

THERMOLUMINESCENCE PROPERTIES OF STRONTIUM-COPPER
CO-DOPING LITHIUM-BORATE GLASS FOR IONIZING
RADIATION APPLICATION

HAYDER KHUDHAIR OBAYES

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الحمد لله رب العالمين

Special dedication to:

To my beloved parents

Father. KHUDHAIR OBEYES KHUDHAIR and great Mother.

AZHAR HAMED MOUSA

Whose love, kindness, patience and prayer have brought me this far

For their love, understanding and support through my endeavours

To my siblings

(ENAS, ORAS & HASSANEEN)

Whose presence fills my life with joy and success

For their endless encouragement, laughs and cares,

Never forget to dedicate my full appreciation for my
beloved country that is dear to my heart in every step forward in my

life

(Iraq)

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ABSTRACT

Thermoluminescent dosimetry (TLD) has become a reliable and promising method used in ionizing radiation-dose measurements nowadays. There are wide range of TLD materials in use and among them the glassy matrix structure represents a potentially attractive system due to the outstanding properties such as good thermal stability, human tissue equivalent properties, relatively low cost, easily shaped and good ability to host luminescent activators in elevated concentrations. Many efforts have been devoted to develop tissue equivalent suitable glassy scintillator materials for ionizing radiation measurements. The aim of the present studies was to determine the influences of dopant (SrCO_3) and co-dopant (Cu_2O , Mg_2O , Na_2O , P_2O_5) on lithium-borate (LB) glasses upon their physical, structure, thermal and TLD properties. Six series of glass compositions $x\text{Li}_2\text{CO}_3-(100-x)\text{H}_3\text{BO}_3$, $15\text{Li}_2\text{CO}_3-(85-y)\text{H}_3\text{BO}_3-y\text{SrCO}_3$, $15\text{Li}_2\text{CO}_3-(83-z)\text{H}_3\text{BO}_3-2\text{SrCO}_3-z\text{Cu}_2\text{O}$, $15\text{Li}_2\text{CO}_3-(83-u)\text{H}_3\text{BO}_3-2\text{SrCO}_3-u\text{Mg}_2\text{O}$, $15\text{Li}_2\text{CO}_3-(83-v)\text{H}_3\text{BO}_3-2\text{SrCO}_3-v\text{Na}_2\text{O}$ and $15\text{Li}_2\text{CO}_3-(83-w)\text{H}_3\text{BO}_3-2\text{SrCO}_3-w\text{P}_2\text{O}_5$ with varying concentrations of x , y , z , u , v , w (in mol%) were synthesized using melt quenching technique. The amorphous phase, structure, composition, morphologies, thermal and physical properties of synthesized glass samples were characterized using X-ray diffraction (XRD), Fourier transform infrared (FTIR), Energy-dispersive X-ray (EDX) spectroscopy, Field emission scanning electron microscope (FESEM) and Differential thermal analysis (DTA) respectively. The TLD properties were measured in terms of thermoluminescence (TL) response, sensitivity, linearity, fading, reusability, minimum detectable dose, and Z-effective. These synthesized glass systems were exposed to various types of ionizing radiations such as Co-60 gamma ray, 6 and 10 MeV electrons, 6 and 10 MV X-ray photons. The XRD patterns confirmed the true amorphous state of all prepared glass samples. The FTIR results show that the structure of the glass samples is that of LB glass. The dopant (SrCO_3) and co-dopant (Cu_2O) in LB glass were not changing the main feature of the structure. The EDX analyses of samples show that the composition of the glasses is that of LB, its doped and co-doped. The FESEM results show homogeneous morphology. The DTA shows that the prepared glass samples are physically and thermally stable. Samples doped with 2.0 mol% of SrCO_3 and 0.01 mol% of Cu_2O concentration showed the highest TL efficiency. Furthermore, the co-doped glasses exhibited very significant TL properties such as linear dose response, good reusability, low minimum detectable dose and high sensitivity. The samples also showed good dose linearity characteristic and TL sensitivity in the dose range of 0.5-4.0 Gy when irradiated with 10 MeV electrons. The achieved effective atomic number of glass samples was found to be 9.69 and 11.08 for LB doped with 2.0 mol% of SrCO_3 and co-doped with 0.01 mol% of Cu_2O , respectively. The relative energy response of both doped and co-doped samples have been calculated theoretically and the results obtained are in good agreement with the experimental ones. In conclusion, the studied glass samples were found to have excellent properties required in TLD applications.

