DOSIMETRIC PROPERTIES OF LITHIUM MAGNESIUM BORATE GLASSES DOPED WITH DYSPROSIUM AND PHOSPHORUS OXIDE FOR RADIATION DOSE MEASUREMENT

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DEDICATION

To the soul of my friend Abdelrahman Alnajjar who drowned in Desaru beach. I really miss you.

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ABSTRACT

Series of lithium and magnesium oxide modified borate glasses of compositions $30Li_2O - (70 - x) B_2O_3 - xDy_2O_3$ where $0 \leq x \leq 1$ mol % (LB:Dy), $20Li_2O - 10MgO - (70\text{-}x) B_2O_3 - xDy_2O_3$ where $0.3 \leq x \leq 1$ mol % (LMB:Dy) and $20Li_2O - 10MgO - (69.5 - x) B_2O_3 - 0.5Dy_2O_3 - xP_2O_5$ where $0.5 \leq x \leq 2$ mol % (LMB:Dy,P) were prepared using melt-quenching method. The present study was performed with the aim of improving the thermoluminescence (TL) properties of lithium borate glass. The prepared glass samples were characterized by X-Ray diffraction (XRD) and differential thermal analysis (DTA). The room temperature photoluminescence (PL) emission spectra of the glass series at 350 nm excitation consist of two peaks centered at 481 nm and 573 nm corresponding to the transitions ($^4F_{9/2} \rightarrow {}^6H_{15/2}$) and ($^4F_{9/2} \rightarrow {}^6H_{13/2}$), respectively. The TL glow curves of LB:Dy (0.5 mol %) revealed a single prominent peak at a maximum temperature (T_m) of 190 °C. An enhancement of TL response about 1.4 times was observed with the presence of MgO as a second modifier to lithium borate. The addition of P_2O_5 as co-dopant into LMB:Dy (0.5 mol %) enhanced the TL intensity by a factor of 2.2, with the increase of P_2O_5 concentration up to 1 mol % and quenching effects occurred beyond this concentration value. LMB:Dy,P was found to have a good effective atomic number (Z_{eff}=9.05), linear dose response up to 100 Gy and showed a higher TL response compared to LMB:Dy and LB:Dy. The study of fading characteristic showed that LMB:Dy,P glass has lower fading compared to LMB:Dy and LB:Dy. The trap parameters, including the order of kinetics, activation energy (E) and frequency factor (s) for the glass samples were also determined. In conclusion, the prepared glasses have potential as a thermoluminescence material for radiation monitoring and dose measurement.

ABSTRAK

Siri kaca borat litium dan magnesium oksida diubahsuai komposisinya $30Li_2O$ - (70 - x) B_2O_3 - xDy_2O_3 dengan $0 \leq x \leq 1$ mol% (LB:Dy), $20Li_2O$ - 10MgO - (70-x) B_2O_3 - xDy_2O_3 dengan $0.3 \leq x \leq 1$ mol% (LMB:Dy) dan $20Li_2O$ - 10MgO - (69.5 - x) B_2O_3 - $0.5Dy_2O_3$ - xP_2O_5 dengan $0.5 \leq x \leq 2$ mol% (LMB:Dy,P) disediakan dengan menggunakan kaedah sepuh lindap. Kajian ini telah dijalankan dengan tujuan untuk meningkatkan sifat-sifat pendar cahaya terma (TL) kaca litium borat. Sampel kaca yang disediakan telah dipercirikan menggunakan analisis pembelauan sinar-X (XRD) dan analisis pembezaan terma (DTA). Pancaran spektrum fotoluminesens suhu bilik (PL) siri kaca pada pengujaan 350 nm terdiri daripada dua puncak berpusat di 481 nm dan 573 nm masing-masing sepadan dengan peralihan ($^4F_{9/2} \rightarrow {}^6H_{15/2}$) dan ($^4F_{9/2} \rightarrow {}^6H_{13/2}$). Lengkung berbara pendar cahaya LB:Dy (0.5 mol%) menunjukkan puncak tunggal pada suhu maksimum (T_m) 190 °C. Satu peningkatan sambutan TL kira-kira 1.4 kali ganda diperhatikan dengan kehadiran MgO sebagai pengubahsuai kedua litium borat. Penambahan P_2O_5 sebagai ko-dopan ke dalam LMB:Dy (0.5 mol%) meningkatkan keamatan TL dengan faktor 2.2, dengan peningkatan kepekatan P_2O_5 sehingga 1 mol% dan kesan pelindapan berlaku selepas nilai kepekatan ini. LMB:Dy,P didapati mempunyai nombor atom berkesan (Z_{eff} = 9.05), sambutan dos linear sehingga 100 Gy dan menunjukkan sambutan TL yang lebih tinggi berbanding dengan LMB:Dy dan LB:Dy. Kajian ciri kepudaran menunjukkan bahawa kaca LMB:Dy,P mempunyai kepudaran yang lebih rendah berbanding dengan LMB:Dy dan LB:Dy. Parameter perangkap, termasuk aturan kinetik, tenaga pengaktifan (E) dan faktor kekerapan (s) untuk sampel kaca juga telah ditentukan. Kesimpulannya, kaca-kaca yang disediakan ini berpotensi sebagai bahan pendar cahaya terma untuk pemantauan dan pengukuran dos sinaran.

