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ABSTRACT

In this study, the structural, optical and thermoluminescence (TL) properties of lithium sodium borate (LNB) glass doped with Er^{3+} or Nd^{3+} ions were investigated. The glass dosimeters were prepared by using melt-quenching technique. The X – ray Diffraction (XRD) spectra had confirmed the amorphous nature of the glass samples. The absorption spectra of Er^{3+} ion doped LNB (LNB:Er) and Nd^{3+} ion doped LNB (LNB:Nd) glasses revealed hypersensitive transitions at (${}^4\text{I}_{15/2} \rightarrow {}^2\text{H}_{11/2}$) and (${}^4\text{I}_{9/2} \rightarrow {}^4\text{G}_{5/2} + {}^2\text{G}_{7/2}$), respectively. Emission spectra of LNB glasses doped Er^{3+} ions had shown three violet bands centered at 369 nm (${}^4\text{G}_{11/2} \rightarrow {}^4\text{I}_{15/2}$), 396 nm (${}^2\text{P}_{3/2} \rightarrow {}^4\text{I}_{13/2}$) and 423 nm (${}^2\text{H}_{9/2} \rightarrow {}^4\text{I}_{15/2}$) and a blue band at 477 nm (${}^2\text{P}_{3/2} \rightarrow {}^4\text{I}_{11/2}$). Meanwhile, LNB glasses doped with Nd^{3+} ions had exhibited three prominent peaks of photoluminescence spectra which are green, orange and red bands centered at 538 nm (${}^4\text{G}_{7/2} \rightarrow {}^4\text{I}_{9/2}$), 603 nm [${}^4\text{G}_{7/2} \rightarrow {}^4\text{I}_{11/2}$, ${}^4\text{G}_{5/2} \rightarrow {}^4\text{I}_{9/2}$] and 675 nm [${}^4\text{G}_{7/2} \rightarrow {}^4\text{I}_{13/2}$, ${}^4\text{G}_{5/2} \rightarrow {}^4\text{I}_{11/2}$], respectively. The introduction of rare earth ions into the glass system had led to the formation of glow curve with prominent peaks at 180 and 220 °C for LNB:Er and LNB:Nd, respectively. The optimum TL intensity of the prepared glasses was determined at 0.3 mol % of Er_2O_3 and 0.5 mol % of Nd_2O_3 . A good linearity of the TL response against dose was achieved between 0.5 to 4.0 Gy. The sensitivity of 0.3 mol % of Er_2O_3 glass dosimeter was two times higher than the sensitivity of 0.5 mol % of Nd_2O_3 . The calculated values of the effective atomic number (Z_{eff}) for both compositions were 7.55 which is close to that of the human soft tissue ($Z_{\text{eff}} = 7.42$). These promising features had demonstrated the potential of the studied glasses to be used as radiation dosimeter.

