UNIVERSITI PUTRA MALAYSIA

SYNTHESIS, CHARACTERIZATION AND EFFECTS OF THERMAL TREATMENT OF ZnO-AND CdO-BASED NANOMATERIALS

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By

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

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Chairman: Professor Elias Saion, PhD

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Nanoscience can simply be defined as the study and understanding of nanomaterials and their manipulation at atomic, molecular and macromolecular scales where properties vary significantly from those at a macroscopic scale. Nanotechnology on the other hand can be defined as the design, production and application of nanostructured devices and systems by controlling shape and size at a nanometer scale. Nanomaterials could be defined as the materials with at least one of its dimensions in the range of a nanometer. The study of nanomaterials is very interesting and important because at nanoscale, materials have fundamentally unique properties compared to their bulk due to increased surface area to volume ratios. The metallic compounds which formed with metal and oxygen in the form of oxide ion (O^{2-}) are called metal oxide." They a named in two words where first word is the name of metal with oxidation number in parenthesis followed by oxide.

Nanomaterials including metal oxide nanoparticles are of scientific and technological importance due to their unique physical and chemical properties arise from their nanoscale dimension and large number of surface atoms. As their properties are dependent on large surface area to volume ratio and quantum confinement effect, they have potential applications in almost every field of human endeavor. PVP displays capping ability (capping agent) which plays significant role in the synthesis of metal oxide nanoparticles. It is however realized that PVP controls the growth of the nanoparticles with the variation of its concentration, prevents the agglomeration, improves the crystallinity and brings about homogeneity and uniformity in the shape of nanoparticles.

From the prepared ZnO results, the XRD diffraction patterns at calcination temperatures 500-650 ºC showed that the crystallite size was in the range of 18–41 nm with hexagonal structure. These results were in agreement with the transition electron microscopy results which showed that the formation of ZnO in nanoscale size. The average particle size determined by TEM images were found to increase

from 19 to 43 nm with increase in calcination temperatures. The FTIR results confirmed the removal of polymer and the presence of metal oxide nanoparticles at calcination temperatures 500-650 ^{o}C. The elemental composition of the samples obtained by EDX spectroscopy has further evidenced the formation of ZnO nanoparticles. In addition, the optical band gap of the samples was calculated using Kubelka-Munk model for calcination temperatures 500-650 ^{o}C. The band gap varied from 3.27 to 3.23 eV for calcination temperatures 500-650 ^{o}C. A decrease in the energy band gap with increasing calcination temperatures is attributed to the increase in the particle size. It is believed that as the particle size increases, the number of atoms that form a particle also increase, which consequently render the valence and conduction electrons more attractive to the ions core of the particles, and hence decreasing the band gap of the particles. The PL spectra at calcination temperatures 500-650 ^{o}C showed that the increment in the intensity with increasing calcination temperatures is attributed to the increase in the particle size.

From the prepared CdO results, the XRD diffraction patterns at calcination temperatures 500-650 ^{o}C showed that the crystallite size was in the range of 13–47 nm with cubic center face structure. These results were in agreement with the transition electron microscopy results which showed the formation of CdO in nanoscale size. The average particle size determined by TEM was found to increase from 18 to 48 nm with increase in calcination temperature. The FTIR results confirmed the removal of polymer and the presence of metal oxide nanoparticles at calcination temperatures 500-650 ^{o}C. The elemental composition of the samples obtained by EDX spectroscopy has further evidenced the formation of CdO nanoparticles. In addition, the optical band gap of the samples was calculated using Kubelka-Munk model for calcination temperatures 500-650 ^{o}C. The band gap was found to vary from 2.14 to 2.01 eV. 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 500-650 ^{o}C showed that the increment in the intensity with increasing calcination temperatures is attributed to the increase in the particle size.

