UNIVERSITI PUTRA MALAYSIA

SYNTHESIS, STRUCTURAL AND OPTICAL CHARACTERIZATION OF CuO, CeO2 AND (CuO)x(CeO2)1-x NANOPARTICLES VIA THERMAL TREATMENT METHOD

ANWAR ALI BAQER

ITMA 2018 5

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By

ANWAR ALI BAQER

Thesis Submitted to the School of Graduate Studies, Universiti of Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

December 2017

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DEDICATION

Finally, a thesis for;

My father to whom I am much indebted and my loving mother, helpful sisters and brothers.

My helpful supervisors, Associate Professor Dr. Khamirul Amin Matori for his patience, guidance and encouragement that helped in fostering and carrying out this research.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Doctor of Philosophy

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December 2017

Chairman : Associate Professor Khamirul Amin Matori, PhD
Institute : Advanced Technology

Metal oxide semiconductor nanocrystals are regarded as one of the most important inorganic nanomaterials because of their electronic, optical, electrical and magnetic, properties. These properties are dependent on the chemical composition and microstructural characteristics in which the particle size and shape might be controlled in the fabrication processes. Amongst all metal oxide nanoparticles (NPs), copper oxide (CuO), cerium oxide (CeO_2) and $(CuO)_x(CeO_2)_{1-x}$ NPs have intriguing properties for the development of novel electronic devices, solar cell, sensor, catalyst and medical applications due to their excellent optical and electronic properties. Therefore, further study is needed to synthesize by other methods and characterize these properties.

CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs were successfully synthesized by thermal treatment method.The XRD diffraction patterns reveal monoclinic structure for CuO NPs and cubic fluorite structure for CeO_2 NPs. With no other impurities can be detected, indicating the high purity of the final products. The crystallite size was found to increase from 12.64-25.76, 8.71-22.74 and 5.12-15.34 nm for CuO and 6.45-22.18, 7.25-18.76 and 6.15-11.43 nm for CeO_2 with evolution in calcination temperatures 500-800 ^oC at a concentration of PVP 0.03, 0.04 and 0.05 g/ml respectively. These results were in agreement with the transition electron microscopy results which showed the formation of CuO and CeO_2 in nanoscale size. The average particle size estimated by TEM was found to increase from15.53 to 30.00 nm, 9.75 to 23.54 nm and 4.25 to 16.93 nm for CuO and 5.15 to 24.19 nm, 4.32 to 20.24 nm and 3.00 to 10.62 nm for CeO_2 with increase in calcination temperature 500-800 ^oC at a concentration of PVP 0.03, 0.04 and 0.05 g/ml respectively. The FTIR results confirmed the removal of polymer and the presence of metal oxides nanoparticles at

calcination temperatures 500-800 ^{o}C. The elemental composition of the samples obtained by EDX spectroscopy has further evidenced the formation highly pure CuO and CeO_2 NPs. Furthermore, the optical band gap of the samples was calculated using Kubelka-Munk function for calcination temperatures 500-800 ^{o}C. The band gap was found to decrease from 2.56 to 2.34 eV, 2.75 to 2.42 eV and 2.78 to 2.46 eV for CuO and 3.37 to 3.31eV, 3.38 to 3.32 eV and 3.45 to 3.41 eV for CeO_2 at a concentration of PVP 0.03, 0.04 and 0.05 g/ml respectively. A reduction in the energy band gap with increasing calcination temperatures is attributed to the increase in the particle size. The PL spectra at calcination temperatures 500-800 ^{o}C showed that the increment in the intensity with increasing calcination temperatures is attributed to the expansion in the particle size. Due to the control over particle sizes of CuO and CeO_2 that this technique allows by the varying of PVP concentration and calcination temperature, semiconductor materials with wide band gaps can be produced. These materials are able to absorb UV–visible wavelengths of solar energy, making them suitable for use within solar cell applications. Furthermore, CeO_2 materials produced by this method may be acceptable for use in manufacturing UV filters, catalysts and photoelectric devices.

