

**INVESTIGATION OF STRUCTURAL, OPTICAL,
AND RADIATION SHIELDING PROPERTIES OF
BISMUTH-BORO-TELLURITE GLASS DOPED
THULIUM OXIDE**

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**MASTER OF SCIENCE
(PHYSICS)**

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OF BISMUTH-BORO-TELLURITE GLASS DOPED
THULIUM OXIDE**

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Thesis submitted to the Centre for Graduate Studies, Universiti Pertahanan Nasional
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(Physics)

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ABSTRACT

Nowadays, the biggest production in glass manufacturing is silica. The primary ingredients of silica glass production are limestone, soda ash, and silica and sand. Unfortunately, this glass production may raise global temperature because of its melting temperature. Lead is one of the important elements in the production of optical properties and radiation-shielding glass due to its high density, high atomic number, and high degree of stability. However, the toxicity of lead can harm the environment and is carcinogenic to the human body. Lead can bioaccumulate in organisms and the production processes of lead will effect on environmental quality. From the problem, there is a need to replace lead with other elements that safer. The bismuth boro-tellurite based glass doped with thulium oxide was successfully synthesised by the conventional melt quenching method. These series glass compositions are $\{[(\text{B}_2\text{O}_3)_{0.25}(\text{TeO}_2)_{0.75}]_{0.75} [(\text{Bi}_2\text{O}_3)_{0.25}]\}_{1-x} [\text{Tm}_2\text{O}_3]_x$; where $x = 0, 0.005, 0.010, 0.015, 0.020, 0.025, 0.030$ mol %. The effect of Tm_2O_3 in bismuth boro-tellurite glasses on structural, optical properties, and gamma radiation shielding has been studied. The density measurement (Archimedes principle), amorphous phase (X-ray diffraction), structural changes (Fourier Transform Infrared (FTIR)) and theoretical calculation of parameter radiation shielding (WinXCom and Phy-X program) are provided for supportive evidence to gamma shielding and optical properties. The results show the glass densities increase with the increase of Tm_2O_3 content which is due to the high molar weight and also on an atomic radius of the glass modifier compared to the glass network $\text{TeO}_2\text{-B}_2\text{O}_3$. The increase in the densities of glasses is 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/cm³), and Tm₂O₃ (8.6 g/cm³). XRD results show the glasses are amorphous in nature, with slightly tendencies toward crystallinity. From the FTIR spectra, it can be seen that the glasses consist of TeO₃, TeO₄, BO₃, and BO₄ structural units. The optical band gap energy which was calculated from Tauc's plots decreases with an increased amount of Tm₂O₃ and increased the non-bridging oxygen (NBO) atoms. The increasing value of Urbach energy is attributed to the increasing defect in the glass structure. The shift of the absorption edge and decrease of E_g value are attributed to the progressive increase in the concentration of non-bridging oxygen (NBO) atoms. Present results also showed the shielding properties improved with increasing amounts of Tm₂O₃. The theoretical data of shielding parameters showed that Compton scattering is a major interaction in gamma energy. The $\{[(B_2O_3)_{0.25}(TeO_2)_{0.75}]_{0.75} [(Bi_2O_3)_{0.25}]\}_{0.970} [Tm_2O_3]_{0.030}$ glass sample were found to give the lowest mean free path and half value layer compared to other glass samples. The result of this glass sample even outperformed some standard concrete and commercial radiation shielding glasses.

ABSTRAK

Pada masa kini, pengeluaran terbesar dalam pembuatan kaca adalah silika. Ramuan utama dalam pengeluaran kaca silika adalah batu kapur, soda abu, dan silika serta pasir. Malangnya, pengeluaran kaca ini boleh meningkatkan suhu global disebabkan suhu leburannya. Plumbum adalah salah satu elemen penting dalam penghasilan sifat optik dan kaca pelindung radiasi kerana kepadatan tinggi, nombor atom tinggi, dan tahap kestabilan yang tinggi. Namun, toksisiti plumbum boleh merosakkan alam sekitar dan bersifat karsinogenik kepada tubuh badan manusia. Plumbum boleh bioakumulasi dalam organisma dan proses penghasilan plumbum akan memberi kesan kepada kualiti alam sekitar. Dari masalah ini, terdapat keperluan untuk menggantikan plumbum dengan unsur lain yang lebih selamat. Kaca bismut boro-tellurit yang ditambah dengan thulium oksida berjaya disintesis melalui kaedah konvensional pelindapkejutan lebur. Komposisi kaca dalam siri ini adalah $\{[(\text{B}_2\text{O}_3)_{0.25}(\text{TeO}_2)_{0.75}]_{0.75} [(\text{Bi}_2\text{O}_3)_{0.25}]\}_{1-x} [\text{Tm}_2\text{O}_3]_x$; di mana $x = 0, 0.005, 0.010, 0.015, 0.020, 0.025, 0.030$ mol%. Kesan Tm_2O_3 dalam kaca bismut boro-tellurit terhadap sifat struktur, optik, dan pelindung radiasi gamma telah dikaji. Pengukuran ketumpatan (prinsip Archimedes), fasa kehabluran (pembelauan sinar-X), perubahan struktur (Transformasi Fourier Inframerah (FTIR)) dan pengiraan teoretikal parameter pelindung radiasi (program WinXCom dan Phy-X) disediakan sebagai bukti sokongan terhadap pelindung sinaran radiasi dan optik. Hasil menunjukkan peningkatan ketumpatan kaca dengan peningkatan kandungan Tm_2O_3 yang disebabkan oleh berat molekul tinggi pemodifikasi kaca berbanding dengan jaringan kaca $\text{TeO}_2\text{-B}_2\text{O}_3$. Peningkatan ketumpatan kaca juga disebabkan oleh peningkatan bilangan atom oksigen tanpa penititan (NBO) dan penggantian oksida B_2O_3 (2.46 g/cm^3) dan TeO_2 (5.67 g/cm^3) dengan oksida ketumpatan tinggi Bi_2O_3 (8.99 g/cm^3), dan Tm_2O_3 (8.6