ABSTRAK

Dosimetri pendarcahaya terma (TLD) telah menjadi satu kaedah boleh percaya dan berpotensi dalam pengukuran dos sinaran mengion kini. Terdapat pelbagai jenis bahan TLD telah digunakan dan antaranya struktur matriks berkaca, merupakan satu sistem berpotensi yang menarik berdasarkan cirinya yang menonjol seperti kestabilan terma yang baik, sifat kesetaraan tisu manusia, kos yang agak rendah, mudah dibentuk, kemampuan yang baik untuk menjadi hos pengaktif pendarcahaya pada kepekatan tinggi. Banyak usaha ditumpukan untuk membangunkan bahan pengendip berkaca yang sesuai serta setara tisu untuk pengukuran sinaran mengion. Tujuan kajian ini adalah untuk menentukan pengaruh dopan (SrCO_3) dan kodopan (Cu_2O , Mg_2O , P_2O_5 , Na_2O) ke atas kaca litium-borat (LB) terhadap sifat fizikal, struktur, terma, dan TLD. Enam siri komposisi kaca $x\text{Li}_2\text{CO}_3-(100-x)\text{H}_3\text{BO}_3$, $15\text{Li}_2\text{CO}_3-(85-y)\text{H}_3\text{BO}_3-y\text{SrCO}_3$, $15\text{Li}_2\text{CO}_3-(83-z)\text{H}_3\text{BO}_3-2\text{SrCO}_3-z\text{Cu}_2\text{O}$, $15\text{Li}_2\text{CO}_3-(83-u)\text{H}_3\text{BO}_3-2\text{SrCO}_3-u\text{Mg}_2\text{O}$, $15\text{Li}_2\text{CO}_3-(83-v)\text{H}_3\text{BO}_3-2\text{SrCO}_3-v\text{Na}_2\text{O}$, dan $15\text{Li}_2\text{CO}_3-(83-w)\text{H}_3\text{BO}_3-2\text{SrCO}_3-w\text{P}_2\text{O}_5$ dengan kepekatan x , y , z , u , v , w (dalam mol%) yang berbeza telah disintesis menggunakan teknik pelindapan cair. Fasa amorfus, struktur, komposisi, morfologi, sifat terma dan fizikal sampel kaca tersintesis telah dicirikan masing-masing menggunakan pembelauan sinar-X (XRD), inframerah transformasi Fourier (FTIR), spektroskopi serakan tenaga sinar X (EDX), mikroskop elektron imbasan pancaran medan (FESEM), dan analisis terma pembeza (DTA). Sifat TLD diukur dari segi tindak balas pendarcahaya terma (TL), kepekaan, kelinearan, kelunturan, kebolegunaan semula, dos pengesanan minimum, dan Z-efektif. Sistem kaca tersintesis ini didedahkan kepada pelbagai jenis sinaran mengion seperti sinar gama Co-60, elektron bertenaga 6 dan 10 MeV, dan foton sinar X bertenaga 6 dan 10 MV. Pola XRD mengesahkan fasa amorfus sebenar semua sampel kaca yang disediakan. Keputusan FTIR menunjukkan struktur utama sampel kaca ialah kaca LB. Dopan (SrCO_3) dan kodopan (Cu_2O) dalam kaca litium borat tidak mengubah sifat utama struktur. Analisis EDX terhadap sampel menunjukkan komposisi sampel ialah komposisi LB, dopan dan kodopan. Keputusan FESEM menunjukkan morfologi yang homogen. DTA menunjukkan sampel kaca disediakan adalah stabil dari segi fizikal dan terma. Sampel didop dengan kepekatan 2.0 mol% SrCO_3 dan 0.01 mol% Cu_2O menunjukkan kecekapan TL paling tinggi. Tambahan pula, kaca dikodopkan menunjukkan sifat TL yang ketara seperti tindak balas dos yang linear, kebolegunaan semula yang baik, dos pengesanan minimum yang rendah dan sensitiviti yang tinggi. Sampel juga menunjukkan ciri kelinearan dos dan kepekaan dos yang baik dalam julat dos 0.5-4.0 Gy apabila disinarkan dengan elektron bertenaga 10 MeV. Nombor atom berkesan sampel kaca yang diperolehi ialah 9.69 dan 11.08 masing-masing bagi LB yang didopkan dengan 2.0% SrCO_3 dan dikodopkan dengan 0.01 mol% Cu_2O . Respon tenaga relatif bagi kedua-dua kaca LB terdop dan terkodop dikira secara teori dan hasil yang diperolehi bersesuaian dengan hasil eksperimen yang dijalankan. Kesimpulannya, sampel kaca yang dikaji didapati mempunyai ciri terbaik yang diperlukan dalam aplikasi TLD.

