



**UNIVERSITI PUTRA MALAYSIA**

***STRUCTURAL, ELASTIC AND OPTICAL PROPERTIES OF RICE HUSK  
SILICATE BOROTELLURITE GLASS SYSTEM DOPED WITH MICRO  
AND NANOPARTICLES OF ERBIUM OXIDE***

**ALIYU UMAR SA'AD**

**FS 2018 88**



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By

**ALIYU UMAR SA'AD**

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

**April 2018**

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To them I dedicate this work.

My late father Alh. Sa'adu Aliyu (Rahimahullah) and my mother Khadijatu Sa'adu who have set the guide for my success in life. My Aunt and Uncle, Aishatu Aliyu Sa'ad and Saleh Aliyu Yahaya and my Vice-chancellor, Prof. M. S. Liman who have been there all the time I needed them. To my wife, Fatimah and my son for the love you've shown throughout this journey.



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

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**ALIYU UMAR SA'AD**

**April 2018**

**Chairman : Associated Professor Halimah Mohamed Kamari, PhD**  
**Faculty : Science**

Successful extraction of high purity SiO<sub>2</sub> (about 99%) from rice husk (waste) was achieved in this work using the cold acid leaching method. Glass series [(TeO<sub>2</sub>)<sub>0.7</sub> (B<sub>2</sub>O<sub>3</sub>)<sub>0.3</sub>]<sub>1-x</sub> (SiO<sub>2</sub>)<sub>x</sub> were fabricated using the rice husk silicate (RHS) by melt-quenching method. The aim was to extract a very high purity SiO<sub>2</sub> from rice husk with no heat used in leaching to fabricate a system of silicate borotellurite glass system doped with erbium oxide and erbium oxide nanoparticles. The choice of rice husk as silicate source was to add to the commercialization of the rice husk waste management which stand at 30%. Also, to study the structural, elastic and optical properties of the glass systems for Erbium Doped Fiber Amplifier (EDFA) application. The use of rice husk silicate in a borotellurite glass system was expected to improve the glass quality in terms of both elastic and optical properties.

The samples were subjected to X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR) and UV-Vis characterizations to study the structural nature and optical behavior of the glass system. Density, molar volume and ultrasonic velocities were measured to obtain the elastic constants for the various silicate proportions in the glass using ultrasonic data obtained from non-destructive ultrasonic probing technique. The glasses fabricated were found to be transparent at lower RHS concentrations (0.0-0.2) the glasses became more darkened and opaque from 30% to 40% RHS concentrations. 20% RHS was found to be the best SiO<sub>2</sub> composition in terms of transparency, elastic and optical properties of the glasses for Er<sup>3+</sup> ion doping. Using the 0.2 molar fraction of RHS (SiO<sub>2</sub>), a glass system of erbium/erbium NPs doped rice husk silicate borotellurite (Er/Er NPs-doped RHSBT) glass system was fabricated using the chemical composition {[(TeO<sub>2</sub>)<sub>0.7</sub> (B<sub>2</sub>O<sub>3</sub>)<sub>0.3</sub>]<sub>0.8</sub> (SiO<sub>2</sub>)<sub>0.2</sub>}<sub>1-x</sub>

$(\text{Er}_2\text{O}_3)_x$  with  $x = 0.01, 0.02, 0.03, 0.04$  and  $0.05$  mol was prepared using the melt-quenching technique.

The glasses were as well subjected to characterizations as XRD, FTIR, UV-Vis spectroscopy, photoluminescence spectroscopy and non-destructive ultrasonic probing to study the structural, elastic, optical and luminescence properties of the glasses. Theoretical elastic models of Makishima and Mackenzie, Rocherulle, bond compression and ring deformation models were used to study more on the elastic properties and compare with the experimental data. Density and molar volume measurements were also carried out to further provide structural details while transmission electron microscopy was carried out on the erbium nanoparticles doped series to study the morphological features of the glasses.

In all the three samples, the XRD showed no sharp peak indicating a glassy nature. The density and molar volume in the case of RHSBT glass system decreased with increase in  $\text{SiO}_2$  concentration. The ultrasonic velocities, elastic moduli, optical band gaps, Urbach energy, oxygen packing density (OPD), metallization criterion and transmission coefficient increased with RHS concentration increased. The theoretical elastic models confirmed the elastic moduli increase in the silicate borotellurite glass system with increasing  $\text{SiO}_2$  content as shown in the experimental study. Only the Makishima and Mackenzie model appeared to be good for the elastic properties of both the erbium oxide doped and erbium oxide NPs doped glass systems.

