



***PHYSICAL, OPTICAL AND THERMAL PROPERTIES OF
ERBIUM-DOPED BOROTELLURITE SILICATE GLASS SYSTEM
INCORPORATED WITH SILVER OXIDE***

ABDULKARIM MUHAMMAD HAMZA

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By

ABDULKARIM MUHAMMAD HAMZA

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

August 2019

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DEDICATION

This thesis is dedicated to

*my mother; Hajiya Khadijah Abdulkarim Hamza
my father; Alhaji Abdulkarim Hamza Musa II
my step mother; Hajiya Fatima Abdulkarim Hamza and Hauwa Abdulkarim Hamza
my lovely wife; Hauwa Muhammad Aliyu
my daughter; Halimah (Mu'aminah) Muhammad Hamza*

*my brothers and sisters
my generous and supportive supervisor
my helpful lecturers, lab mates, friends and all the members of staff of the Faculty of
Science*

*I also dedicate this thesis to the poor Nigerians who are suffering in the hands of
bad and corrupt leaders*

*Thank you all for your support and understanding
May God Almighty bless and ease your ways*



Abstract of a thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Doctor of Philosophy

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August 2019

Chairman : Professor Halimah Mohamed Kamari, PhD
Faculty : Science

Four series of rare earth doped borotellurite silicate glass were prepared by melt quenching method. The first and second series are a quaternary erbium oxide and erbium oxide nanoparticles doped glass with chemical composition $\{[(\text{TeO}_2)_{0.8}(\text{B}_2\text{O}_3)_{0.2}]_{0.8}(\text{SiO}_2)_{0.2}\}_{1-y}(\text{RE})_y$, $y = 0.01, 0.02, 0.03, 0.04, 0.05$ molar fraction and $\text{RE} = \text{Er}_2\text{O}_3$ / Er_2O_3 NPs, while the third and fourth series are a multicomposition $[\{[(\text{TeO}_2)_{0.8}(\text{B}_2\text{O}_3)_{0.2}]_{0.8}(\text{SiO}_2)_{0.2}\}_{0.99}(\text{Ag}_2\text{O})_{0.01}\}_{1-y}(\text{RE})_y$ where $y = 0.01, 0.02, 0.03, 0.04, 0.05$ molar fraction. This research proposes to extract silica from the rice husk and use it to synthesize a series of selected chemical composition of borotellurite silicate glasses doped with different concentration of Er_2O_3 , Er_2O_3 nanoparticles (NPs) and doped with Ag_2O in order to study their effects on the physical, structural, optical and thermal properties. Addition of Ag_2O into the glass composition modifies the optical properties of the glass system. This will go a long way in turning waste into wealth. A novel type of glasses containing rare earth ions and silver ions has recently emerged and already attracted significant attention, the reason for such interest lies in the efficient enhancement of the fluorescent properties in rare earth doped glasses when appropriate silver ions is introduced to it. Results of X-ray diffraction (XRD) confirmed the amorphous nature of the glass. The X-ray fluorescence (XRF) verified the achievement of 98.6% of silicate from rice husk. Fourier transform infrared (FTIR) has revealed the basic structural units such as TeO_4 , TeO_3 , BO_4 , BO_3 , Si-O-Si and O-Si-O in the glass system. The presence of erbium nanoparticles in the second and fourth series was verified from transmission emission microscopy (TEM) and the size of the nanoparticles were recorded within the range of 25 – 28 nm and 41 – 50 nm. The differential scanning calorimetry (DSC) measurements indicate a good thermal stability of borotellurite silicate glasses with the values of $T_s > 100$ °C, and the transition temperature, T_g in all the glass series are found to increase from 437 to 511 °C, 447 to 498°C, 452 to 482 °C and 469 to 495 °C with the increasing dopants concentrations in all the four glass series. The four glass series have a Hruby parameter