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LIST OF ABBREVIATIONS

CB	-	Conduction band
BI	-	Band gap
VB	-	Valence band
DTA	-	differential thermal analysis
EDX	-	Energy dispersive X-ray
ICRU	-	International commission of radiation unite
ERD	-	Environmental radiation dosimetry
ECC	-	Elemental correction coefficient
FESEM	-	Field emotion scan electron microscopy
LET	-	Low liner energy transfer
FTIR	-	Fourier transform infrared
ICRP	-	international commission on radiological protection
KE	-	Kinetic energy
LINAC	-	Linear accelerator
MeV	-	Million electron volts
MLC	-	Multi leaf collimator
MU	-	Monitor unit
MV	-	Megavolt
NBO	-	Non bridging oxygen
PMT	-	Photomultiplier Tube
PRD	-	personal radiation dosimetry
RD	-	Radiation dosimetry
RF	-	Radio frequency
TL	-	Thermoluminescence
TLD	-	Thermoluminescence dosimetry

TTP	-	Time temperature profile
XRD	-	X-Ray diffraction
TD	-	Threshold dose
TMAC	-	Total mass attenuation coefficient
GFA	-	Glass forming ability
KBR	-	Potassium bromide
CT	-	Computed tomography
MNCI	-	Malaysian national cancer institute
LLD	-	Lowest level of detection

LIST OF SYMBOLS

σ_B	-	Standard deviation
$(RER)_E$	-	Relative Energy Response
^{60}Co	-	Cobalt-60
Cu_2O	-	Copper(I) Oxide
D	-	Absorbed Dose
$D(t)$	-	Radiation dose at time
D_0	-	Minimum detectable dose
E_g	-	Energy band gap
F	-	Calibration factor of TL system
F	-	Field Strength
$f(D)$	-	Linearity index
H_3BO_3	-	Boric Acid
Li_2CO_3	-	Lithium Carbonate
m	-	Mass
MgO	-	Magnesium Oxide
M_i	-	average molecular weight
M_T	-	Total molecular weight
n	-	Refractive index
N_A	-	Avogadro's number
Na_2O	-	Sodium Oxide
P_2O_2	-	Phosphorus
$r_1(\text{\AA})$	-	inter-nuclear distance
R_m	-	Molar refraction
$r_p(\text{\AA})$	-	Polaron radius
$S_E(E)$	-	Photon energy response

SrCO_3	-	Strontium Carbonate
CC, R^2	-	Correlation coefficient
t	-	Time
V_M	-	Molar volume
V_m	-	molar volume
W_i	-	Weight fraction of element
x	-	Mole fraction
Z	-	Atomic number
Z_{eff}	-	Effective atomic number
P	-	Density
T_c	-	Glass crystallization temperature
T_g	-	Glass transition temperature
T_m	-	Glass melting temperature
Trg	-	Glass forming ability
Hr	-	Hruby parameter

