***EFFECT OF HEAT TREATMENT ON OPTICAL, ELASTIC AND THERMAL PROPERTIES OF SILICA BOROTELLURITE GLASS DOPED WITH MANGANESE***

**ZAITIZILA BINTI ISMAIL**

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By

**ZAITIZILA BINTI ISMAIL**

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

**April 2019**

Abstract of 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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**Chairman : Professor Halimah Mohamed Kamari, PhD**
**Faculty : Science**

The properties and applications of various glass materials have been improving in daily life. Rice husk is the major source of biomass waste and offer an immense potential to create bioenergy. Rice husk have been used as a source of silica in this research. In order to unveil the effect of heat treatment and manganese on physical, structural, optical, elastic and thermal properties of silica borotellurite glass system, the quaternary glass system $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.8}(SiO_2)_{0.2}\}_{1-x}(MnO_2)_x$ where x = 0.00, 0.01, 0.02, 0.03, 0.04 and 0.05 molar fraction were successfully fabricated through melt quenching technique. The physical, structural, optical, elastic, and thermal properties of the glass samples were investigated by using densimeter, X-ray Diffraction (XRD) spectrometer, Fourier Transform Infrared Spectrometer (FTIR), ultrasound technique and UV-Visible Spectrophotometer (UV-VIS) as well as Differential Scanning Calorimetry (DSC). The samples underwent heat treatment process at temperature of 250 ℃, 350 ℃, 450 ℃ and 600 ℃ for 3 hours. The density of the glass is found to decrease for samples before and after heat treatment as more dopant were added into the glass system which suggest the substitution of a heavier element in the glass system by a lighter dopant. Meanwhile, the molar volume of fabricated glass increases as the concentration of manganese increase due to the growth of free volume in the glass network. The minimum and maximum values for molar volume are $2.823 \times 10^{-5}$ and $3.141 \times 10^{-5}$ $m^3$/mol respectively. Structural unit of $TeO_4$, $TeO_3$, $BO_4$ and $BO_3$ were detected in samples before and after heat treatment procedure while Mn structural unit was only found in samples after heat treatment process at 600 ℃. The detection of Mn structural unit indicates that heat treatment procedure causes the glass network start to restructure. The amorphous and crystalline nature of the glass is confirmed by using XRD analysis. SEM images show the agglomeration of the particle after heat treatment process. In terms of optical band gap, refractive index, and Urbach energy, all of the samples possess the same trends as well as some variations in the values as more dopants are added into silica borotellurite glass system. The inconsistent values as well as trends for the investigated parameter

suggest that the formation of bridging and non-bridging oxygen take place in the glass system. The largest value of refractive index is 3.57 while the smallest Urbach energy value among all the synthesized samples is 0.39 eV. The current value of refractive index in the market is 1.98. The small Urbach energy hints the less disorderness in the glass structure. On the other hand, the elastic moduli and other elastic parameters of the prepared glass samples are found to be inconsistent with the addition of more manganese element in the silica borotellurite glass system. The decreasing trends in the ultrasonic velocities have contributed to the decrement in rigidity and compactness of the glass system. The value of Poisson's ratio indicated that the glass sample have high cross-link density since the values lie on the range 0.1 to 0.2 for glass samples before and after heat treatment glass samples. Furthermore, the thermal properties of the glasses show that the glass sample have increasing thermal stability and glass transition temperature with the introduction of manganese in the silica borotellurite glass system. The increment in both thermal stability as well as glass transition temperature after the addition of manganese proved that the introduces dopant increased the rigidity and the stiffness of the bond in the glass network. The prepared glass samples can be categorized as strong glass former since the fragility index for samples before and after heat treatment lies in the range of 0.84 to 0.90. In conclusion, the addition of manganese and the heat treatment process done on the silica borotellurite glass have altered the properties of the glasses in different ways. This research is able to provide new knowledge regarding transition metal doped silica borotellurite glass and effect of heat treatment on the glass system. The results from the refractive index suggests that silica borotellurite glass doped with manganese have potential to be used as UV absorbance glass.