From the XRD diffraction patterns results, the prepared $(CuO)_x(CeO_2)_{1-x}$ NPs at different calcination temperatures range from 500-800 ^{o}C showed that the crystallite size was increased in the range of 11.25-34.17 nm for $(CuO)_{0.6}(CeO_2)_{0.4}$ with monoclinic and cubic fluorite structures together with no other impurities can be detected, indicating the high purity of the final products. These results were in agreement with the transition electron microscopy results which showed the formation of $(CuO)_x(CeO_2)_{1-x}$ in nanoscale size. The average particle size determined by TEM was found to increase 11.96-31.83 nm for $(CuO)_{0.8}(CeO_2)_{0.2}$ and 2.97-10.70 nm for $(CuO)_{0.2}(CeO_2)_{0.8}$ with increase in calcination temperature 500-800 ^{o}C respectively. At the lower concentration of CuO and with calcination temperature, the particle size smaller and consistent for binary $(CuO)_x(CeO_2)_{1-x}$. The FTIR results confirmed the removal of polymer and the presence of metal oxide nanoparticles at calcination temperatures 500-800 ^{o}C. The elemental composition of the samples obtained by EDX spectroscopy has further evidenced the formation of $(CuO)_x(CeO_2)_{1-x}$ nanoparticles. In addition, the optical band gap of the samples was calculated using Kubelka-Munk function for calcination temperatures 500-800 ^{o}C. The band gap was found to decrease from in the range of 2.82, 3.22 to 2.72, 3.13 eV for $(CuO)_{0.8}(CeO_2)_{0.2}$ and 2.90, 3.30 to 2.83, 3.24 eV for $(CuO)_{0.2}(CeO_2)_{0.8}$. A decrease in the energy band gap with increasing calcination temperatures is attributed to the increase in the particle size. The PL spectra at calcination temperatures 500-800 ^{o}C showed that the increment in the intensity with increasing calcination temperatures is attributed to the increase in the particle size. Due to the control over $(CuO)_x(CeO_2)_{1-x}$ particle sizes that this technique allows by the varying of PVP concentration and calcination temperature, semiconductor materials with multiple band gaps can be produced. These materials are able to absorb specific wavelengths of solar energy, making them very suitable for use within solar cell and sensor applications.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENCIRIAN SINTESIS, STRUKTUR DAN OPTIK PARTIKEL NANO CuO, CeO_2 DAN $(CuO)_x(CeO_2)_{1-x}$ MELALUI KAEDAH RAWATAN TERMAL

Oleh

ANWAR ALI BAQER

Disember 2017

Pengerusi : Profesor Madya Khamirul Amin Matori, PhD
Institut : Teknologi Maju

Hablur nano semikonduktor oksida logam dianggap sebagai salah satu bahan nano tak organik yang paling penting kerana ciri-ciri elektronik, optik, elektrik dan magnetik mereka. Ciri-ciri ini bergantung kepada komposisi kimia dan ciri-ciri mikrostruktur di mana saiz dan bentuk zarah mungkin boleh dikawal dalam proses fabrikasi. Antara bahan zarah nano (NPs) oksida logam, oksida tembaga (CuO), cerium oxide (CeO_2) dan $(CuO)_x(CeO_2)_{1-x}$ NPs mempunyai ciri-ciri menarik untuk pembangunan peranti elektronik novel, sel suria, sensor, pemangkin dan aplikasi perubatan disebabkan oleh sifat optik dan elektronik mereka yang sangat baik. Oleh itu, kajian lanjut diperlukan untuk mensintesiskan menggunakan kaedah lain dan mencirikan sifat-sifat ini.

CuO, CeO_2 dan dedua $(CuO)_x(CeO_2)_{1-x}$ NPs telah berjaya disintesis oleh kaedah rawatan haba. Corak pembelauan XRD mendedahkan struktur monoklinik untuk CuO NPs dan struktur fluorit padu untuk CeO_2 NPs. Tanpa sebarang bendasing lain dapat dikesan, menunjukkan ketulenan tinggi bagi produk akhir. Saiz kristal didapati meningkat dari 12.64-25.76, 8.71-22.74 dan 5.12-15.34 nm untuk CuO dan 6.45-22.18, 7.25-18.76 dan 6.15-11.43 nm untuk CeO_2 dengan evolusi dalam suhu kalsinasi 500-800 ºC masing-masing pada kepekatan PVP 0.03, 0.04 dan 0.05 g/ml. Keputusan ini sepadan dengan hasil mikroskopi elektron peralihan yang menunjukkan pembentukan CuO dan CeO_2 dalam skala nano saiz. Saiz zarah purata yang dianggarkan oleh TEM dijumpai meningkat dari 15.53 ke 30.00 nm, 9.75 ke 23.54 nm dan 4.25 ke 16.93 nm untuk CuO dan 5.15 ke 24.19 nm, 4.32 ke 20.24 nm dan 3.00 ke 10.62 nm untuk CeO_2 dengan peningkatan suhu kalsinasi 500-800 ºC masing-masing pada kepekatan PVP 0.03, 0.04 dan 0.05 g/ml. Hasil FTIR mengesahkan penyingkiran polimer dan kehadiran zarah nano oksida logam pada suhu kalsinasi 500-800 ºC. Komposisi unsur sampel yang diperolehi oleh