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LIST OF ABBREVIATION

Al_2O_3:C	-	Aluminium oxide doped with carbon
ATR	-	Attenuated total reflectance
B_2O_3	-	Boron oxide
CB	-	Conduction band
CaF:Mn	-	Calcium fluoride doped with manganese
CaF:Dy	-	Calcium fluoride doped with dysprosium
DTA	-	Differential thermal analysis
Dy_2O_3	-	Dysprosium oxide
FOM	-	Figure of merit
FTIR	-	Fourier transform infrared
FWHM	-	Full width at half maxima
GCD	-	Glow curve deconvolution
IR	-	Initial rise method
LB:Dy	-	Lithium borate doped with dysprosium
$Li_2B_4O_7$:Mn	-	Lithium tetraborate doped with manganese
Li_2CO_3	-	Lithium carbonate
LiF:Mg,Ti	-	Lithium fluoride doped with magnesium and titanium
LINAC	-	Linear accelerator
LMB:Dy	-	Lithium magnesium borate doped with dysprosium
MDD	-	Minimum detectable dose
NBO	-	Non-bridging oxygen
PL	-	Photoluminescence
PMT	-	Photomultiplier tube
PS	-	Peak shape method
P_2O_5	-	Phosphorus oxide
RC	-	Recombination center

RE	-	Rare earth
RER	-	Relative energy response
SSD	-	Source surface distance
SSDL	-	Secondary standard dosimeter laboratory
TL	-	Thermoluminescence
TLD	-	Thermoluminescent dosimeters
UV	-	Ultraviolet
VB	-	Valence band
XRD	-	X-ray diffraction
XPS	-	X-ray photoelectron spectroscopy

LIST OF SYMBOLS

D_h	-	Demarcation for holes
D_e	-	Demarcation for electrons
T_m	-	Melting temperature
T_g	-	Transition temperature
BO_3	-	Triangular units
BO_4	-	Tetrahedral unit
Z	-	Atomic number
T_o	-	Irradiation temperature
E_g	-	Trap depth or activation energy
m	-	Concentration of holes
s	-	Frequency factor
b	-	Kinetics order
k	-	Boltzmann constant
A	-	Area under glow curve
μ_g	-	Geometric factor
β	-	Linear heating rate
P	-	Transition probability
$f(D)$	-	Linearity index
σ_B	-	Standard deviation of background
F	-	Conversion factor
B^*	-	Background signal
Z_{eff}	-	Effective atomic number
$S(E)$	-	Energy response
(μ_{en}/ρ)	-	Mass energy absorption coefficient
ρ	-	Density
V_m	-	Molar volume

M	-	Molecular weight
N_A	-	Avogadro’s number
X_B	-	Mole fraction
N	-	Ion concentration
r_p	-	Polaron radius
r_i	-	Inter-nuclear distance
T_c	-	Crystalline temperature
T_{rg}	-	Glass forming ability
H_R	-	Glass stability
f_{exp}	-	Oscillator strength
w	-	Fractional weight
$<d_{B-B}>$	-	Boron-boron separation

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Research Background

Radiation is an energy that is transmitted into the form of waves or particles. There are two kinds of radiations: ionizing and non-ionizing. In this study, some ionizing radiations will be discussed such as X-rays, gamma-rays, beta-rays, and other forms of penetrating radiations. Ionizing radiations are found in varying amounts in the environment and in increased amounts in hospitals, clinics, laboratories, and other establishments. It is desirable to guard against the probability of exposing being exposed to unsafe quantities of such ionizing radiations. So that, ionizing radiations require to be monitored and detected because the penetration power for ionizing radiation can cause cancer.

Ionizing radiation is involved in our lives and in many ways such as medical, nuclear and industrials fields. As a result of radiation applications, special protective considerations should be taken to reduce radiation hazards. A radiation dosimeter is one of the significant methods used to monitor and determine the absorbed dose. The most common types of radiation dosimeters that are used to monitor and detect ionizing radiation, e.g., ionization chambers, Geiger-Mueller counters, scintillation detectors, proportional counters, semiconductor diode detectors, thermoluminescence dosimeters (TLD) and X-ray film (Podgorsak, 2003).