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

**KESAN RAWATAN HABA KEPADA SIFAT OPTIK, KENYAL DAN TERMA BAGI KACA SILIKA BOROTELLURIT TERDOP DENGAN MANGAN**

Oleh

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Sistem kaca kauter $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.8}[SiO_2]_{0.2}\}_{1-x}\{MnO_2\}_x$ dimana x = 0.00, 0.01, 0.02, 0.03, 0.04 and 0.05 pecahan molar telah berjaya dihasilkan menggunakan teknik lebur lindap. Sekam padi adalah sumber utama sisa biomass dan menawarkan potensi besar untuk menghasilkan biotenaga. Sekam padi telah digunakan sebagai sumber silika di dalam kajian ini. Sifat fizikal, struktur, optik, kenyal dan terma sampel kaca diuji dengan menggunakan analisis pembelauan sinar-X (XRD), Spektrometer Inframerah Jelmaan Fourier (FTIR), teknik ultrabunyi dan Spektofotometer UV-nampak (UV-VIS), dan Kalorimeter Imbasan Perbezaan (DSC). Sampel menjalani proses pengolahan haba pada suhu 250 ℃, 350 ℃, 450 ℃ dan 600 ℃ selama 3 jam. Kepadatan sampel kaca didapati menurun untuk sampel sebelum dan selepas pengolahan haba apabila lebih dopan ditambahkan ke dalam sistem kaca di mana mencadangkan bahawa penggantian unsur yang lebih berat dengan dopan yang lebih ringan di dalam sistem kaca. Sementara itu, isipadu molar kaca bertambah apabila pertambahan dopan mangan kerana pertumbuhan isipadu bebas dalam rangkaian kaca. Nilai minima dan maksima untuk isipadu molar masing-masing adalah 2.823 x $10^{-5}$ dan 3.141 x $10^{-5}$ $m^3$/mol. Unit struktur $TeO_4$, $TeO_3$, $BO_4$ and $BO_3$ dapat dikesan sebelum dan selepas prosedur pengolahan haba sementara struktur unit Mn hanya dijumpai dalam sampel selepas rawatan haba pada suhu 600 ℃. Pengesanan unit struktur Mn menunjukkan pengolahan haba menyebabkan rantaian kaca mula meyusun semula. Sifat amorfus dan kristal pada sampel kaca dapat dipastikan dengan menggunakan analisis XRD. Imej SEM menunjukkan penggumpulan partikel berlaku selepas proses pengolahan haba. Dari segi jurang jalur optik, indeks biasan, dan tenaga Urbach, kesemua sampel mempamerkan variasi yang sama dan juga beberapa variasi dalam nilai apabila dopan ditambahkan di dalam sistem kaca silika borotelurit. Ketidakseragaman nilai dan juga trend untuk parameter yang dikaji mencadangkan pembentukan penitian oksigen dan penitian bukan oksigen berlaku dalam sistem kaca. Nilai terbesar indeks biasan adalah 3.57 sementara nilai terendah tenaga Urbach di antara semua sampel yang disintesis adalah 0.39 eV. Nilai indeks biasan di pasaran pada masa sekarang adalah 1.98. Nilai yang rendah pada tenaga Urbach

menunjukkan pengurangan dalam ketidak seragaman dalam struktur kaca. Selain itu, modulus kenyal dan parameter kenyal yang lain pada kaca didapati tidak seragam dengan pertambahan unsur mangan di dalam sistem silika kaca borotelurit. Penurunan trend pada halaju ultrasonik menyumbang pada pengurangan ketegaran dan kepadatan sistem sampel kaca. Nilai nisbah Poisson's menunjukkan bahawa sampel kaca mempunyai kepadatan tautan silang yang tinggi di mana berada pada julat 0.1 dan 0.2 untuk sebelum dan selepas pengolahan haba sampel kaca. Tambahan pula, sifat terma pada kaca menunjukkan pertambahan dalam kestabilan terma dan suhu peralihan kaca dengan pengenalan kandungan mangan di dalam sistem silika borotelurit kaca. Pertambahan dalam kedua-dua kestabilan terma dan juga suhu peralihan kaca selepas penambahan mangan membuktikan pengenalan pendopan menaikan ketegaran dan kekakuan ikatan dalam rantaian kaca. Sampel kaca boleh dikategorikan sebagai pembentukan kaca kuat untuk sebelum dan selepas pengolahan haba di mana nilai index kerapuhan sampel sebelum dan selepas pengolahan haba berada dalam nilai 0.84 dengan 0.90. Kesimpulannya, penambahan mangan dan proses pengolahan haba dijalankan di atas silika borotelurit kaca telah mengubah ciri-ciri kaca dengan cara yang berbeza. Kajian ini boleh menyumbang ilmu baharu mengenai logam peralihan didop dengan kaca silika borotelurit dan kesan pengolahan haba kepada sistem kaca . Keputusan dari indeks biasan mencadangkan bahawa silika borotelurit didop dengan mangan mempunyai potensi digunakan sebagai kaca serapan UV.