From the prepared $(ZnO)_x(CdO)_{1-x}$ nanosheets results, the XRD diffraction patterns at calcination temperatures 500-650 ^{o}C showed that the crystallite size was in the range of 15-25 nm for $(ZnO)_{0.2}(CdO)_{0.8}$ and 13-32 nm for $ZnO)_{0.8}(CdO)_{0.2}$ with hexagonal and cubic structures respectively. The average particle size determined by TEM were found to increase with calcination temperatures from 14-26 nm for $(ZnO)_{0.2}(CdO)_{0.8}$ and 16-40 nm for $ZnO)_{0.8}(CdO)_{0.2}$. The FTIR results confirmed the removal of polymer and the presence of metal oxide nanoparticles at calcination temperatures 500-650 ^{o}C. The elemental composition of the samples obtained by EDX spectroscopy has further evidenced the formation of $(ZnO)_x(CdO)_{1-x}$ nanosheets In addition, the optical band gap of the samples was calculated using Kubelka-Munk model for calcination temperatures 500-650 ^{o}C. The band gap varied from 2.83-3.22 to 2.68-3.09 eV for calcination temperatures 500-650 ^{o}C. A decrease in the energy band gap with increasing calcination temperatures is attributed to the increase in the particle size. It is believed that as the particle size increases, the number of atoms that form a particle also increase, which consequently render the valence and conduction electrons more attractive to the ions core of the particles, and hence decreasing the band gap of the particles. The PL spectra at calcination

temperatures 500-650 ºC showed that the increment in the intensity with increasing calcination temperatures is attributed to the increase in the particle size.
A thermogravimetric analyser (TGA) was used to study thermal stability and the temperature at which polymer could be remove from the samples during calcination. The maximum decomposition of the polymer was found at 485 ºC.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi syarat keperluan Ijazah Doktor Falsafah

SINTESIS , PENCIRIAN DAN KESAN RAWATAN HABA DARIPADA ZnO , DAN CdO -BASED BAHAN NANO

Oleh

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Nanosains hanya boleh ditakrifkan sebagai kajian dan pemahaman nanobahan dan manipulasi mereka pada skala atom, molekul dan makromolekul di mana sifat-sifat berbeza dengan ketara daripada mereka yang berada di skala yang makroskopik. Nanoteknologi pula boleh ditakrifkan sebagai reka bentuk, pengeluaran dan penggunaan alat-alat dan sistem bernanostruktur dengan mengawal bentuk dan saiz pada skala nanometer. Bahan Nano boleh ditakrifkan sebagai bahan yang mempunyai sekurang-kurangnya salah satu dimensi dalam julat yang nanometer. Kajian nanobahan adalah sangat menarik dan penting kerana pada skala nano, bahan-bahan mempunyai ciri-ciri asasnya yang unik berbanding dengan sebahagian besar mereka disebabkan oleh peningkatan kawasan permukaan untuk nisbah kelantangan. Sebatian logam yang dibentuk dengan logam dan oksigen dalam bentuk oksida ion (O^{2-}) dipanggil oksida logam . "Mereka yang bernama dalam dua perkataan di mana perkataan pertama adalah nama logam dengan nombor pengoksidaan dalam kurungan diikuti oleh oksida.

Bahan Nano termasuk nanopartikel oksida logam mempunyai kepentingan sains dan teknologi kerana sifat mereka yang unik fizikal dan kimia timbul dari dimensi nano dan nombor atom besar permukaan. Sebagai sifat-sifat mereka adalah bergantung kepada kawasan permukaan yang besar kepada nisbah jumlah dan kesan pantang kuantum, mereka mempunyai aplikasi yang berpotensi dalam hampir setiap bidang endeaver manusia. Memaparkan PVP menghadkan keupayaan (ejen menetapkan siling) yang memainkan peranan penting dalam sintesis nanopartikel oksida logam. Namun ia menyedari bahawa PVP mengawal pertumbuhan nanopartikel dengan pengubahan kepekatannya, menghalang penumpuan, meningkatkan penghabluran dan membawa homogenity dan keseragaman dalam bentuk partikel nano.

