# STRUCTURAL AND OPTICAL PROPERTIES OF ERBIUM DOPED PHOSPHATE GLASS CONTAINING ZINC OXIDE NANOPARTICLES

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Master of Science (Physics)

Faculty of Science
Universiti Teknologi Malaysia

JULY 2014

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Alhamdulillah

To my beloved mother (Kalthom Salleh)
father (Mohamad Zulkifeli Mohamad Zain),
siblings (Mohamad Zahin, Nur Najihah, Mohamad Hazim and Nur Syahirah), and
friends.

# ACKNOWLEDGEMENT

Alhamdulillah, first and foremost Thanks to Allah SWT, whom with His willing giving me the opportunity to complete this master study. I would like to express my sincere appreciation to my supervisor, Dr. Ramli bin Arifin and Associate Professor Dr. Sib Krishna Ghoshal for their supervision, advices, guidance and encouragement to enable me to develop an understanding of the project.

I would like to thank all lecturers who share their knowledge and effort throughout my research journals. My great appreciation goes to all colleagues in Advance Optical Material Research Group (AOMRG), Faculty of Science, UTM for their pleasant and friendly working atmosphere that I will never forget.

Deepest thanks and appreciation goes to my parent, family and postgraduate friends and other who has provides assistance for me while completing this project. Last but not least, special thanks to the financial support from the Grant GUP (Vot 00J76, 06J71 and 06J75), Ministry of Higher Education (MoHE).

# ABSTRACT

Understanding and examining the influence of metallic nanoparticles (NPs) on structural and optical properties of phosphate glass has become tremendous topical interests. A series of glasses with composition (78.5-x) $P_2O_5$–$10Li_2O$–10ZnO–$1.5Er_2O_3$–(x) ZnO (with $0 \leq x \leq 1.2$ mol%) were prepared using melt quenching technique and their detailed characterizations were performed. The amorphous nature of the glass was confirmed using X-ray diffraction (XRD) technique. The estimated NPs average sizes are 8.54 nm by using Debye-Scherrer formula. The glass stability *S*, the onset of $T_g$ and $T_c$ were measured by using differential thermal analyser (DTA). The mode of vibrations for these glasses was analyzed using Fourier transform infrared (FTIR) spectroscopy. FTIR measurements revealed that the presence of ZnO NPs increased the depolymerisation of the glasses at $Q^3$ tetrahedral sites. FTIR spectra exhibit five peaks which are assigned to P=O group at $Q^3$ tetrahedral site (1310 $cm^{-1}$), $PO_2$ group (1260–1170 $cm^{-1}$), P-$O^-$ groups (1080 and 900 $cm^{-1}$) and P-O-P groups (775–710 $cm^{-1}$). The results affirmed that the incorporation of ZnO NPs improved the chemical and physical stability of the phosphate glass. The UV-Vis absorption spectra comprised of eight absorption bands corresponding to the transitions $^4F_{3/2}$, $^4F_{3/2}$, $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4F_{9/2}$ and $^4I_{9/2}$. The band gap energy was found to increase from 3.54 to 3.59 eV and the Urbach energy decreased from 0.258 to 0.246 eV as obtained from the UV-Vis absorption measurements. The influence of ZnO NPs on the luminescence property of $Er^{3+}$ as dopant was studied and the mechanisms involved in the enhancement on emission intensity was determined using photoluminescence spectroscopy at 357 nm excitation. The emission spectra consist of eight peaks in which the peak intensity of the violet band centered at 413 nm for $^4F_{3/2} \rightarrow {^4I_{15/2}}$ transition and blue band at 458 nm due to $^4F_{7/2} \rightarrow {^4I_{15/2}}$ transition showed gradual increment with increased concentration of ZnO NPs added to the glass. The enhancement in the emission peak intensity for the transition is attributed to the effect of quantum confinement and local field of ZnO NPs in the vicinity of $Er^{3+}$ ion. The correlation between structural and optical properties in the presence of NPs is established.

