**UNIVERSITI PUTRA MALAYSIA**

***STRUCTURAL, OPTICAL AND GAMMA RADIATION SHIELDING PROPERTIES OF $Bi_2O_3/BaO-B_2O_3-TeO_2$ DOPED $CeO_2$ GLASS SYSTEM***

**AZURAIDA BINTI AMAT**

**FS 2018 89**

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By

**AZURAIDA BINTI AMAT**

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

**April 2018**

# DEDICATION

This thesis is specially dedicated to my husband, Shaiful Azman Abdul Kadir, my sons, Luqman Syafi, Aqil Syafi, Muhammad Syafi and Syafia Az Zahraa, my family, lecturers, and friends.

Thank you for encouraging me to finish this journey

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

**STRUCTURAL, OPTICAL AND GAMMA RADIATION SHIELDING PROPERTIES OF $Bi_2O_3$/BaO-$B_2O_3$-$TeO_2$ DOPED $CeO_2$ GLASS SYSTEM**

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**April 2018**

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

Lead is one of the important elements in production of radiation shielding glass due to its high density, high atomic number and high degree of stability. However, toxicity of lead can cause negative impact to the environment and carcinogenic to human body.

Three lead-free series of boro-tellurite based glass were successfully synthesized by conventional melt quenching method. These three glass compositions are: $[(TeO_2)_{70}(B_2O_3)_{30}]_{100-x}(Bi2O3)_x$ with x = 0, 5, 10, 15, 20, 25 and 30 mol%), $[(TeO_2)_{70}(B_2O_3)_{30}]_{100-x}(BaO)_x$ with x = 5, 10, 15, 20, 25 and 30 mol%) and $\{[(TeO_2)_{70}(B_2O_3)_{30}]_{75}(Bi_2O_3)_{25}\}_{100-y}(CeO_2)_y$ with y = 1, 2, 3, 4 mol%). The effect of $Bi_2O_3$, BaO and $CeO_2$ in boro-tellurite glasses on gamma shielding and optical properties has been studied. Gamma shielding properties measurement were conducted using "Lead equivalent thickness" equipment while optical properties were determined using UV-VIS spectrophotometer. The density measurement (Archimedes principle), amorphous phase (X-ray diffraction), structural changes (Fourier Transform Infrared (FTIR)) and theoretical calculation of parameter radiation shielding (WinXcom program) are provided for supportive evidence to gamma shielding and optical properties. The results show, the glass densities increases with the increase of $Bi_2O_3$, BaO and $CeO_2$ content which due to the high molecular weight of glass modifier compared to glass network $TeO_2$–$B_2O_3$. The increase in densities of glasses are also due to the increase in the number of non-bridging oxygen (NBO) atoms and the replacement of a low–density oxide $B_2O_3$ (2.46 g/cm$^3$) and $TeO_2$ (5.67 g/cm$^3$) by a high–density oxide $Bi_2O_3$ (8.99 g/cm$^3$), BaO (5.72 g/cm$^3$) and $CeO_2$ (7.65 g/cm$^3$). XRD result show all glasses are amorphous in nature. The absorption peak changes shown in FTIR spectra are depended on the glass composition. FTIR analysis show the glass sample consist of $TeO_3$, $TeO_4$, $TeO_6$, $BO_3$ and $BO_4$ structural units. The attenuation coefficients were measured for gamma ray photon energy of 662 keV.