## ACKNOWLEDGEMENTS

Firstly, thank you Allah and all praises to Allah the most beneficent for allowing me to complete my study and this research. I can only finish this study with His grace and mercy.

I would like to express my highest gratitude to my main supervisor, Prof. Dr. Halimah Mohamed Kamari as well as my co-supervisors, Dr. Farah Diana Muhammad and Dr. Nurisya Mohd Shah for their helpful guidance as well as suggestion and supportive encouragement along the journey. I would like to give my sincere gratitude to my fellow lab colleagues, seniors and friends, Dr. Faznny Fudzi, Dr. Hasnimulyati Laoding, Dr. Eevon Chua, Suzliana Muhamad, Ami Hazlin Mohd Nor, Dr. Umar Saad Aliyu, Nurhayati Mohd Nor, Abdullahi Usman, Abdul Baset, Abdulkarim Mohammad Hamza, Suzliyana Muhammad, Nazirul Nazrin Shahrol Nidzam, Nurul Asyikin Ahmad Sukri for their willingness in guiding and helping in in carrying the experiment, taking measurement, discussing about the results, analyzing data and giving support when I face a hard time. My greatest appreciation to dearest my mother, Zaharah Abdul Aziz, my late father, Ismail Hashim, my sister, Zaidah Ismail and my brothers, Badrul Nizam and Badrul Hisham for their support, understanding and prayers for me. Last but not least, many thanks to the staff of Faculty of Science as well as Institute of Bioscience for allowing me and assisting me to carry out the measurement for the required equipment.

Without the support from all of you, I maybe could not finish this research.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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| | |
|---|---|
| $\theta_D$ | Debye temperature |
| ℃ | Degree celcius |
| $\Delta T$ | Thermal stability |
| Å | Armstron |
| $Ag_2O$ | Silver oxide |
| AgI | Silver iodide |
| $Al_2O_3$ | Aluminium oxide |
| $As_2O_3$ | Arsenic trioxide |
| ASTM | American Society for Testing Materials |
| $B_2O_3$ | Boron oxide |
| BaO | Barium oxide |
| BO | Bridging oxygen |
| $BO_3$ | Trioxoborate |
| $BO_4$ | Tetrahydroxyboranuide |
| $CaCO_3$ | Calcium carbonate |
| CaO | Calcium oxide |
| $cm^{-1}$ | Centimeter |
| CO | carbon monoxide |
| $CO_2$ | carbon dioxide |
| $Co^{2+}$ | Cobalt ion |
| DSC | Differential scanning electron |
| DTA | Differential thermal analysis |
| $Dy_2O_3$ | Dysprosium oxide |
| $E_{opt}$ | Optical band gap |
| $Er^{3+}$ | Erbium ion |
| eV | electron volt |
| F | Fragility index |
| $F_2$ | Fluorine |
| $Fe^{3+}$ | Ferum oxide |
| FTIR | Fourier transform infrared spectroscopy |
| $Gd_2O_3$ | Gadolinium oxide |
| $Gd^{3+}$ | Gadolinium ion |
| $GeO_2$ | Germanium |
| h | Plank's constant |
| H | Microhardness |
| $H_2O$ | Water |
| HCI | Hydrochloric acid |
| HgO | Mercury oxide |
| IUPAC | The International Union of Pure and Applied Chemistry |
| K/min | Kelvin/minute |
| $K_2O$ | Potassium oxide |
| Kgl | Hruby value |
| KI | Potassium iodide |
| $Li_2O$ | Dilithium oxide |
| $M_w$ | Molecular weight |
| MgO | Magnesium oxide |
| MHz | Mega hertz |
| Mn | Manganese |