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CHAPTER 1

INTRODUCTION

1.1 Study Background

In radiation safety related health physics, the term so called radiation dosimetry (RD) refers to the determination of the radiation dosage that is received by any matter including tissue upon exposing it to direct or indirect ionizing radiation. It indeed measures and evaluates directly or indirectly the amount of exposure in terms of equivalent or absorbed effective dose or some other quantities related to ionization radiation. The dosimetric dose range of interest depends on the source and nature of radiation. The specification provided by International Commission of Radiation Units (ICRU) are for personal dosimetry it ranges from 0.01 - 1.0 mSv, for X-ray diagnosis the range is 0.1 - 100 mGy and for radiotherapy it varies between 1 - 5 Gy [1].

Radiation monitoring devices are the only way to detect and measure the presence of radiation, which cannot be detected by sensor. In the environmental and medical dosimetry, different types of radiation detectors are used for quantifying the radiation dose. However, a single detector cannot measure all kinds of radiation or useful in all situations. The most popular dosimeters used for detecting ionization

radiation include ionizing chamber, film, luminescent and semiconductor materials. Other devices that are also used for this purpose are diamond dosimeter, plastic dosimeter, gel dosimeter etc.

Borate glass system is an interesting and potential material in radiation dosimetry. This glass system was first used for precious metal working and later in ceramic industries. It is well known that Boron and its compounds find extensive applications from glass to fibers, flame-retardants to nuclear applications and several others. Present developments in the area of radiation dosimetry for the protection from radiation exposure allowed the researchers to exploit borate glass as a novel thermoluminescence (TL) material, which is greatly potential for ionizing radiation dose measurement. Borate glasses are chemically stable compounds and can easily be doped with impurities such as rare earths, transition and alkaline metals. Such doped materials exhibit high sensitivity, linearity and good fading properties suitable for dose measurement [2]. In this regard, lithium borate (LB) is one of the appropriate materials for radiation dosimetry, particularly for clinical and radiation therapeutic application. It is because, the effective atomic number ($Z_{eff}=7.3$) of LB system is nearly equivalent to human tissue and easy to handle.

The LB based TL detectors are first commercially developed in 2001 [3]. The dosimetric properties of these materials ($\text{Li}_2\text{B}_4\text{O}_7$: Cu, In, Ag and $\text{Li}_2\text{B}_4\text{O}_7$: Cu) in the sintered pellet form are widely studied to determine their potential as tissue equivalent TLDs. These materials revealed glow curves with prominent dosimetric peaks and higher TL sensitivity. Furetta [4] examined the dosimetric properties of LB systems based TLDs. The annealing procedure of $\text{Li}_2\text{B}_4\text{O}_7$: Cu system is thoroughly examined. In addition, the TL sensitivity, glow-curve shape, minimum detectable dose, photon dose response, relative photon energy response, fading, reproducibility and precision of dose measurements of such material is inspected. Numerous natural and synthetic borates are exploited for diverse industrial applications. Generally, natural borates are cleaned from their impurities in processing plants and further treated to more qualified end products including anhydrous borax, anhydrous and hydrous boric acid, borax penta- and deca-hydrate, as well as sodium per-borate in re-crystallization process.

Inconsistency of borate chemistry [5], allows researchers to synthesize numerous borate structures usable in high technology areas. Despite all these several shortfalls in terms of efficiency, stability and accuracy remain utter challenges to overcome.

The choice of dosimetric materials critically depend on their several essential characteristics such as the good sensitivity and linearity between dose and TL response, low fading rate, inexpensive to manufacture, good stability through multiple readout cycles and a near-tissue equivalent of effective atomic number (Z_{eff}). The later one is very important because materials with higher or lower Z_{eff} than human tissue may misjudge the contributions of high energy photons leading to radiation damage. The search for the best material in terms of these characteristics added further impetus to the discovery and testing of a number of various novel glass compounds.

