UNIVERSITI PUTRA MALAYSIA

STRUCTURAL, OPTICAL, ELASTIC AND GAMMA RADIATION SHIELDING PROPERTIES OF RICE HUSK-DERIVED SILICA BOROTELLURITE GLASS SYSTEM DOPED WITH BISMUTH OXIDE

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By

GEIDAM IBRAHIM GANA

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DEDICATION

This thesis is dedicated to my beloved late parent, Alhaji Muhammad Gana Geidam and Hajiya Halima Abdullahi. I sincerely missed them a lot and I pray to Allah (God) the Exalted to bless their souls and make them among the dwellers of highest place in paradise.

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

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Faculty : Science

Rice husk (RH) is a byproduct of rice mills reached in silica (SiO_2) contain. Milling industries discarded the RH in open field which causes serious environmental pollution. Previous investigation proved that the RH contain over 90% pure SiO_2 which could serve as alternative source of commercial silica in glass fabrication. Various aggregate of concrete and lead-based glasses are traditionally used for gamma radiation shielding application. But, degradation of concrete over time and toxicity of lead limited their function as an effective shielding materials. Silica of 98.36% purity was extracted from the RH using acid treatment method. A system of silica borotellurite glasses containing bismuth oxide (Bi_2O_3) with empirical chemical relation $[\{(TeO_2)_{0.7}(B_2O_3)_{0.3}\}_{0.8}(SiO_2)_{0.2}]_{1-x}(Bi_2O_3)_x$ (where x = 0.01 to 0.05 molar fraction in step of 0.01) were synthesized via melt-quenching procedure. Structural, optical, elastic and gamma radiation shielding properties of the glasses were investigated through XRD, FTIR, UV-Vis spectroscopies, ultrasonic pulse-echo and gamma transmission techniques. The density and molar volume increased simultaneously from 3.90 to 4.30 g/cm^3 and 30.785 to 31.508 cm^3/mol. with an increase in Bi_2O_3 content. The values of band gaps increased by the progressive addition of Bi_2O_3 from 1% to 3% and 5%, but decreased slightly at 4%. Metallization criterion (M_C) of the glasses matched with the values of non-metallic and nonlinear optical glasses. The refractive index (3.013-2.622), dielectric constant (9.078-6.875), optical dielectric (8.078-5.875), and electronic polarizability (α_m) were observed to decrease with the incorporation of more Bi_2O_3 content. The longitudinal (44.657 to 54.263 GPa), shear (19.119 to 19.946 GPa), bulk (17.784 to 27.668 GPa) and Young (43.886 to 49.156 GPa) moduli of the glasses' demonstrated non-uniform behaviour. Theoretical elastic models (Makishima-Mackenzie, Rocherulle and Bond Compression models) were calculated and compared with experimental data. The results revealed that only Makishima-Mackenzie model fitted excellently with the experimental elastic moduli and Poisson's ratio. The values of linear and mass attenuation coefficients (LAC and MAC), effective atomic number and effective density increased in order of increasing content of Bi_2O_3, $0.00Bi_2O_3 > 0.01Bi_2O_3 > 0.02Bi_2O_3 > 0.03Bi_2O_3 > 0.04Bi_2O_3 > 0.05Bi_2O_3$. Mean free path (MFP),

half-value layer (HVL) and tenth-value layer (TVL) decrease at the expense of increasing Bi_2O_3 concentration. Maximum values of density, MAC, LAC, effective atomic number, effective electron density and minimum values of MFP, HVL and TVL were achieved with sample containing greater % of Bi_2O_3, which demonstrates its superior shielding efficacy against samples with less bismuth. This indicates bismuth addition enhanced the overall shielding properties of the present glasses. The data of experimental shielding measurement was compared with XCOM and Phy-X/PSD software, and shows a very good correlation. Conclusively, the study succeeded in extracting 98.36% SiO_2 from RH. The optimum dopant concentration achieved at 0.05 molar fraction, which indicate possibility of adopting the glass sample $0.05Bi_2O_3$ as a new lead-free glass for radiation shielding application.