| | |
|---|---|
| $Mn^{2+}$ | Manganese ion |
| $MnO_2$ | Manganese dioxide |
| MSW | municipal solid waste |
| Mw | Molecular weight |
| n | Oxygen atom per formula unit |
| $N_2O$ | nitrous oxide |
| $Na_2O$ | Sodium oxide |
| NBO | Nonbridging oxygen |
| $NH_4$ | methane |
| nm | Nano meter |
| OH | Hydroxyl |
| OPD | Oxygen packing density |
| $P_2O_5$ | Phosphoric oxide |
| $P_2O_5$ | Phosphate oxide |
| PL | photoluminescence |
| RE | Rare earth |
| RHA | Rice husk ash |
| Se | Selenium |
| SEM | Scanning electron microscopy |
| $Si_3N_4$ | silicon nitride |
| SiC | Silicon carbide |
| $SiO_2$ | Silica dioxide |
| $SiO_4$ | Orthosilicate |
| SrO | Strontium oxide |
| $T_c$ | Crystallization temperature |
| $TeO_2$ | Tellurium oxide |
| $TeO_3$ | Tellurium trioxide |
| $TeO_4$ | Trioxotellurate |
| $T_f$ | Fictive temperature |
| $T_g$ | Glass transition temperature |
| TGA | Thermogravimetric analysis |
| $T_m$ | Melting temperature |
| $T_o$ | Onset temperature |
| UV | Ultra violet |
| UV-Vis | Ultra violet visible |
| $V_2O_5$ | Vanadium |
| $V_m$ | Molar volume |
| XRD | X-ray diffraction spectroscopy |
| ZnO | Zinc oxide |
| $\alpha$ | Alpha |
| $\beta$ | Beta |
| $\gamma$ | Gamma |
| $\delta$ | Delta |
| $\Delta E$ | Urbach energy |
| $\mu m$ | Micro meter |
| ρ | Density |

# CHAPTER 1

# INTRODUCTION

This chapter deals with the research background of the present study which consists of the definition of glass, types of glass, glass network theory and the crucial information on silica that has been extracted from waste material, rice husk. Moreover, other valuable components in present study such as the problem statement, research objective, hypothesis, outline, scope and limitations are elaborated in this chapter.

## 1.1 Definition of the glass

Glasses have been used from the ancient times until present day with changes in the appearance of glass material from transparent glass to glasses possessing a variety of colour. Glasses are traditionally made of inorganic materials such as silica sand, feldspars, borates, phosphates, sodium and calcium carbonates that were melt together and cooled to form solid transparent materials (Morey, 1954; Doremus, 1994).

According to American Society for Testing Materials (ASTM) in 1945, glass can be defined as inorganic product of fusion which has been cooled to a rigid condition without undergoing crystallization process. According to Jones (1956), glass is a material formed by a cooling process from the normal liquid state that shows no discontinuous change at any temperature range to glass material which become more or less rigid through a progressive increase in its viscosity. There are several methods that can be employed in order to achieve glass formation such as melting-quenching, vapour deposition, sol-gel processing and neutron irradiation of crystals.

Glass can exist in amorphous and crystalline phase as can be seen in Figure 1.1 Amorphous state materials basically do not have long range order structure, short periodic atomic arrangement and possessing time-dependent glass transformation behaviour. The arrangement of elements in amorphous solids is mostly random and disorderly. Example of amorphous materials includes window glass, various polymers, rubber, plastic and glass. Crystalline glass can be described as materials with atoms, ions and molecules that are arranged in a highly ordered manner that formed the crystal lattice. The crystalized solids are formed through a process known as crystallization process whereby the lattice structure gets extended in all directions. Some example of crystalline materials in our daily life are snowflakes, diamonds and table salt. In addition, crystalline solid is anisotropic in nature whereby material with anisotropy behavior hints that the material will have some difference in its properties when its properties are measured along a different axes or different directions. Crystalline solid usually show different values for some of its properties such as electrical resistance, mechanical strength, thermal conductivity and refractive index when the measurement takes place along different directions.