Radiation dose measuring instruments are essential for any environment having existing ionizing radiation. Apart from the specific properties possessed by TL materials for a particular application, some other basic and general conditions must fulfill. These include isolated glow curve around 200 °C (180 – 250 °C), Z_{eff} near to the human tissue, high signal per unit of dose, low fading characteristics, good linear dose response, easy annealing procedure, stability to chemical and environmental effects, non-toxicity, abundance and cheap composite materials. To date, a promising TL phosphor fulfilling all of the above mentioned characteristics is far from being achieved. Although several TL materials are developed and some reached the commercial level but they suffer from many disadvantages. Continuous efforts are made to either develop a new phosphor with an enhanced TL characteristics for improving the already existing TL material. This is achieved by modifying the host with other elements, changing or varying the doping element or co-doping the phosphor with new impurities.

Complex TL materials processing and lack of reusability has discouraged the use of film in radiation dosimetry [6]. Studies on lithium fluoride (LiF) by Harshaw Company revealed a very small TL sensitivity of this material due to the removal of unknown impurities during the processing procedure of the crystal. Titanium was found to be one of these impurities and was incorporated in this material to fabricate a phosphor having enhanced TL response. LiF is now considered as the standard TL phosphor known as 'TLD-100'. However, LiF phosphor has the disadvantage of complex annealing procedure and supra-linearity trend after the first 10 Gy dose [7, 8].

Over the years, several techniques are developed to prepare glassy TL host. Melt quenching technique is a promising method for preparing high quality glass easily and economically. This method is based on the melting process and subsequent rapid quenching of a metal oxide, where the viscosity is increased very quickly to a high value without forming the crystalline phase. Upon increasing the temperature in dark some minerals emit a transient glow called TL. It is this TL process that is exploited in radiation dosimetry. Denial *et al.* used these TL phenomena and measured the amount of radiation exposure on LiF. They acknowledged that this material is the most suitable phosphor for assessing the ionizing radiation exposure [9]. Currently, borate glass system owing to their lower fabrication cost, high sensitivity and easy availability received focused industrial attention for developing TL products.

Inspired by these notable attributes of LB phosphor, this work intends to develop a new Sr doped LB glass dosimeter for the precise measurement of ionizing radiation dosage. The TL properties of strontium (Sr) doped lithium borate glass and co-doped with copper (Cu), phosphorous (P), sodium (Na) and magnesium (Mg) are thoroughly examined. This is achieved by synthesizing a series of LB glasses via melt quenching method. The composition of co-doped LB: Sr, M (where M: Cu, P, Na and Mg) glass system are optimized and the prepared glasses are characterized to determine the effects of Sr contents on their structural and TL properties. Attempts are made to determine a relationship between co-dopants concentration and TL

response of the synthesized TLD. Experimental results are analyzed, interpreted and compared with other findings.

1.2 Problem Statement

Commercially available TLD-100 has some problem such as complex annealing procedure and supra-linearity trend at 10 Gy dose of exposure. Literature showed that the dosimetric properties of lithium borate system with Sr /Cu co-doping are not widely studied. Furthermore, the hygroscopic nature of lithium borate based glass system as well as the quenching effect, less sensitivity and fading behavior of the doping materials contributed to the setback of earlier developed phosphor. Thus, as a possible solution to the existing limitations the modification of strontium doped lithium borate system with copper co-doping is proposed.

1.3 Objectives of the Study

The main goal is to determine the fundamental TLD properties of Sr-Cu, Sr-Mg, Sr-Na and Sr-P co-doped Lithium borate glass system. Based on the problem statement the following objectives are set:

- 1) To evaluate the effects of Sr doping and Cu co-doping on the structure, chemical composition, thermal, morphological, TL and physical properties of Lithium borate glass systems.
- 2) To determine the influence for annealing temperature, annealing time and heating rate on the TL intensity of synthesized co-doped Lithium borate glass systems as proposed TLD.

- 3) To optimize the concentration of the Sr dopant and Cu, P, Na and Mg co-dopants in the Lithium borate glass best in TL intensity.
- 4) To determine the influence of TL properties of the Sr-Cu co-doped of Lithium borate glass system subjected to different types of irradiations (photons, electrons and cobalt-60 gamma) useful for TLD.