spektroskopi EDX telah membuktikan pembentukan CuO dan CeO_2 NPs yang sangat tulen. Selain itu, jurang jalur optik sampel dikira menggunakan fungsi Kubelka-Munk untuk suhu kalsinasi 500-800 ºC. Jurang jalur didapati menurun dari 2.56 ke 2.34 eV, 2.75 ke 2.42 eV dan 2.78 ke 2.46 eV untuk CuO dan 3.37 ke 3.31eV, 3.38 ke 3.32 eV dan 3.45 ke 3.41 eV untuk CeO_2 masing-masing pada kepekatan PVP 0.03, 0.04 dan 0.05 g/ml. Pengurangan dalam jurang jalur tenaga dengan peningkatan suhu kalsinasi adalah disebabkan peningkatan saiz zarah. Spektrum PL pada suhu kalsinasi 500-800 ºC menunjukkan bahawa kenaikan dalam keamatan dengan peningkatan suhu kalsinasi adalah disebabkan oleh peningkatan saiz zarah. Dengan kebolehan pengawalan ke atas saiz zarah CuO dan CeO_2 menggunakan teknik ini dengan mengubah kepekatan PVP dan suhu kalsinasi, bahan semikonduktor dengan jurang lebar boleh dihasilkan. Bahan-bahan ini dapat menyerap panjang gelombang UV tenaga solar, menjadikannya sesuai digunakan dalam aplikasi sel solar. Selain itu, bahan CeO_2 yang dihasilkan menggunakan kaedah ini boleh diterima untuk digunakan dalam pembuatan penapis UV, pemangkin dan peranti fotografi.

Dari hasil corak pembelauan XRD, $(CuO)_x(CeO_2)_{1-x}$ NPs yang dihasilkan pada suhu kalsinasi berbeza dari 500-800 ºC menunjukkan bahawa saiz hablur meningkat dalam lingkungan 11.25-34.17 nm untuk $(CuO)_{0.6}(CeO_2)_{0.4}$ dengan struktur fluorit monoklinik dan kubik hadir bersama-sama dengan tiada bendasing lain dapat dikesan, menunjukkan ketulinan tinggi produk akhir. Keputusan ini sepadan dengan hasil mikroskopi elektron peralihan yang menunjukkan pembentukan $(CuO)_x(CeO_2)_{1-x}$ dalam saiz nano. Saiz zarah purata yang ditentukan oleh TEM didapati meningkat dari 11.96-31.83 nm untuk $(CuO)_{0.8}(CeO_2)_{0.2}$ dan 2.97-10.70 nm untuk $(CuO)_{0.2}(CeO_2)_{0.8}$ dengan peningkatan suhu kalsinasi masing-masing dari 500-800 ºC. Pada kepekatan CuO dan suhu kalsinasi yang rendah, hampir kesemua saiz zarah mengecil dan konsisten bagi binari $(CuO)_x(CeO_2)_{1-x}$. Hasil FTIR mengesahkan penyingkiran polimer dan kehadiran nanopartikel oksida logam pada suhu kalsinasi 500-800 ºC. Komposisi elemen sampel yang diperolehi dari spektroskopi EDX telah membuktikan pembentukan nanopartikel $(CuO)_x(CeO_2)_{1-x}$. Di samping itu, jurang jalur optik sampel dikira menggunakan fungsi Kubelka-Munk untuk suhu kalsinasi 500-800 ºC. Jurang jalur didapati berkurang dari julat 2.82, 3.22 ke 2.72, 3.13 eV bagi $(CuO)_{0.8}(CeO_2)_{0.2}$ dan 2.90, 3.30 ke 2.83, 3.24 eV bagi $(CuO)_{0.2}(CeO_2)_{0.8}$. Penurunan dalam jurang jalur tenaga dengan peningkatan suhu kalsinasi adalah disebabkan peningkatan saiz zarah. Spektrum PL pada suhu kalsinasi 500-800 ºC menunjukkan peningkatan intensiti dengan peningkatan suhu kalsinasi adalah disebabkan oleh peningkatan saiz zarah. Disebabkan oleh pengawalan saiz zarah $(CuO)_x(CeO_2)_{1-x}$ yang diperolehi dari teknik disebabkan oleh penggunaan pelbagai kepekatan PVP dan suhu kalsinasi, bahan-bahan semikonduktor dengan pelbagai jurang jalur boleh dihasilkan. Bahan-bahan ini dapat menyerap panjang gelombang khusus tenaga solar, menjadikannya sangat sesuai digunakan dalam sel solar dan aplikasi sensor.