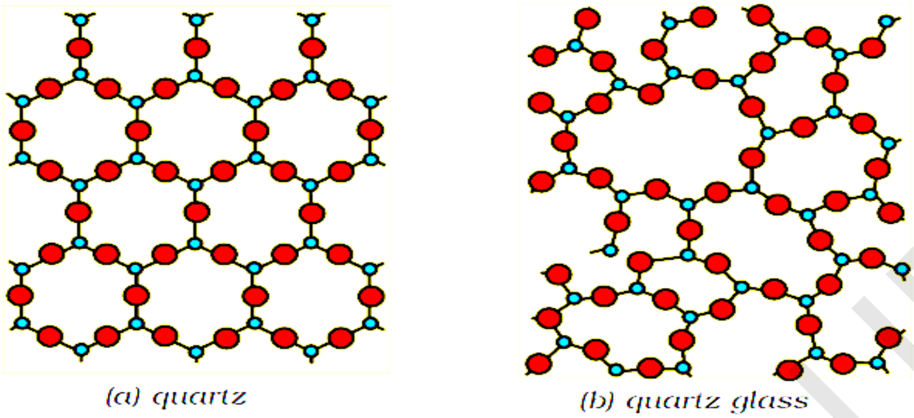


**Figure 1.1: Comparison of the structure in two-dimensional of crystalline and amorphous materials. (a) crystalline quartz ($SiO_2$); (b) amorphous quartz ($SiO_2$)** (Esquinazi, 2013)

## 1.2 Glass components

The components of a glass material can be mainly divided into three categories depending upon their function and general characteristics which include network former, network modifiers and intermediate compounds. The main difference between the components is their respective bond strength.

### 1.2.1 Network formers or glass formers

Glass former also known as a network former or glass forming oxides in many oxide glasses. Glass former can form a glass network alone and usually possesses covalently bonded compounds. Some important glass formers in current commercial oxide glasses are silicon dioxide ($SiO_2$), phosphoric oxide ($P_2O_5$), boric oxide ($B_2O_3$), germanium oxide ($GeO_2$), tellurium oxide ($TeO_2$), vanadium pentoxide ($V_2O_5$) and arsenic trioxide ($As_2O_3$). All the oxides capable of forming tetrahedral units which share corners in crystalline compounds except for $B_2O_3$ that forms triangle structure and can readily produce a glass. Usually glass formers have a high melting temperature and have high resistance against chemical attack due to their continuous rigidly held oxygen bonds.

### 1.2.2 Network modifiers

Glass modifier such as alkaline earth element, transitional metal oxide, alkali oxides and silver ions can alter the properties of a glass when they are added into the glass system. Network modifiers unable to form glass alone, but usually react with glass formers and induce changes in the glass network (Macfarlane and Martin, 2004). The incorporation of network modifier causes the rupturing of oxygen or chalcogenide bond which is connected to two glass cations. Ionic bonds are created between the positively charged interstitial modifiers cations and negatively charged covalent network as a result of broken bonds in the glass matrix. The presence of modifier not only changes structure and bonding of the network, but also affect the rigidity, net charge and distribution of interconnected interstices in the glass network. Examples of network modifier are sodium oxide ($Na_2O$), potassium oxide ($K_2O$), magnesium oxide (MgO), mercury oxide (HgO), calcium oxide (CaO), barium oxide (BaO), lithium oxide ($Li_2O$), strontium oxide (SrO) and zinc oxide (ZnO). Generally, network modifiers possess great potential application as sensor application, optoelectronic devices, bioactive materials and solid-state ionic conductors.

### 1.2.3 Intermediate compounds

In contrast with network former and network modifier, intermediate compound can either takes part as a network former or network modifier but it cannot form glass by itself as the bond strength of atoms or ions in the chain of intermediate compound are insufficient to prevent and hinder the orderly orientation effectively, however it able to act as glass former when combined with the other glass formers. Aluminum, titanium, iron and zirconium oxides are example of intermediate compounds that having single bond strength. In addition, the glass intermediate tend to occupy the holes in between network formers and in another way around, substitutes the network former cations in the macromolecular chain after it has been incorporated into the glass matrix.