Using McCumber theory analysis from the UV-Vis data, Absorption and Emission cross-section data was obtained and used to calculate the simple fiber gain of each sample in the two micro and nano erbium doped RHSBT glass systems. The gains were higher in the case of micro erbium doped system than the nano erbium doped system. With high refractive index of between 2.452 to 2.521 and wide effective band with in the S and C bands of between 76 to 106 nm, the micro erbium doped RHSBT glass system provides a good potential for application in the erbium doped fiber amplifiers (EDFA).

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

**SIFAT STRUKTUR, KENYAL DAN OPTIK SISTEM KACA SEKAM PADI SILIKA BOROTELURIT DIDOP DENGAN ERBIUM OKSIDA DAN NANOZARAH ERBIUM OKSIDA**

Oleh

**ALIYU UMAR SA'AD**

**April 2018**

**Pengerusi : Profesor Madya Halimah Mohamed Kamari, PhD**  
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Penghasilan  $\text{SiO}_2$  yang mempunyai ketulenan yang tinggi (kira-kira 99%) daripada sekam padi berjaya dicapai di dalam kajian ini menggunakan kaedah larut lesap asid sejuk. Siri kaca  $[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{1-x} (\text{SiO}_2)_x$  telah dihasilkan menggunakan sekam padi silika (RHS) dengan kaedah sepuh lindap. Tujuan kajian ini adalah untuk menghasilkan  $\text{SiO}_2$  dengan ketulenan yang tinggi daripada sekam padi tanpa menggunakan haba dalam larut lesap untuk menghasilkan sistem silika borotelurit didop dengan erbiium oksida dan nanozarah erbiium oksida. Pensusunan sisa sekam padi pada masa kini sebanyak 30% boleh dikomersial apabila sekam padi chipisin sebagai sumber silica. Selain itu, ia juga adalah untuk mempelajari sifat struktur, kenyal dan optik sistem kaca itu bagi aplikasi EDFA. Penggunaan sekam padi silika di dalam sistem kaca borotelurit dijangkakan boleh meningkatkan kualiti kaca itu dalam kedua-dua sifat kekenyalan dan optik.

Sampel-sampel itu diuji dengan pembelauan sinar-X (XRD), spektroskopi jelmaan Fourier Inframerah (FTIR) dan pencirian UV-nampak untuk mengkaji keadaan struktur dan sifat optik kaca tersebut. Ketumpatan, isipadu molar dan halaju ultrabunyi diukur untuk mendapatkan pemalar kenyal bagi kandungan silika yang pelbagai di dalam kaca tersebut menggunakan data ultrabunyi yang diperolehi daripada kaedah pemproban ultrabunyi tanpa musnah. Kaca-kaca yang dihasilkan itu ditemui lutsinar pada kepekatan RHS yang rendah (0.0-0.2 mol) dan kaca-kaca itu menjadi lebih gelap dan legap daripada 30% hingga 40% kepekatan. Komposisi yang terbaik untuk transparansi, elastik dan sifat optik untuk pendopan  $\text{Er}^{3+}$  pada gelas adalah 20% RHS. RHS Dengan menggunakan 0.2 mol RHS ( $\text{SiO}_2$ ), satu sistem kaca erbiium/nanozarah erbiium didop dengan sekam padi silika borotelurit (Er/Er NPs-doped RHSBT) dihasilkan menggunakan komposisi kimia  $\{[(\text{TeO}_2)_{0.7} (\text{B}_2\text{O}_3)_{0.3}]_{0.8} (\text{SiO}_2)_{0.2}\}_{1-x}$

$(\text{Er}_2\text{O}_3)_x$  dengan  $x = 0.01, 0.02, 0.03, 0.04$  dan  $0.05$  mol dihasilkan menggunakan teknik sepuh lindap.

