

PHYSICAL, STRUCTURAL AND OPTICAL PROPERTIES OF ERBIUM
DOPED TELLURITE GLASS WITH COPPER OXIDE NANOPARTICLES
EMBEDMENT

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UNIVERSITI TEKNOLOGI MALAYSIA

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NUR E'ZZATI NABILAH SYAQILAH BINTI ABDUL HAMID

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Specially dedicated to

My beloved father and mother

Abdul Hamid Bin Bagong

Norwati Binti Husin

My dearest siblings

Along, Nazrah, Jeha

My dedicated lectures,

My supportive friends,

Maisalmah, Zulaiha, Fiona, Alina, Syifa, SOTO members

My labmates,

And all my friends.

Thank you so much....

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ABSTRACT

Two series of glass with composition of $(70-x)\text{TeO}_2 - 10\text{ZnO} - 10\text{MgO} - 10\text{Na}_2\text{O} - (x)\text{Er}_2\text{O}_3$ where $0.0 \leq x \leq 2.0$ mol% and $(69-y)\text{TeO}_2 - 10\text{ZnO} - 10\text{MgO} - 10\text{Na}_2\text{O} - 1\text{Er}_2\text{O}_3 - (y)\text{CuO}$ where $0.05 \leq y \leq 1.50$ mol% are prepared using melt quenching technique. The physical properties are measured in terms of glass density, ionic packing density and molar volume while the glass hardness is determined by using Vickers Microhardness. The amorphous nature of the glass is determined by X-ray Diffractometer (XRD). The occurrence of copper oxide nanoparticles (CuO NPs) is verified by using High-Resolution Transmission Electron Microscopy (HRTEM). The structural and optical properties are characterized by using Fourier Transform Infrared (FTIR), UV-Visible-NIR spectrophotometer and Photoluminescence (PL) spectrometer, respectively. The glass density, molar volume, ionic packing density and hardness are found in the range of $(4.799 - 4.951)$ g cm⁻³, $(26.575 - 27.202)$ cm³ mol⁻¹, $(0.457 - 0.467)$ and $(235 - 349)$, respectively. It is found that all glasses are amorphous in nature while the presence of CuO NPs with lattice spacing 0.23 nm at (111) plane is verified by HRTEM analysis. FTIR spectrum exhibited three major bands which are attributed to Te – O – Te, TeO₄ and TeO₃ in the range of $400 - 4000$ cm⁻¹. Meanwhile, UV-Visible-NIR spectra reveal nine absorption peaks centered at about 407, 449, 488, 522, 545, 652, 799, 976 and 1530 nm originating from $^4\text{I}_{15/2}$ to $^2\text{H}_{9/2}$, $^4\text{F}_{5/2}$, $^4\text{F}_{7/2}$, $^2\text{H}_{11/2}$, $^4\text{S}_{3/2}$, $^4\text{F}_{9/2}$, $^4\text{I}_{9/2}$, $^4\text{I}_{11/2}$ and $^4\text{I}_{13/2}$ transitions respectively. Down-conversion emission spectra under 380 nm excitations shows four peaks centered at about 408, 530, 550, and 660 nm allocated to $^4\text{F}_{3/2} \rightarrow ^4\text{I}_{15/2}$, $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$, $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ transition. Meanwhile, up-conversion emission spectra under 980 nm excitations shows peaks three centered at about 530, 550, and 660 nm allocated to $^2\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$, $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ transition.