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

SIFAT STRUKTUR, OPTIK, KENYAL DAN PERISAIAN SINAR GAMMA BAGI SISTEM KACA SILIKA BOROTELLURIT DIDOP DENGAN BISMUTH OKSIDA

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Sekam padi (RH) adalah hasil sampingan kilang padi yang dicapai dalam kandungan silika (SiO_2). Industri pengilangan membuang RH di padang terbuka yang menyebabkan pencemaran alam sekitar yang serius. Siasatan sebelum ini membuktikan bahawa RH mengandungi lebih 90% SiO_2 tulen yang boleh berfungsi sebagai sumber alternatif silika komersial dalam fabrikasi kaca. Pelbagai agregat konkrit dan cermin mata berasaskan plumbum digunakan secara tradisional untuk aplikasi perisai sinaran gamma. Tetapi, degradasi konkrit dari semasa ke semasa dan ketoksikan plumbum mengehadkan fungsinya sebagai bahan perisai yang berkesan. Silika dengan ketulenan 98.36% telah diekstrak daripada RH menggunakan kaedah rawatan asid. Sistem gelas borotellurit silika yang mengandungi bismut oksida (Bi_2O_3) dengan hubungan kimia empirik $[\{(TeO_2)_{0.7}(B_2O_3)_{0.3}\}_{0.8}(SiO_2)_{0.2}]_{1-x}(Bi_2O_3)_x$ (di mana x = 0.01 hingga 0.05 pecahan molar dalam langkah 0.01) telah disintesis melalui prosedur pelindapkejutan cair. Ciri-ciri pelindung sinaran struktur, optik, elastik dan gamma bagi cermin mata telah disiasat melalui spektroskopi XRD, FTIR, UV-Vis, gema nadi ultrasonik dan teknik penghantaran gamma. Ketumpatan dan isipadu molar meningkat secara serentak daripada 3.90 kepada 4.30 g/cm^3 dan 30.785 kepada 31.508 cm^3/mol. dengan peningkatan kandungan Bi_2O_3. Nilai jurang jalur meningkat dengan penambahan Bi_2O_3 secara progresif daripada 1% kepada 3% dan 5%, tetapi menurun sedikit pada 4%. Kriteria pemekatan (MC) cermin mata dipadankan dengan nilai cermin mata bukan logam dan bukan linear. Indeks biasan (3.013-2.622), pemalar dielektrik (9.078-6.875), dielektrik optik (8.078-5.875), dan kebolehpolaran elektronik (α_m) diperhatikan berkurangan dengan penggabungan lebih banyak kandungan Bi_2O_3. Moduli longitudinal (44.657 hingga 54.263 GPa), ricih (19.119 hingga 19.946 GPa), pukal (17.784 hingga 27.668 GPa) dan Young (43.886 hingga 49.156 GPa) menunjukkan tingkah laku tidak seragam. Model anjal teori (model Makishima- Mackenzie, Rocherulle dan Bond Compression) telah dikira dan dibandingkan dengan data eksperimen. Keputusan menunjukkan bahawa hanya model Makishima-Mackenzie yang sesuai dengan moduli anjal eksperimen dan nisbah Poisson. Nilai pekali pengecilan linear dan jisim (LAC dan MAC), nombor atom berkesan dan ketumpatan berkesan meningkat mengikut tertib peningkatan kandungan Bi_2O_3, $0.00Bi_2O_3 > 0.01Bi_2O_3 > 0.02Bi_2O_3 > 0.03Bi_2O_3 > 0.04Bi_2O_3 > 0.05Bi_2O_3$. Purata laluan bebas (MFP), lapisan separuh nilai (HVL) dan lapisan nilai kesepuluh (TVL) berkurangan dengan mengorbankan peningkatan kepekatan Bi_2O_3. Nilai maksimum ketumpatan, MAC, LAC, nombor atom berkesan, ketumpatan elektron berkesan dan nilai minimum MFP, HVL dan TVL dicapai dengan sampel yang mengandungi % lebih besar Bi_2O_3, yang menunjukkan keberkesanan perisai yang unggul terhadap sampel yang kurang bismut. Ini menunjukkan penambahan bismut meningkatkan sifat pelindung keseluruhan cermin mata sekarang. Data pengukuran perisai eksperimen dibandingkan dengan perisian XCOM dan Phy-X/PSD, dan menunjukkan korelasi yang sangat baik. Secara konklusif, kajian ini berjaya mengekstrak 98.36% SiO_2 daripada RH. Kepekatan dopan optimum dicapai pada 0.05 pecahan molar, yang menunjukkan kemungkinan untuk menggunakan sampel kaca $0.05Bi_2O_3$ sebagai kaca bebas plumbum baharu untuk aplikasi perisai sinaran.