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I certify that a Thesis Examination Committee has met on 7 December 2017 to conduct the final examination of Anwar Ali Baqer on his thesis entitled "Synthesis, Structural and Optical Characterization of CuO, CeO_2 and $(CuO)_x(CeO_2)_{1-x}$ Nanoparticles via Thermal Treatment Method" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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I hereby confirm that:

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This is to confirm that:

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LIST OF ABBREVIATIONS

A	Absorbance
CCP	Cubic close-packing
CeO_2	Cerium oxide
CuO	Copper oxide
D	Distance
E_g	Optical band gap
eV	Electron volte
FESEM	Field emission scanning electron microscopy
FTIR	Fourier transforms infrared spectroscopy
FWHM	Full-width at half-maximum
h	Hour
KM	Kubelka-Munk
min	Minutes
nm	Nanometer
NPs	Nanoparticles
PL	Photoluminescence spectroscopy
PVP	Poly (vinyl pyrrolidone)
R.T.	Room temperature
T	Transmittance
TEM	Transmission electron microscopy
Temp.	Temperature
TGA	Thermo gravimetric analysis
UV-Vis	Ultraviolet-Visible absorption spectroscopy

XRD	X-ray diffraction
θ	Bragg angle
^{o}C	Degree Celsius
a	Lattice parameter
λ	Wavelength
Å	Unit of length
λ_{max}	maximum absorbance wavelength

CHAPTER 1

INTRODUCTION

1.1 Background of the study

The field of nanotechnology and nanoscience has recently received a growing interest in the literature. The notion of "nanotechnology" was first introduced by Richard Feynman in 1959, in his well-known Caltech lecture titled "there is plenty of room at the bottom: an invitation to enter new field of physics" and made notes on the consequences of measuring and manipulating materials at the nanoscale level. In 1974, Nario Tanigushi coined the term "nanotechnology" and suggested using the name to indicate all the processes that occur in the materials of less than 1 micrometer in size. The prefix "nano" means one-billionth part of something. In general, a nanostructure refers to objects whose size range from individual atoms to a large clusters or molecules (Bardosova and Wagner 2015). The transition of material structure from macroscale to nanoscale results in a dramatic change in the physical, chemical, electrical and optical properties of the new materials. The main reason beyond this change is thought to be due to an increase in the proportion of surface atoms and surface volume ratio, as the size of the particle further decreases.

More importantly, the quantum size effect comes into play when the dimension of the particle is reduced. The carriers (electron-hole) pairs in semiconductor particles are said to be in potential wells defined by the conduction and valence bands of the solid. The quantization effect simply arises from the confinement of charge carriers in semiconductors with potential wells of small dimensions. Depending on the dimensionality of the quantum system, these structures can exist as quantum dots (0-D), quantum wires (1-D) or quantum wells (2-D) (Martienssen and Warlimont, 2005). The relationship between surface area and particle size is inversely proportional with one another (See Figure 1.1).

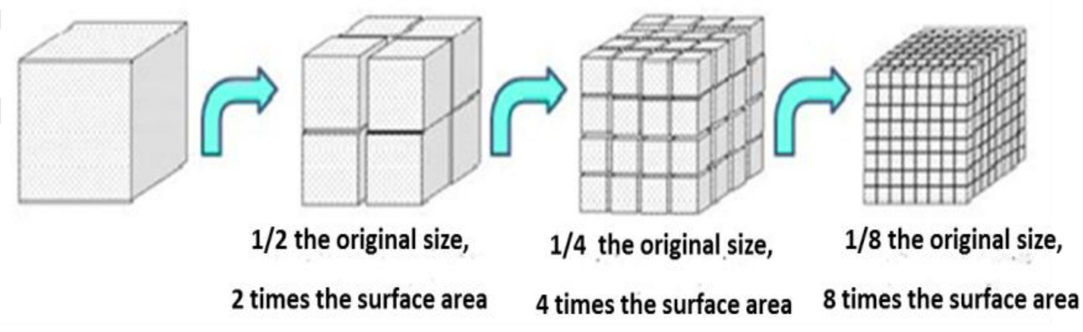

Figure 1.1 : Relationship between particle size and surface area (Meşin, 2012)

