SYNTHESIS, STRUCTURAL, MORPHOLOGICAL AND OPTICAL PROPERTIES OF (ZnO)X(TiO2)1-X NANOPARTICLES PREPARED THROUGH THERMAL TREATMENT TECHNIQUE

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By

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Numerous studies have researched the chemical, physical, and photochemical characteristics of zinc oxide titania ZnO-TiO_2 nanoparticles resulting through various routes of fabrication. The properties of nanoparticles rely upon the chemical composition and microstructural features in which the shape and the particle size might be controlled in the preparation procedure. Several techniques have been applied previously to create ZnO-TiO_2 nanoparticles with potential physical and chemical properties. However, no previous research has been found in utilizing thermal treatment method to convey ZnO-TiO_2 nanoparticles. Relative to most other techniques, thermal treatment method has a more straightforward methodology, shorter reaction time, lower reaction temperature, utilize less number of synthetic substances, does not utilize any toxic reagent and furthermore does not produce earth unsafe waste. In this thesis, the current study applied thermal treatment method to synthesize $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles with x = 0.25, 0.50 and 0.75 by using only metal precursors (zinc nitrate and titanium (IV) propoxide), capping agent polyvinylpyrrolidone (PVP) and deionized water. The aqueous solution containing the metal precursors, the capping agent PVP and deionized water was dried for 24 hours at 80 °C, ground to turn out to be fine powder and lastly calcined at temperature ranging from 500 to 800 °C. The structural, morphological and optical properties of the synthesized samples were examined by utilizing different characterizations such as X-ray diffraction (XRD), Fourier-transform infrared (FTIR) spectroscopy, transmission electron microscopy (TEM), field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) spectroscopy, ultraviolet-visible (UV-Vis) spectrophotometry and photoluminescence (PL) spectroscopy. Likewise, thermogravimetric analyzer (TGA) was used to study the thermal stability and to determine the ideal calcination temperature at which polymer could be removed from the samples. The TGA measurement demonstrated that the capping agent PVP was completely deteriorated at temperatures higher than 500 °C which is in concurrence with the vanishing of PVP absorption peaks in the FTIR spectra. XRD spectra of the nanoparticles that have been calcined at 500−800 °C uncovered that the mean crystallite size for $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles were in the range of 24.90 to 92.82 nm with the structure of hexagonal zinc oxide, tetragonal titania, hexagonal ecandrewsite and cubic titanium dizinc oxide. The XRD results were additionally affirmed by TEM analysis which confirmed the development of nanoscale samples. As the calcination temperature increased, the average nanoparticle size determined through TEM image also increased from 25.922 to 90.056 nm. Furthermore, the optical behavior of the prepared nanoparticles was considered by means of UV-Vis spectrophotometer whereby the optical band gaps were determined by using Kubelka-Munk equation. The various band gaps showed up in the absorption spectra essentially demonstrated an increasing trend ranging from 3.238 to 3.741 eV as the calcination temperature increased from 500 to 800 °C. PL spectral investigations of the nanoparticles demonstrated emission peaks at around 420 nm (violet) and 480 nm (blue) for an excitation wavelength of 280 nm. The procured information on the synthesized $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles through thermal treatment method propose that the samples have extraordinary potential in semiconductor applications, for example, photocatalytic and wide band gap power devices.

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

SINTESIS, SIFAT STRUKTUR, MORFOLOGI DAN OPTIKAL NANOPARTIKEL $(ZnO)_X(TiO_2)_{1-X}$ DISEDIAKAN MELALUI TEKNIK RAWATAN TERMA

