UNIVERSITI PUTRA MALAYSIA

OPTICAL, PHYSICAL AND STRUCTURAL PROPERTIES OF ZINC-BOROTELLURITE GLASS DOPED WITH SAMARIUM OXIDE AND SAMARIUM OXIDE NANOPARTICLES

HAJER SAAD SALEH ABUZLILIA

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By

HAJER SAAD SALEH ABUZLILIA

Thesis is Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

July 2015

DEDICATION

Always, there have been some hard circumstances during my life which were relieved by love, patient, blessing, encouragement and sacrifice. To evince my feeling, I would like to dedicate my dissertation work to my darling husband for his love and patient, my lovely children for making my life complete, my precious sisters for their blessing, my gentle brothers for their encouragement. And to my devoted parents which nothing can express my gratitude toward their love, patient, blessing, encouragement, sacrifice and all supports.

Abstract of thesis presented to the senate of Universiti Putra Malaysia, in Fulfilment of the requirements for the degree of Master of Science

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Chairman: Assoc. Prof. Halimah Mohamed Kamari, PhD.

Faculty: Science

Tellurite glasses are very promising materials for laser and non-linear applications in optics, due to some of their important characteristic features, such as high refractive index, low phonon maxima and low melting point. Addition of small amount of TeO_2 into borate glass network enhance glass quality with an improvement in transparency and refractive index. Addition of ZnO into boro-tellurite glass network produce low rates of crystallization and increase glass forming ability. When other metals oxides are added in tellurite glass system, it can enhance the optical and physical behaviour. It is known that there is only a few reasearch on samarium and samarium nanoparticles doped tellurite glass system. Samarium oxide has high potential for optical applications. Meanwhile, samarium nanoparticles affect the optical properties of the glass system which is due to their smaller size of particles. For this research, two series of zinc-borotellurite glass: Samarium oxide and Samarium oxide nanoparticles doped zinc-borotellurite glass were successfully synthesized using a conventional melt-quenching method. XRD results for both Samarium oxide and Samarium oxide nanoparticles doped zinc-borotellurite glass show that the glasses are in amorphous phase. The physical and optical properties measurements were investigated for both series of zinc-borotellurite glass system.

In case of Samarium doped zinc-borotellurite glass system, the density of glass samples was in the range of 3.693-4.285 kg/m^3 and was found to be increased with an increasing content of samarium which is due to the formation of non-bridging oxygen. The molar volume of these glasses was in the range of 31.689-30 057 m^3 /mol and it was increasesed which is due to the large value of ionic radii and bond length of Sm_2O_3 compared to TeO_2 and increasing in oxygen packing density which results the structure becomes more compact. The non-

linear trend of refractive index 1.871-2.005 and polarizability 5.716-5.981×$10^{-24}$$cm^3$ are due to the substitution of ZnO oxides which acts as a modifier and former at certain values of mol %. The FTIR analysis consists of several bands which indicate the characteristic of Te-O and B-O vibrational groups. The optical absorption spectra of these glasses revealed that fundamental absorption edge shifts to longer wavelength as the content of Sm_2O_3 increases. The decreasing value of the band gap energy from 2.780 eV to 2.528 eV is due to the increasing number of non-bridging oxygen. The value of Urbach energy was in the range 0.689-0.660 eV and it was found to be increased with increasing content of samarium. This is due to the increasing number of TeO_4 pyramids as the content of Sm_2O_3 oxide increases. The presence of TeO_4 pyramids results the structure to become less stable.