ABSTRAK

Dua siri kaca dengan komposisi $(70-x)$ TeO₂ – 10 ZnO – 10 MgO – 10 Na₂O – (x) Er₂O₃ dengan $0.0 \leq x \leq 2.0$ mol% dan $(69-y)$ TeO₂ – 10ZnO – 10MgO – 10Na₂O – 1 Er₂O₃ – (y) CuO dengan $0.05 \leq y \leq 1.50$ mol% disediakan menggunakan teknik pelindap-kejutan leburan. Ciri-ciri fizikal diukur dari segi ketumpatan kaca, isipadu molar dan ketumpatan kepadatan ionik sementara itu kekerasan kaca ditentukan dengan menggunakan kekerasan mikro Vickers. Sifat semulajadi amofus kaca ditentukan menggunakan pembelauan sinar-X (XRD). Kewujudan nanopartikel kuprum oksida (CuO NPs) disahkan dengan menggunakan transmisi electron mikroskopi yang beresolusi tinggi (HRTEM). Sifat-sifat struktur dan optikal dicirikan dengan menggunakan spektroskopi Transformasi Fourier infra merah (FTIR), spektfotometer UV-Visible-NIR dan spectrometer fotoluminesen (PL). Ketumpatan kaca, isipadu molar, ketumpatan padatan ionic dan kekerasan masing-masing didapati dalam julat $(4.799 - 4.951)$ g cm⁻³, $(26.575 - 27.202)$ cm³ mol⁻¹, $(0.457 - 0.467)$ dan $(235 - 349)$. Didapati semua kaca adalah adalah bersifat amofus sementara kewujudan CuO NPs dengan jarak kekisi 0.23 nm pada satah (111) disahkan dengan menggunakan analisis HRTEM. Spektrum FTIR memaparkan tiga jalur utama iaitu Te – O – Te, TeO₄ dan TeO₃ dalam julat 400 – 4000 cm⁻¹. Sementara itu, spektrum UV-Visible-NIR mendedahkan sembilan puncak serapan berpusat di sekitar 407, 449, 488, 522, 545, 652, 799, 976 and 1530 nm yang masing-masing berasal dari transisi $^4I_{15/2}$ kepada $^2H_{9/2}$, $^4F_{5/2}$, $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4I_{9/2}$, $^4I_{11/2}$ dan $^4I_{13/2}$. Spektra pancaran penukaran turun pada pengujaan 380 nm menunjukkan empat puncak berpusat di sekitar 408, 530, 550, and 660 nm dengan masing-masing berasal dari transisi $^4F_{3/2} \rightarrow ^4I_{15/2}$, $^2H_{11/2} \rightarrow ^4I_{15/2}$, $^4S_{3/2} \rightarrow ^4I_{15/2}$ dan $^4F_{9/2} \rightarrow ^4I_{15/2}$. Sementara itu, spektra pancaran penukaran naik pada pengujaan 980 nm menunjukkan tiga puncak berpusat di sekitar 530, 550, and 660 nm dengan masing-masing berasal dari transisi $^2H_{11/2} \rightarrow ^4I_{15/2}$, $^4S_{3/2} \rightarrow ^4I_{15/2}$ dan $^4F_{9/2} \rightarrow ^4I_{15/2}$.

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LIST OF ABBREVIATIONS

BO	-	Bridging oxygen
CB	-	Conduction band
CCD	-	Charge-couple device
CuO NPs	-	Copper oxide nanoparticles
DC	-	Down-conversion
Er ₂ O ₃	-	Erbium oxide
eV	-	Electron Volt
FFT	-	Fast Fourier Transform
FTIR	-	Fourier Transform Infrared
g	-	gram
HRTEM	-	High-Resolution Transmission Electron Microscopy
KBr	-	Potassium Bromide
LSPR	-	Localized surface plasmon resonance
MgO	-	Magnesium Oxide
NR	-	Non-radiative
Na ₂ O	-	Sodium Oxide
NBO	-	Non-bridging Oxygen
NIR	-	Near Infrared
nm	-	Nanometer
PL	-	Photoluminescence
RE	-	Rare earth
TeO ₂	-	Tellurium Dioxide
tbp	-	Trigonal bipyramids

tp	-	Trigonal pyramid
UV	-	Ultraviolet
UC	-	Up-conversion
Vis	-	Visible
VB	-	Valence band
XRD	-	X-ray Diffraction
Xe	-	Xenon
ZnO	-	Zinc oxide

LIST OF SYMBOLS

θ	-	Bragg angle
\AA	-	Angstrom
A	-	Absorbance
α_e	-	Electronic polarizability
α	-	Absorption coefficient
β	-	Nephelauxetic ratio
$\bar{\beta}$	-	Nephelauxetic effect
c	-	Speed of light
d	-	Indention diagonal length / interatomic spacing
E	-	Energy
E_g	-	Optical band gap energy
E_u	-	Urbach energy
F	-	Test load
h	-	Planck constant
H	-	Hardness
H_V	-	Vickers hardness
I	-	Intensity of light transmitted from sample
I_o	-	Intensity of light entering the sample
M	-	Molecular weight
n	-	Refractive index
N_A	-	Avogadro's number
ρ_l	-	Density in immersion fluid
ρ	-	Density
ρ_a	-	Density of air