Kaca-kaca itu juga dihantar untuk ujikaji XRD, FTIR, spektroskopi UV-Vis, spektroskopi fotopendarcahayaan dan pemprobaan ultrabunyi tanpa musnah untuk mempelajari sifat struktur, kekenyalan, optik dan pendarcahayaan kaca tersebut. Model teori kenyal Makishima dan Mackenzie, Rocherulle, pemampatan ikatan dan model canggaaan gegelang digunakan untuk lebih mempelajari mengenai sifat kenyal dan membandingkannya dengan data eksperimen. Pengukuran ketumpatan dan isipadu molar juga dijalankan untuk membekalkan butiran struktur manakala mikroskopi penghantaran electron dilakukan pada siri yang didop nanozarah erbium untuk mempelajari sifat morfologi kaca-kaca tersebut.

Di dalam ketiga-tiga sampel, XRD menunjukkan tiada puncak yang tajam, menandakan keadaan kaca. Ketumpatan dan isipadu molar di dalam kes sistem kaca RHSBT menurun apabila kepekatan  $\text{SiO}_2$  bertambah. Halaju ultrabunyi, modulus-modulus kenyal, jurang jalur optik, tenaga Urbach, ketumpatan padatan oksigen (OPD), kriteria penglogaman dan pekali penghantaran menaik dengan kenaikan kepekatan RHS. Model teori kenyal mengesahkan kenaikan modulus-modulus kenyal di dalam sistem silika borotelurit dengan kenaikan kandungan  $\text{SiO}_2$  seperti yang ditunjukkan di dalam kajian eksperimen. Hanya model Makishima dan Mackenzie dilihat sesuai untuk sifat kenyal bagi kedua-dua sistem kaca didop dengan erbium oksida dan nanozarah erbium oksida.

Dengan menggunakan teori analisis McCumber daripada data UV-nampak, data keratan rentas penyerapan dan pancaran diperolehi dan digunakan untuk mengira gandaan gentian ringkas untuk setiap sampel di dalam kedua-dua sistem kaca RHSBT didop mikro dan nano erbium. Gandaannya lebih tinggi bagi sistem didop mikro erbium berbanding dengan sistem didop nano erbium. Dengan indeks biasan yang tinggi di antara 2.452 hingga 2.521 dan lebar jalur efektif di dalam jalur S dan C di antara 76 hingga 106 nm, system kaca RHSBT didop mikro erbium memberikan potensi yang baik untuk aplikasi di dalam gentian amplifier didop erbium (EDFA).



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I certify that a Thesis Examination Committee has met on 26 April 2018 to conduct the final examination of Aliyu Umar Sa'ad on his thesis entitled "Structural, Elastic and Optical Properties of Rice Husk Silicate Borotellurite Glass System Doped with Micro and Nanoparticles of Erbium Oxide" 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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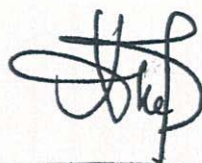
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## LIST OF ABBREVIATIONS

Symbols	Description	Units
RHA	Rice Husk Ash	-
RHS	Rice Husk Silicate	-
XRD	X-ray diffraction	-
FTIR	Fourier transform infrared	-
UV-Vis	Ultraviolet-Visible	-
TEM	Transmission Electron Microscopy	-
BO	Bridging Oxygen	-
NBO	Non-Bridging Oxygen	-
$\rho$	Density	g/cm <sup>3</sup>
$V_m$	Molar Volume	cm <sup>3</sup>
OPD	Oxygen Packing Density	cm <sup>-3</sup>
$V_L$	Longitudinal Velocity	m/s
$V_s$	Shear Velocity	m/s
$L$	Longitudinal Modulus	GPa
$G$	Shear Modulus	GPa
$E$	Young Modulus	GPa
$K$	Bulk Modulus	GPa
$\sigma$	Poisson Ratio	-
$T_s$	Softening Temperature	K
$\theta_D$	Debye Temperature	K
$C_i/V_i$	Packing Density	-
$G_i$	Dissociation Energy	kJ/cm <sup>3</sup>
$N_c$	Bond Number per Unit Formula	-
$F$	Average Stretching Force Constant	(Nm <sup>-1</sup> )
$R$	Bond Length	(nm)