Dari disediakan keputusan ZnO, corak belauan XRD pada suhu pengkalsinan 500-650 ºC menunjukkan saiz hablur tersebut adalah dalam lingkungan 18-41 nm dengan struktur heksagon. Keputusan ini adalah selaras dengan keputusan

mikroskopi elektron peralihan yang menunjukkan bahawa pembentukan skala nano oksida saiz logam. Saiz zarah purata ditentukan oleh imej TEM telah didapati untuk meningkatkan 19-43 nm dengan peningkatan suhu pengkalsinan. Keputusan FTIR mengesahkan penyingkiran polimer dan kehadiran partikel nano oksida logam pada suhu pengkalsinan 500-650 ºC. Komposisi unsur sampel diperolehi oleh EDX spektroskopi telah dibuktikan lagi pembentukan partikel nano ZnO. Di samping itu, jurang jalur optik bagi sampel telah dikira menggunakan model Kubelka - Munk untuk suhu pengkalsinan 500-650 ºC. Jurang jalur diubah 3,27-3,23 eV untuk suhu pengkalsinan 500-650 ºC . Penurunan dalam jurang jalur tenaga dengan meningkatkan suhu pengkalsinan adalah disebabkan oleh peningkatan dalam saiz zarah. Adalah dipercayai bahawa peningkatan saiz zarah, bilangan atom yang membentuk zarah yang juga meningkat, yang seterusnya menyebabkan valens dan elektron konduksi lebih menarik kepada teras ion zarah, dan dengan itu mengurangkan jurang jalur zarah. PL spektrum pada suhu pengkalsinan 500-650 ºC menunjukkan bahawa kenaikan dalam keamatan dengan suhu pengkalsinan meningkat adalah disebabkan oleh peningkatan dalam saiz zarah.

Dari disediakan keputusan CdO, corak belauan XRD pada suhu pengkalsinan 500-650 ºC menunjukkan saiz hablur tersebut adalah dalam lingkungan 13-47 nm dengan padu struktur muka pusat. Keputusan ini adalah selaras dengan keputusan mikroskopi elektron peralihan yang menunjukkan pembentukan oksida logam bersaiz nano. Saiz zarah purata ditentukan oleh TEM telah didapati untuk meningkatkan 18-48 nm dengan peningkatan suhu pengkalsinan. Keputusan FTIR mengesahkan penyingkiran polimer dan kehadiran partikel nano oksida logam pada suhu pengkalsinan 500-650 ºC. Komposisi unsur sampel diperolehi oleh EDX spektroskopi telah dibuktikan lagi pembentukan partikel nano CdO. Di samping itu, jurang jalur optik bagi sampel telah dikira menggunakan model Kubelka - Munk untuk suhu pengkalsinan 500-650 ºC. Jurang band didapati berbeza-beza 2,14-2,01 eV . Penurunan dalam jurang jalur tenaga dengan meningkatkan suhu pengkalsinan adalah disebabkan oleh peningkatan dalam saiz zarah. PL spektrum pada suhu pengkalsinan 500-650 ºC menunjukkan bahawa kenaikan dalam keamatan dengan suhu pengkalsinan meningkat adalah disebabkan oleh peningkatan dalam saiz zarah.

Dari disediakan $(ZnO)_{x} (CdO)_{1-x}$ nanosheets keputusan , corak belauan XRD pada suhu pengkalsinan 500-650 ºC menunjukkan saiz hablur tersebut adalah dalam lingkungan 15-25 nm untuk $(ZnO)_{0.2} (CdO)_{0.8}$ dan 13-32 nm untuk $ZnO)_{0.8} (CdO)_{0.2}$ dengan struktur heksagon dan padu. Saiz zarah purata ditentukan oleh TEM telah didapati meningkat dengan suhu pengkalsinan 14-26 nm untuk $(ZnO)_{0.2} (CdO)_{0.8}$ dan 16-40 nm untuk $ZnO)_{0.8} (CdO)_{0.2}$. Keputusan FTIR mengesahkan penyingkiran polimer dan kehadiran partikel nano oksida logam pada suhu pengkalsinan 500-650 ºC. Komposisi unsur sampel diperolehi oleh EDX spektroskopi telah dibuktikan lagi pembentukan $(ZnO)_{x} (CdO)_{1-x}$ nanosheets Di samping itu, jurang jalur optik bagi sampel telah dikira menggunakan model Kubelka - Munk untuk suhu pengkalsinan 500-650 ºC . Jurang jalur diubah 2,83-3,22 2,68-3,09 kepada eV untuk suhu pengkalsinan 500-650 ºC . Penurunan dalam jurang jalur tenaga dengan meningkatkan suhu pengkalsinan adalah disebabkan oleh peningkatan dalam saiz zarah. Adalah dipercayai bahawa peningkatan saiz zarah, bilangan atom yang membentuk zarah yang juga meningkat, yang seterusnya menyebabkan valens dan elektron konduksi lebih menarik kepada teras ion zarah, dan dengan itu mengurangkan jurang jalur zarah. PL spektrum pada suhu pengkalsinan 500-650 °C menunjukkan bahawa kenaikan dalam keamatan dengan suhu pengkalsinan meningkat adalah disebabkan oleh peningkatan dalam saiz zarah.