r_M	-	Ionic radius of metal
r_o	-	Ionic radius of oxygen
T_g	-	Glass Transition temperature
T_m	-	Glass Melting temperature
T_c	-	Glass Crystallization temperature
T	-	Transmittance
\bar{v}	-	Wavenumber
v	-	Frequency
V_M	-	Molar volume
V_i	-	Ionic packing density
v_a	-	Wavenumber in aquo ion system
v_c	-	Wavenumber in host matrix
W_a	-	Weight of sample in air
W_l	-	Weight of sample in immersion liquid
x_i	-	Molar Fraction
δ	-	Bonding parameter
λ	-	Wavelength

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Nowadays, glass plays important role in our daily life. It has attracted strong attention in both technologically and scientifically. The evolution of glass expended and the advent of technology provide new opportunities for glass applications such as in electronics and optical fibres for telecommunications industry. Glasses can be defined as an amorphous solid completely lacking in long range, periodic atomic structure and more important exhibits a region of glass transformation behaviour [1]. It is hard, brittle and usually transparent. It also can be obtained by quenching of melts or rapid cooling [2]. Lately, an extensive research has been done to obtain the best compositions for host, glass modifier and rare earth (RE) to improve the properties of the glass. The use of tellurite glass as a host material is demanded for the past few years due to its various and unique properties compare to other oxide glasses. It has high refractive index, good transmission in infrared region, low melting temperature ($\sim 800\text{ }^{\circ}\text{C}$) and low phonon energy that make tellurite glass suitable to be used in laser applications [3]. Moreover, tellurite glass has Te-O bond that can break easily and suitable as a host to be doped with heavy metal or RE ions [4].

However, under normal quenching conditions, pure tellurium dioxide (TeO_2) cannot easily form glasses by itself [5]. By introducing other oxide name as glass modifiers such as alkaline oxide, alkaline earth oxide or transitions metal oxides, high transparent of tellurite glass can be obtained. Among alkali oxides, sodium oxide (Na_2O) is classified as the best modifier due to the most glass forming ability [6]. Meanwhile, addition of zinc oxide (ZnO) as a modifier improved the opacity, chemical durability and melting properties of glass that is necessary for optical properties in making glass filter [7] and increase the glass transition temperature (T_g) of the glass [8]. Furthermore, addition of another modifier which is magnesium oxide (MgO) disturb the glass network and promotes the formation of large number of non-bridging oxygen (NBO) groups [9].

Tellurite glass doped with RE elements has great interest on their properties that leads toward worldwide applications [10]. Er^{3+} is one of the most important RE materials used in the research. Introduction of erbium oxide (Er_2O_3) help to improve the optical properties of tellurite glasses. By using high concentrations of RE ions luminescence enhancement can be obtained [11]. Tellurite glass doped with erbium oxide are extensively used in optical and photonic applications such as in making waveguide amplifiers and optical fibre [12]. In addition, Yousef *et al.* reported Er^{3+} doped tellurite glasses which are very useful for nonlinear optical device and up-conversion lasers [4]. Thus, Er^{3+} ions doped tellurite glass will present good performance in optical applications. On the other hand, some of the researcher reported that high concentrations of RE ions can cause luminescence quenching. So, the content of RE ions must be low in order to enhance the optical characteristic of the glass. For this reason, low concentration of RE ions doped to the glass consist of nanoparticles (NPs) are of considerable interest [13].

Addition of NPs into the glass network leads to the strong structural changes of the glass as a result of changing the environment felt by RE ions. Thus, introduction of NPs help to modify the spectral behaviour and improved the luminescence efficiency of the glasses. NPs play a role as a transmitter, meanwhile excited Er^{3+} ions become emitters due to the energy transfer between NPs and Er^{3+}

ions [14]. Transition metal ions are the simplest and perfect dopants for glass since they are identified by the presence of partly filled d shells and are strongly related with the magnetic, electrical and optical features of glasses [15]. Recently, transition metal oxide such as copper oxide (CuO) are of considerable interest due to its behaviour that present two different valence states in the glass system which are Cu^{1+} monovalent and Cu^{2+} divalent where Cu^{2+} ions present as the stable element [16]. Interestingly, luminescence quenching of Cu^{2+} ions with various RE-doped glasses have attracted attention. Addition of CuO NPs into the glass network make the glass electrically good semiconductor and super-paramagnetic [15]. CuO NPs have good thermal stability and low bulk resistivity despite of its cost effective material compare to silver and gold [17]. CuO NPs doped glasses have received significant attention in the field of materials science in their potential for various optical device applications such as lighting systems, optical fibres and photonic waveguides [18].