These coefficients were used to obtain the values of mass attenuation coefficients, mean free path, half value layer and effective atomic number. Results showed the shielding properties improved as increasing amount of glass modifiers ($Bi_2O_3$ and BaO). But, the shielding properties decrease with the increasing of $CeO_2$. Results from theoretical calculation also showed that Compton scattering is a major interaction in gamma energy of 662 keV. Half value layer and mean free path of the glass systems has been compared with some standard radiation shielding materials. The $[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(Bi_2O_3)_{0.3}$ glasses were found to give the lowest mean free path (2.9265) and half value layer (2.0281) compared to other glass samples and even better than some standard concretes and commercial radiation shielding glasses. This indicates that $[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(Bi_2O_3)_{0.3}$ glass is more efficient to attenuate the 662 keV gamma ray and provides a better shielding glass. The optical band gap energy, $E_{g,indirect}$ which was calculated from Tauc' plots decreases from 3.08 to 2.78 eV and 3.49 to 3.23 with the increased amount of $Bi_2O_3$ and BaO, respectively. The shift of the absorption edge to higher wavelength are also observed. The shift of the absorption edge and decrease of $E_g$ value attributed the restructure of glass network and modifier, and it can be related to the progressive increase in the concentration of non-bridging oxygen (NBO) atoms. The refractive indices of glass were found to increase from 1.767 to 2.093, 1.767 to 1.839 and 1.955 to 2.098 with the increase amount of $Bi_2O_3$, BaO dan $CeO_2$, respectively. It is due to the high polarization and density of host material and glass modifier. In order to observe the ability of studied glasses against gamma irradiation, selected glass samples from each series were exposed to $^{60}Co$ radioisotope to the overall dose of 45 kGy. The changes of peak intensity of FTIR spectra after exposed to gamma radiation are due to breaking of network bonding and displacement of lattice atoms. The $E_g$ values of the glasses keep decreasing after exposed to gamma irradiation. Gamma irradiation causes the breaking up of three dimensional networks leading to the transformation of bridging oxygen (BO) to non-bridging oxygen (NBO). Negative charge from NBO have larger magnitude compared to BO. These NBO ions raise the top of the valence band resulting in the reduction of optical band gap.

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

**SIFAT STRUKTUR, OPTIK DAN PERLINDUNGAN SINAR GAMA SISTEM KACA $Bi_2O_3$/BaO-$B_2O_3$-$TeO_2$ DIDOPKAN $CeO_2$**

Oleh

**AZURAIDA BINTI AMAT**

**April 2018**

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Plumbum merupakan satu unsur penting dalam pembuatan kaca perlindungan radiasi kerana ianya berketumpatan tinggi, nombor atom tinggi dan tinggi darjah kestabilan. Namun, ketoksikan plumbum boleh memberikan impak negatif kepada persekitaran dan karsinogenik kepada tubuh manusia. Tiga siri kaca tiada - plumbum berasaskan boro-telurit telah berjaya disintesis melalui kaedah konvensional lebur pelindapkejutan. Komposisi tiga siri kaca tersebut adalah $[(TeO_2)_{70} (B_2O_3)_{30}]_{100-x} (Bi_2O_3)_x$ dengan x = 0, 5, 10, 15, 20, 25 dan 30 mol%), $[(TeO_2)_{70} (B_2O_3)_{30}]_{100-x} (BaO)_x$ dengan x = 5, 10, 15, 20, 25 dan 30 mol%) dan $\{[(TeO_2)_{70} (B_2O_3)_{30}]_{75} (Bi_2O_3)_{25}\}_{100-y} (CeO_2)_y$ dengan y = 1, 2, 3 dan 4 mol%). Kesan $Bi_2O_3$, BaO dan $CeO_2$ dalam kaca boro-telurit pada pencirian perlindungan gama dan optik telah dikaji. Pengukuran pencirian perlindungan gama telah dijalankan menggunakan peralatan "Kesetaraan Ketebalan Plumbum" manakala pencirian optik telah ditentukan menggunakan spektrofotometer UV-VIS. Pengukuran ketumpatan (prinsip Archimedes), fasa kehabluran (pembelauan sinar-X), perubahan struktur (Fourier Transform Inframerah (FTIR)) dan pengiraan parameter perlindungan sinaran secara teori (program WinXcom) terhadap sampel kaca juga dijalankan untuk menyokong pencirian optik dan perlindungan gama. Keputusan menunjukkan, ketumpatan kaca meningkat dengan peningkatan kandungan $Bi_2O_3$, BaO dan $CeO_2$ yang mana disebabkan oleh jisim molekul pengubahsuai kaca yang tinggi berbanding dengan rangkaian kaca $TeO_2$-$B_2O_3$. Peningkatan ketumpatan kaca juga disebabkan oleh peningkatan dalam bilangan atom oksigen tanpa penitian (NBO) dan penggantian oksida $B_2O_3$ (2.46 $g/cm^3$) dan $TeO_2$ (5.67 $g/cm^3$) yang berketumpatan rendah oleh oksida $Bi_2O_3$ (8.99 $g/cm^3$), BaO (5.72 $g/cm^3$) dan $CeO_2$ (7.65 $g/cm^3$) yang berketumpatan tinggi. Keputusan XRD menunjukkan semua kaca adalah berkeadaan amorfus. Perubahan keamatan puncak yang ditunjukkan dalam spektrum FTIR adalah bergantung kepada komposisi kaca. Analisis FTIR menunjukkan sampel kaca terdiri daripada unit struktur $TeO_3$, $TeO_4$ dan $TeO_6$, $BO_3$ dan $BO_4$. Pekali pengecilan kaca diukur