Radiation dosimeters are commercially available for radiation dose measurement. These dosimeters are classified into two main categories: immediate read-out and delayed read-out. Ionization chamber dosimeters, proportional and Geiger Mueller counters are immediate and self-reading. However, ionization chamber dosimeters are small electronic dosimeters but they have some disadvantages like being expensive, the need for batteries and not being resistant to severe conditions such as very high or low temperatures and humidity. Proportional and Geiger Mueller counters are radiation detectors. In high doses, these counters are overwhelmed and cannot monitor the total dose exposure. TLD and X-ray film are delayed read-out and they require to be read out in laboratory for the determination of the dose. TLD and X-ray film can monitor as well as detect radiation over a very wide dose range, e.g., 0.1 mGy-10 Gy (Podgorsak, 2003).

As mentioned above, TLD is considered a very common type of delayed read-our dosimeters. Thermoluminescence (TL) is the phenomenon of light emission from solid materials formerly subjected to ionizing radiation under circumstances of increased temperature. A thermoluminescent material or phosphor has the features of taking up and storing energy in traps when subjected to ionizing radiation. Afterward, this energy is liberated from these traps by the heating of the material, with the production of a luminescent glow curve. The intensity of the emitted light released from the TL emission in the material depends on the nature and the quantity of impurities (dopants); the size of component particles of material; the network of defects present in the material and the effect of radiation interaction (McKeever, 1988).

TLD is generally used in personal monitoring (i.e. to monitor the radiation dose experienced by a person working in such a radiation environment) and in medical applications, e.g. radiation therapy. TLD used for the previous described applications is outstanding to other dosimeter systems because of its some properties like stability response in widely varying environmental conditions, reusability and the accordance of low average cost per dose measurement.

A diversity of TLD such as lithium fluoride (LiF, TLD-100 and TLD-600) and calcium sulphate ($CaSO_4$:Dy and $CaSO_4$:Tm) are known to be used in X-ray and gamma ray dosimetry. However, these dosimeters are used in low-level radiation detection because of their low-level saturation limit. The TLD 100 exhibits good sensitivity to radiation, but has two drawbacks: dose linearity (supra-linearity) and a complex annealing procedure (Furetta, 2003).

Through reviewing previous studies, these afore mentioned drawbacks were overcome by some researchers like Schulman *et al.* (1967) through the use the borate instead of fluoride in their studies. Schulman *et al.* (1967) was the first researchers to propose the use of borate as a TLD and to overcome these problems. Lithium tetraborate was activated with manganese oxide and manufactured in crystalline form. This phosphor material has attractive properties due to its effective atomic number (7.3) but shows low radiation sensitivity. This drawback was attributed to the incompatibility between the wavelength of the emitted light (600 nm) and the photomultiplier tube response of the TL reader (TLD reader).

An excellent work to enhance the sensitivity by using dysprosium oxide with a borate dosimeter was carried out by (Kazanskaya *et al.*, 1974). The dysprosium oxide shifts the luminescence light to shorter wavelengths (475 and 580 nm) which match the photomultiplier tube (PMT) response (Kazanskaya *et al.*, 1974). Anishia and colleagues (2011) studied the glow curve property of lithium magnesium borate doped with dysprosium (LMB:Dy). This dosimeter exhibited two intense peaks at 180 and 350 °C. The peak at 180 °C displayed an intensity three times higher than that at 350 °C.

Any TLD should possess several properties such as good linearity, high sensitivity, low energy dependence, low fading, a simple TL glow curve, and good reproducibility.

1.2 Problem Statement

This study encompasses an investigation of the performance of three series of TLD detector namely LB:Dy, LMB:Dy and LMB:Dy,P. In general, this study investigates these dosimeters in terms of their preparation, characterization and thermoluminescence properties.

As mention in the research background, Schulman was the first researcher used the borate in the dosimeters. This dosimeter was activated by manganese, which shows desired properties like effective atomic number but it has low radiation sensitivity (Schulman *et al.,* 1967). The sensitivity was improved using different transition elements and rare earth as an activator instead of manganese that shifted the red-light emission (600 nm) to the blue-light emission (Takenaga *et al.,* 1980; Kazanskaya *et al.,* 1974). Indeed, the emitted light with 480 nm wavelength (red emission spectra) enhanced the sensitivity more than ten times, and overcame the sensitivity drawback (Kazanskaya *et al.,* 1974).