ABSTRAK

Dalam kajian ini, sifat-sifat struktur, optik dan termopendarcahaya bagi kaca litium natrium borat (LNB) didopkan dengan ion Er^{3+} atau ion Nd^{3+} telah disiasat. Dosimeter kaca telah disediakan dengan menggunakan teknik lebur lindap. Spektrum pembelauan sinar-X telah mengesahkan sifat amorfus sampel kaca. Spektrum penyerapan kaca LNB didopkan dengan ion Er^{3+} (LNB:Er) dan kaca LNB didopkan dengan ion Nd^{3+} (LNB:Nd) telah menunjukkan peralihan hipersensitif di (${}^4\text{I}_{15/2} \rightarrow {}^2\text{H}_{11/2}$) dan (${}^4\text{I}_{9/2} \rightarrow {}^4\text{G}_{5/2} + {}^2\text{G}_{7/2}$). Spektrum pancaran kaca LNB didopkan dengan ion Er^{3+} menghasilkan tiga jalur ungu berpusat di 369 nm (${}^4\text{G}_{11/2} \rightarrow {}^4\text{I}_{15/2}$), 396 nm (${}^2\text{P}_{3/2} \rightarrow {}^4\text{I}_{13/2}$), 423 nm (${}^2\text{H}_{9/2} \rightarrow {}^4\text{I}_{15/2}$) dan jalur biru di 477 nm (${}^2\text{P}_{3/2} \rightarrow {}^4\text{I}_{11/2}$). Manakala, kaca LNB didopkan dengan ion Nd^{3+} mempamerkan tiga puncak utama dalam spektrum fotoluminesens iaitu hijau, jingga dan merah berpusat di 538 nm (${}^4\text{G}_{7/2} \rightarrow {}^4\text{I}_{9/2}$), 603 nm [${}^4\text{G}_{7/2} \rightarrow {}^4\text{I}_{11/2}$, ${}^4\text{G}_{5/2} \rightarrow {}^4\text{I}_{9/2}$] dan 675 nm [${}^4\text{G}_{7/2} \rightarrow {}^4\text{I}_{13/2}$, ${}^4\text{G}_{5/2} \rightarrow {}^4\text{I}_{11/2}$]. Pengenalan ion nadir bumi ke dalam sistem kaca telah membawa kepada pembentukan lengkung berbara dengan puncak utama pada 180 °C untuk dosimeter kaca LNB:Er dan 220 °C untuk dosimeter kaca LNB:Nd. Termopendarcahaya dosimeter kaca telah mencapai bacaan optimum pada kepekatan 0.3 mol% daripada Er_2O_3 dan 0.5 mol% daripada Nd_2O_3 . Kelinearan termopendarcahaya terhadap dos dicapai antara 0.5-4.0 Gy. Kepekaan dosimeter kaca bagi kepekatan 0.3 mol% daripada Er_2O_3 ialah dua kali lebih tinggi daripada kepekaan dosimeter kaca bagi kepekatan 0.5 mol % daripada Nd_2O_3 . Nilai nombor atom berkesan (Z_{eff}) yang dihitung untuk kedua-dua komposisi ialah 7.55, iaitu berhampiran dengan nilai bagi tisu lembut manusia ($Z_{eff} = 7.42$). Gambaran yang meyakinkan ini menunjukkan kaca yang dikaji berpotensi untuk digunakan sebagai pengukur dos sinaran.

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LIST OF SYMBOLS

D_o	–	Lowest detected dose
B^*	–	Mean TL background signals obtained from a group of dosimeters after annealing
σ_B	–	Standard deviation obtained from the mean background signals
F	–	Calibration factor
Z_{eff}	–	Effective atomic number
λ	–	Wavelength
d	–	Distance between atom layers
T	–	Temperature
P	–	Density
V_m	–	Molar volume
N	–	Ion concentration
r_p	–	Polaron radius
r_i	–	Inter-nuclear distance
d_{b-b}	–	Boron-boron separation
n	–	Refractive index
R_m	–	Molar refractivity
α_e	–	Polarizability
Z	–	Atomic number

N_A	–	Avogadro number
w	–	Fractional content
n_i	–	Number of electrons in one mole
a_i	–	Fractional numbers of electron

CHAPTER 1

INTRODUCTION

1.1 Overview

The growing applications of ultraviolet, X and gamma radiations, in various processes associated with industrial, medical and agriculture fields, has led to numerous on-going researches in the search of new compositions thermoluminescence dosimeter (TLD) material with desirable dosimetric properties (Rojas *et al.*, 2006). One of the techniques used for radiation measurements is known as thermoluminescence (TL). TL phenomenon works on the basis of forming a visual indication, in which the energy stored in an irradiated material, when heated, will be released in the form of light emission. The plot of intensity of the emitted light as a function of temperature forms a glow curve. The position, shape and intensity of the glow peaks are related to the properties of the trapping states responsible for TL. The parameters of producing a dosimeter of high precision include high sensitivity, good linearity, high chemical and thermal stability and low hygroscopicity (Furetta, 2003). In addition, it is desirable for the dosimetric material that is used in a personal dosimetry to be near tissue-equivalent. The dosimetric material that has a similar effective atomic number as that of human tissue ($Z_{eff} = 7.42$) will contribute to a more accurate determination of the absorbed dose in soft biological tissue exposed to ionizing radiation (Annalakshmi *et al.*, 2013).