In confined electronic systems, the size of the particle is comparable to de Broglie wavelength or less; thus, the carrier confinement creates discrete levels in the conduction and valence bands (Grimes et al., 2008). Therfore, the band gap energy and the separation between available states for an excited electron in a nanocrystal become noticeably bigger with diminishing size. This is another particular nanosize impact, wherein, under a specific material based critical size, the electrons in nanocrystal becomes ‘quantum confined’ leading to new size-dependent interactions of the valence electrons to certain energies of excitation (especially photons and electric field) (Archana, 2011).

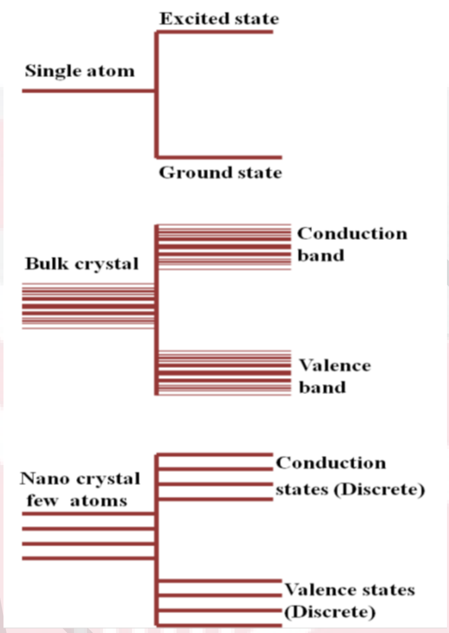

Figure 1.2 : A schematic representation of excited states available to valance electrons (Archana, 2011)

The development of nanotechnology has aroused a renewed in nanocrystalline materials, which have the ability to improve the properties of the material by controlling the microstructure (Maensiri et al., 2007). Metal oxide nanoparticles and binary oxide nanoparticles are of great interest because of their extensive applications ranging from basic research to applications. For instance, the metal oxide nanoparticles are used in cosmetics, structural materials, biomaterials, paint pigments, electronics, membranes and filters, medical diagnostics, pharmaceuticals, catalysts and supports, flat panel displays, batteries and fuel cells, magnetic and optical devices and in protective coatings (Holmberg, 2001).

1.2 Problem statement

Nanoscale metal oxide materials have attracted a lot of researchers attention because of their unique physical and chemical properties depending on their size and dimensionality, as well as their promising applications as key components of micro/nanometric devices. This group has profound applications in areas such as nanophotonics, gas sensor, conversion catalysis and biomedical applications (Mirzaei and Darroudi, 2017; Tyagi et al., 2016). Copper oxide (CuO) and cerium oxide (CeO_2) NPs are considered crucial elements of metal oxide semiconductors due to their p-type and n-type conductivity with band gaps 1.5 eV and 3.2 eV, respectively (Tuller and Nowick, 1979; Mohamed et al., 2014). This renders these materials more appropriate for current technologies. CuO and CeO_2 have potential applications in catalysts (Cao et al., 2008; Lin et al., 2012), gas sensors (Li et al., 2008; Umar et al., 2015), and solar cells (Lira-Cantu and Krebs, 2006; Kidowaki et al., 2011). CuO and CeO_2 NPs have monoclinic and cubic fluorite phases, respectively.

Furthermore, binary CuO-CeO_2 NPs semiconductors which consist of two types of materials can usually improve certain functions when compared with their individual components such as catalysis (Chen et al., 2015), water–gas shifts (Li et al., 2007) and solid oxide fuel cell (SOFC) (Ye et al., 2009).