$n_b$	Bond Number per Unit Volume	( $\text{cm}^{-3}$ )
$\alpha$	Coefficient of Absorption	( $\text{cm}^{-1}$ )
$\lambda_c$	Cut-off Wavelength	(nm)
$E_{opt}$	Optical Energy Band Gap	(eV)
$\Delta E$	Urbach Energy	(eV)
$n$	Refractive Index	-
$R_m$	Molar Refractive Index	-
$\alpha_m$	Molar Polarizability	( $\text{\AA}^3$ )
$\alpha_e$	Electronic Polarizability	( $\text{\AA}^3$ )
$\alpha_{O^{2-}}^{(n)}$	Refractive Index Based Oxide Ion Polarizability	( $\text{\AA}^3$ )
$\alpha_{O^{2-}}^{(E)}$	Band Gap Based Oxide Ion Polarizability	( $\text{\AA}^3$ )
$X$	Linear Dielectric Susceptibility	-
$A_{th}$	Optical Basicity	-
$E$	Dielectric Constant	-
$\epsilon_{opt}$	Optical Dielectric Constant	-
$R_i$	Ion-Ion Separation	( $\text{\AA}$ )
$R_p$	Polaron Radius	( $\text{\AA}$ )
$\sigma_{ab}$	Absorption Cross-section	( $\text{cm}^2$ )
$\sigma_{em}$	Emission Cross-Section	( $\text{cm}^2$ )
$g$	Simple Gain	-
$G$	Fiber Gain	dB
$\Delta\lambda_{eff}$	Effective Band Width	nm

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

Over the last decades, tellurite glasses have been under comprehensive investigation due to their high refractive index, better transparency in mid-infrared (0.35–6  $\mu\text{m}$ ), thermal and chemical stability, rare earth (RE) ion solubility, relatively low-phonon energy ( $\sim 700\text{--}800\text{ cm}^{-1}$ ), better glass stability, high dielectric constant and good corrosion resistance. Because of their large refractive index, good semiconducting properties, and low melting point, tellurite glasses as semi-conducting glasses are used for a wide range of applications when containing oxides of ferroelectric materials, transition metal oxides and rare earth oxides (Saddeek, 2005).

Among the oxide glasses, tellurite glasses have advantage over other oxide glasses due also to their good mechanical strength, chemical durability, malleability and low processing temperatures among others (Hesham et al., 2003). High hyper polarity of the lone electron pair in the 5s orbit of the tellurium atom makes the tellurite-based glasses excellent nonlinear optical glasses. Weak Te-O bond in the tellurite based glasses which are easily broken during glass formation gives the ability to accommodate rare earth metal ions and heavy metal oxides (Maheshvaran et al., 2011).

Tellurite glasses have found many applications in non-linear optical devices such as high speed optical- switches, up-conversion frequency systems and others like production of optical fibers and planar waveguides, high performance optics, laser technology and in optical data transmission, detection and sensing (Joshi et al., 2012). Because of their high third order nonlinear susceptibility, tellurium oxides glasses are considered excellent for optical amplifiers (Pavani et al., 2011). Their applications in fields of science, technology and even industrial are informed by their high linear and non-linear refractive indices as well as their physical, chemical and mechanical qualities (Linda et al., 2013).

Because tellurium oxide ( $\text{TeO}_2$ ) alone does not form glass, and is considered a conditional glass former, another oxide is required to form a tellurite based glasses (Gaafar et al., 2014). Previous researches have proven boron oxide ( $\text{B}_2\text{O}_3$ ), another glass former to be the best candidate for combination with tellurium oxide. Normally, borate glasses are considered in glass science and technology because of their high transparency, relatively high thermal stability, good rare earth (RE) ions solubility, low melting point and variable coordination numbers (Kesavulu et al., 2016).

Borate glasses in the face of science and technology has found special attention due to their application in electrochemical and optics. These applications include fabrication of solid-state lasers, optical waveguide, Bio-materials and optical luminescent materials (Janek et al., 2016). In borate glasses, borate matrix  $\text{BO}_3$  triangles and  $\text{BO}_4$  tetrahedra forms, diborate, triborate, tetraborate and so on, which are stable borate groups (Maheshvaran et al., 2013).

To improve the glass quality in terms of thermal and chemical stability, hardness, glass solidification and also the forming ability, boron oxide ( $\text{B}_2\text{O}_3$ ) is used in combination with tellurium oxide ( $\text{TeO}_2$ ) to form what is called borotellurite glass (Hajer et al., 2014). The incorporation of tellurium oxide with boron oxide in the glass network also reduces the hygroscopic nature of the glass (Azlan et al., 2017). Due to their advantage in terms of optical and electrical properties, borotellurite glasses have been excellent in microelectronics and opto-acoustics applications (Gaafar et al., 2014).

