enables energy saving because microwave energy is not wasted to heat the equipment walls or the environment. Heat is only developed where it is needed, such as material or precursors that are used for the process.

Other advantages include space saving and fast process or production startup and shutdown. This is because microwave oven is compact equipment, and it does not affect the processing rate prior to its building space. Therefore, production capacity can be increased without the need for any additional space. Nowadays, many researchers are trying to design, evaluate, and optimize their microwave to achieve the desirable, higher yield result. There are several type of equipment used in microwave synthesis, as listed below.

1.3.1 Household microwave oven

This is the cheapest of equipment used especially in organic synthesis process and is limited to 1000 W only. Its electric field distribution is homegenous and samples are often subjected to the maximum levels for varying time period. However, organic syntheses often require control of reaction parameters such as pressure and temperature, which cannot be controlled in this type of setup.

1.3.2 Modified microwave oven

This setup is very similar to the domestic microwave oven, but has been modified with a slight variation prior to maintain its accuracy and safety during synthesis. The only difference in modified microwave oven is that it has a cavity at the top of the microwave, which allows the introduction of air cooler for solvent reflux as well as allowing researchers to follow various process parameters.

1.3.3 Commercially available microwave reactor.

This category is specialized equipment, which is equipped with various features such as build-in magnetic stirrer, direct temperature controller to maintain fixed temperature and pressure of the reaction mixture with the aid of fiber optic probes.

1.4 Problem Statement

ZnO is a valuable material with wide applications; however, current production to produce zinc oxide at nano-sized is complicated, time-consuming, tedious process and it requires expensive design and setup. Most of the methods also require post treatment such as calcination of nanopowders. Also, current continuous method for ZnO at nano-sized production requires expensive laboratory setup and it can only work at very low concentrations.

The purpose of this research is to build and evaluate a continuous reactor for the production of ZnO nanopowders. The proposed continuous microwave flow reactor will make use of cheap precursors and will not involve expensive and elaborate setup. It mainly involves a modified household microwave oven which easily available at the market, and the modification of microwave is made from inexpensive laboratory equipment as well as easily the equipment is easily made from local laboratory.

1.5 Objectives of the Study

Below are the main objectives for this research:

1. Design and build a continuous microwave reactor for the production of ZnO.
2. Control process parameters to obtain phase pure ZnO nanoparticles. All process control will be observed in terms of process flow rates, pumps velocity, effect on process time and microwave power and effect on precursor concentration,
3. Study the effect of process parameters on the crystallinity of nanopowder, crystallite size and its effect on the optical properties as well as antibacterial activities of ZnO nanopowder.

1.6 Scope of the Study

This research addresses the continuous production of ZnO nanoparticles using continuous microwave flow reactor. The main reactor consists of 800 W modified household microwave operating at 2.45 GHz frequency. This project will be assessed prior to the efficiency design setup and the quality of the ZnO produced. Therefore, it will be evaluated according to the degree of crystallinity, phase purity, crystallite size, particle morphology and optical properties. Techniques such as XRD, FTIR, TEM, UV-Vis, and Photoluminescence will be used to fully characterize the ZnO nanostructures.

1.7 Significance of the Study

This study will enable us to continuously produce crystalline ZnO nanoparticles, where physicochemical and optical properties of the nanoparticles could be controlled by adjusting the process parameters. This customized continuous microwave flow reactor would be a cheaper alternative to the elaborate and expensive setup of continuous hydrothermal flow synthesis reactor.

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