menggunakan foton sinaran gama bertenaga 662 keV. Pekali ini digunakan untuk mendapatkan nilai pekali pengecilan jisim, laluan bebas min dan lapisan nilai separuh. Keputusan menunjukkan sifat perlindungan kaca bertambahbaik dengan peningkatan pengubahsuai kaca ($Bi_2O_3$, BaO). Tetapi sifat perlindungan kaca berkurang dengan peningkatan $CeO_2$. Keputusan daripada pengiraan teori juga telah menunjukkan bahawa serakan Compton merupakan interaksi utama di dalam 662 keV tenaga gama. Laluan bebas min dan lapisan nilai separuh bagi semua sistem kaca telah dibandingkan dengan sebilangan bahan perlindungan radiasi piawai. Kaca $[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(Bi_2O_3)_{0.3}$ didapati telah memberikan nilai laluan bebas min (2.9265) dan lapisan nilai separuh (2.0281) yang terendah berbanding sampel kaca lain dan lebih baik dari standard konkrit dan kaca perlindungan radiasi komersial. Ini menunjukkan kaca $[(TeO_2)_{0.7}(B_2O_3)_{0.3}]_{0.7}(Bi_2O_3)_{0.3}$ lebih effektif untuk menyerap 662 keV tenaga gama dan memberikan kaca perlindungan yang lebih baik. Tenaga jalur optik, $E_{g,indirect}$ yang dikira dari plot Tauc' berkurangan dari 3.08 kepada 2.78 eV dan 3.49 kepada 3.23 dengan peningkatan kandungan $Bi_2O_3$ dan BaO, masing-masing. Peralihan penyerapan pinggiran ke panjang gelombang yang lebih tinggi juga diperhatikan. Peralihan penyerapan pinggiran dan pengurangan nilai $E_g$ disebabkan oleh penyusunan semula struktur rangkaian kaca dan pengubahsuai, dan ia boleh dikaitkan dengan peningkatan progresif kandungan atom oksigen tanpa penitian (NBO). Indeks biasan kaca didapati meningkat daripada 1.767 kepada 2.093, 1.767 kepada 1.839 dan 1.955 kepada 2.098 dengan peningkatan kandungan $Bi_2O_3$, BaO dan $CeO_2$, masing-masing. Ianya disebabkan oleh polarisasi dan ketumpatan yang tinggi oleh bahan utama dan pengubahsuaian kaca. Dalam usaha untuk melihat keupayaan kaca yang dikaji terhadap sinaran gama, sampel kaca yang dipilih dari setiap siri telah didedahkan kepada $^{60}$Co radioisotop dengan dos keseluruhan 45 kGy. Perubahan keamatan puncak spektrum FTIR selepas terdedah kepada sinaran gama adalah disebabkan oleh pemutusan ikatan rangkaian dan anjakan atom kekisi. Nilai $E_g$ kaca semakin berkurangan selepas didedahkan kepada sinaran gama. Sinaran gama menyebabkan pemutusan rangkaian tiga dimensi yang membawa kepada transformasi oksigen penitian (BO) kepada oksigen tanpa penitian (NBO). Cas negatif dari NBO mempunyai magnitud yang lebih besar dibandingkan dengan BO. Ion-ion NBO ini meningkatkan bahagian atas jalur valens dan mengakibatkan pengurangan jurang jalur optik.

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I certify that a Thesis Examination Committee has met on 19 April 2018 to conduct the final examination of Azuraida binti Amat on her thesis entitled "Structural, Optical and Gamma Radiation Shielding Properties of $Bi_2O_3$/BaO-$B_2O_3$-$TeO_2$ Doped $CeO_2$ Glass System" 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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| | |
|---|---|
| $\alpha$ | Absorption coefficient |
| $N_A$ | Avogadro's number |
| BO | Bridging oxygen |
| kGy | Kilo Gray |
| mCi | Mili Curie |
| NBO | Non-bridging oxygen |
| $\langle d_{B-B} \rangle$ | Boron-boron separation |
| $^{60}Co$ | Cobalt 60 |
| Cs-137 | Caesium 137 |
| $B_2O_3$ | Boron trioxide |
| $TeO_2$ | Tellurium dioxide |
| $CeO_2$ | Cerium oxide |
| $Bi_2O_3$ | Bismuth oxide |
| BaO | Barium oxide |
| FTIR | Fourier Transform Infrared |
| XRD | X-ray diffraction |
| UV-VIS | Ultra violet-visible |
| LET | Lead equivalent thickness |
| $E_{opt}$ | Optical band gap |
| $\alpha_m$ | Molar polarizability |
| $\alpha_o^{2-}$ | Oxide ion polarizability |
| $R_m$ | Molar refraction |
| $V_m$ | Molar Volume |