Several studies were carried out to improve the properties of borate dosimeters in terms of their preparation methods, modifiers, and activator modification (Prokic 1980; Campos and Fernandes Filho 1990; Furetta *et al.,* 2000; Prokic 2000; Li *et al.,* 2004; Liu *et al.,* 2007; Jiang *et al*., 2008; Anishia *et al.,* 2010; Jiang *et al.,* 2010; Alajerami *et al.,* 2013a). The preparation methods are divided into three types: the single crystal technique (Fernandes *et al.,* 2008; Patra *et al.,* 2013; Ekdal *et al.,* 2014), the polycrystalline technique (Li *et al.,* 2005; Anishia *et al.,* 2010; Anishia *et al.,* 2011; Annalakshmi *et al.,* 2013; Kawashima *et al.,* 2014), and the glass system technique (Rao *et al.,* 2002; Nageswara Rao *et al.,* 2006; Yoshimura *et al.,* 2009; El-Adawy *et al.,* 2010; Elkholy, 2010; Ayta *et al.,* 2011; Alajerami *et al.,* 2012b; Aboud *et al.,* 2014).

Different types of alkali and alkaline earth metals were used as modifiers to reduce the hygroscopic properties and improve the mechanical stability. The addition of another modifier reagent improved the intensity, created disruption in the lattice, opened the network structure, weakened the bond strength, and lowered the viscosity of glass (Li *et al.,* 2004; Liu *et al.,* 2007; Jiang *et al.,* 2008; Jiang *et al.,* 2010; Ayta *et al.,* 2011; Alajerami *et al.,* 2013a; Aboud *et al.,* 2014; Hashim *et al.,* 2014). During the last decades, several alkaline earth metals oxides (such as BaO, CaO, MgO, ZnO, PbO, TeO, Bi_2O, and SrO) were used as modifiers to improve physical, optical, and TL properties (Santiago *et al.,* 2001; Li *et al.,* 2004;

Li *et al.,* 2008; Anishia, *et al.,* 2010; Aboud *et al.,* 2012; Alajerami *et al.,* 2012a; Alajerami *et al.,* 2012b; Annalakshmi *et al.,* 2013).

Various transition metals and rare earths were used as activators and co-activators to enhance the luminescence via the electrons' transition and to increase the number of trap centers (Furetta *et al.*, 2000; Prokic, 2000; Elkholy, 2010; Jiang *et al.*, 2010; Alajerami *et al.*, 2013; Alajerami *et al.*, 2013a; Hashim *et al.*, 2014).

One of the challenges confronting researchers is the quenching state resulting from the dopant activation. The co-dopant technique is an effective method used to overcome this drawback (Furetta *et al.,* 2000; Furetta *et al.,* 2001; Prokic 2001; Alajerami *et al.,* 2012a; Alajerami *et al.,* 2013a).

The problem statement is illustrated in the schematic shown in Figure 1.1.