## 1.3 Waste product

Waste products around the world have caused significant health and environmental problems especially during the management and disposal step that ultimately lead to direct economic costs and a problem issues to the communities. However, recently many researchers have found out that waste product actually contain valuable minerals for the production of glass, ceramics and glass-ceramics. (Cornejo et al., 2014; Yoon et al., 2013). There are many waste products that can be used to replace the commercial raw material such as rice husk, peanuts shells, egg shells, corn husk and banana peels. Waste product such as rice husk can produce silicon oxide or silica by undergoing a drying process in an oven before being heated to a temperature in order to produce white rice husk ash which also been widely known as silica. On the other hand, peanut shells that contain about 93% of organic material and water can produce CaO, $K_2O$ and $Al_2O_3$ while CaO or $CaCO_3$ can be extracted from eggshell waste. The extracted minerals are among the valuable minerals that can be used in the production of glass and glass-ceramic materials in the industries (Demiral et al., 2014; Li et al., 2015).

Rice husk as a biomass waste originated from agricultural field whereby this waste product is getting a lot of attention all over the world since rice husk is environmental friendly source, renewable, widely available and cheap. Moreover, rice is the second most important crop in the world after wheat with Asia being the largest producer and consumer for as much as 90 percent (Matthews, 1995; Banik, 1999; Gumma et al., 2011). Rice is a staple food for Malaysian and according to the Department of Statistics Malaysia, the paddy production for 2015 was 2674.4 tones. Meanwhile according to United States Department of Agriculture, rice imported from Malaysia reaches a total of 900,000 tons in 2017/2018 and 1.0 million tons in 2016/2017.

Malaysia currently has been facing a serious problem about municipal solid waste (MSW) whereby about 2500 ton of MSW is collected every day for only the city of Kuala Lumpur (Kathirvale et al., 2003). If this waste management problem is not solved day by day, it would definitely become a crucial problem in the ecological ecosystem and brings about health and environment problem to human, animals and plants. Under the Malaysia Solid Waste and Public Cleaning Management Act 2007 (Act 672), disposal of any solid waste could be by any means of destruction, incineration and deposit or decomposing the waste (Nagapan et al., 2012). In order for Malaysia to move toward green or sustainable environment, countries like Japan and Hong have been a good benchmark to be followed in the precaution or steps taken by the countries on handling waste issues.

Rice husk is extremely prevalent in Asian countries due to the rich land and tropical climate that make a perfect condition to cultivate rice. Disposal of rice husk ash is an important issue in these countries that cultivates large quantities of rice. Therefore, the 100 million tons of rice husk waste generated globally begins to bring about impact on the environment if the waste has not been disposed properly. The recycling of rice husk is an excellent alternative in order to minimize the environmental impact as well as issues due to improper disposal and to reduce the disposal costs. The rice husk ash is a by-product from the burning of rice husk. The burning of the rice husk can produces various elements such as silicon (Si), silica ($SiO_2$), silicon carbide (SiC), silicon nitride ($Si_3N_4$) and graphene (Liu et al., 2013; Liou and Yang, 2011; Krishnarao and Godkhindi,1992; Pavarajarn et al., 2010; Muramatsu et al., 2014). Burning the rice husk in air can produce approximately white ash with one-fifth of the weight of the rice husk. The extracted white ash has about 80% of silica with only a small quantities of metallic elements such as alumina, sulfur trioxide, iron oxide, calcium oxide, magnesium oxide, sodium oxide, and potassium oxide (Sankar et al., 2016). In this research, silica element in waste materials can be extracted by using several methods which include vapor-phase reaction, sol-gel method and coprecipitation techniques (Nishiyama et al., 2003; Lee et al., 2007; Esquena et al., 2006). However, these preparation methods are quite costly with limited application. In contrast, the combustion of rice husk in order to extract silica is relatively inexpensive. Rice husk will usually undergo burning process at different temperature that varies from 500 ℃ to 1100 ℃ along with acid leaching treatment to get white rice hush ash or silica (Real et al., 1996; Della et al., 2002; Zhang et al., 2010).