In case of Samarium nanoparticles doped zinc-borotellurite glass system, the density of the prepared glass samples was found in the range 3.644-4.080 kg/m^3 and it was to be increased with an increase content of samarium nanoparticles which is due to the low solubility of samarium nanoparticles. The value of molar volume of these glasses was in the range 32.515-31.570 m^3/mol and it was found to be decreased with increasing content of samarium nanoparticles because of the dual nature of zinc oxide. The range of refractive index of these glasses are 2.056-2.051 and it was decreased to 0.02 mol% of Sm_2O_3 NPs which is due to the restriction movement of the electrons with respect to their small size of particles. The increasing trend of refractive index at 5% of samarium nanoparticles is due to the increasing number of non-bridging oxygen. The presence of Sm_2O_3 nanoparticles was confirmed by using TEM technique with the average size of 22.5 nm. The FTIR spectra revealed the shifting and new absorption peak in the presence of nanoparticles. The absorption edge is found to be shifted to longer wavelength. The decreasing value of the band gap energy from 2.956 eV to 2.980 eV is due to the broadening of the valence band or multivalence structures in the presence of Sm electrons. The value of Urbach energy is 0.361-0.354 eV and it was found to be increased with increasing concentration of samarium nanoparticles. It is known that materials which possess large value of Urbach energy have higher tendency to convert the weak bonds into defects.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SIFAT OPTIK, FIZIKAL DAN STRUKTUR KACA ZINK-BOROTELURIT DOP DENGAN SAMARIUM OKSIDA DAN NANOPARTIKEL SAMARIUM OKSIDA

Oleh

HAJER SAAD SALEH ABUZLILIA

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Kaca telurit adalah bahan yang sangat berpotensi untuk aplikasi laser dan optik tidak linear, disebabkan oleh sebahagian ciri-ciri yang penting padanya seperti indeks biasan yang tinggi, tenaga fonon yang rendah dan takat lebur yang rendah. Penambahan kadar jumlah TeO_2 yang rendah ke dalam sistem kaca borat meningkatkan kualiti kaca dengan penambahbaikan di dalam transparensi dan indeks biasan. Penambahan ZnO ke dalam sistem kaca boro-telurit menghasilkan kadar penghabluran yang rendah dan meningkatkan kebolehan pembentukkan kaca. Apabila logam oksida yang lain di masukkan ke dalam sistem kaca telurit, ianya boleh meningkatkan sifat optik dan fizikal. Telah diketahui bahawa hanya sedikit sahaja kajian mengenai samarium dan samarium nanopartikel terdop sistem kaca zink-borotellurit. Samarium oksida memiliki potensi yang tinggi untuk aplikasi optik. Manakala, samarium nanopartikel memberi kesan kepada sifat optik sistem kaca zink-borotellurit yang disebabkan oleh saiz partikel yang kecil. Bagi kajian ini, dua siri kaca zink-borotelurit telah berjaya disintesis dengan menggunakan kaedah lebur lindap. Keputusan XRD untuk kedua-dua samarium dan samarium nanopartikel terdop kaca zink-borotelurit menunjukkan bahawa kaca tersebut adalah di dalam fasa amorfus. Pengukuran sifat fizikal dan optik telah dikaji untuk kedua-dua sistem kaca zink-borotelurit.

Bagi sistem kaca samarium dop zink-borotelurit, ketumpatan sampel kaca di dalam julat 3.693-4.285 kg/m^3 di dapati meningkat dengan penambahan kandungan samarium yang disebabkan oleh pembentukkan bukan penyambung oksigen. Isipadu molar bagi kesemua kaca di dalam julat 1.689- 30.057 m^3/mol telah meningkat disebabkan oleh nilai jejari ionik yang besar dan stuktur panjang ikatan Sm_2O_3 berbanding TeO_2 dan peningkatan tumpat kepadatan oksigen yang menghasilkan struktur yang lebih padat. Corak indeks biasan 1.871-2.005

yang tidak linear dan kebolehan berkutub 5.716-5.981×10^{-24} cm^3 adalah disebabkan oleh penggantian ZnO oksida yang bertindak sebagai pengubah dan pembentuk pada nilai mol% yang tertentu. Analisa FTIR merangkumi beberapa jalur yang menunjukkan ciri-ciri Te-O dan B-O kumpulan bergetar. Spektra penyerapan optik pada kaca-kaca ini menunjukkan bahawa pinggir asas penyerapan beralih kepada gelombang yang lebih tinggi apabila kandungan Sm_2O_3 bertambah. Penurunan nilai jurang jalur tenaga dari 2.780 eV ke 2.528 eV adalah disebabkan oleh peningkatan nilai bukan penyambung oksigen. Nilai tenaga Urbach adalah di dalam julat 0.689-0.660 eV telah didapati bertambah dengan penambahan kandungan samarium. Ini disebabkan oleh peningkatan jumlah piramid TeO_4 apabila kandungan Sm_2O_3 bertambah. Kehadiran piramid TeO_4 menyebabkan struktur menjadi tidak stabil.