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LIST OF ABBREVIATIONS

$\alpha_{O^{2-}}^{(E)}$	Band gap based oxide ion polarizability
$\alpha_{O^{2-}}^{(n)}$	Refractive index based oxide ion polarizability
ΔE	Urbach Energy
B_2O_3	Boron oxide
Bi_2O_3	Bismuth oxide
BO	Bridging oxygen
BSBT	Bismuth silicate borotellurite
CS	Compton Scattering
C_t/V_t	Packing Density
DBO	Double bonded oxygen
D_i	Diffusion constant
E	Young's modulus
EDXRF	Energy dispersive X-ray fluorescence
E_{opt}	Optical energy band gap
F	Average stretching force constant
FTIR	Fourier transform infrared
G	Shear modulus
G_t	Dissociation Energy
HVL	Half value layer
K	Bulk modulus
L	Longitudinal modulus
LAC/μ	Linear attenuation coefficient
MAC/μ_m	Mass attenuation coefficient

M_C	Metallization criterion
MFP	Mean free path
n	Refractive Index
N_A	Avogadro's number
n_b	Number of bond per unit volume
NBO	Non-bridging oxygen
N_c	Bond Number per Unit Formula
OPD	Oxygen packing density
PE	Photoelectric effect
PP	Pair production
R	Bond Length
RH	Rice husk
RHA	Rice Husk Ash
RHS	Rice Husk Silicate
R_i	Inter-ionic distance
R_m	Molar Refractive Index
R_P	Polaron radius
SBO	Single bounded oxygen
SBT	Silica borotellurite
SiO_2	Silicon oxide
TEM	Transmission Electron Microscopy
TeO_2	Tellurium oxide
T_s	Softening Temperature
TVL	Tenth value layer
U_m	Mean ultrasonic velocity

UV-Vis	Ultraviolet-Visible
V_L	Longitudinal velocity
V_m	Molar volume
V_o	Oxygen molar volume
V_S	Shear Velocity
XRD	X-ray diffraction
XRF	X-ray fluorescence
Z_i	Acoustic impedance
α	Coefficient of Absorption
α	Alpha
α_e	Electronic Polarizability
α_m	Molar polarizability
α_p	Thermal expansion coefficient
β	Beta
γ	Gamma
$\Delta\chi^*$	Optical electronegativity
ε	Dielectric Constant
ε_{opt}	Optical Dielectric Constant
θ_D	Debye Temperature
Λ_{th}	Optical Basicity
ρ	Density
σ	Poisson's ratio
χ	Linear Dielectric Susceptibility
χ_{av}	Average electronegativity