(H_r) in the range of 0.32 – 0.51, 0.20 – 0.37, 0.55 – 0.64 and 0.47 – 0.64 greater than 0.4 which verified that the glasses were easily formed with moderate quenching rate. The reduced glass transition (T_{rg}) are also within the range of 1/2 – 3/2. The DSC profiles show an improvement of thermal stability of the glasses with addition of Ag₂O to 273 °C. The theoretical complications in understanding the structures and properties of amorphous solids are amplified by the lack of precise experimental information. Absorption spectrum from near infrared to visible light was obtained and the Judd–Ofelt (J–O) intensity parameters ($\Omega_\lambda, \lambda = 2,4,6$) were calculated. The calculated values of the parameters fall within the range of $(4.178 \text{ to } 6.507) \times 10^{-20} \text{ cm}^2$, $(1.384 \text{ to } 2.143) \times 10^{-20} \text{ cm}^2$ and $(0.893 \text{ to } 2.451) \times 10^{-20} \text{ cm}^2$ for Ω_2 , Ω_4 and Ω_6 respectively. Also, the J–O parameter of Ω_2 decreases from $5.366 \times 10^{-20} \text{ cm}^2$ to $4.529 \times 10^{-20} \text{ cm}^2$ and Ω_6 from $1.349 \times 10^{-20} \text{ cm}^2$ to $1.212 \times 10^{-20} \text{ cm}^2$ with increasing Er³⁺ NPs/Ag concentration.

Spontaneous emission probabilities of some important transitions, branching ratio, and radiative lifetimes of some excited states of Er³⁺ and Er³⁺ NPs are predicted using J–O intensity parameters. The branching ratio β for $^4I_{15/2} \rightarrow ^2H_{11/2}$, and $^4I_{15/2} \rightarrow ^4F_{9/2}$ transitions are within the range 65 – 95% indicating that there is a possibility for red and green emissions. The glass samples with 0.01 Er and 0.01 Er NPs for the two glass series exhibits the smallest value of τ which are 0.025 and 0.204 ms respectively corresponding to $^2H_{11/2} \rightarrow ^4I_{15/2}$ transition. Absorption cross-section, calculated emission cross-section and gain, were evaluated using the McCumber theory for $^4I_{13/2} \rightarrow ^4I_{15/2}$ Er³⁺ transition. A schematic energy level diagram is proposed in this work. The full width at half maximum (FWHM), the gain band width (GBW) and figure of merit (FOM) for $^4I_{13/2} \rightarrow ^4I_{15/2}$ transitions of the glasses have also been studied and reported. All the glass samples show a positive gain above 50% population inversion and the samples exhibit a flat gain bandwidth in the range of 1400 - 1700 nm, which covers the S band (1460 - 1530 nm) and C band (1530 - 1565 nm) in the optical communication window. The gain-bandwidth and gain cross-section profile suggest that these glasses could be found as more promising materials for broadband amplification of both S and C-band signals. The large values of thermal stability of the glass unveils that these glasses are stable against devitrification and are suitable for potential application in fiber drawing. The spectroscopic quality factors in the range of 0.79 – 1.87 and 0.29 – 0.43 for Er₂O₃ and Er₂O₃ NPs doped glasses and 0.48-0.79 and 0.80-0.89 for the glasses incorporated with silver oxide obtained from the Judd-Ofelt parameters suggest the fact that the glasses are suitable for solid state laser application.

The optimum concentration of dopants is achieved is achieved at 0.03 molar fraction as shown in the PL analysis of all the sample glasses. Beyond that concentration, the PL intensity begins to drop with increasing dopant concentration. In view of that, the glasses with 0.03 molar fraction are considered suitable for solid state laser application.

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

**SIFAT FIZIKAL, OPTIKAL DAN TERMA SISTEM KACA ERBIUM
TERDOP DENGAN BOROTELURIT SILIKA DITAMBAHKAN DENGAN
ARGENTUM OKSIDA**