Bagi sistem kaca samarium nanopartikle dop zink-borotelurit, ketumpatan telah didapati ditemui di dalam julat 3.644-4.080 kg/m^3 dan menunjukkan bertambah dengan penambahan kandungan samarium nanopartikel disebabkan oleh kelarutan samarium nanopartikel yang rendah. Isipadu molar bagi kaca-kaca adalah di dalam julat kadar 32.515-31.570 m^3/mol dan telah didapati berkurang dengan penambahan kandungan samarium nanopartikel yang disebabkan oleh sifat semula jadi pendua bagi zink oksida. Indeks biasan bagi kesemua kaca-kaca adalah di dalam julat 2.056-2.051 dan telah menunjukkan berkurangan sehingga 0.02 mol% Sm_2O_3 nanopartikel. Corak peningkatan bagi indeks biasan pada 5% samarium nanopartikle adalah disebabkan oleh peningkatan nilai bukan penyambung oksigen. Kehadiran Sm_2O_3 nanopartikel telah dipastikan dengan menggunakan teknik TEM dengan saiz purata 22.5 nm. Spektra FITR telah menunjukkan peralihan dan puncak penyerapan yang baru di dalam kehadiran nanopartikel. Pinggir penyerapan telah didapati beralih kepada gelombang yang lebih panjang. Penurunan nilai jurang jalur tenaga dari 2.956 eV ke 2.980eV adalah disebabkan oleh pelebaran jalur valens atau struktur berbilang valens di dalam kehadiran elektron Sm^{3+}. Nilai tenaga Urbach adalah 0.361-0.354 eV dan telah didapati bertambah dengan penambahan kepekatan samarium nanopartikel. Telah diketahui bahawa bahan yang memiliki jumlah tenaga Urbach yang besar memiliki potensi yang tinggi untuk menukarkan ikatan yang lemah kepada kecacatan.

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I certify that an Examination Committee has met on 13 July 2015 to conduct the final examination of HAJER SAAD SALEH ABUZLILIA on her Master thesis entitled "Optical, Physical and Structural Properties of Zinc-Borotellurite Glass doped With Samarium oxide and Samarium oxide Nanoparticles" 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 Master of Science.

Members of the Examination Committee were as follows:

Chen Soo Kien, PhD
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Date: 22 September 2015

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirements for the degree of Master of Science. The members of the supervisory Committee were as follows:

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Date: 13/07/2015

DECLARATION

Declaration by graduate student

I hereby confirm that:

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- the research conducted and the writing of this thesis was under our supervision;
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Name of Chairman of Supervisory Committee: Associate professor. Halimah Mohamed Kamari, PhD

Signature:
Name of Member of Supervisory Committee: Professor. Azmi Zakaria, Phd

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LIST OF ABBREVIATIONS

TeO_2	Tellurite oxide	-
B_2O_3	Boron oxide	-
ZnO	Zinc oxide	-
Sm_2O_3	Samarium oxide	-
Sm_2O_3 NPs	Samarium oxide nanoparticles	-
NBO	Non-bridging oxygen	-
BO	Bridging oxygen	-
XRD	X-ray diffraction	-
FTIR	Fourier transform infrared	-
d	Interplanar distance	m
ɵ	Scattering angle	°
λ	Wavelength	m
n	Refractive index	-
θ_i	Angle of incidence	°
θ_r	Angle of refraction for a beam of light	°
α_e	Polarizability	cm^3
V_m	Molar volume	m^3/mol
N	Avogadro number	-
ρ_{sample}	Density of sample	g/cm^3
M	Molecular weight of the material	-
R_M	Molar refraction	cm^3
α	Absorption coefficient	cm^{-1}
dI	Fraction absorption of light	eV
dx	Thickness	m
T	Transmission	%
A	Absorbance	-
I	Intensity of light back of the flow cell is defined as reference light	-
I_0	Intensity of light which pass through the flow cell	-
$\hbar\omega$	Energy of the incident photon	eV
ħ	Plank constant	-
E_{opt}	Optical band gap energy	eV
B	A constant	-
ΔE	Band tail of the electron stated	eV
ρ_L	Density of distill water	1.0 g/m^3
M_{air}	Mass in air	g
M_{water}	Mass in distill water	g