| | |
|---|---|
| $n$ | Refractive index |
| $I_o$ | Incident intensity |
| $I$ | Transmitted intensity |
| $T$ | Time |
| $\rho$ | Density |
| $\mu$ | Linear attenuation coefficient |
| $\mu_m$ | Mass attenuation coefficient |
| HVL | Half-value layer |
| $\Lambda$ | Basicity |
| M | Metalization |
| MFP | Mean free path |
| $\lambda$ | Wavelength |
| $\gamma$-ray | Gamma-ray |
| $N_{el}$ | Electron density |
| $Z_{eff}$ | Effective atomic number |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background

Developments of tellurite glasses started since early 1950s especially in optoelectronics field such as fiber optic and laser technology. Tellurite glasses are excellent in glass stability among other oxide glass formers and they show high linear and nonlinear refractive index compared with the fluoride and silicate glasses (Dorofeev *et al*., 2011; El-Malawany, 2002). It is also reported that the density of tellurite glass compared to others is in this order: tellurite > germinate > phosphate > silicate (El-Malawany, 2002). This makes the tellurite glasses a potential candidate for radiation shielding glass as shown in previous report, such as Bi-Te (Ooi *et al*., 2012), Bi-Ba-Te (Xu *et al*., 2011) and Pb-$NaO_2$-Te (Limkitjaroeporn *et al*., 2011). As reported earlier, tellurite glass system is made up of three basic structural units: $TeO_3$, $TeO_4$ and $TeO_{3+1}$. Existence of these structural units depends on $TeO_2$ concentration and addition of modifier in the glass system.

Borate is also one of the most popular glasses, possessing high chemical durability, good solubility of rare earth and is easy for synthesis. In addition, combining with heavy metal oxide make it possible to use as electronic sensor (Bobkova, 2016), electronic device (Insitiponga *et al*., 2011) and radiation shielding material (Kirdsiri *et al*., 2009; Marzouk and ElBatal, 2014). Usually, borate glass consists of [$BO_4$] and [$BO_3$] structural units, but it can be changed to other $B_xO_y$ structural group together with non-bridging oxygen when combined with alkali or heavy metal oxide (Bobkova, 2016; Rejisha and Santha, 2011; Kamitsos and Chryssikos, 1991).

Investigation on borotellurite glass system has been growing yearly. Combination of two glass formers, borate and tellurite has found to affect the physical properties which can produce good quality glass. Structural unit of $TeO_4$ from tellurite and $BO_4$ from borate have strong tendency to link each other and can produce high connectivity in glass network. In addition, with suitable ratio of both glass formers will reduce their hygroscopic nature, increase the IR transmission and refractive index (Burger *et al*., 1984; Kaur *et al*.,2014; Halimah *et al*., 2007).