Oleh

ABDULKARIM MUHAMMAD HAMZA

Ogos 2019

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Empat siri nadir bumi borotelurit kaca didopkan dengan bio-silika telah disediakan dengan kaedah sepuh lindap. Siri pertama dan kedua ialah kaca yang didop erbium oksida dan nanopartikel erbium oksida dengan kandungan komposisi kimia $[(\text{TeO}_2)_{0.8}(\text{B}_2\text{O}_3)_{0.2}]_{0.8}(\text{SiO}_2)_{0.2}\}_{1-y}(\text{RE})_y$, $y = 0.01, 0.03, 0.04, 0.05$ pecahan molar dan $\text{RE} = \text{Er}_2\text{O}_3 / \text{Er}_2\text{O}_3$ NPs, manakala siri ketiga dan keempat ialah komposisi pelbagai jenis kaca $[[(\text{TeO}_2)_{0.8}(\text{B}_2\text{O}_3)_{0.2}]_{0.8}(\text{SiO}_2)_{0.2}\}_{0.99}(\text{Ag}_2\text{O})_{0.01}]_{1-y}$ di mana $y = 0.01, 0.02, 0.03, 0.04, 0.05$ pecahan molar. Penyelidikan ini telah mencadangkan untuk mengeluarkan silika daripada sekam padi dan menggunakannya untuk mensintesis satu siri komposisi kimia terpilih bagi kaca borotelurit silika yang telah didopkan dengan Ag_2O serta Er_2O_3 , Er_2O_3 NPs dengan kepekatan yang berbeza untuk mengkaji kesan mereka terhadap sifat fizikal, struktur, optik dan terma terhadap sistem kaca tersebut. Penambahan Ag_2O ke dalam komposisi kaca mengubah sifat optik pada sistem kaca. Ini akan membantu untuk menukarkan bahan buangan kepada bahan berguna dalam jangka masa yang panjang. Sejenis kaca baharu yang mengandungi ion nadir bumi dan ion perak telah muncul kebelakangan ini serta sudah pun menarik perhatian yang ketara, sebab disebalik perhatian sedemikian terletak pada peningkatan kecekapan sifat pendarfluor dalam kaca yang telah didop nadir bumi apabila ion perak yang sesuai telah diperkenalkan kepadanya. Hasil daripada penyinaran sinar-x (XRD) mengesahkan sifat amorfus kaca yang telah dihasilkan. Pendarfluor X-ray (XRF) mengesahkan pencapaian sehingga 98.6 % silika daripada sekam padi. Transformasi Fourier inframerah (FTIR) telah mendedahkan unit struktur asas sistem kaca seperti TeO_4 , TeO_3 , BO_4 , BO_3 , Si-O-Si and O-Si-O. Kehadiran nanopartikel erbium dalam siri kedua dan keempat telah diverifikasi daripada TEM dan saiz nanopartikel dicatatkan dalam lingkungan 25 - 28 nm dan 41 - 50 nm. Pengukuran kalorimetri pengimbang perbezaan (DSC) menunjukkan kestabilan terma yang baik daripada kaca silika dengan nilai $T_s > 100^\circ\text{C}$, dan suhu peralihan, T_g dalam semua siri kaca didapati meningkat dari 437 ke 511°C, 447 ke 498°C, 452 ke 482°C dan 469 ke 495°C dengan