CHAPTER 1

INTRODUCTION

1.1 Overview

Glasses have distinctive properties as compared to other matters. It is high in hardness and strength but transparent at room temperature. At the same time glasses have excellent resistance to corrosiveness. Glass has a significant impact in the research field as it has various potential and applications in engineering, technology as well as day to day applications. In other words, it is worthwhile to do research based on the properties of glass in order to explore and unveil the potential of glass applications. There are many advantages in using glassy materials such as the absence of grain boundaries, the ability in continuous variable composition which perfectly making them prominent in optical applications.

The types of glass that is considered the oldest in history are based on chemical compound silica (silica dioxide), the main component in sand. Glass is the common term used to describe the material that is usually used as window glass and bottles.

Most silica-free glass like fluoride glasses, aluminosilicates, borate glasses, phosphate glasses, and glasses may contain physico-chemical properties that make it suitable for usage in fiber optics and various specific technical applications.

The three types of oxide glasses are network intermediates, formers, and modifiers. Network intermediates like zirconium, aluminium, beryllium, titanium, magnesium, zinc play the role of network formers and modifiers, based on the composition of the glass. Network formers like boron, silicon and germanium produce an extremely cross-linked chemical bonds network. Modifiers like calcium, lithium, lead, sodium, potassium generally exist as ions modifying the arrangement of the network. They are compensated by adjacent non-bridging oxygen atoms which were bound by one covalent bond to the glass network and holding one negative charge to compensate for the positive ion. Some elements can perform numerous roles. For example, lead can act both as a network former (Pb^{4+} replacing Si^{4+}), or as a modifier.

The non-bridging oxygen reduces the number of strong bonds relatively in the material and disrupts the glass network system. The viscidness of the melt decreases and lowers the melting temperature.

Glasses are a unique range of ceramic materials defined principally by their atomic structure. Glasses do not display the ordered crystalline structure of most other ceramics but instead have a highly disordered amorphous structure. This gives them very diverse properties to other crystalline ceramics. The most widely utilised glasses are silicate glasses, formed from silica, SiO_2. Silica consists of a 3D network of tetrahedral where every corner oxygen atom is shared with the adjacent tetrahedron. This SiO_2 tetrahedral unit is also incorporated into chains and sheets (clays), forming diverse ceramics as shown in Figure 1.1.

Pure silica can be made to exist as a glass, and is called fused silica. A glass is a material that has hardened and become rigid without crystallising, making it amorphous. Silicate glasses are the most widely used glasses. All of the most important glasses are based on silica SiO_2. Two glasses of special interest are soda-lime glass, used in bottles and windows, with composition $70SiO_2.10CaO.15Na_2O$, and borosilicate glass, used in chemical glassware and cooking, with composition $80SiO_2.15B_2O_3.5Na_2O$.

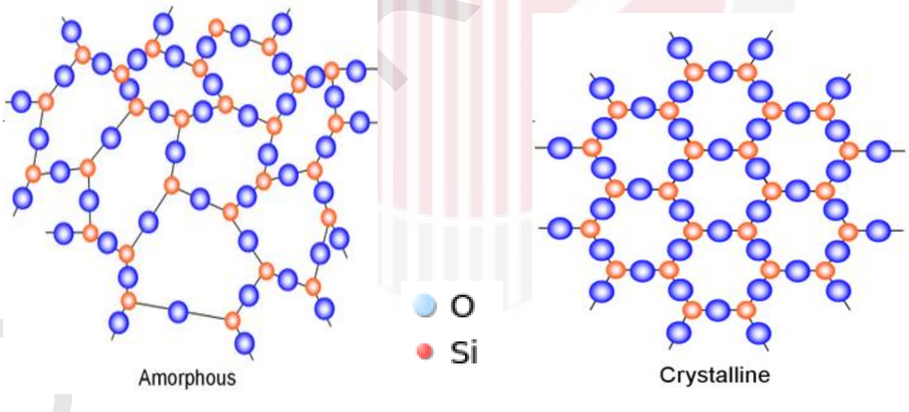

Figure 1.1: The most widely used glasses are silicate glasses, formed from silica, SiO_2. Silica consists of a 3D network of tetrahedral where every corner oxygen atom is shared with the adjacent tetrahedron. This SiO_2 tetrahedral unit is also incorporated into chains and sheets (clays), forming different ceramics.