CHAPTER 1

INTRODUCTION

1.1 Background

Glass is a transparent solid material characterized by hardness, brittleness and rigidity. The estimated global demand for glass has risen to almost 150 million tons in recent years (Westbroek et al., 2021). Therefore, through proper selection and adjustment of chemical composition, glass can be fabricated into various shapes and sizes with better properties that will enable its vast applications. Tellurite glass is the most widely studied glass system, having numerous properties when compared with other glasses (Al-Hadeethi & Sayyed, 2019; Gaikwad et al., 2018). It is distinguished by its remarkable high rare earth (RE) ion solubility, optical nonlinearity, low melting temperature, broad optical transmission in the infrared (IR) region and low phonon energy (Aliyu et al., 2021; Halimah et al., 2017; Queiroz et al., 2019; Sayyed et al., 2020). In addition, they have a high linear and nonlinear index of refraction, resistance to corrosion, a high thermal expansion coefficient and less hygroscopic compared to boron, phosphate and other oxide glasses, which are accountable for their broad applications in science and engineering (Rao et al., 2016; Tanko et al., 2016). In tellurite composition, TeO_2 is the main host glass material but could not form a stable glass without the addition of another glass former/network modifier oxide (Kurudirek et al., 2018a; Sayyed & Lakshminarayana, 2018). Therefore, the weak Te–O bond in TeO_2 accommodates the incorporation of other oxides to form a stable glass.

Boron trioxide (B_2O_3) is a perfect glass former highly hygroscopic with superior thermal stability, optical transparency, smaller cation size, chemical durability, varying coordination numbers, lower melting and working temperature (Aljewaw et al., 2020; Hazlin et al., 2017; Faznny et al., 2019; Stalin et al., 2021). Previous studies justified that incorporation of B_2O_3 into tellurite glass will form a stable borotellurite glass system with enhanced properties (Al-Buriahi et al., 2020; Halimah et al., 2007; Halimah & Eevon, 2019). Borotellurite glass is a good compromise between low phonon energy, hygroscopic, thermal stability, chemical durability and ease of fabrication.

Rice husk-derived silicon dioxide (SiO_2) was introduced into borotellurite glass network considering its good mechanical strength, high viscosity, transparent to visible light and very low crystallization (Fanderlik, 1991; Issa & Mostafa, 2017), which will enrich the glass mechanical strength and resistance to chemical attack (Aliyu et al., 2021). The report of Asyikin et al. (2020), Umar et al. (2019) and Umar & Ibrahim (2020), showed that the addition of SiO_2 improved the glass mechanical properties. Fernandes et al. (2017) obtained high purity silica of about 98.7% to 99.6% from rice husk (RH). In another scholarly work by Tuscharoen et al. (2012), it is revealed that the silica extracted from rice husk ash (RHA) has better visible light transmission compared to commercial SiO_2.

Recent researches on silica borotellurite doped glasses includes the spectroscopic investigation of Er^{3+} ions doped SiO_2-B_2O_3-TeO_2 glasses by Aliyu et al. (2021), reported the possibility of utilizing the glass for laser, EDFA and optoelectronic applications. Meanwhile, Halimah et al. (2019) studied Er_2O_3-Ag_2O-SiO_2-B_2O_3-TeO_2 glasses and revealed the potentiality of using the glasses for solid state laser devices. Asyikin et al. (2020) synthesized SiO_2-B_2O_3-TeO_2 glasses doped with Sm_2O_3 nanoparticles and investigated their optical, structural and physical properties. Series of studies related to heavy metal oxides (HMO) in silicates (Hammad et al., 2017; Tekin et al., 2019), tellurite (Al-Hadeethi & Sayyed, 2019; Gaikwad et al., 2018), and borates (El-Mallawany et al., 2018) glass systems were reported.

Bi_2O_3 is eco-friendly HMO known with short field strength, high atomic number and density (Sayyed, 2016b). Glasses containing Bi_2O_3 have attracted great number of research scholars owing to their properties including high optical susceptibility, high polarizability, high optical basicity, high density and radioactive resistance (Dong et al., 2017; Madheshiya et al., 2020). The existence of BiO_6 and BiO_3 pyramidal structural units in Bi_2O_3 accounts for the dual behavior of bismuth oxide to act as a glass former/modifier at high and low concentration, respectively (Elsafi et al., 2021). In a previous research, Kaur et al. (2016) reported insertion of HMO upgrade the optical application of tellurite glasses. Kumar et al. (2018) and Elsafi et al. (2021) reported that incorporating Bi_2O_3 into glass network structure enhances the physical, optical, elastic and radiation shielding properties. Mariyappan et al. (2018) studied the effect of Bi_2O_3 on physical, structural and radiation shielding properties of Er^{3+} ions doped B_2O_3-Bi_2O_3-Na_2O-CaF_2 glasses.