g/cm^3). keputusan XRD menunjukkan kaca adalah pada fasa amorfus secara semulajadi, dengan kecenderungan sedikit kepada kristaliniti. Dari spektra FTIR, dapat dilihat bahawa kaca terdiri daripada unit struktur TeO_3 , TeO_4 , BO_3 , dan BO_4 . Tenaga jalur optik yang dikira dari plot Tauc berkurang dengan peningkatan kandungan Tm_2O_3 dan peningkatan atom oksigen tanpa penitian (NBO). Peningkatan nilai tenaga Urbach dikaitkan dengan peningkatan kecacatan dalam struktur kaca. Perubahan pinggiran serapan dan pengurangan nilai E_g dikaitkan dengan peningkatan progresif dalam kepekatan atom oksigen tanpa penitian (NBO). Hasil kini juga menunjukkan sifat pelindung meningkat dengan peningkatan kandungan Tm_2O_3 . Data teoretikal parameter perlindungan menunjukkan bahawa penyerakan Compton adalah interaksi utama dalam tenaga Gama. Sampel kaca $\{[(\text{B}_2\text{O}_3)_{0.25}(\text{TeO}_2)_{0.75}]_{0.75} [(\text{Bi}_2\text{O}_3)_{0.25}]\}_{0.970} [\text{Tm}_2\text{O}_3]_{0.030}$ didapati memberikan laluan bebas min dan lapisan nilai separuh terendah berbanding dengan sampel kaca lain. Hasil dari sampel kaca ini bahkan melebihi beberapa konkrit piawai dan kaca pelindung radiasi komersial.

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APPROVAL

The Examination Committee has met on **7 May 2024** to conduct the final examination of **Nor Falihan Binti Ramli** on his degree thesis entitled **Investigation of Structural, Optical, and Radiation Shielding Properties Bismuth-Boro-Tellurite Glass Doped with Thulium Oxide**.

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Academic session : 2024/2025

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LIST OF ABBREVIATIONS

XRD	-	Xray Diffraction
FTIR	-	Fourier Transform Infrared
UV-Vis	-	Ultraviolet & Visible
m	-	Mass
<i>M</i>	-	Molecular mass
BO	-	Bridging Oxygen
NBO	-	Non-Bridging Oxygen
RE	-	Rare Earth
tp	-	Trigonal pyramidal
tbp	-	Trigonal bipyramidal
UHPC	-	Ultra-high-performance concrete
UPNM	-	Universiti Pertahanan Nasional Malaysia

LIST OF SYMBOLS

α	- Absorption coefficient
NA	- Avogadro's number
BO	- Bridging oxygen
NBO	- Non-bridging oxygen
kGy	- Kilo Gray
mCi	- Mili Curie
$\langle d_{B-B} \rangle$	- Boron-boron separation
^{60}Co	- Cobalt 60
Cs-137	- Caesium 137
B_2O_3	- Boron trioxide
TeO_2	- Tellurium dioxide
Bi_2O_3	- Bismuth oxide
XRD	X-ray diffraction
FTIR	- Fourier Transform Infrared
UV-Vis	- Ultraviolet-visible
V_m	Molar Volume
ρ	Density
T	Time
LET	- Lead equivalent thickness
E_{opt}	- Optical band gap
α_m	- Molar polarizability
$\alpha_{\text{O}^{2-}}$	- Oxide ion polarizability
R_m	- Molar refraction
n	- Refractive index
I_0	- Incident intensity
I	- Transmitted intensity
μ	- Linear attenuation coefficient
μ_m	- Mass attenuation coefficient
Λ	- Basicity
M	- Metallization
HVL	- Half value layer

MFP	-	Mean free path
λ	-	Wavelength
γ -ray	-	Gamma-ray
N_{el}	-	Electron density
Z_{eff}	-	Effective atomic number

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CHAPTER 1

INTRODUCTION

1.1 Background

Modification of the glass surface composition is the most regular function of the material used in many industries (Stary et al., 2019). Glass is commonly used in many industries, largely due to the development of glass formulations that are opaque to visible light. On the other hand, polycrystalline materials do not usually transmit visible light (Barsoum, 2003). Enhancements in the usage of the glass surface play a crucial role in combining glass with different materials (Xiong et al., 2019). The structure of glass has been expanded to rationalize the apparent ease of different non-crystalline solid substances. It can be seen that non-crystalline solids can be classified into two different classes of thermodynamics: glasses and amorphous solids (Gupta, 1996).