1.4 Scope of the Study

A new LB glass system doped with Sr and co-doped with Cu, P, Na and Mg are synthesized via melt-quenching method to evaluate their TL performance. These glasses are subjected to various photons, electrons and cobalt-60 gamma irradiations. The amorphous nature of all the un-doped, doped and co-doped glass are confirmed by X-ray diffraction (XRD) analysis. Glass morphology is analyzed using field emission scanning electron microscope (FESEM), where the fractional percentage of the composite elements is determined for effective atomic number of the proposed TLD. Thermal properties in terms of glass stability, transition temperatures and Hruby parameter are determined using differential thermal analyzer (DTA). The identification of elemental traces is performed using energy dispersive X-ray (EDX) measurement. The weight fraction obtained via EDX is further used for the calculation of effective atomic number. The effect of the modifier and the co-dopant concentration variation on the structure (bonding vibrations) of the proposed dosimeters is investigated by Fourier transform infrared (FTIR) spectroscopy. Physical properties including glass density, molar volume, ion concentration, polaron radius, inter-nuclear separation and field strength are calculated.

The TL measurements involves the determination of the dosimetric properties of Sr doped LB glass system co-doped with Cu, P, Na and Mg. The optimum concentration for doping and co-doping is evaluated in terms of TL response of this glass system. These optimum glass compositions are further selected to evaluate their dosimetric properties. The best combination of TL set up (annealing procedure, time in the rang 15-60 minutes and Annealing temperature in the range 100-400 °C, heating rate in the range 1-7 °C/s) are determined using the optimized Sr doped and Cu co-doped glass sample that give higher response compared to other compositions. These TL parameters remained constant throughout all TL characterization. The TL properties such as glow curve, dose response, sensitivity, fading, minimum detection dose, reproducibility, relative energy response, bleaching, accuracy of dose performance are evaluated under the exposure of various irradiations. The kinetic parameters including the activation energy and frequency factor are estimated using peak shape method and initial rise method for understanding of TL phenomenon. This method is shown to better justify the trap nature of the present glass system.

1.5 Significance of the Study

The proposed new materials can be used as good TLD system for clinical, personal and environmental dose monitoring applications. The performance of these TLD materials would ensure the dose delivered to the patients and workers more accurately in order to improve the level of safety in line with the guidance by the International Commission on Radiological Protection (ICRP).

The current study is expected to promote a better understanding on:

- 1) New material properties with optimize composition for accurate dose measurement leading to human safety.

- 2) Influence of co-doping and strontium on the TL properties of newly proposed glass system will be understood.
- 3) New glass composition may have commercial potential.
- 4) Fundamental understanding of the TL response.
- 5) Easy glass preparation, simple composition and low cost material would help large scale industrial production.
- 6) Accurate and efficient dose measurement using dosimeter based on this new composition.

1.6 Thesis Organization

This thesis is organized into seven chapters. Chapter 1 highlights the background of the study and the research gap to justify the need of this research. It includes the hypothesis and research question, research objectives, scope and significance of the study.

Chapter 2 provides a critical literature review relevant to this study. The general concept of luminescence and TL in particular as well as the theories and models associated with TL are emphasized. The dosimetric properties of TL phosphor are also discussed.

Chapter 3 describes the detail methodology and procedures in terms of instrumentation and analysis that are needed to fulfill the proposed objectives. It encloses the identification of material, glass sample preparation and description of sample characterizations.

Chapter 4 provides the results of various characterizations on thermal, physical, morphological and structural properties towards the fulfillment of the proposed objectives.

Chapter 5 presents the experimental results and analysis. The evaluation of TL properties, discussion, interpretation and comparisons. The results obtained from the characterization, temperature time profile settings and optimization of dopants and co-dopants are discussed in depth.

Chapter 6 depicts the main dosimetric properties of the proposed TLDs and their analysis in terms of various mechanisms.

Chapter 7 concludes the thesis with further information of this research in terms of recommendations for future work.

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