1.2 Problem Statement

It is well reported that RE ions doped glasses have many applications and become interest based on active glass material especially in optical devices in laser applications, waveguide amplifier and fibre for optical transmission [5]. In the last decade, luminescence properties of RE doped tellurite glass have been investigated. Increase the concentrations of Er^{3+} ions causing quenching effect through energy transfer between Er^{3+} ions. Despite much effort, optimization the luminescence enhancement of Er^{3+} ions remains challenges and need further improvement. Therefore, it is important to know the best composition of the glass. Thus, the luminescence properties of the glass can be enhanced [13].

Glasses containing RE ions and metallic NPs attracted much attention due to its enhancement in optical properties [19]. Many studies had been done involving transition metal ions with various glasses. CuO NPs is one of the transition metal

that is practically importance due to its unique properties [17]. Yet, most of the study focuses of doping gold and silver NPs to the host glass but embedment of CuO NPs doped glass are far from being understood and require further attention.

In addition, interactions of CuO NPs with the atoms inside the glass matrix embedded into Er³⁺ doped tellurite glass are at present lacking in the literature and not much explored. The detailed studies on the effect of CuO NPs on the absorption and luminescence emission are important to be determined and should be done for further investigation. Therefore, due to the lack of reports in this study, it is very important to study the effect of CuO NPs doped with RE especially Er³⁺ doped tellurite glass in the physical, structural and optical properties to give better information with beneficial features.

1.3 Objective of the Study

The objectives of this study are:

- i. To prepare two series of RE doped tellurite glass of composition (70–x–y)TeO₂ – 10ZnO – 10MgO – 10Na₂O – (x)Er₂O₃ – (y)CuO with and without CuO NPs by melt quenching technique.
- ii. To verify the presence of CuO NPs nucleated inside the glass.
- iii. To determine the influence of different concentration of Er³⁺ ions and CuO NPs on the physical, structural and optical properties into tellurite glass.

1.4 Scope of the Study

In order to achieve the objectives, this study has been divided into several scopes as follows:

- i. Preparations of two glass series via melt quenching technique.
 - (a) Glass with different concentration of RE with molar composition $(70-x)\text{TeO}_2 - 10\text{ZnO} - 10\text{MgO} - 10\text{Na}_2\text{O} - (x)\text{Er}_2\text{O}_3$ where $(0.0 \leq x \leq 2.0 \text{ mol\%})$.
 - (b) Glass with different concentrations of CuO NPs with molar composition $(69-y)\text{TeO}_2 - 10\text{ZnO} - 10\text{MgO} - 10\text{Na}_2\text{O} - 1\text{Er}_2\text{O}_3 - (y)\text{CuO}$ where $(0.05 \leq y \leq 1.5 \text{ mol\%})$.
- ii. Determination of the amorphous nature of glass using X-ray Diffraction (XRD).
- iii. Determination of physical and mechanical properties of both glass series.
- iv. Confirmation of the existence CuO NPs using HRTEM.
- v. Determination of the types of bonding vibration in glass with and without CuO NPs using Fourier Transform Infrared spectroscopy (FTIR).
- vi. Determination of the influence of different concentration of Er^{3+} ions and CuO NPs on the absorption spectra using UV-Vis-NIR spectroscopy and emission spectra using Photoluminescence spectroscopy (PL).

1.5 Significance of the Study

Studies on the glass material become important due to the large potential in diverse applications such as in optics and laser system. In this study, the role of Er³⁺ doped tellurite glass embedded with CuO NPs will be explained by controlling and optimizing the content of RE and NPs. Understanding the physical, structural and optical properties of this glass is important to determine the optimum composition of CuO NPs. This is due to the CuO NPs present their unique properties with extremely small dimensions and magnetic behaviour. Therefore, this research is done to give better understanding and to enhance the efficiency of tellurite glass for development wide range applications especially in nano-glass technology.

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