Because of their low stability in terms of chemical durability pure borotellurite absorbs atmospheric water vapors to form crystalline precipitates of  $\text{TeO}_2$  in the glass matrix. This informs the need for glass modification to correct or reduce such effect (Azlan et al., 2017). Although they are limited by their low solubility of rare earth ions, low infrared transparency and high phonon energy, silicon oxide glasses possess strong mechanical strength, high laser threshold and good thermal stability. Hence, silicon oxide is a good choice for borotellurite combination for use in optical applications and high gain photonic crystal fibers (PCFs) (Tingting et al., 2016).

Rare earth oxides are used sometimes in high concentrations (depending on the need and application) in oxide glasses for wide range of applications from optical data transmission, detection, sensing to laser technologies and many others (El-Mallawany, 2000). The choice of the RE ions depends on the relationship between the radiative or non-radiative RE ions and the host glass composition (Ashur et al., 2014). Hence, the phonon energy must be low to attend high efficiency in terms of luminescence and the tunability between the host and the dopant ions is necessary. Of the rare earth metal oxide ions, erbium ions were found to be excellent for doping in borotellurite glasses due to their high solubility in the network and unique properties they possess (Azlan et al., 2017).

Through their transition from  $^4\text{S}_{3/2}$  to  $^4\text{I}_{15/2}$  at around  $18300\text{ cm}^{-1}$  and subsequent emission due to the transition,  $\text{Er}^{3+}$  ions are preferable for use in green up-conversion lasers.  $\text{Er}^{3+}$  ions, when excited with 980 nm signal exhibits in the NIR region of 1400-1600 nm an optically transparent window. This window possesses high signal to noise ratio, high detector sensitivity, low auto-fluorescence background, as well as high penetration depth in biological tissues (Annappoorani et al., 2016).

Due to their potentials for applications in the areas of microchip lasers, infrared lasers, medical eye-safe lasers and erbium doped fiber amplifiers (EDFA) in the communication technology. EDFA used in the communication technology (Swapna et al., 2015). Nippon Telephone and Telegraph (NTT) produced a tellurite-based  $\text{Er}^{3+}$  doped fiber amplifiers (EDFA) that showed a gain-flattened amplification of 20 dB across 1530 to 1610 nm wavelength range. Because of the quality in terms of efficiency, tellurite glasses doped with  $\text{Er}^{3+}$  ions are used in many radiative and non-radiative applications as optical fiber amplifiers, optical devices, up-conversion lasers, nonlinear and many others (Sayed et al., 2016).

This work presents the utilization of rice husk (waste from rice milling) to obtain high purity silicon oxide for use in fabrication of silicate borotellurite glasses. The optical, structural and elastic properties of the glasses were studied to obtain the best amount of  $\text{SiO}_2$  for borotellurite glasses. The best proportion of  $\text{SiO}_2$  was used to fabricate erbium and erbium nano-particles doped silicate borotellurite glasses. The optical, structural and elastic properties of the glasses were studied to ascertain the applicability of the glasses from the studied properties.

## 1.2 Problem Statement

Scientists and technologists have been engaged over the years in the study of utilization of tellurite glasses in various areas of science, technology and industry. Since  $\text{TeO}_2$  alone does not form glass, it must be combined with one or more other oxides to form glass (El-Mallawany, 2002). Structural, optical, electrical and elastic properties of tellurium oxide-based glasses have been and are still under study.

The work of Halimah *et al.* (2005) studied the ultrasonic and physical properties of borotellurite glass. From the work, study was made of the proportion of tellurium oxide and boron oxide combination in terms of physical and mechanical properties for the various scientific, technological and industrial applications of the glasses (Halimah et al., 2005).

For their properties that made them promising materials in various applications for photonics and opto-electronics, tellurite based glasses have been given special research attention (Bahari Poor et al., 2013). Applications particularly in fabrication of both active and passive optical fibers as well as high-gain optical amplifiers used in the communication technology has made  $\text{TeO}_2$  based glasses of high research interest (Chillice et al., 2011).

In the report of Damas *et al.*, (2013), tellurium oxide as a host material in glasses have the lowest phonon energy of around  $780 \text{ cm}^{-1}$  with high transparency in the visible, near and mid infrared spectral region. Having refractive indices that are larger than those of quartz, phosphate glasses or borosilicate glasses, they appeared to be better

in terms of losses due scattering and absorption and possesses higher chemical durability (Damas et al., 2012).