Oo et al. (2012) reported that the addition of bismuth in tellurite glasses enhanced the optical properties. Research by Kurudirek et al. (2018), synthesized and investigated HPSg-Na_2O-Bi_2O_3 glasses. The findings unveiled that the addition of Bi_2O_3 improved physical and shielding parameters of the glasses and indicated the applicability of utilizing the glass for radiation protection. According to Sayyed et al. (2020), incorporation of Bi^{3+} ions into TeO_2-GeO_2-ZnO-BaO glasses increased density and effective atomic number, which correlated with better shielding effectiveness

This study is designed toward extraction of SiO_2 from waste RH to be utilized for the synthesis of eco-friendly bismuth-doped silica borotellurite glass systems. Density measurement, XRD, FTIR, UV-Vis, ultrasonic pulse-echo techniques and photon transmission alongside with WINXCOM, Phy-X/PSD were employed to examine the influence of Bi_2O_3 doping on the physical, structural, elastic, optical and radiation shielding properties of the RH-derived silica borotellurite glass system.

1.2 Problem Statement

Rice is the third-most-produced agricultural commodity in the world, with roughly 761.5 million tons (1,000 kilograms) produced in 2018 and the production is projected to increase due to global population growths (WPR, 2021). Rice is produced in approximately 120 countries across globe, although China (211 million tons) and India

(173 million tons) are the global leading producers of rice paddy in 2019, accounting for more than 50 percent total global production (WPR, 2021). Rice husk (RH) is a by-product of milled white rice, and it constitutes ~ 20% of the bulk grain weight (Hossain et al., 2018). RH disposal in landfills causes serious environmental contamination (Umar et al., 2020). To address the issue of waste disposal and environmental pollution, proper utilization of the waste in the mainstream production should be strictly adhered. This study will emphasis on the conversion of waste materials (rice husk) into wealth through simple and efficient dilute hot acid-leaching (dilute hydrochloric acid) method of silica extraction from rice husk.

In a modern globalization, the exposure to ionizing radiation (examples, X- and gamma-rays) has been increasing rapidly because of its broad applications in medical diagnostics, radiotherapy, sterilization of food and medical apparatus. Additionally, nuclear industries use radiation for research purpose, reactor fuel and power generation (El-bashir et al., 2017; Lakshminarayana et al., 2017; Zakaly et al., 2021). Consequently, a frequent personnel or occupational exposure to radioactive sources/materials might trigger serious health disorder such as human organs malfunctions, cardiovascular diseases, genetic mutation/cancer that led to death (Kebaili et al., 2021; Temir et al., 2021). In this vein, concerns have been raised by various radiation regulatory agencies and experts on how to reduce the risks of excessive human and environmental exposure to hazardous radiation.

For decades, different aggregates of concrete have been widely used as traditional shielding materials because of their density, cost, compressive strength and excellent attenuation features against X-rays and γ-rays. But investigations confirmed that prolonged exposure of concrete material to radiation resulted in the decrease in density, loss of water content, which led to crack formation (Sayyed et al., 2020). Additionally, the opaque nature of concrete to visible light limits its use as a radiation shield. Previous researchers proposed the use of alloys, polymers, ceramics, glass and glass-ceramics for shielding applications (Kavaz et al., 2020; Sayyed et al., 2018; Singh et al., 2018; Toyen et al., 2018; Vadavathi et al., 2021). Among other materials, glass is the most suitable material owing to its high stability, transparency, high capability of absorbing high gamma energy and flexibility in modifying its composition to suit specific applications. In all the different varieties of glasses, lead-based glasses were widely investigated and adopted as being highly effective and reliable in providing protection against a source of ionizing radiation (Issa et al., 2020; Matori et al., 2017; Tarim & Gürler, 2018). Despite early recognition and utilization of Pb-glasses as a perfect conventional radiation shield, radiation experts recently challenge the usage of Pb due to its toxicity. Therefore, it is a necessity to search for a new eco-friendly material that could effectively replace Pb for shielding applications.