peningkatan kepekatan dopan. Empat siri kaca mempunyai parameter Hruby dalam julat 0.32 – 0.51, 0.20 – 0.37, 0.55 – 0.64 dan 0.47 – 0.64 yang lebih besar daripada 0.4 yang telah mengesahkan bahawa kaca mudah dibentuk dengan kadar pendinginan sederhana. Peralihan kaca berkurang (T_{rg}) juga dalam julat 1/2 – 3/2. Profil DSC menunjukkan peningkatan kestabilan terma kaca dengan penambahan Ag_2O hingga 273 °C. Komplikasi teori dalam memahami struktur dan sifat pepejal amorfus telah diperbesarkan dengan kekurangan maklumat eksperimen yang tepat. Spektrum penyerapan daripada inframerah berhampiran cahaya nampak telah diperoleh dan parameter intensiti Judd-Ofelt (J-O) ($\Omega_\lambda, \lambda = 2,4,6$) telah dikira. Nilai-nilai yang dikira dari parameter tersebut berada dalam lingkungan $(4.178 \text{ hingga } 6.507) \times 10^{-20} \text{ cm}^2$, $(1.384 \text{ hingga } 2.143) \times 10^{-20} \text{ cm}^2$ dan $(0.893 \text{ hingga } 2.451) \times 10^{-20} \text{ cm}^2$ untuk Ω_2, Ω_4 serta Ω_6 . Sebagai tambahan, Ω_2 parameter J-O menurun daripada $5.366 \times 10^{-20} \text{ cm}^2$ kepada $4.529 \times 10^{-20} \text{ cm}^2$ dan Ω_6 daripada $1.349 \times 10^{-20} \text{ cm}^2$ kepada $1.212 \times 10^{-20} \text{ cm}^2$ dengan peningkatan kepekatan Er^{3+} NP/Ag. Kebarangkalian pancaran spontan pada beberapa peralihan penting, nisbah cawangan, dan jangka hayat radiasi pada beberapa paras yang teruja pada Er^{3+} dan Er^{3+} NPs telah diramalkan menggunakan parameter intensiti J-O. Nisbah cawangan β untuk peralihan ${}^4I_{15/2}$ - ${}^2H_{11/2}$, and ${}^4I_{15/2}$ - ${}^4F_{9/2}$ berada dalam lingkungan 65 - 95% yang menunjukkan kemungkinan terdapatnya pancaran merah dan hijau. Sampel kaca yang mempunyai 0.01 Er dan 0.01 Er NPs bagi kedua-dua siri kaca mempamerkan nilai τ terkecil iaitu 0.025 dan 0.204 ms masing-masing yang bersamaan dengan peralihan ${}^2H_{11/2} \rightarrow {}^4I_{15/2}$. Keratan silang penyerapan, pengiraan keratan rentas pancaran dan perolehan telah dinilai menggunakan teori McCumber untuk peralihan ${}^4I_{13/2} \rightarrow {}^4I_{15/2} Er^{3+}$. Rajah paras tenaga skematik juga telah dicadangkan. Lebar penuh pada separuh maksimum (FWHM), lebar jalur peroleh (GBW) dan angka merit (FOM) untuk ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$ peralihan kaca juga telah dikaji dan dilaporkan. Semua sampel kaca menunjukkan perolehan positif atas 50% penyongsangan populasi dan sampel memperlihatkan lebar jalur perolehan rata dalam julat 1400 - 1700 nm, yang meliputi jalur S (1460 - 1530 nm) dan jalur C (1530 - 1565 nm) dalam tettingkap komunikasi optik. Perolehan jalur lebar dan perolehan profil keratan rentas menunjukkan bahawa kaca ini berpotensi sebagai bahan penguatan jalur lebar bagi kedua-dua isyarat jalur S dan C. Nilai besar kestabilan terma kaca mengungkapkannya bahawa kaca ini stabil terhadap penyusunan dan sesuai untuk aplikasi potensi dalam lukisan serat. Faktor-faktor kualiti spektroskopi dalam lingkungan 0.79 – 1.87 dan 0.29 – 0.43 bagi kaca yang didopkan Er_2O_3 dan Er_2O_3 NPs manakala 0.48-0.79 dan 0.80-0.89 bagi kaca yang telah digabungkan dengan argentum oksida yang diperoleh daripada parameter Judd-Ofelt mencadangkan fakta bahawa kaca yang telah dikaji sesuai untuk aplikasi laser keadaan pepejal.

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LIST OF SYMBOLS AND ABBREVIATIONS

TeO ₂	Tellurium dioxide
B ₂ O ₃	Boron oxide
SiO ₂	Silicon oxide
RE	Rare earth
Er ₂ O ₃	Erbium oxide
IR	Infrared
EDFA	Erbium doped fibre amplifiers
FTIR	Fourier Transforms Infrared Spectroscopy
XRD	X-ray diffraction
TEM	Transmission Electron Microscopy
Er ³⁺ NPs	Erbium ion nanoparticle
Er ³⁺	Erbium ion
J-O	Judd-Ofelt
Ag ₂ O	Silver oxide
Ag ⁺	Silver ion
GBW	Gain band width
FOM	Figure of merit
MIR	Mid infrared
RDFs	Radial distribution functions
PL	Photoluminescence
HCl	Hydrochloric acid
h	Hour
M	Molar
XRF	X-ray fluorescence
µm	Micro meter

DSC	Differential scanning calorimetry
NBO	Non-bridging oxygen
Sm ₂ O ₃	Samarium oxide
eV	Electron volt
Ω ₂	Omega two
Ω ₄	Omega four
Ω ₆	Omega six
M	Metallization criterion
R _m	Molar refraction
V	Molar volume
β _n	Nephelauxetic ratios
δ	Bonding parameter
T _g	Transition temperature
T _c	Crystallization temperature
T _m	Melting temperature
T _s	Thermal stability
H _r	Hruby parameter
T _{rg}	Reduced glass transition
F	Thermodynamic fragility
E _a	Activation energy
β	Heating rate
α	Absorption coefficient
λ	Wavelength
d	Thickness of the sample
A	Absorbance
B	Constant related to the extent of the band tailing