Seorang penganalisis Termogravimetri (TGA) telah digunakan untuk mengkaji kestabilan haba dan suhu di mana polimer boleh mengalihkan dari sampel semasa proses mengapur. Penguraian maksimum polimer didapati di 485 °C.

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APPROVAL

I certify that a Thesis Examination Committee has met on 12 January 2015 to conduct the final examination of Naif Mohammed Al-Hada thesis entitled " SYNTHESIS, CHARACTERIZATION AND EFFECTS OF THERMAL TREATMENT OF ZNO- AND CDO-BASED NANOMATERIALS" 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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KM	Kubelka-Munk
DI	Deionize water
NPs	Nanoparticles
SEM	Scanning electron microscopy
nm	Nanometer
eV	Electron volte
Θ	Bragg angle
h	Hour
min	Minutes
E_g	Optical band gap
ºC	Degree celsius
λ	Wavelength
d	Distance
λ	Wavelength
T	Transmittance
λν	Energy
β	FWHM
ZnO	Zinc oxide
CdO	Cadmium oxide
UV	Ultraviolet- visible absorption spectroscopy
PL	Photoluminescence
a	Lattice parameter
EDX	Energy dispersive X-Ray
TEM	Transmission electron microscopy
FTIR	Fourier transforms infrared spectroscopy
XRD	X-ray diffraction
TGA	Thermo gravimetric analysis
PVP	Poly (vinyl pyrrolidone)

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Nanoscience has started when Herman Staudinger developed the concept of macromolecules during 1920s and later he received the Nobel Prize in 1953. Nanoparticles have long history of usage in pottery and medicine since ancient days. Historical evidences show that gold nanoparticles were used as drug by Chinese during 2500 BC. Red colloidal gold is still in use under the name of Swarna Bhasma and Makaradhwaja" in traditional medicine system of India called Ayurveda, which dates back to 1st millennium BC (Bhattacharya and Mukherjee, 2008). Recent scientific study of a vessel of Roman period (4th century AD) called "Lycurgus Cup," kept in British Museum London, shows the use of nanoparticles of Gold-Silver alloy for its decoration (Freestone et al., 2007). Similarly, churches of Middle Ages used gold in colloidal state trapped within the matrix of glass to make aesthetically pleasant ruby coloured glasses of different hues and colours (due to the formation of nanoparticle of different sizes). In 16th Century Europe an aqueous form of colloidal gold called "Aurum Potabile (drinkable gold)" was thought to have curative properties for many diseases (Caseri, 2000). In 1857 Michael Faraday described methods for synthesis of stable aqueous dispersions and optical properties of gold nanoparticles (Faraday, 1857). In 1915, in his famous book "The World of Neglected Dimensions", Wolfgang Ostwald recognized colloidal particles as unique state of matter, whose particles "are so small that they can no longer be recognized microscopically, while they are still too large to be called molecules." However the credit of realizing the enormous potential of nanoparticles and their possible implications in different fields is given to Richard P. Feynman. In his classical lecture in 1959 at California Institute of Technology (Caltech) during Annual meeting of the American Physical Society Feynman has stated: "............I would like to describe a field, in which little has been done, but in which an enormous amount can be done in principle. This field is not quite the same as the others in that it will not tell us much of fundamental physics (in the sense of, what are the strange particles?).......