# ABSTRAK

Memahami dan meneliti pengaruh zarah nano logam (NP) ke atas sifat struktur dan optik kaca fosfat telah menjadi topik yang menarik pada masa kini. Satu siri kaca dengan komposisi (78.5-x) $P_2O_5$–10$Li_2O$–10ZnO–1.5$Er_2O_3$–(x) ZnO (dengan $0 \leq x \leq 1.2$ mol%) telah disediakan menggunakan teknik pelindapan leburan dan pencirian terperinci telah dijalankan. Sifat amorfus kaca disahkan dengan menggunakan teknik pembelauan sinar-X (XRD). Anggaran saiz purata NP ialah 8.54 nm menggunakan formula Debye-Scherrer. Kestabilan kaca, *S*, permulaan $T_g$ dan $T_c$ diukur menggunakan penganalisis terma pembezaan (DTA). Mod getaran bagi kaca dianalisis menggunakan spektroskopi Inframerah transformasi Fourier (FTIR). Hasil pengukuran FTIR menunjukkan bahawa kehadiran NP ZnO telah meningkatkan penyahpolimeran kaca pada tapak tetrahedron $Q^3$. Spektrum FTIR mempamerkan lima puncak yang menunjukkan kumpulan P=O pada tapak tetrahedron $Q^3$ (1310 $cm^{-1}$), kumpulan $PO_2$ (1260–1170 $cm^{-1}$), kumpulan P-$O^-$ (1080 dan 900 $cm^{-1}$) dan kumpulan P-O-P (775–710 $cm^{-1}$). Keputusan ini mengesahkan bahawa penggabungan NP ZnO dapat menambah baik kestabilan kimia dan fizikal kaca fosfat. Spektrum penyerapan UV-Vis terdiri daripada lapan jalur penyerapan yang berpadanan dengan peralihan $^4F_{3/2}$, $^4F_{3/2}$, $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4F_{9/2}$ and $^4I_{9/2}$. Jurang jalur tenaga didapati meningkat daripada 3.54 kepada 3.59 eV dan tenaga *Urbach* menurun daripada 0.258 kepada 0.246 eV seperti yang diperoleh daripada pengukuran penyerapan UV-Vis. Pengaruh NP ZnO ke atas sifat luminesens dopan $Er^{3+}$ telah dikaji dan mekanisme yang terlibat dalam peningkatan keamatan pancaran telah ditentukan menggunakan spektroskopi fotoluminesens pada pengujaan 357 nm. Spektrum pancaran terdiri daripada lapan puncak yang mana keamatan puncak jalur ungu yang berpusat pada 413 nm untuk peralihan $^4F_{3/2} \rightarrow {}^4I_{15/2}$ dan jalur biru pada 458 nm bagi peralihan $^4F_{7/2} \rightarrow {}^4I_{15/2}$ menunjukkan peningkatan secara beransur-ansur dengan pertambahan kepekatan NP ZnO yang ditambahkan ke dalam kaca. Peningkatan keamatan puncak pancaran bagi peralihan tersebut disebabkan oleh kesan kurungan kuantum dan medan setempat terhadap NP ZnO di sekitar ion $Er^{3+}$. Korelasi antara sifat struktur dan optik dengan kehadiran NP telah dapat ditentukan.

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| | | |
|---|---|---|
| $A$ | - | Absorbance |
| $B_2O_3$ | - | Boron trioxide |
| BCE | - | Before the Common Era |
| BO | - | Bridging Oxygen |
| $d$ | - | Distance between each adjacent crystal planes |
| D | - | Nanocrystal diameter |
| $d_2$ | - | Thickness sample |
| DTA | - | Differential Thermal Analyzer |
| $e$ | - | Electron charge |
| E | - | Energy |
| $E_f$ | - | Energy upper band |
| $E_c$ | - | Conduction band energy |
| $E_g$ | - | Energy band gap |
| $E_i$ | - | Energy lower band |
| $E_{opt}$ | - | Optical energy gap |
| $E_v$ | - | Valence band energy |
| $Er_2O_3$ | - | Erbium oxide |
| $Er^{3+}$ | - | Trivalent erbium ion |
| $eV$ | - | Electron Volt |
| FTIR | - | Fourier Transform Infrared |
| IR | - | Infrared |
| $k$ | - | Force constant of the bond |
| $Li_2O_3$ | - | Lithium oxide |
| m | - | Mass of atom |
| n | - | Integer |

| | | |
|---|---|---|
| NBO | - | Non-bridging Oxygen |
| NIR | - | Near-infrared |
| NPs | - | Nanoparticles |
| $O_T$ | - | Terminal oxygen |
| P | - | Poise |
| P=O | - | Double bond |
| $P_2O_5$ | - | Phosphorus pentoxide |
| $P_4O_{10}$ | - | Tetraphosphorusdecaoxide |
| PL | - | Photoluminescence |
| $PO_4$ | - | Phosphate |
| RE | - | Rare earth |
| $SiO_2$ | - | Silicon dioxide |
| $T_c$ | - | Crystallization temperature |
| $T_g$ | - | Glass formation temperature |
| $T_m$ | - | Melting temperature |
| UV-Vis | - | Ultraviolet Visible |
| v-$P_2O_5$ | - | vitreous phosphoric oxide |
| XRD | - | X-Ray Diffraction |
| ZnO | - | Zinc oxide |
| $\alpha$ | - | Absorption coefficient |
| β | - | Full width half at maximum (FHWM) |
| $\Delta E$ | - | Urbach energy |
| θ | - | Bragg angle |
| λ | - | Wavelength of incident X-ray beam |
| $\mu$ | - | Reduced mass |
| $\sigma$ -bonds | - | Sigma bond (strongest covalent bond) |
| υ | - | Vibrational frequency |
| $\omega$ | - | Frequency |
| $\hbar\omega$ | - | Photon energy |