E_{opt}	Optical band gap energy
$h\omega$	Incident photon energy
ΔE	Urbach energy
\ln	Natural logarithm
K	extinction coefficient
δ_s	Skin depth
σ_c	Optical conductivity
n	Refractive index
NBO	Non-bridging oxygen
BO	Bridging oxygen
χ	Optical electronegativity
α_m	Electronic polarizability
Λ	optical basicity
f_{exp}	Experimental oscillator strength
f_{cal}	Calculated oscillator strength
m	Electron mass
e	Electronic charge
c	Speed of light
N_o	Avogadro's number
ε	Molar extinction coefficient
aJ	Ground state
aJ'	Excited state
X_{md}	Lorentz localize field correction responsible for magnetic dipole transitions
X_{ed}	Lorentz localize field correction responsible for electric dipole transitions
h	Planck's constant

S_{md}	Magnetic dipoles line strength
S_{ed}	Electric dipoles line strength
RMS	Root mean square
$A(J-J')$	Spontaneous transition probability
A_{ed}	Average electric dipole
A_{md}	Average magnetic dipole
β_r	Branching ratio
τ_r	Radiative lifetime
σ_{abs}	Absorption cross section
σ_{em}	Emission cross section
OD	Optical density
G	Gain
P	Population inversion
Dq	Crystal field strength
R and C	Racah parameters
ESA	Excited state absorption
GSA	Ground state absorption
m_{object}	Mass of object
m_{liquid}	Mass of liquid
ρ_{object}	Density of object
ρ_{liquid}	Density of liquid
W_{object}	Weight of object
W_{liquid}	Weight of liquid
$W_{apparent}$	Apparent weight
REI	Rare earth ion
UV-Vis	Ultra violet visible

FWHM

Full width half maximum

$\sigma_{em_{max}}$

Peak emission cross section



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CHAPTER 1

INTRODUCTION

This chapter deals with the overview of the study that is made up of the basic nature of glass, glass formation process and the chemical compositions used in the glass formation. The chapter also includes the objective of the research, scope of the study, problem statement, hypothesis and the thesis outline.

1.1 Overview

Generally, glasses are formed by melting crystalline material at high temperatures which are afterward rapidly cooled to rigid conditions without crystallization. It is a super cooled liquid with solid form appearance. The glass appearance depends mainly on the rate of cooling (Schawe, 1995). Glasses have irregular and disordered arrangements with short range order in periodic arrangements. Thus, a unit cell is insufficient to describe the glass structure.

Chalcogens are one of the families of interest among glass researchers and tellurium is one among them, along with oxygen, sulphur, and selenium from group VI A of the periodic table. From the year 1980, when glass research begun to be tailored towards the various properties of glasses to be utilized in several application, Tellurite glasses became of special interest to glass researchers. Tellurium dioxide (TeO_2) is the most stable oxide of tellurium (Te). The stability nature of TeO_2 is one of the properties that fascinated researchers to tellurite glasses. Some of the properties of tellurite glasses that enable them to attain stability are their chemical durability, high refractive index, high thermal stability, good non-hygroscopic and low phonon energy (Pandarinath et al., 2016).

Most materials that have borate as one of its constituents, have high propensity for glass formation because B_2O_3 does not crystallize by itself, the glasses have high viscosity. The attention given to borate glasses is due to their electrochemical and optical applications, such as optical waveguides, solid-state batteries and luminescent materials. Borate glass is particularly suitable for optical material due to its high transparency, low melting point, high thermal stability, different coordination numbers, and good solubility of rare-earth ions (Ravi et al., 2015). Pure borate glasses possess low refractive index, high melting point and high phonon energies in the range of $1300\text{--}1500\text{ cm}^{-1}$ (Ramadevudu et al., 2017).

Boro-tellurite glasses are formed when tellurite combine with borates. They are intensively studied for their practical applications in terms of enhancing the quality, transparency and refractive index of the glass (Pavani et al., 2011). Borate and tellurite glasses each have a dual coordination numbers of 3 and 4 with oxygens.

As for silica, it can be found in agricultural waste such as rice husk and palm ash. The large amount of this waste can be a new source of silica production in Malaysia in contrast to the one produced from quartz or sand by the extraction process. Silica (SiO_2) is an excellent glass former that easily forms glassy phases even at very low melt-quenching rates (Leenakul et al., 2016). Silicate glasses have good transmission to visible light. They are used for windows and bottle containers.

One example is borosilicate glasses which have excellent chemical durability, high resistance and low thermal expansion coefficient. They are used in laboratory, sealed headlights, pharmaceutical containers, pipelines etc. (Zhao et al., 2019).