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Banyak kajian telah meneliti ciri-ciri kimia, fizikal, dan fotokimia zink oksida titania ZnO-TiO_2 nanopartikel yang dihasilkan melalui pelbagai kaedah fabrikasi. Ciri-ciri nanopartikel bergantung kepada komposisi kimia dan ciri-ciri mikrostruktur di mana bentuk dan saiz zarah dapat dikawal dalam prosedur penyediaan. Beberapa teknik telah digunakan sebelum ini untuk menghasilkan nanopartikel ZnO-TiO_2 dengan ciri-ciri fizikal dan kimia yang berpotensi. Walaubagaimanapun, tiada kajian terdahulu yang ditemui menggunakan kaedah rawatan terma untuk menghasilkan nanopartikel ZnO-TiO_2. Berbanding dengan kebanyakan teknik lain kaedah rawatan terma mempunyai metodologi yang lebih ringkas, masa reaksi yang lebih pendek, suhu tindak balas yang lebih rendah, menggunakan kurang bahan kimia, tidak menggunakan sebarang reagen toksik dan juga tidak menghasilkan sisa buangan bumi yang berbahaya. Dalam kajian ini, penyelidikan semasa menggunakan kaedah rawatan terma untuk mensintesiskan nanopartikel $(ZnO)_x(TiO_2)_{1-x}$ di mana x = 0.25, 0.50 dan 0.75 dengan menggunakan hanya prekursor logam (zink nitrat dan titanium (IV) propoksida), polyyinylpyrrolidone (PVP) dan air deionized. Larutan akueus yang mengandungi prekursor logam, PVP agen pelindung dan air deionized dikeringkan selama 24 jam pada suhu 80 °C, dilenyek menjadi serbuk halus dan kemudian dikalsinasi pada suhu antara 500 hingga 800 °C. Ciri-ciri struktur, morfologi dan optik sampel yang disintesis diperiksa dengan menggunakan ciri-ciri yang berbeza seperti pembelauan sinar-X (XRD), spektroskopi inframerah transformasi Fourier (FTIR), mikroskopi elektron transmisi (TEM), mikroskopi elektron pengimbasan pancaran medan (FESEM), spektroskopi X-ray dispersif tenaga (EDX), spektrofotometri ultralembayung-cahaya nampak (UV-Vis) dan spektroskopi fotopendarcahaya (PL). Tambahan pula, penganalisis termogravimetri (TGA) digunakan untuk menyelidik kestabilan terma dan untuk menentukan suhu kalsinasi optimum yang dapat menyingkirkan polimer dari sampel. Pengukuran TGA menunjukkan bahawa agen pelindung PVP telah disingkirkan

sepenuhnya pada suhu lebih tinggi daripada 500 °C setara dengan kehilangan puncak penyerapan PVP dalam spektrum FTIR. Spektrum XRD nanopartikel yang telah dikalsinasi pada suhu 500−800 °C mendapati bahawa saiz hablur bagi $(ZnO)_x(TiO_2)_{1-x}$ nanopartikel berada dalam lingkungan 24.90 hingga 92.82 nm dengan struktur heksagon zink oksida, tetragonal titania, heksagonal ecandrewsite dan kubik titanium dizink oksida. Hasil XRD juga disahkan oleh analisis TEM yang membuktikan penghasilan sampel bersaiz nano. Apabila suhu kalsinasi meningkat, saiz purata nanopartikel yang ditentukan melalui imej TEM juga meningkat dari 25.922 hingga 90.056 nm. Tambahan pula, sifat optik nanopartikel yang disediakan telah dipertimbangkan melalui spektrofotometer UV-Vis di mana jurang jalur optik ditentukan dengan menggunakan persamaan Kubelka-Munk. Pelbagai jurang jalur yang muncul dalam spektrum penyerapan pada dasarnya menunjukkan peningkatan dari 3.238 hingga 3.741 eV apabila suhu kalsinasi bertambah dari 500 kepada 800 °C. Penyiasatan spektrum PL terhadap nanopartikel menunjukkan puncak pancaran di sekitar 420 nm (ungu) dan 480 nm (biru) untuk panjang gelombang pengujaan 280 nm. Maklumat yang diperolehi mengenai nanopartikel $(ZnO)_x(TiO_2)_{1-x}$ mencadangkan bahawa sampel yang dihasilkan melalui kaedah rawatan terma mempunyai potensi yang luar biasa dalam aplikasi semikonduktor, contohnya, fotomangkin dan peranti kuasa jurang jalur lebar.

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

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ZnO	zinc oxide
UV	Ultraviolet
TiO_2	titanium dioxide or titania
VB	valence band
CB	conduction band
DNA	deoxyribonucleic acid
E_g	band gap
TEOT	tetraethyl titanate
$Zn(NO_3)_2{\cdot}6H_2O$	zinc nitrate hexahydrate
TEM	transmission electron microscopy
TTIP	tetraisopropyl orthotitanate
SEM	scanning electron microscopy
XRD	X-ray diffraction/diffractometer
FTIR	Fourier-transform infrared
PL	photoluminescence
TGA	thermogravimetric analyzer/analysis
UV-Vis	ultraviolet-visible
HRTEM	high resolution transmission electron microscope /microscopy
FESEM	field emission scanning electron microscope/microscopy
$C_{12}H_{28}O_4Ti$	titanium (IV) isopropoxide
$C_4H_6O_4Zn{\cdot}2H_2O$	zinc acetate dihydrate
H_2S	hydrogen sulfide
NaOH	sodium hydroxide
rpm	revolution per minute
NH_4OH	ammonium hydroxide
$Ti(OC_4H_9)_4$	titanium (IV) butoxide
Pa	pascal
Zn_2TiO_4	zinc orthotitanate
$ZnTiO_3$	zinc metatitanate or ecandrewsite
$Zn_2Ti_3O_8$	zinc titanate
min	minutes
$TiCl_4$	titanium tetrachloride
$TiCl_3$	titanium trichloride
eV	electron volt
$Ti(SO_4)_2$	titanium (IV) sulfate
CH_4N_2O	urea
PEG	polyethylene glycol
PVP	polyvinylpyrrolidone
$CoFe_2O_4$	cobalt ferrite
$ZnCr_2O_4$	zinc chromite
ZnSe	zinc selenide
NiO	nickel oxide
CdSe	cadmium selenide
$Fe(NO_3)3{\cdot}9H_2O$	iron nitrate
$Co(NO_3)_2{\cdot}6H_2O$	cobalt nitrate
CdO	cadmium oxide