The bold advantages of borate glass have become prominent in the TL field due to its properties of near tissue-equivalent, good rare earth (RE) ion solubility, transparency and easily handled process (Elkholy, 2010). However, the hygroscopic nature and relative instability of borate glass adversely affect its performance and often limit its practical uses (Elkholy, 2010). These drawbacks are overcome by incorporating suitable modifiers into the glass network. The introduction of modifiers enhances the stability and reduces the hygroscopic properties of the glass. Lithium oxide is one of the most frequently used modifiers to improve the stability of borate. The incorporation of lithium ions into the glass system leads to the creation of vacancy by increasing the dislocation and improving the strength of the host (Alajerami *et al.*, 2013a). Moreover, lithium borate appears to be one of the most attractive materials in personal dosimetry due to their effective atomic number, Z_{eff} of 7.3 (Singh *et al.*, 2011). Sodium oxide is also used as a modifier in borate networks. Following the electronic configuration of [Na] $3s^1$, the free electron in the outer L-shell of sodium with isoelectric sequence makes it extremely stable, particularly after the loss of extra electron on the 3s level (Alajerami *et al.*, 2013a).

The changes in TL features can be observed due to the presence of RE impurities in the host (Santiago *et al.*, 1998). The variation of dopant sites with different ion-host interactions results in differences of spectral properties due to the incorporation of RE in the glass. It could also form large complex defects and long range interaction which involves charge trapping and the recombination components of TL process, which ultimately enhance the TL response (Zhijian *et al.*, 2013). Erbium oxide (Er_2O_3) and neodymium oxide (Er_2O_3) are the examples of RE used as a dopant in the glass network due to its ability to enhance the TL and create new defects (Laxmi *et al.*, 2004 and Soliman, 2006).

1.2 Glass and Thermoluminescence

The efficiency of glass dosimeter in the field of radiation detection and thermoluminescence theory had been confirmed by literature studies. Silicon glass (SiO_2), boron glass (B_2O_3), phosphorus glass (P_2O_5) and germanium glass (GeO_2) are some of the compounds that can be used to synthesize pure glasses. The present study focuses on the formation of glass by using boron oxide as a host. Different types of material with modifiers and dopants can be used in radiation detection. Beside the glass form, the TL materials are also available in other forms such as hot pressed chips, pellets, powder and impregnated teflon disks. One of the conventional methods used to fabricate a glass is melt-quenching technique. Mel-quenching technique explains the process of melting the mixture under the melting point, before being annealed for three hours under the transition temperature of the host and finally being cool down for 12 hours at room temperature.

1.3 Problem Statements

The benefits of radiation are enormous and continuously increasing. It is also well known that the ionizing radiation can induce cancer and other genetic defects. Therefore, the estimation of risks using ionizing radiation is becoming much important. Dosimeter is one of the simple instruments that is used to measure or evaluate, either directly or indirectly, the absorbed dose of ionizing radiation. A radiation dosimeter must exhibit desirable characteristics such as tissue-equivalence, high sensitivity and excellent stability in order to function efficiently (Furetta, 2003). However, most dosimeters are failing in fulfilling the aforementioned characteristics. Therefore, the looking for new and high performance TL materials are becoming more intense.