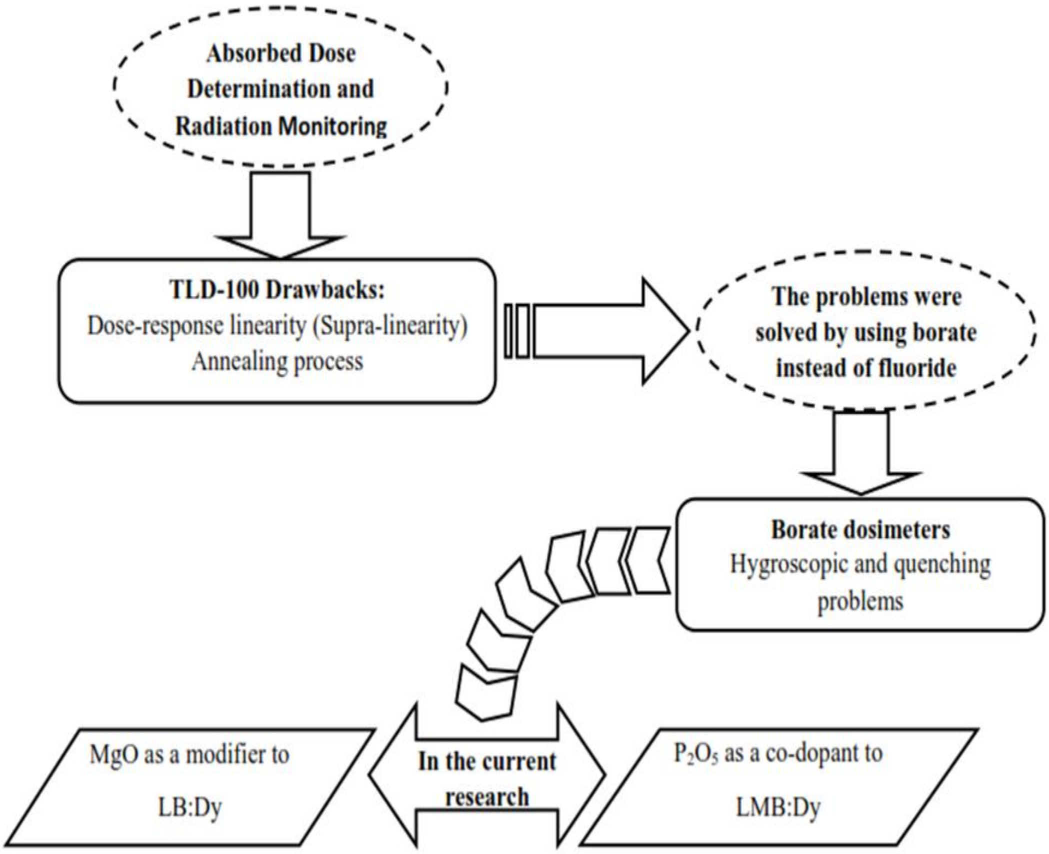

Figure 1.1 Schematic of the problem statement of the current study

1.3 Research Objectives

1. To determine the physical properties and characterization of the proposed dosimeters (LB:Dy, LMB:Dy and LMB:Dy,P) for understanding their structure

2. To examine the photoluminescence properties of the proposed dosimeters that contributes to the TL signals

3. To investigate the TL features such as the TL glow-curve, annealing procedure, reproducibility, fading, photon dose response, minimum detectable dose, TL sensitivity, effective atomic number, energy dependence, and kinetic parameters of the proposed dosimeters in order to evaluate their dosimetric properties.

4. To explore the role of magnesium oxide (MgO) as a second modifier and phosphorous oxide (P_2O_5) as a co-dopant in the TL properties of the proposed dosimeters which can be useful for their applications in radiation dosimetry

1.4 Scope of the Study

Lithium borates are attractive dosimeter hosts due to their tissue equivalent, good linearity, high sensitivity to external dose, low cost, and easy preparation. In this study, the melt-quench technique is exploited to prepare three series of glass dosimeters (LB:Dy, LMB:Dy and LMB:Dy,P). The amorphous nature of the proposed dosimeters are examined by X-ray diffractometer. The stability of the proposed dosimeters are checked by DTA. Physical properties such as density and molar volume; ion concentration (N); Polaron radius (r_p); internuclear distance (r_i) and field strength (F) are calculated. Photoluminescence of prepared glasses is measured. In addition, the dosimetric properties of the proposed dosimeters are studied.

1.5 Significance of the Study

The development of new glass dosimeters with their attractive dosimetric properties are of a great interest at the present time in radiation dosimeters. Such dosimeters can be provided by applying the rare earth (RE) ions in crystals and glasses. Among the REs, the Dy_2O_3 ion presents good dopants with borate in TL field. The simple glow curve of lithium borate glass system doped with Dy_2O_3 (LB:Dy) is the key to improve the dosimetric properties when compared to other dosimeters. In the current study, the introduction of MgO as second modifier and P_2O_5 as co-dopant in prepared dosimeters is the main interest to enhance its TL intensity and reduce the hygroscopic. This study provides great knowledge on the roles of the second modifier and the co-dopant on dosimetric properties of LB:Dy. The ideal glass dosimeters (LMB:Dy,P) can be used radiation dosimetry.

1.6 Thesis Outline

This thesis contains five chapters. A brief outline of each chapter is given below. Chapter 1 includes a research background. In addition, the problem statement and objectives of the study are presented

Chapter 2 presents the literature review. This chapter is divided into two parts. The first part includes the scientific data (i.e. TL materials, glass formation, borate glass, materials used in this study, luminescence phenomena, and theory of TL). Previous studies and related work are discussed in the second part of this chapter.

Chapter 3 provides a brief demonstration of experimental techniques utilized in the current study. This demonstration includes the preparation methods, the optimization process for current samples, and the instruments. These instruments are split into three parts: the characterization includes X-Ray Diffraction (XRD) and Differential Thermal Analysis (DTA), the photoluminescence (PL) and the TL study includes annealing furnaces, irradiation sources, and TLD readers.

Chapter 4 presents the results of the experiments described in Chapter 3. These results include the outputs from the characterization analysis and PL properties as well as the TL measurements.

Finally, Chapter 5 is devoted to the conclusions drawn from this study and recommendations for future studies.

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