Previously, silica glass has been widely explored in owing to its potential as optical, electrical and medical devices. Silica glass with a wide band gap (~9 eV) has resulted in a glass material possessing high native transparency that extends from UV to infrared region in the electromagnetic spectrum (Kim et al., 2013; Tolba et al., 2016). $SiO_2$ based composite are important material for clinical uses such as in bio-glass and various glass ceramics. Silica also has been used in the production of various ceramic materials which is mainly utilized in cement industry as inorganic filler of polymers composites and for processing silicon carbide ceramics (Rodriguez de Sensale, 2006; Ishak and Bakar, 1995; Singh et al., 2002). On the other hand, tellurite have always been the most popular element to be chosen as the main component in glass material due to its ability in accepting rare earth ions in its environment matrices which make tellurite based glass a glass material with favorable lasing properties to be utilized as optical component (Yang et al., 2004; Ohishi et al., 1998). Tellurite glasses possess interesting glass forming ability and glass structure that enable the tellurite based glasses to have various properties and function. Tellurite based glass is also well known for its high density, high refractive index and high transparency in the far infrared region. In contrast, boron oxide is one of the host materials or glass formers that has been incorporated into various kinds of glass systems due to its random configuration (Yawale et al., 2000). The interaction between the anion and the covalent boron-oxygen network which is $BO_3$ groups and tetrahedral $BO_4$ unit tends to increase the glass transition temperature and chemical durability of the glass system.

## 1.4 Problem statement

Due to the fast growing technology and materials science field all over the world, abundant studies have been done on the properties and potential applications of various glass material with different chemical composition. Various glass system possess unique properties that have different potential in applications such as glass fiber, composite materials, radiation shielding glass, laser material and smart window glass (Kelestemur et al., 2014; Liu et al., 2017; Yang et al., 2004; Jones 2002). Different chemical composition and elements that have been added into a glass material is capable of enhancing the properties of the glass system such as giving great toughness, high chemical stability and large temperature resistance.

Biomass is waste material from plants or animal that has not been used for feed or food. Nowadays biomass from farming field such as wheat stalks, food processing process such as corn cobs and rice husk, animal farming (manure) and human waste from sewage plants is reconsidered as valuable source of renewable energy. According to Saxena et al., (2008), they predicted that biomass will account about 15% of the world's total energy supply. Rice husk is a type of biomass that provides highly efficient thermal energy which can be used in bio-oil preparation procedure (Zheng and Kong, 2010).The combustion of rice husk to be reused as a bio resource will surely give impact to environmental issue. Rice husk are often burned at the rice mill just get rid of the husks. The burning process usually releases carbon dioxide ($CO_2$), carbon oxide (CO), methane ($NH_4$) and nitrous oxide ($N_2O$) into the atmosphere whereby these gases are known as the greenhouse gas that bring danger to the ecosystem (Sun and Gong, 2001; Arai et al., 2015). The utilization of recyclable waste product can decrease the disposal problem since the amount of rice husk continues to increase each year.

In addition, the act of doping transition metal in glass system has been the subject of great interest recently due to the broad radial distribution of outer d-electron orbital functions in transition metal (Durga and Veeraiah, 2002). Among all the transition metal ions, manganese (Mn) ion is particularly interesting since it is able to exist in different valence states in various glass matrices (Lee, 1996; Van Die et al., 1988; Margaryan et al., 2004).

However, some problem arises when a high percentage of manganese are added into a glass system whereby the glass will tend to be dark in colour until the light cannot transmit through the sample. Heat treatment procedure is used in the present study in order to alter the properties of the glass. By using heat treatment method, the characteristic of the glass may be further modified while maintaining a low percentage of manganese at the same time. In this research, heat treatment temperature was 250 ℃, 350 ℃, 450 ℃ and 600 ℃. This range of heating temperature has been chosen because to see the changes in the glass structural from amorphous to crystalline when the fabricated glass are heat treated near, around, at and higher than glass transition temperature. Heat treatment of the samples can ulter the physical properties such as density of the glass sample.

Up to this day, there are less studies that were conducted on silica borotellurite glass by previous researches. Umar et al., (2017) and Hamza et al., (2018) mentioned that doping the most famous rare earth element, erbium into silica borotellurite glass is able produce glasses with high optical band gap, molar refraction, metallization criterion, transmission coefficient and small Urbach energy as well as refractive index. In this research, they used chemical composition of $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.8}(SiO_2)_{0.2}\}_{1-x}(Er_2O_3)_x$ and this chemical composition is the best formula to fabricate a glass. Furthermore, no research has been done on heat treated silica borotellurite glasses that have been doped with manganese. The influence of heat treatment to the physical, structural, optical, elastic and thermal properties glass system also have not been unveiled or studied by any researcher. In addition, the act of using waste product, in this case, rice husk as silica source in this study rather than the commercial silica might able to help to reduce cost in fabricating useful glass that capable of benefiting human.