Lithium fluoride (LiF:Mg,Ti), which is also known as TLD-100, has been used as a standard TLD phosphor and radiotherapy dose measurements due to its properties; tissue equivalence ($Z_{eff} = 8.04$), simple glow curve structure, sensitivity to small doses and has limited fading over a certain period of time (Seth *et al.*, 2012). Despite these remarkable properties, it shows two significant drawbacks; the complex annealing and supralinearity in the first 10 Gy doses. Similarly, lithium borate ($\text{Li}_2\text{B}_4\text{O}_7\text{:Mn}$) also shows low sensitivity due to incompatibility between the wavelength of the emitted light (600 nm) and the response of the TLD's reader (Schulman *et al.*, 1967).

Silicates, borates, phosphates and telluride glasses are identified as conventional glass forming systems due to their good glass-forming ability (Mahamuda *et al.*, 2013). Amongst the subjected glasses, borate glass is conceived as the preferred glass. The inclination towards borate glass is due to its properties of high transparency, low melting point, high thermal stability and good rare earth ion solubility (Venkatramu *et al.*, 2006). However, despite its superior advantages, borate alone easily crystallized and possesses hygroscopic nature (Alajerami *et al.*, 2012), and thus, countless studies have been conducted to improve its properties. Efforts are focused in introducing modifiers to fabricate the glass which is relatively moisture-resistant and mechanically strong compared to the pure borate glass (Balaji *et al.*, 2004).

The studies of RE doped borate glass have improved its potential applications in the field of radiation dosimeters based on TL (Anishia *et al.*, 2011). The addition of RE enhances the luminescence efficiency as discovered by Rojas *et al.* (2006) when the host matrices is activated with Dy^{3+} ions compared to the undoped glass. The experiments by Anishia *et al.* (2011) by using different RE ions (Tb^{3+} , Pr^{3+} , Ce^{3+} and Eu^{3+}) also showed similar findings. By considering the aforementioned potentials and possible applications, the present work is aimed at reporting the physical, structural, optical and thermoluminescence properties of lithium sodium borate glass doped with erbium or neodymium ions.

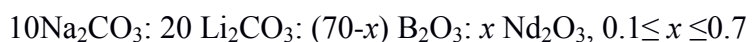
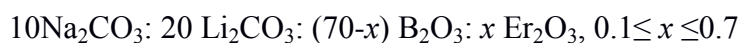
1.4 Objectives

The objectives of this study are:

- a. To study the physical and structural properties of newly proposed glass dosimeter lithium sodium borate doped with erbium (LNB:Er) and lithium sodium borate doped with neodymium (LNB:Nd).
- b. To examine the optical properties of LNB:Er and LNB:Nd (photoluminescence and absorption spectra).
- c. To investigate the thermoluminescence properties of LNB:Er and LNB:Nd (glow curve analysis, heating rate, annealing temperature and time, dose response and linearity, sensitivity, minimum detectable dose (MDD) and effective atomic number).

1.5 Significance of Study and Scope of Study

In the present study, two series of new glass compositions were synthesized by melt-quenching technique. The new composition of $\text{Li}_2\text{CO}_3\text{-Na}_2\text{CO}_3\text{-B}_2\text{O}_3$ (LNB) doped with different concentrations of erbium oxide or neodymium oxide were prepared, in which, four samples were prepared for each series in order to draw a comparison between both series. The glass was prepared based on the following composition;



This study will cover the following subjects:

- a. The characterization of amorphous phase of the glass dosimeter by using X-Ray diffraction (XRD).
- b. The characterization of vibrations mode of the glass dosimeter by using fourier transform infrared (FT-IR) spectroscopy.
- c. The evaluation of glass-forming ability and stability by using differential thermal analysis (DTA) measurement.
- d. The determination of absorption spectra and energy band gap by using UV-visible (UV-vis) spectroscopy.
- e. The determination of emission spectra by using photoluminescence (PL) spectroscopy.
- f. The determination of dosimetric properties of glass dosimeter according to basic thermoluminescence characteristics.

The present study attempted at fabricating a newly prepared glass which would serve as a promising candidate for radiation sensor and dosimetric applications.

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