Nanoscience is a multi-discipline field of science that has been drastically expanding since 1980s (Nalwa, 2004). Nanoscience surmount with numerous essential issues, in which many of them having potential technological applications. Putting the nanoscience into applications is described as nanotechnology. The main research areas of nanotechnology include among others physics, chemistry, materials science, biology, medicine, bioengineering, agriculture and the environmental science. Nanoscience involves a variety of submicron size materials, which are described as nanoparticles. Nanoparticles are particles with one or more dimensions at the order of 100 nm or less. It is a critical length scale at which certain novel nanosize acquires different properties compared to its molecules or bulk form. Besides "strictly nano" (1-100 nm) all submicron colloidal particles/mesoscale, i.e. particles with at least one dimension in the scale of 1-1000 nm, are referred to as nanoparticles as well, to include organic polymers and vesicles widely used in the area of drug delivery (Uchegbu et al., 2013; Azarmi et al., 2006; Kreuter, 2007).

Metal oxide semiconductor nanoparticles possess unique morphological, structural, and optical properties at nanoscale. With a decrease in particle size, a remarkable high surface area to volume ratio is inevitable, leading to an even distribution of the particles and increase in surface active sites for chemical reactions to enhance the reaction and absorption efficiency. The enhanced surface area also increases the surface states, which changes the activity of charge carries and affects the chemical reaction dynamics. Moreover the decrease of particle size resulted in quantum size effect because of the confinement of charge carriers especially the electrons. The quantum size effect splits both conduction and valence bands into discrete electronic states which influence the optical and electronic properties of the nanoparticles.

At present, ZnO and CdO semiconductor nanoparticles are regarded as two of the most important inorganic semiconductor nanomaterials because of their n-type conductivity with a wide band gap (3.3 eV and 2.2 eV respectively) which make these materials more suitable for modern technologies. ZnO and CdO have promising applications in catalysts (Elseviers and Verelst, 1999; Abd El-Salaam and Hassan, 1982), gas sensors (Mochinaga et al., 1998, Shchukin et al., 2001), and solar cells (Mane et al., 2006, Gal et al., 2000; Cai et al., 2010). Binary oxide of $(ZnO)_x(CdO)_{1-x}$ nanoparticles have display hexagonal and face-centered-cubic (fcc) strucures respectively (Yousef et al., 2012). The present of binary oxide $(ZnO)_x(CdO)_{1-x}$ semiconductor nanoparticles could improve further their optical performance, excellent chemical stability, and mechanical hardness, which are good contender for optoelectronic, photocatalytic, and solar cell applications.

1.2 Problem Statement

In the past decade, nanoscale research has opened revolutionary opportunities for a wide number of technological applications. Due to their special optical, magnetic, electrical and catalytic properties, and improved physical properties like mechanical hardness, thermal stability or chemical passivity (Feldmann and Jungk, 2001). Metal oxide nanoparticles and binary oxide nanoparticles are attracting significant interest due to their extensive applications, ranging from fundamental research to applications. For example, metal oxide nanostructures are extensively used as paint pigments, cosmetics, pharmaceuticals, medical diagnostics, catalysts and supports, membranes and filters, batteries and fuel cells, electronics, magnetic and optical devices, flat panel displays, biomaterials, structured materials and protective coatings (Holmberg et al., 2002).