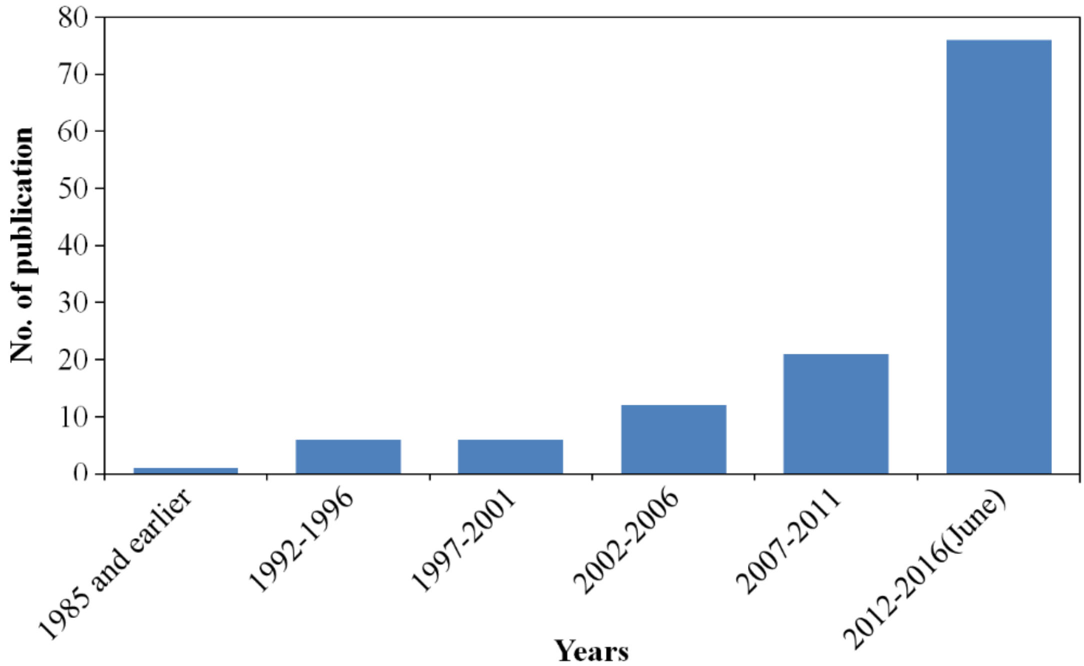


**Figure 1.1 : Number of publication for "boro-tellurite glasses" reported by ScienceDirect database**

ScienceDirect is a famous database among researchers and scientist which provides them an abstract and full-text from many publishers. It offers articles from over 3800 journals and more than 35,000 book titles in four main sections, which are Life Sciences, Physical Sciences and Engineering, Social Sciences and Humanities and Health Sciences. From Figure 1.1, it can be seen that the publication list from ScienceDirect database for the keywords of "boro-tellurite glasses" have dramatically increase in the last five years (2012-2016). However, the total numbers of publication for boro-tellurite glass are still small compared to research on other base glass system such as silicate, phosphate, boro-silicate, boro-phosphate etc. The properties cover for these glasses include physical, optical, thermal, dielectric, spectroscopy and shielding properties. While, the spectroscopy and linear and nonlinear optical properties are the most topic reports for boro-tellurite glass publication. Unfortunately, there are no reports found on shielding properties for boro-tellurite glass system.

Therefore, this work offers a new insight for boro-tellurite glass system which focuses on radiation shielding properties by adding selected heavy metal oxide as their glass modifiers. The densities, structural and optical properties of this glass system are also explored to see their relation with radiation shielding properties.

## 1.2 Problem Statement and Research Aim

Nowadays, the uses of radioactive isotope find increasing interest in various fields such as medical, food safety, nuclear power plant and industry. Improper handling of radioactive isotope can cause harm to human body and damage the laboratory equipment. The effect of the radioactivity on human body also cannot be immediately recognized. Therefore, a good quality of protective material is required to limit the radiation from radioactive isotope such as gamma ray to safety and acceptable levels. One of the famous materials used for protection from gamma irradiation is high density transparent glass which contains lead. This type of lead glass is commonly used for observing windows in medical x-ray imaging room and radiation source containers. Lead is one of the important elements in production of radiation shielding glass due to its high density, high atomic number and high degree of stability. However, toxicity of lead can cause negative impact to the environment and carcinogenic to human body.

Some researchers concluded that lead poisoning affects almost all major organ system in human body such as nervous, hematopoietic, cardiovascular systems and renal, including intellectual and neurological development. It also causes critical damage to enzymes, proteins, DNA and antioxidant defense system (Flora *et al.*, 2012; Panda, 2009). In addition, lead are found to be the most toxic metal compared with bismuth, antimony, silver, tin, copper and indium. Lead can bioaccumulate in organisms and the production processes of lead will effect on environmental quality (Ku *et al.*, 2003).

From the aforementioned problem, there is a need of replacement element which was encountered the requirement of the lead-free shielding material from unsafe irradiation. In this situation, bismuth and barium can be selected as suitable element to replace lead for radiation shielding glass production. Both of these elements are expected to enhance the attenuation of gamma irradiation due to its high atomic number. The toxicity of bismuth and barium and its environmental effect are less than lead element. Moreover, the addition of $Bi_2O_3$ and BaO in glass system can improve chemical durability, transparency, refractive index and also optical properties of glass system (Yasaka *et al.*, 2014; Saeed *et al.*, 2014; Bootjomchai *et al.*, 2012). Cerium oxide, $CeO_2$ can also be considered to be added in glass system as a protective material for reducing the gamma irradiation effect. Existence of $Ce^{3+}$ and $Ce^{4+}$ ions (from $CeO_2$) in glass system have been found to help in improving the gamma irradiation resistance (Fu *et al.*, 2014; Qian *et al.*, 2007; Baccaro *et al.*, 2002).