Currently, doping glasses with rare-earth ions is getting much consideration from researchers across the globe. This is due to the extensive use of the rare-earth ions to improve the optical and physical properties of host glasses because of their unique spectroscopic properties resulting from their optical transitions in the intra 4f shell (Nandi et al., 2009). In addition, it is very essential to know the relationship between the host composition and radiative or non-radiative characters of the RE ions in order to design lasing glasses with high performance (Shen et al., 2007). Quite recently, glasses doped with RE ions are found to be potential in the development of luminescent devices suitable for Infrared (IR) lasers, infra-red to visible up-convertors, fibre and waveguide amplifiers for optical transmission network (Miniscalco, 2001; Xu et al., 2004). Erbium (Er_2O_3) ions are considered to be the most important among all the RE ions, due to their potential applications in the fields of optical fibre amplifiers, microchip lasers, infrared lasers, erbium doped fibre amplifiers (EDFA) in the wavelength division multiplexing (WDM) System, eye-safe lasers in medical fields (Swapna et al., 2015).

1.2 Statements of the research problem

The investigation on the new glass of different compositions so as to obtain the most suitable host glasses that fit in to various applications by researchers have been made for some decades.

Around 90% of rice husk are discarded as waste products and either burned in open air or left to settle in waste ponds which causes damage to the land and environmental pollution. Moreover, the open burning of rice husk can lead to heavy air pollution (Lee et al., 2013). Therefore, useful applications of rice husk are desirable to solve this problem. Extensive several works had been conducted on the extraction of silica from agricultural wastes (e.g. rice husk), because silica is an important raw material for industrial application. Although, silica production from agricultural wastes is relatively less compared to that from quartz or sand, it can still serve the needs of the industries. This research will focus on non-destructive and cost-effective method to utilize rice husk for production of SiO_2 using a dilute hydrochloric acid leaching, instead of the conventional strong acid process. This will go a long way in turning waste into wealth.

Tellurite glasses are of interest for both scientific and technological point of view, because of their exceptional and promising physical properties. While the crystalline structure of solids as well as their physical properties are now understood, the reverse is the case for amorphous materials including glass. The theoretical complications in understanding the structures and properties of amorphous solids are amplified by the lack of precise experimental information. Therefore, there is a need for research to fill this gap.

A novel type of glasses containing both rare earth (RE) ions and Ag ions has recently emerged and already attracted significant attention (Liu et al., 2018). The reason for such interest lies in the efficient enhancement of the fluorescent properties in RE-doped glasses when an appropriate Ag ion is introduced to it.

The possibility of obtaining new laser transition wavelengths for specific applications (such as biomedical, surgery, sensing and data storage) has driven the investigation of laser materials made with rare earths. Laser action has been demonstrated in optical fibres doped with erbium, ytterbium, neodymium, thulium, samarium holmium and praseodymium. Laser action at multiple wavelengths within the same fibre species has also been demonstrated (Nguyen, 2014).

However, there is no information and knowledge on the effect of silver oxide on the thermal properties of erbium and erbium nanoparticles doped borotellurite silicate glass system.

The Judd-Ofelt intensity parameters and spectroscopic properties of erbium oxide, erbium oxide nanoparticles and silver oxide doped borotellurite silicate glass system have never been investigated to reveal their possible application in optical communication and solid state laser. Thus, this research is aimed at the studying the influence of erbium oxide, erbium oxide nanoparticles and silver oxide on the physical, structural, linear optical and thermal properties of borotellurite silicate glasses as well as its potential application in fibre drawing, optical communication window and solid state laser.

1.3 Scope and limitation of this thesis

The scope of this research is limited to the physical, structural, linear optical and thermal properties. For the physical properties, the density of the glass was measured using Archimedes' principle while the molar volume, polaron radius and interionic distance of the glasses were calculated. Fourier Transforms Infrared Spectroscopy (FTIR), X-ray diffraction (XRD) spectroscopy and Transmission Electron Microscopy (TEM) were used to characterize the structural properties of the glasses. The linear optical properties comprise of the optical absorption, band gap energy, refractive index, Urbach energy, electronic polarizability, optical basicity, metallization criterion and electronegativity. Thermal properties (thermal stability, activation energy and Hruby parameters) were also analysed. Additionally, the radiative properties of several

excited states of Er^{3+} and Er^{3+} NPs ions in the glass were calculated using J-O parameters. Absorption cross-section and calculated emission cross-section, using the McCumber theory, for the ${}^4\text{I}_{13/2} \rightarrow {}^4\text{I}_{15/2}$ transition was determined and the spectroscopic properties (gain band width, full width half maximum and figure of merit) were evaluated.