Metal oxide nanostructures can be prepared using various methods, such as precipitation, solvothermal, hydrothermal, sol-gel, microemulsion, combustion, electrochemical, sonochemical etc but with some imperfections for example the need for catalyst, oxidizing or reducing agents and longer reaction times, high reaction temperatures, toxic reagents and by-products which are potentially harmful to the environment. It is worth nothing that the application of CdO and ZnO nanoparticles and their optical properties depending on the preparation method used. In order to achieve materials that have the desired physical and chemical properties, the preparation of CdO and ZnO nanoparticles through different routes has become an essential focus of the related research and development activities namely ZnO and CdO nanoparticles such as sol–gel method (Kaur et al., 2006; Zhang et al., 2006; Karami et al., 2010), microemulsion method (Dong and Zhu, 2003; Sarkar et al., 2011), precipitation method, thermal decomposition (Ristić et al., 2004), hydrothermal method (Zhang et al., 2008; Wang and Li, 2006), chemical co-precipitation method (Waghulade et al., 2007), thermal evaporation (Lu et al., 2008), etc. Most of these methods have achieved particles of the required sizes and shapes, but they are difficult to employ on powder form especially in CdO nanoparticles synthesized, high purity, a large scale because of their expensive and complicated procedures, high reaction temperatures, long reaction times, toxic reagents, and their potential harm to the environment. The thermal treatment method can be considered as one of the best methods in nanoparticles formation because it is fast and cheap, high purity and characterization of metal oxide nanoparticle can be improved.

1.3 Significant of The study

Metal oxide semiconductor nanoparticles and binary metal oxide semiconductor nanoparticles are attractive subjects of continuous scientific interest and have been deeply investigated in materials sciences, because of their physical-chemical properties and their wide range of applications as sensor, solar cell, semiconductors, magnetic materials, catalysts, super hard materials, high temperature ceramics, among others. In particular, ZnO and CdO nanoparticles and binary $(ZnO)_x(CdO)_{1-x}$ nanosheets are commonly used as catalytic materials, sensors and solar cell.

In this study, the synthesis of ZnO and CdO nanoparticles and binary oxide $(ZnO)_x(CdO)_{1-x}$ nanosheets by means of thermal treatment method from an aqueous solution containing metal nitrates, poly(vinyl pyrrolidone), and deionized water was described. The solution was dried at 80 °C for 24 h before grinding and calcination at temperatures ranging from 500 to 650 °C. This method has the advantages of simplicity, less expensive, no unwanted by-products, and it is environmentally friendly. Possibly this method is employable on a large scale production.

1.4 Scope of The present Study

The present research work is limited to the preparation of ZnO, CdO nanoparticles and binary $(ZnO)_x(CdO)_{1-x}$ nanosheets using metal nitrate as precusor and PVP as capping material via thermal treatment route. . Furthermore, the study involves the morphological, structural and optical characterization of the as-prepred nanomaterials.

1.5 Objectives of The study

The purpose of this work is to employ thermal treatment technique to synthesize ZnO, CdO nanoparticles and binary $(ZnO)_x(CdO)_{1-x}$ semiconductor nanosheets in PVP as capping agent. The nanomaterials produced are expected to have improve physical and chemical properties. The objectives are further splitted as follow:

1. To produce high purity CdO and ZnO semiconductor nanoparticles and binary $(ZnO)_x$ $(CdO)_{1-x}$ semiconductor nanonsheets via thermal-treament method.

2. To study the influence of PVP concentration on the structural, morphological and optical properties of ZnO and CdO nanoparticles.
3. To investigate the influence of calcination temperature on the structural, morphological and optical properties of ZnO and CdO nanoparticles.
4. To investigate the influence of calcination temperature on the structural, morphological and optical properties of $(ZnO)_x$ $(CdO)_{1-x}$ semiconductor nanosheets.

1.6 Outline of Thesis

This dissertation is structured as follow:
Chapter 1 presents the general introduction about the research background, scope, problem statement, significant of the study and objectives of the study. Chapter 2 reports the previous works carried out by other researchers including the current and past literatures in terms of the background materials and method, also includes the application of ZnO ,CdO and $(ZnO)_x(CdO)_{1-x}$ nanomaterials. Chapter 3 provides theoretical background to the thesis, which includes the structural and optical properties of study. Chapter 4 discusses the methodology of the study, including materials and preparation of samples. This chapter also provides a set-up of the experimental apparatus such as TGA, FTIR, EDX, XRD, TEM, SEM, UV-Visible spectroscopy and PL. In Chapter 5, detailed results and discussion on characterization of metal and binary oxide nanomaterials by using the above mentioned microscopic andspectroscopy techniques were reported. Chapter 6 contains the conclusions of the study and suggestions for future works.

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