In order to obtain materials with the desired physical and chemical properties, the preparation of the CuO and CeO_2 and binary (CuO)(CeO_2) NPs through different methods has become an essential axis of the related research and development activities, namely CuO and CeO_2 NPs such as sol–gel method (Xiao et al., 2009; Mallick and Sahu, 2012), micro emulsion method (Nagy and Dékány, 2009; Zhang et al., 2013), precipitation method (Chung and Yeh, 2008; Phiwdang et al., 2013; Heidari and Irankhah, 2014), thermal decomposition (Zhang et al., 2008c; Gabal et al., 2012), hydrothermal method (Lu et al., 2009; Zeng et al., 2012; Sonia et al., 2015), and solvothermal method (Song et al., 2010; Yu et al., 2012b; Xu et al., 2016). Most of these methods could be used to synthesize nanoparticles of the desired shape and size, but, they are difficult to use on powder form prepared, high purity, a large scale because of their expensive and complex procedures, high reaction temperatures and an addition of toxic reagents which could harm the environment. The thermal treatment method could be deemed as one of the best approaches of forming nanoparticles because it is cheap, fast synthesized pure nanoparticles and also led to an improved characterization of the metal oxide nanoparticles.

Although, many methods could produce copper oxide (CuO) and cerium oxide (CeO_2) nanoparticles separately with small size but to the best of our knowledge until now, none could produce pure binary $(CuO)_x(CeO_2)_{1-x}$nanoparticles in powder form with small size and good distribution of particles. In this method thermal treatment could produce highly pure binary $(CuO)_x(CeO_2)_{1-x}$ nanoparticles in powder form with small particle, narrow size distribution and uniform morphology.

1.3 Significance of study

The metal oxide semiconductors nanoparticles are appealing subjects of constant scientific interest. They have been intensively investigated in the field of materials science, due to their physical-chemical characteristics and their broad range of applications. More specifically, CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs semiconductors are usually used as catalytic substances, ultraviolet (UV) filter and absorbents, fuel cells, solar cells, opto-electronic devices, super capacitors, field-emission emitters, sensors, energy storage, electrolyte, biomedical science and several other applications.

In the present study, CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs were synthesized from an aqueous mixture containing deionised water, metal nitrates and poly (vinyl pyrrolidon), using a low temperature thermal treatment process. Further processes of grinding and calcinations were applied. No other materials were added to the mixture. This process is eco-friendly in that it neither utilises nor does it create toxic materials, and it offers the benefits of low cost, simplicity, and low reaction temperatures.

1.4 Scope of study

The present research work is limited to the synthesis of CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ Nps semiconductors by employing PVP as capping agent, and metal nitrate as precursors through the thermal treatment method. In addition, the study includes structural, morphological and optical characterization of the as-synthesized nanoparticles.

1.5 Hypothesis

The purity of metal oxide nanoparticles produced by thermal treatment method is depended on the interaction between Cu or Ce metallic ions and capping agent (polyvinylpyrrolidone) that undergo to the calcination temperature and causes the growth of the nanoparticles.In this method, the effect of PVP is to establish a distance between the metal atoms, so the agglomeration of oxide particles could be diminished depending upon the PVP concentration. Higher concentration of PVP leads to more capping of metal atoms from each other and manufactured smaller particle size after calcination.

1.6 Objectives of study

The general aim of the present study is to use the thermal treatment method to synthesize CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs with the aid of PVP acting as a capping agent. It is expected that the nanomaterials synthesized via this technique will improved the physical and chemical properties of the nanoparticles. The specific objectives are stated as follows:

1. To synthesize high purity CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs via thermal-treatment method.
2. To investigate the effect of calcination temperature on the structural, morphological and optical properties of CuO and CeO_2 NPs.
3. To study the influence of different PVP concentration on the structural, morphological and optical properties of CuO and CeO_2 NPs.
4. To examine the impact of different concentrations of precursor (metal nitrate) and calcination temperatures on the structural, morphological and optical properties of binary $(CuO)_x(CeO_2)_{1-x}$ NPs semiconductor.

1.7 Thesis outline

This thesis consists of six chapters. Chapter 1 begins with a general introduction of nanotechnology and nanoscience with physical properties of CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs metal oxides semiconductor. It also discusses the problem statement, research questions and objectives of the study. Chapter 2 provides a comprehensive review of the array of various preparatory techniques used to synthesize CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs semiconductor with their technological applications. Chapter 3 provides a theoretical background of the present study. This includes the structural, optical, luminescence properties of the as-prepared nanomaterials. Chapter 4 presents the synthesis and characterization of the prepared nanomaterials by thermal treatment method. Chapter 5 presents the results and discussion on the various characterizations of CuO, CeO_2 and binary $(CuO)_x(CeO_2)_{1-x}$ NPs Chapter 6 summarizes the study and suggests recommendations for future work.

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