The combination of  $B_2O_3$  with  $TeO_2$  in the borotellurite glass represents wonderful compromise between the requirements of the two oxides with each complimenting the other. Thus making it easy to achieve a better quality of low phonon energy and a relatively high thermal stability, high chemical durability and ease of fabrication that none of the two has combined (Maheshvaran et al., 2013). Because of their near white luminescence characteristics,  $Dy^{3+}$  ion doping on tellurite or borotellurite glass medium has been under study nowadays. They are considered very promising as laser active material that emits radiation at around  $\sim 3\mu m$  and in radiation dosimetry, they are used in identifying new materials on glass (Ab et al., 2014).

Different researchers studied different effects of  $Er^{3+}$  ions doping on many different borotellurite glass mediums. Most recent works include the work that study the doping effect of erbium oxide NPs on the green emission, Linear and non-linear optical properties, polarizability and optical basicity and spectral intensity of zinc borotellurite glass (Azlan et al., 2013; Azlan et al., 2014; Azlan et al., 2015; Azlan et al., 2017). The erbium doping effect on optical, elastic and DTA of borotellurite glasses containing lead and zinc were also studied (Sayed et al., 2016).

There was no report found in the literature reporting a study of the optical, structural and elastic properties of erbium or erbium NPs doped rice husk silicate borotellurite glasses. Hence in this work, silicate is extracted from rice husk and utilized to prepare erbium and erbium NPs doped silicate borotellurite glasses for the study of the optical, structural and elastic properties.

### 1.3 Scope of Study

The study includes the use of rice milling waste called “rice husk” to extract high purity silica ( $SiO_2$ ). The percentage purity of the extracted silica was determined using the XRF characterization. The silica extracted was used to fabricate silicate borotellurite glasses using the chemical formula  $[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{1-x}(SiO_2)_x$  with  $x=0.0-0.4$ . The silicate boro tellurite glasses were subjected XRD, FTIR, UV-Vis and Ultrasonic characterization for optical, structural and ultrasonic study to obtain the best  $SiO_2$  proportion for optical glass applications. The best amount of  $SiO_2$  was used to fabricate two series of erbium doped silicate borotellurite and erbium NPs doped silicate borotellurite glasses using the chemical formula  $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{1-x}(SiO_2)_x\}_{1-y}(Er_2O_3)_y$  with  $y=0.01-0.05$  for both the series. The two series were also subjected to XRD, FTIR, UV-Vis characterizations and Photoluminescence spectroscopy to study the erbium or erbium NPs doping effects on the optical, structural and ultrasonic properties of the silicate borotellurite glasses.

## 1.4 Aim and Objectives

The aim of the research is to study the optical, structural and elastic properties of micro and nanoparticle erbium oxide doped rice husk silicate borotellurite glasses. The objectives through which the aim will be achieved are as follows;

1. To extract high purity silicate from rice husk and fabricate silicate borotellurite glass.
2. To study the optical, structural and elastic properties of silicate (RHA) borotellurite glasses.
3. To fabricate micro/nanoparticle erbium oxide doped glasses using the best Silicate boro-tellurite proportion obtained and study the structural, optical and elastic properties of the two glass systems.
4. To determine the applicability of the two erbium oxide doped series in the EDFA technology using the McCumber theory.

## 1.5 Hypotheses

High purity  $\text{SiO}_2$  can be extracted from the rice husk using the simple cold acid leaching method (Abu, Yahya, & Neon, 2016; Sharifnasab & Alamooti, 2017).

The pattern of glass XRD exhibits no sharp peaks but a broad hump indicating an amorphous nature of the material (Tagiara et al., 2017)

The FTIR spectra for silicate borotellurite glass system shows structural units such as  $\text{SiO}_4$ ,  $\text{TeO}_4$ ,  $\text{TeO}_3$ ,  $\text{BO}_4$ ,  $\text{BO}_3$  units.

The use of silicate in a borotellurite glass system improves the glass mechanical quality and thermal resistance (Oana et al., 2017).

$\text{Er}_2\text{O}_3$  and  $\text{Er}_2\text{O}_3$  NPs doping on silicate borotellurite glass system might increase the elastic moduli of the glass by increasing the rigidity of the glasses through increase in network connectivity (Yousef, 2013).