CHAPTER 1

INTRODUCTION

1.1 Nanoscience and nanomaterials

Nano is a measurement prefix alluding to a factor of 10^{-9} that is originated from Greek word nanos meaning dwarf (Hagens et al., 2007). Nanoscience is the investigation of structures and materials in nanometer size range though science, designing and innovation directed at the nanoscale is called nanotechnology. Nature nanoscale structures have existed in nature even some time before individuals began to understand the presence of such structures for instance a single deoxyribonucleic acid **(**DNA) strand, the cuticle on scales of Morpho butterflies' wings, layers of peacock plumes and thickness of soap bubbles.

A long time before Professor Norio Taniguchi concoct the term nanotechnology in 1974, Nobel physicist Richard Feynman, which is later known as the father of nanotechnology, triggers the possibility of nanoscience in 1959 from a discussion entitled "There's Plenty of Room at the Bottom" at California Institute of Technology (Toumey, 2008). Unfortunately, the nanoparticles can only be seen in 1981 when scanning tunneling microscope has begun being created.

Materials that have at least one nanoscale measurement of approximately 1 nm to 100 nm are called nanomaterials (Sekhon, 2014). Currently, numerous researchers are finding the most ideal approaches to make nanoscale materials and actualizing the materials in instruments to exploit their novel upgraded optical, chemical, mechanical and electrical features such as higher strength, lighter weight, better control of light spectrum and greater chemical reactivity. The explanations behind the marvel are the small size, huge surface area to volume ratio and the mechanical impacts of nanomaterials (Yahya et al., 2009).

Some examples of nanoscience advancements that effect human lives in an everyday schedule are nanoscale magnetic strips in computer hard drives that store data, carbon nanotubes in the circuit that guarantees faster, smaller and longer-enduring batteries computers, nanofibers in textures improve recolor obstruction, water and fire resistance, sunscreens that are produced using nanoparticles are effectively spread over the skin and adequately ingest risky ultraviolet range of light. The uniqueness of nanomaterials and the huge developments of nanoscience spark the enthusiasm of considering nanoparticles in this research.

1.2 Metal oxides semiconductor

Over the last decades, the synthesization and investigation of inorganic metal oxide semiconductors has raised consideration enormously. Among various oxides, numerous studies claim that wide band gap semiconductors ZnO and TiO_2 are the two greatest technologically significant materials because of their extraordinary electronic, chemical and optical properties (Shaheen et al., 2013) which narrow down this research to the two semiconductors.

1.2.1 ZnO metal semiconductor

ZnO, an astounding *n*-type semiconductor, is a II-VI transparent conductive metal oxide semiconductor with a wide direct band gap E_g ~3.37 eV which is reasonable for retention of ultraviolet (UV) light (Xu et al., 2010). Up until now, different ZnO nanostructure, for example, nanodots, nanorods, nanobelts and nanotubes have been synthesized by utilizing various techniques such as solvothermal, precipitation, sol-gel, hydrothermal, self-combustion and laser ablation method. ZnO can produce laser radiation at room temperature and even above room temperature because of the semiconductor large exciton binding energy ~60 meV (Özgür et al., 2005). Due to ZnO referenced intriguing properties, the metal oxide has potential applications in short wavelength optoelectronics (Bae et al., 2003), powerful photocatalysts, degradation of organic compounds (Wang et al., 2007), antibacterial agent (Padmavathy et al., 2008) and biological cytotoxic (Hanley, 2009).

1.2.2 TiO_2 metal semiconductor

TiO_2 is a IV-VI transparent conductive oxide (TCO) semiconductor with a wide band gap of ~3.2 eV. TCO are n-type materials with wide band gap and have high carrier concentrations once being doped (Ellmer et al., 2018). TiO_2 exists in three polymorphs which are rutile (α-TiO_2), anatase and brookite (Wang et al., 2016). Among them, anatase regularly shows the best photoactivity contrasted with the other two sorts of TiO_2 (Liu et al., 2010). To date, different structures of TiO_2 have been manufactured, for example, wires, rods, tubes, flakes, spheres and porous structures through various methods for instance solvothermal, sol-gel, microwave, hydrothermal and sonochemical technique.