The aim of the present research is to continue the investigation of lead-free glass system containing bismuth oxide and barium oxide as glass modifier and cerium oxide as dopant for exploring their probability for new candidate as gamma shielding material especially for observation windows in radiotherapy room and nuclear power plant control room. $B_2O_3$-$TeO_2$ based glass are selected as a glass former due to their low melting temperature, high transparency and exceedingly stable against devitrification. Furthermore, lead-free glass from this work are expected to possess higher density, higher refractive index and large polarizability compare $B_2O_3$-$TeO_2$ glass and can improve optical properties which makes them suitable use for photonic devices.

## 1.3 Research Objectives

The above research aim will be accomplished by fulfilling the following objectives:

1. To synthesis new composition of boro-tellurite based glass with addition of $Bi_2O_3$, BaO and $CeO_2$ and also to study the relation between their density and structural properties.
2. To determine the radiation shielding properties such as half value layer, mean free path and mass attenuation coefficient for gamma irradiation and compare it with theoretical.
3. To analyze the variation of optical band gap and Urbach energy of glasses with respect to glass composition.
4. To investigate the effect of gamma irradiation on structural and optical properties for selected sample.

## 1.4 Scope of Study

i. Melt quenching method was chosen as technique for preparation of glass sample which based on stoichiometric equation: $[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{1-x} (Bi_2O_3)_x$, $[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{1-x} (BaO)_x$ and $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.75} (Bi_2O_3)_{0.25}\}_{1-y} (CeO_2)_y$. Where x = 0, 0.05, 0.10, 0.15, 0.20, 0.25 and 0.30 mol fraction, while y = 0, 0.01, 0.02 and 0.03 mol fraction. $Bi_2O_3$ and BaO are function as modifier and $CeO_2$ as dopant. As references, boro-tellurite glass with stoichiometric equation of $(TeO_2)_{0.7}(B_2O_3)_{0.3}$ will also be prepared using same technique.

ii. The measurement of radiation shielding properties of glass sample will be conducted using the lead equivalent thickness setup with Cs-137 as gamma source. Samples which are transparent and only have stable condition will be used in this measurement. The intensity of gamma ray before and after going through the sample will then be used to determine shielding parameter such as mass attenuation coefficient, half value layer, mean free path and effective atomic number. The experimental value of this shielding parameter will then be compared with theoretical value, predicted from Winxcom programme.

iii. The optical properties of all glasses will be measured using UV-Vis spectrophotometer. The absorbance data collected only from wavelength of 200 to 800 nm will be used to calculate their band gap, Urbach energy and refractive index.

iv. In order to study the influence of gamma radiation on the glass, the sample are only exposed to Co-60 gamma source in five different gamma doses: 5 kGy, 15 kGy, 25 kGy, 35 kGy and 45 kGy under room temperature. The samples which only have high density, transparent and in good condition are selected for gamma exposure. Any changes on structural and optical properties and color induced of sample will be measured within one week after completing the irradiation.

## 1.5 Thesis Organization

This thesis is structured in five chapters. Chapter 1 deals with introduction, problem statement, research aims and scope of study. It gives brief explanation about research topic and the rationale of this study. Chapter 2 provides information on literature from previous work relevant to tellurite based glass which focuses on radiation shielding and properties. The chapter also summarizes literature on physical, structural and optical properties and their effect when exposed to gamma radiation. The rest of subtopics includes detailed explanation on theory, concept and formula used in present study.

Chapter 3 presents chemicals involved and experimental technique used for synthesis of glass samples. Some information of equipment used for sample characterization such as XRD, FTIR and UV-VIS spectrophotometer are given. The details of experimental setups for gamma shielding properties which are specially proposed only for sample size in this research are also described.

Chapter 4 focuses on result obtained from experimental and sample characterization including general physical characteristic of glass such as color, density and molar volume. The relationship between structural, density, non-bridging oxygen (NBO) and band gap energy along with shielding parameter value are explored. Effects of gamma irradiation on some properties are also discussed for selected samples. In addition, the detail discussions for new finding in this research are also compared with related previous researches. Last chapter (Chapter 5) concludes the main results produced from this research and some suggestion for future research.

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