## 1.5 Objective of study

This research was conducted based on four clear and concise objectives. The purpose of this research are as stated below;

1. To extract high purity silica from waste product, rice husk.
2. To analyze the effect of $MnO_2$ on the physical, structural, optical, thermal and
3. elastic properties of silica borotellurite glass system**.**
4. To determine the influence of heat treatment on $MnO_2$ doped silica borotellurite

## 1.6 Scope of study

In order to achieve the objectives of the study, the scope of the study is stated below;

1. The preparation of the glasses was done by using conventional method which is the melt-quenching technique and based on the stoichiometric equation of $\{[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.8}[SiO_2]_{0.2}\}_{1-x}\{MnO_2\}_x$ where x = 0.00, 0.01, 0.02, 0.03, 0.04 and 0.05 molar fraction.
2. The structure of the glasses was studied by using X-ray Diffraction (XRD) to confirm the amorphous nature as well as crystalline state in the sample and Fourier Transform

Infrared Spectroscopy (FTIR) that is utilized to study the chemical bonding in the studied material.
3. The ultrasonic wave velocity of the glass samples has been measured by using pulse-echo technique which will be used to determine the elastic properties of the glass samples.
4. The optical properties such as optical band gap, Urbach energy and refractive index of the glasses were investigated by using UV-Vis Spectrophotometer.
5. The effect of heat treatment on the glasses were studied by heating glass samples with different heat-treatment temperature ranging from 250 ℃, 350 ℃, 450 ℃ to 600℃.

## 1.7 Hypotheses

Based on the five objectives, the hypotheses for this research are;

1. The extracted silica are expected to possess high purity since the optimum temperature and acid leaching treatment is used in this study.
2. The addition of $MnO_2$ into the glass sample should produce glass with amorphous nature that can be verified by using XRD. After heat treatment process, crystalline peak should be present in the XRD pattern. FTIR should detect the structural unit of $TeO_4$, $TeO_3$, $BO_4$ and $BO_3$ in the glass sample. The agglomeration of particles in the glass system after heat treatment is expected to be observed in the SEM image. The density values are expected to decrease as lighter dopant are added into the glass matrix.
3. It is expected that the elastic properties for silica borotellurite glass doped with $MnO_2$ will be enhanced. This might be due to the formation of bridging oxygen that will eventually increase the rigidity of the glass samples. The addition of dopant, $MnO_2$ are expected to improve the optical properties of the glass by decreasing the energy band gap and the Urbach energy while increasing the refractive index value of the prepared glass samples. The incorporation of manganese that plays the crucial role as a modifier in the glass system might be capable of creating more highly polarizable non-bridging oxygen which contributes to the decrement in energy band gap and Urbach energy.
4. Heat treatment for manganese doped silica borotellurite glasses at different temperature is expected to possess physical, structural, elastic, optical and thermal properties that are different from those of untreated glass samples. Heat treatment procedure may restructure the network bond and lead to the changes in its properties.

## 1.8 Outline of thesis

The thesis is arranged in the following manner. Chapter 1 consists of the definition of the glass, glass components, waste product that is used in this research, problem statement, objectives and possible outcome of this study. Chapter 2 comprises the literature review from previous researchers that is related to this current study. Next, Chapter 3 highlights the theory and equations that are used in this research. The process and technique used to fabricate the glass samples in this study is as reported in Chapter 4. Next, Chapter 5 discusses the obtained results and trends which includes the physical, structural, optical, elastic and thermal properties of the glass samples. The comparative studies between some parameters in this study and the previous studied glass are also discussed in this chapter. Chapter 6 also provides the conclusion and suggestion for future works.

This chapter unveils the research background of this study that consists of a simple history as well as an introduction to glass, the formation of glass material and the chemical components that made up the glass. Crucial component of this study such as problem statement, scope of study, research objective, hypothesis and the outline of this thesis are also included in this chapter.

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