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# CHAPTER 1

# INTRODUCTION

## 1.1 General Introduction

Glass is an amorphous solid that has been around in various forms for thousands of years and has been manufactured for human since 12, 000 BC. Glass is a super cooled liquid, meaning that it is rigid and static but does not change molecularly between melting and solidification into a desired shape. Glass is one the most versatile substances on earth, used in many applications and in wide variety of forms, from plain clears glass to tempered and tinted varieties.

Some oxides, called glass former, have the ability to form glasses by themselves or by mixing with other network formers. Examples of the oxides are $SiO_2$, $P_2O_5$ and $B_2O_3$. They are capable of forming a 3D network with oxygen which will provide strong covalent bond. The glass is formed by heating the oxides up to the glass melting temperature and quickly cooled to ensure the glass will not crystallize. That is why the glass is also called “super-cooled liquid”.

Recently, phosphate glasses have received a great deal of attention due to their potential application in optical data transmission, detection, sensing and laser technology, waveguide and fibre optical amplifier devices (Martin *et al.*, 2006). Compared to silica glasses, phosphate glasses offer distinct properties such as good a.c conductivity (Mariappan *et al.*, 2005), large infrared transmission window (Govindaraj *et al.*, 2002), good chemical durability (Brow, 2000), high gain density (Toyoda *et al.*, 2003), wide bandwidth emission spectrum and low up-conversion characteristics. Other than that, phosphate glasses also have properties such as low melting point, high thermal expansion coefficient, and optical properties make these glasses potential candidates for many technological applications such as sealing materials (Toyoda *et al.*, 2003), medical use, and solid state electrolytes (Brow, 2000).

The successful development of optical amplifiers for long-distance communication system has increased the interest on rare earth doped materials (Miniscalco, 1991). Therefore, rare earth doped materials have been studied and used for the last 40 years in a variety of photonics applications (Santos *et al*., 2010), such as frequency up-converters, optical amplifiers and lasers. In particular, erbium-doped phosphate glasses are interesting materials for active planar waveguide fabrication and potential application in integrated optic devices since they have comparably large emission cross-section and weak interaction among active ions (Delavaux *et al*., 1997).

The trivalent erbium ion, $Er^{3+}$ is the most attractive choice to optically activate glass matrices is due to the fact that $Er^{3+}$ doped phosphate glass emits a broad green and red luminescence. Depending on the crystalline phase appearing, rare earth ions can be incorporated or not in the crystalline structure if an appropriate site for the trivalent ions is available (Petit *et al*., 2005). Ananthamohan *et al*. (1990) studied absorbed molecules on a solid surface and they found out that the addition of very small amount of $Er^{3+}$ also affects the absorption band positions compared with binary copper phosphate glasses.

Nanoparticles (NPs) are frequently used as substrates for signal enhancement and can be thought of as inorganic chromophores with strong extinction. NPs exhibit many unique chemical and physical properties and have several applications, including as bridges for energy transport, media for electronic reactions, probes for microscopy, and active surfaces for surface enhanced Raman spectroscopy, fluorescence scattering, and biological sensing (Cattaruza *et al*., 2007). While, the properties of NPs can be examined in a wide range because it possess desired optical properties owing to the high local fields in the NPs which result in enhancement of some optical properties (Lysenko *et al*., 2006).

The ternary lithium zinc phosphate glasses are not widely explored. Ardelean *et al.* (2007) has studied ternary vanadium lithium phosphate glass system to obtain further information about the local symmetry and interaction between vanadium ions in the glass matrix. Meanwhile, Ghauri *et al.* (2009) evaluated band gap energy, structural changes, molar volume, density and chemical durability of the ternary zinc molybdenum phosphate glass.