The Optical band gap of the  $\text{Er}_2\text{O}_3$  and  $\text{Er}_2\text{O}_3$  NPs doped silicate borotellurite glasses might increase with increase in the  $\text{Er}_2\text{O}_3$  content. This is due to the possible changes in the glass structure leading to the formation of bridging oxygens (BOs)(Yousef, 2013). The glasses are expected to have high refractive index as the presence of  $\text{TeO}_2$  enhances refractive index.

The  $\text{Er}_2\text{O}_3$  and  $\text{Er}_2\text{O}_3$  NPs doped glasses are expected to have good signal power amplification ability for fiber optics application. This is expected as the use of rice



husk ash and borotellurite is expected to improve both optical and mechanical quality of the glasses.

## 1.6 Significance of the Study

The study was carried out to study the optical, structural and the elastic properties of erbium and erbium doped silicate borotellurite glasses. With over 120 tonnes of rice husk produced globally every year without proper management, the world is risk of environmental pollution and land damages (Mohanta et al., 2012). Various scientific and technological approaches are being employed by scientist to manage and recycle the agricultural waste and control the possible environmental hazard that may be caused. These includes the use of the rice husk in cement production (Khan et al., 2014), as an alternative fuel in power generation plants, as a raw material for production of silica ( $\text{SiO}_2$ ) and silicon compounds in chemical industries (Kumar et al., 2013) and in the production of activated carbon (Abu et al., 2016). The utilization will there for bring about ease in disposal and save cost (Nagrале et al., 2012). Erbium doped glasses has been under study due their variety of applications in optical data transmission and optical fiber amplifiers. The work attempts to determine the possibility of utilization of rice husk in the fabrication of glasses for optical glass technology.  $\text{SiO}_2$  based glasses are known to have chemical durability, thermal stability, high viscosity and optical transparency at excitation and lasing wavelength (Berwal et al., 2017). Hence, the rice husk silicate ( $\text{SiO}_2$ ) was employed to help in managing the rice milling waste and to improve the physical, mechanical, optical and life span of the erbium doped borotellurite glasses used in the fabrication optical fiber and optical fiber amplifiers (Sayed et al., 2016).

To improve the glass quality in terms of thermal and chemical stability, hardness, glass solidification and also the forming ability, boron oxide ( $\text{B}_2\text{O}_3$ ) is used in combination with tellurium oxide ( $\text{TeO}_2$ ) to form what is called borotellurite glass (Hajer et al., 2014). The incorporation of tellurium oxide with boron oxide in the glass network also reduces the hygroscopic nature of the glass (Azlan et al., 2017). Due to their advantage in terms of optical and electrical properties, borotellurite glasses have been excellent in microelectronics and opto-acoustics applications (Gaafar et al., 2014).

## 1.7 Thesis Organization

The thesis consists of six chapters, with each chapter describing the sequence of study as described as follows;

**Chapter 1** shows the introduction to tellurite, borotellurite, silicate and erbium doped glasses with their applications in the background. It contains the problem statement, scope of the study, aim and objective, significance of study and thesis organization. **Chapter 2** shows an over view of related literatures on rice husk, glass, tellurite

glasses, borate glasses, silicate glasses, borotellurite glasses, doping and erbium doped glasses and applications. **Chapter 3** describes the principles and theories employed in the study. The theories applied in some of the characterizations as XRF, XRD, FTIR, UV-Vis, Photoluminescence and the Ultrasonic non-destructive characterizations. The chapter also shows the theory used in density measurement. Also discussed here is the concept of glass free volumes, molar crystalline volume, molar crystalline volume based free volume, molar ionic volume, molar ionic volume based free space/ glass cubic interstitial space and the glass degree of compactness. **Chapter 4** describes the materials, methods and the experimental techniques employed in the study. This includes the rice husk ash (silica) extraction and XRF characterization, Silicate borotellurite glass fabrication, density measurements and characterizations and Erbium/Erbium NPs dope silicate borotellurite glass fabrication, density measurements and characterizations. Also mentioned are the equipment and chemicals used in the process of the study. **Chapter 5** presents and discusses the results from the XRF analysis result, through the optical, structural and elastic results of the various characterizations, measurements and calculation carried out on the three series of glass under study. **Chapter 6** presents the overall conclusion based on the obtained results and gives recommendations on some future study in the field.

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