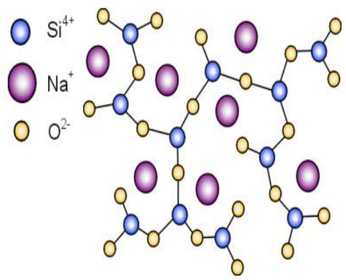

Figure 1.2: Glass with added sodium as a network modifier.

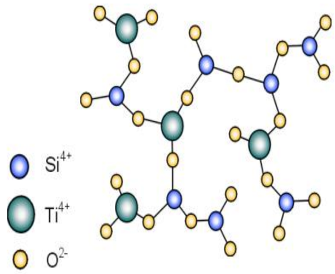

Figure 1.3: Glass with added titanium as an intermediate.

The ability of an oxide to form a glass upon cooling, i.e. an amorphous structure, depends on the structural relationship between the oxygen atoms and the cations of the oxide compound. In order to achieve a glass structure the oxide cations will bind with the oxygen atoms to form a tetrahedral network.

Certain other oxides can be added to substitute for Si atoms in the tetrahedral structure. These oxides become part of the network and act as a stabiliser as exposed in Figure 1.2 and Figure1.3.

Tellurite based glasses are a fascinating system to study as the glass phase can be moulded over a huge range of concentration in addition to its exceptional properties. Tellurite glass have been researched on for more than150 years. However TeO_2 glasses of purities exceeding 98.5 mol% have been made recently. (El-Mallawany, 1998).

Tellurite glass used in electric, optical, electronic industries and other areas are vast due to their good physical properties. Tellurite glasses have been used broadly for laser as well as non-linear applications in optics. They have some excellent features which are required in the optical application such as low phonon maxima, high refractive index allowing infrared radiation at high levels conducted and thus applicable as materials for optical devices and lasers or photochromic glasses, as well as low melting point. The non-appearance of hygroscopic properties restricts phosphate and borate glasses application. Furthermore, they own high density and low transformed temperature (El-

Mallawany, 1998). In glass formation process, TeO_2 needs a modifier to enhance the glass formation process easily. In other word, TeO_2 is a conditional glass former. The glass formation process on two glass formers such as Tellurite glass has been a focus of interest from both practical and academic perspective whereby the technical applications and microscopic mechanism are studied for research and industrial undertakings. In addition, researchers are also optimistic on the possibility on new structural formation and discovery in glass research which potentially brings more breakthroughs in glass technology.

Borate oxide is commonly used to solidify the glass and strengthen the glass quality with better reflective index, amelioration in transparency and even rare earth ion solubility and hardness. The formation of borate matrix that consists of BO_3 triangles and BO_4 tetrahedra create stable borate groups form for instance diborate, triborate and tetraborate.

Heavy metal oxides in borate glasses require specific care owing to the glass' applications like laser hosts, lamp phosphors and other photonic devices. Alkaline earth borate glasses contain heavy metal oxides display rare-earth ions good solubility. TeO_2 glasses give fascinating non-linear optical properties, good chemical durability, and low melting point. Alkaline earth oxides increase glass forming ability whereas heavy metal oxides intensify the optical properties' ability of the second harmonic generation. Glass formation ability are the region, stability, chemical durability of the glass forming oxide materials like B_2O_3, SiO_2, GeO_2, P_2O_5, As_2O_3 etc, can be boosted with the addition of metal oxides such as ZnO, TeO_2, PbO, Bi_2O_3 etc, which are called conditional glass modifiers.

Zinc oxide or zinc white is the white hexagonal crystal or white powder. It is odourless, has a better taste and non-water soluble. ZnO tolerates high heat and conductivity. The low coefficient of expansion is advantage in ceramic body (Porter, 1991). Moreover, ZnO is an n-type semiconductor with band gap energy of approximately 3.2 eV (Pearton et al., 2003). It shows both photoconductivity and photocatalytic activity (Liu et al., 2001) as a vital semiconductor material, ZnO is applied in catalysis (King and Nix, 1996), rubber and paint industries, ceramics bodies, fertilizers and in cosmetics (Porter, 1991). The addition of zinc oxide in glass formation process is important to decrease the melting point, reduce the rate of crystallization and finally increase the glass forming ability. Past studies have proven that the zinc oxide can decrease the optical energy gap and thus increase the refractive index.