Past research have revealed that TiO_2 is the most appropriate material for the degradation of organic compounds contrasted with other various semiconductors in light of the fact that TiO_2 is relatively inexpensive, nontoxic, chemically and thermally stable under various conditions, environmentally friendly and has high photocatalytic activity (Liqiang et al., 2004; Braun, 1991). Likewise, TiO_2 has additionally been uncovered as one of the most dependable material for many applications such as optoelectronic and photoelectrochemical devices (Yu et al., 2014; Crossland et al., 2013), air and water decontamination (Machado et al., 2006), water splitting through hydrogen evolution (Swierk et al., 2016), energy conversion in solar cell (Campbell et al., 2004) and cancer treatment (Cai et al., 1992).

1.3 Problem statement

As the above discussion attests, there have been a significant number of studies on the individual ZnO and TiO_2 nanoparticles. To date, the coupling of titania, TiO_2 and zinc oxide, ZnO semiconductors has been seriously utilized as a photocatalyst for the degradation of organic pollutants (Hussein et al., 2013; Wang et al., 2013). The extraordinary impact of ZnO-TiO_2 nanoparticles system to the degradation is due to the improvement of photocatalytic efficiency that is caused by a couple of reasons including the small particle size, the lower band gap energy, the low electron-hole recombination rate and the presence of more surface OH groups than that of the pure TiO_2 and ZnO photocatalysts. Unfortunately, numerous past investigations produce ZnO-TiO_2 in the form of core-shell as well as thin film and constrained research focus around creating high purity ZnO-TiO_2 powder form semiconductor nanoparticles.

Out all of the methods that are being mentioned in chapter two, none of the research use thermal treatment method to produce pure ZnO-TiO_2 nanoparticles in powder form even though Al-Hada et al. (2014) and Keiteb et al. (2016) have successfully synthesized ZnO and TiO_2 nanoparticles respectively by using the same method. The success of the two researchers leads to the application of thermal treatment route to produce $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles in this work.

1.4 Objectives of study

The general purpose of this research is to produce ZnO-TiO_2 nanoparticles with enhanced structural and optical properties relative to the bulk materials by using the most efficient method. Meanwhile, the specific objectives of this work are:

- To synthesize $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles with molar ratio x = 0.25, 0.50 and 0.75 by using thermal treatment method.
- To study the structural and morphological properties of $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles at different calcination temperatures.
- To investigate the optical properties of $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles at different calcination temperatures.

1.5 Scope of study

In order to achieve the objectives of the study, the scope of this thesis are stated below.

- The preparation of ZnO-TiO_2 nanoparticles was done by using thermal treatment method based on the equation of $(ZnO)_x(TiO_2)_{1-x}$ with molar ratio x = 0.25, 0.50 and 0.75.
- The structure of the samples was analyzed by using XRD to confirm the amorphous as well as the crystalline nature in the samples and FTIR was utilized to study the chemical bonding in the studied nanoparticles.
- The nanoparticles structure of the samples was proven by using TEM and the particle size can be determined from the analyzed data.
- The optical properties such as optical band gap and Urbach energy were investigated by using UV-Vis spectrophotometer.
- The effect of thermal treatment on the structural, morphological and optical properties of the nanoparticles was studied by calcining the samples at different temperature of 500, 600, 700 and 800 °C.

1.6 Hypotheses

Based on the three study objectives, the hypotheses for this study are;

- Thermal treatment method should produce nanoparticles sample with crystalline nature that can be verified through the present of sharp peaks in the XRD spectra.
- FTIR is expected to detect the existence of Zn-O, Ti-O and Zn-O-Ti bonding in the samples to prove the formation of ZnO, TiO_2, $ZnTiO_3$ and Zn_2TiO_4 compounds as well as the total removal of PVP after the calcination treatment.
- The agglomeration of the nanoparticles after calcination treatment is predicted to be observed in the FESEM and TEM images along with the increment of the crystallite and particle size as the temperature rises.
- $(ZnO)_x(TiO_2)_{1-x}$ nanoparticles being produced are anticipated to have better optical properties relative to individual ZnO and TiO_2 nanoparticles by producing multiple band gaps and changing the Urbach energy of the samples.

1.7 Thesis outline

The thesis starts with an introduction in chapter one which interweaves the background of the study, the problem statement, the study objectives, the research scopes and the hypotheses. The thesis continues with chapter two which generally is the narrative review of reported findings that are related to the previous methods in producing $ZnO-TiO_2$ nanoparticles and also the current thermal treatment method. Then, chapter three discusses the related theories that can help to understand more into the conceptual parts of the work. Next, chapter four explains the methodology of the research that includes the list of materials being used, the step by step process in preparing the samples and the detailed process of the samples characterizations. After that, chapter five presents the results of the characterizations and explains the reasons behind the findings. The thesis ends with chapter six which concludes the work with the potential applications of the samples and suggestions for further research of this work.

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