1.4 Significance of the study

The significance of this research will include providing the fundamental bases of new optical glasses with many new applications, particularly borotellurite silicate -based optical fibers. These function and properties are achieved through a simplified melt quenching technique by utilizing a new material as composites in glass. Hence, the significance of the current study is to find a possible solution to some of the aforementioned challenges through synthesizing erbium, erbium nanoparticles and silver oxide doped borotellurite silicate glasses with enhanced thermal, optical and spectroscopic properties.

1.5 Objectives of the study

The main objective of this research is to study the physical, structural, optical and thermal properties of erbium doped borotellurite silicate glass system with silver oxide incorporation. In order to achieve this, five objectives have been designed which are as follows;

1. To extract silica (SiO_2) from rice husk and utilize it to fabricate a borotellurite silicate glasses doped with erbium, erbium nanoparticles and silver oxide.
2. To examine the structural and optical changes in borotellurite silicate glasses doped with erbium/erbium nanoparticles by adding an appropriate mass of silver oxide.
3. To evaluate the radiative properties such as lifetime, branching ratio and transition probabilities of the glass using Judd-Ofelt theory.
4. To determine the emission cross section, spectroscopic properties and gain using McCumber theory.
5. To investigate the thermal properties such as the transition temperature, crystallization temperature, melting temperature, thermodynamic fragility and glass forming ability of the prepared borotellurite silicate glasses.

1.6 Hypothesis

The hypothesis of this research is listed as follows;

1. The glass density of the prepared glass is expected to increase with Er_2O_3 and Er_2O_3 NPs contents. This could be due to the higher molecular weight of Er_2O_3 and Er_2O_3 NPs when compared to $\text{TeO}_2\text{-B}_2\text{O}_3\text{-SiO}_2$ (Umar et al., 2017a).
2. The addition of Er_2O_3 , Er_2O_3 NPs and Ag_2O are expected to enhance the linear optical properties of the borotellurite silicate glasses. the energy band gap is expected to decrease with increasing Er_2O_3 and Er_2O_3 NPs concentration and with introduction of and Ag_2O .
3. The incorporation of silver oxide into the glass system would modify the glass structure and increase the refractive index due to the creation of more non-bridging oxygen which has higher polarity than the bridging oxygen (Awang et al., 2013).
4. The Ag^+ ions will play a role of sensitizer; thus, it will clearly promote an efficient energy transfer among the Er^{3+} ions with longer radiative lifetime of the $^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$ transition.
5. Bonding nature between Er^{3+} ions and their surrounding ligands would be observed through Judd-Ofelt parameters and bonding parameter. Moreover, the Judd-Ofelt parameters are expected predict the spectroscopic quality of the glasses and as well use to compute the radiative properties of the glass samples.
6. By incorporating Ag_2O in to Er_2O_3 and Er_2O_3 NPs doped borotellurite silicate glass, it is expected that the stimulated emission cross-section, gain band width (GBW) and the figure of merit (FOM) obtained from the McCumber theory would be enhanced.

1.7 Thesis outline

This thesis comprises of six chapters. The first chapter consists of the overview of the research, and short introduction of the individual oxides used in the glass fabrication and their significant roles in the glass compositions. The chapter also focuses on the problem statements, objectives, scope and limitations, significance and expected outcomes of this research. The second chapter gives a brief review of the earlier researches that are related to the present study. These literatures give reports on silica borotellurite glasses, rare-earth doped glasses, erbium doped glasses, physical and structural properties, thermal properties, optical properties, spectroscopic properties, bonding parameters, Judd-Ofelt and McCumber theory. Chapter three gives the existing theories and equations used in this study. The derivation of some theoretical equations are also shown in this chapter. The fourth chapter of this thesis describes the method used in the extraction of silica from rice husk and techniques employed by the conventional melt quenching process for sample fabrication. The chapter also highlights the basic techniques used in this research for sample characterization. Chapter five discusses the analysed results and trends of the findings in the present

work. This covers the structural, physical, thermal and non-linear optical properties of the synthesized glasses. Judd-Ofelt analysis, McCumber theory and spectroscopic parameters are also discussed in this chapter. The comparative studies between some of the parameters of the studied glass with the previous researches are also discussed while chapter six summarizes the important findings of this research, with some suggestions and recommendations for future study.



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