However, the physical properties of glasses might exhibit discontinuous changes when structural changes of cation happens after the addition of zinc oxide as it able to fill up the network forming and modify the position in borate network glasses. Depending on the application, this property is a functional property.

In year 2013, Sailaja et al. published that Samarium (Sm), one of the rare earth ions can be utilised as a dopant in various crystal hosts. Besides, Samarium (Sm) is also used as glass hosts for intense emissions in the visible region. Samarium especially the reddish orange emission region from Sm-doped materials is prominently used in laser applications since it has properties such as strong luminescence intensity, large emission cross section and high quantum efficiency. In addition, Samarium is used widely in fluorescence applications for its $^4G_{5/2}$ level that is high in quantum efficiency.

At the same time, samarium oxide (Sm_2O_3) is widely used in electrical and optical application and has greatly contributed in lasers, neutron capture, masers, magnets, thermoelectric devices productions. More attention is paid to the nano size samarium oxide compared to the micron size samarium oxide because of the nano size effects that give rise to better interfacial influence, quantum and tunnelling effects. Nanoparticles are known to have unique quantum nature which modifies the materials properties. Nowadays, nanotechnology research has focused on enhancing the properties of materials. It is strongly essential to lengthen the research on doped-glass nanoparticles to acquire exceptional photonic devices properties. The nanomaterials exceptional organization has opened up a new dimension for development of nanotechnology applications. The nano size particles have been known to give important effect on semiconductor due to their dependency on size particles.

Due to its vast application and functions Rare Earth doped materials have the market value driven motivation to develop photonic system for even more applications. Samarium oxide could be one of the best choices from the various lanthanide groups in these photonic materials studies. The trivalent electron in Samarium has the exact properties needed for the fiber amplifier applications. However, there is no recent research reported to the best of our knowledge on Sm_2O_3 nanoparticles doped tellurite glass system. Thus, this is a motivation to conduct a study on this potential system in samarium nanoparticles doped Tellurite glasses.

1.2 Problem statement

Rare earth doped glasses have successfully gathered much attention in research and development due to their potentials in optical area. It is well known that rare earth materials have excellent potential in optical switches as well as optical communications. There are extensive research on erbium doped tellurite glass since it has good optical properties. Meanwhile, samarium oxide is an excellent materials for optical applications especially to enhance the properties of optical switches and communications. There is only a few research have been conducted for samarium oxide doped tellurite glass system. Moreover, the interest of nanoparticles to improve the optical properties of tellurite glass is in high demand. Hence, it is important to investigate the optical properties of samarium and samarium nanoparticles doped tellurite glass system.

1.3 Objective of research

The aims of this study are:

a. To fabricate the quaternary composition glass: $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}\}]_{0.7}$ $[ZnO]_{0.3}\}_{1-x}\{Sm_2O_3\}_x$ where : x= 0, 0.005, 0.01, 0.02, 0.03, 0.04, 0.05, and $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}\}]_{0.7}$ $[ZnO]_{0.3}\}_{1-x}\{Sm_2O_3$ NPs$\}_x$, where: x=0.005, 0.01, 0.02, 0.03, 0.04, 0.05.
b. To determine the structural and optical properties of Samarium and Samarium nanoparticles doped zinc borortellurite.

1.4 Thesis outline

This thesis paper consists of six chapters whereby chapter one is the introduction to glasses and material background, followed by problem statement, definition of scope and objective of research. Second chapter reinstated literature review of the study where important findings and properties that are published by past researchers are discussed and related to this study. Third chapter explains further on the background of the glass properties including the theory of structural, physical and optical properties measurements. Fourth chapter discusses the methodology of the study whereby the procedure of glass sample preparation methods and related tools used for the synthesis and characterization of glasses are reported. Fifth chapter interprets the results obtained and further discussion on the parameter of physical properties, structure properties and optical properties of the study. Lastly, sixth chapter summarizes the conclusion of the study and suggestions for future work.

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