## 1.2 Statement of Problem

Research in glasses has extensively been done where the effect of modifiers, dopants or co-dopants incorporated into the system especially in modifying the structures has been well studied. However, majority of the experiments uses binary and ternary tellurite, halide, sulphide and phosphate glass focus mostly on preparation, optimization and characterization techniques. Phosphate glasses have limitations in their low absorption and emission cross-section. Enhancing the absorption and emission properties is the most challenging task. Studies on optical enhancement by incorporating different nanostructures are still lacking. Somehow, embedding NPs in rare earth doped phosphate glasses and examining their optical behaviour is more recent development and gained renewed interests. Few studies have been dedicated on embedding NPs in this system and they are limited to certain nobel metallic NPs and rare earth dopant. Improving the structural and spectroscopic properties of phosphate glass by minimizing the effect of concentration quenching is the key issue. The role of ZnO NPs in modifying the optical properties of phosphate glasses is far from being understood. In view of the above, the aim of this research is in order to study the effect of embedded ZnO NPs to $P_2O_5$–ZnO–$Li_2O$–$Er_2O_3$ on the optical properties. In addition, other properties such as structural, thermal and size of ZnO NPs will also be examined.

## 1.3 Glass System Chosen

In these studies, a series of glass system are chosen as below:

$$(78.5\text{-}x)\ P_2O_5\text{–}10Li_2O\text{–}10ZnO\text{–}1.5Er_2O_3\text{–}(x)\ ZnO(NPs)$$

$$(\text{with } 0 \leq x \leq 1.2 \text{ mol\%})$$

Through detailed characterizations of these series, the role played by the ZnO NPs can be determined and quantified by calculating structural and optical parameters.

Phosphate has been used as glass host because of its low melting point, wide glass formation region and good chemical properties (Doremus, 1973; Agus, 2003). Two modifier ions that have been used in this study are $Li_2O$ from group IA and ZnO from group 12. These modifier ions act as network modifier where they modified the glass chain structure during melting process.

Erbium oxide is used as a dopant since it is relatively stable in air, not quickly oxidizing like other rare earth. Erbium is group of metallic rare-earth element. It is suitable for glass, optic and porcelain enamel. It can be applied as colouring, as amplifier in fibre optics and in laser for medical and dental use. It exhibits a sharp absorption bands in the visible, ultraviolet and near infrared (Lide, 2004).

The main advantages with ZnO NPs are its low price, good gas sensing, photo-catalytic and antibacterial features. Zinc oxide also has wide direct band gap (3.37 eV) semiconductors that have potentials for blue-ultraviolet light emitters and detectors, transparent high-power electronics, and piezoelectric transducers (Sreeja *et* al., 2010). It is possible to prepare structures with interesting optical properties like photonic crystals in small amount of ZnO NPs (Nohavica and Gladkov, 2010). Chen *et al*. (2008) in their studies indicate that the effect of adding the ZnO NP degenerated the crystalline quality, associated with the stress generated which resulted in lattice disorder.

## 1.4 Objectives of the Study

The objectives of this study are:

1. To synthesize the erbium doped phosphate glass containing varying concentration of ZnO NPs from 0.0 to 1.2 mol%.
2. To determine the amorphous state and size of ZnO NPs in glass samples.
3. To identify the effect of vibrational frequency mode on the glass system via FTIR spectroscopy.

4. To estimate the thermal parameters (glass transition temperature ($T_g$), crystallization temperature ($T_c$) and glass melting temperature ($T_m$) of each glass sample.

5. To examine the optical properties of glass by means of their absorption and photoluminescence phenomena in the presence of NPs.

## 1.5 Scope of the Study

In order to achieve the proposed objectives, the immediate scopes are:

1. Preparation of glass samples based on phosphorous pentaoxide ($P_2O_5$); zinc oxide (ZnO) and lithium oxide ($Li_2O$) as modifier; erbium oxide ($Er_2O_3$) as dopant, with different composition of ZnO as NPs by using melt quenching technique.

2. The amorphous phase of the glass and size of ZnO NPs are determined using X-Ray Diffraction.

3. The structural characterization is made via FTIR spectroscopy.

4. The thermal properties of the glass sample are determined by using DTA.

5. The optical properties are determined by means of ultraviolet-visible spectroscopy and luminescence spectroscopy.

## 1.6 Significance of the Study

The study of unique characteristics of phosphate glass has contributed so much in developing new knowledge and technology in this world. For example, the study of spectroscopy of rare earth of phosphate glass has helped to develop material for optical data transmission, detection and sensing, laser, waveguide and fibre optical amplifier. Due to few studies exist based on erbium doped phosphate glass containing NPs, the present study has been carried out to understand further the structural and optical features of the glass. We expect that the combination of $Er_2O_3$ and ZnO NPs can enhance the optical characteristic of the phosphate glass. Hence, a direct examination of relationship between rare earth ions and effect of NPs can also be understood. As a result, the structural and optical features of the glass will act as new information in this field for the purpose of further study. A clear understanding of embedding ZnO NPs in the glass system will emerge.

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