In this research, the choice of SiO_2-B_2O_3-TeO_2 glass containing bismuth as a possible replacement of lead based-glasses for shielding energetic radiation emerged due to the superb properties of Bi_2O_3 such as; radioactive resistance, non-toxic, high density (8.9 g/cm^3), high atomic weight (465.96 g/mol), high refractive index, optical transmission and long infrared cut-off (Hussein et al., 2021). The above-mentioned outstanding properties of Bi^{3+} ions favor multiple applications in mechanical sensors, reflecting windows, radiation protection, medical and electronic devices (Gupta et al., 2017). In this end, the glasses were expected to serve as novel lead-free shielding materials. No available report in literature concerning the study of structural, optical, elastic and gamma shielding properties of rice husk-derived silica borotellurite glasses doped with bismuth oxide.

1.3 Aim and Objectives

The goal of this study is to synthesize and investigate the structural, optical, elastic, and gamma-ray shielding properties of bismuth oxide-doped silica borotellurite glasses. The aim will be fulfilled through the following objectives:

1. To extract silica from rice husk and utilize it for the synthesis of Bi_2O_3 doped rice husk-derived silica borotellurite glass system via melt-quenching technique.
2. To study the physical, structural, and optical properties of the prepared glasses.
3. To examine the role of Bi_2O_3 doping on ultrasonic velocities, elastic constants, Poisson's ratio, microhardness and compare between experimental elastic moduli with their corresponding values obtained using theoretical models.
4. To determine gamma-ray shielding effectiveness of the prepared glasses experimentally and compare with the computed value from XCOM and Phy-PSD software.

1.4 Scope of the Study

1) The study comprises of the extraction of SiO_2 from waste biomass (rice husk) and evaluation of percentage purity of the extracted SiO_2 using EDXRF spectroscopy.
2) Using the extracted SiO_2 and pure chemical oxides (Bi_2O_3, TeO_2 and B_2O_3) for the synthesis of bismuth doped bio-silica borotellurite glasses with empirical formula $\{[(TeO_2)_{0.7} (B_2O_3)_{0.3}]_{0.8} (SiO_2)_{0.2}\}_{1-x} (Bi_2O_3)_x$, where x = 0.01, 0.02, 0.03, 0.04 and 0.05 mol. fractions.
3) The influence of the Bi_2O_3 addition on physical, structural, optical and elastic properties will be analyzed via density measurement, XRD, FTIR, UV-Vis and non-destructive ultrasonic pulse-echo technique.
4) Finally, experimental approach will be employed to study X-ray/gamma-rays shielding properties of the glasses and the result will be compared with theoretically obtained values (using WinXCom program and Phy-X/PSD software).

1.5 Organization of thesis

This thesis comprises of chapter one up to chapter five. The first chapter presents the background of the research, problem statement, objectives and scope of the study. The second chapter contains previous literatures concerning glass, silicate glasses, borate glasses, tellurite glasses, borotellurite glasses, bismuth doped glasses, radiation shielding glasses, ionizing radiation and summaries of some related literatures. The third chapter presents and discusses the experimental procedures and materials utilized for SiO_2 extraction, glass fabrication and various characterization techniques. The fourth chapter contains results and discussions of the influence of Bi^{3+} ions addition on structural, physical, elastic, optical and gamma radiation shielding properties of silica borotellurite glass system. The fifth chapter contains conclusion in form of a summary of some essential findings of the present work, alongside recommendations for future research.

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