To form glass, it is necessary to prevent crystallization. In certain conditions, the melts of the many substances can be cooled to reach a glass state. The amount and type of substances that are ready during a glassy or 'amorphous solid' condition have been greatly inflated with techniques within which the material is condensed onto a surface control well below its glass temperature. There is a minimum of some glass

former in each category of material, in step with bond sort, i.e., covalent, ionic, and metallic (Turnbull, 1969). A few preparation steps were used to create the glass. There is a method to select the powdery glass to melt when heated. Some methods need to be calculated, mixed, melted, and glass formed (Francis & Processing, 2016).

Heavy metal oxide Bismuth Oxide, Bi_2O_3 based on glasses has been investigated for a wide variety of applications in the field of glass ceramics, thermal and mechanical sensors, layers for optical and electronic devices, and reflected windows (Baia et al., 2002). They are satisfactory for the joining of rare earth particles and metallic nanoparticles and interesting materials for photonics (Tekin et al., 2019), and applications for white light generation systems, optical amplifiers, down converters, and upwards converter materials have been demonstrated.

Due to the limited field strength of Bi^{3+} ions, Bi_2O_3 cannot be regarded as a former network, but in combination with Boron Trioxide, B_2O_3 , glass arrangement is conceivable in a relatively large composition range (Baia et al., 2002). Glass modifiers such as Bi_2O_3 are applied to boro tellurite glasses to enhance the thermal, physical, and optical properties of these glasses. Bi_2O_3 is a heavy metal oxide and is valuable in electronics; structure applications, optical applications, and radiation shielding (Shaaban & Yousef, 2020).

Borate and tellurite glasses are commonly used as the host matrix among all the glasses due to their outstanding properties that are desirable for specific applications. Boron oxide or borate glasses are recognized as having structural units when monovalent alkali oxide or divalent alkaline earth oxide is combined with B_2O_3

of short-range order (SRO), and complex intermediate range order (IRO) (Pimentel et al, 2018). The structural units show the presence of BO_3 triangle boron and BO_4 tetrahedral boron units (Ibrahim et al., 2018). Glass system known as boro-tellurite glasses with a two-combination glass former of B_2O_3 and TeO_2 is important for research purposes as the coexistence of borate and tellurite in a single glass system will vary the number of network connectivity and coordinates that will change the properties of the glass. This combination of two glasses has been found to relate to the physical properties the properties of glass can produce. Boro-tellurite glasses produce non-linear optical properties, a high refractive index, and a wide infrared transmission window for different applications (Larink & Eckert, 2015; Tijani et al., 2019). Former B_2O_3 and TeO_2 network mixing have a negative impact on ionic conductivity. The structural concepts for mixed glass former effects in systems with different network former properties are also gaining great interest in boro-tellurite glass (Larink & Eckert, 2015). The existence of B-O stretching vibrations and $\text{O}_3\text{B-O-BO}_3$ bond bending vibration along the Te-O bending vibration in the boro-tellurite glasses has been found in the previous report (Maheshvaran & Marimuthu, 2012).

Thulium (Tm^{3+}) is generally considered to be one of the strongest active ions in the world (Gebavi et al., 2009). The thermal formation of metal oxides from inorganic precursors is broadly used commercially for the manufacture of technologically significant materials. Tm is an essential metal oxide, and its properties depend on the quality of the precursor used for its preparation, as well as on the pre-treatment conditions that establish its basicity (Hussein et al., 2000).

Rare earth (RE) metal oxides, such as thulium oxide, Tm_2O_3 have recently attracted a lot more interest from researchers, as RE metal oxides have shown high surface basicity, accelerated oxygen ion mobility, effective load transfer capability, and interesting catalytic properties, and are actively engaged in a range of scientific disciplines, including materials science, bioscience, and nanotechnology (Singh et al., 2014) and among the different RE metal oxides, Tm_2O_3 is being utilised in X-ray equipment, as an active agent of phosphors, as a control element of nuclear reactors, as a luminescent element, and in therapeutic and semiconductor applications.

At present, the bismuth-boro-tellurite glass doped thulium oxide, $\{[(\text{B}_2\text{O}_3)_{0.25}(\text{TeO}_2)_{0.75}]_{0.75} [(\text{Bi}_2\text{O}_3)_{0.25}]\}_{1-x} [\text{Tm}_2\text{O}_3]_x$ that focus on structural, optical and radiation shielding properties will be investigated by X-ray diffraction (XRD), Infrared Spectroscopy (FTIR) of Fourier Transforms, UV-Vis spectroscopy measurement and evaluate shielding by using WinXCom and Phy-X program. Since tellurite-based glass remains non-commercialized, prompting this study further investigates its shielding and optical properties. This study also many contributes additional insights into tellurite-based glass for future applications.