OPTICAL AND THERMOLUMINESCENCE PROPERTIES OF SAMARIUM OR DYSPROSIUM DOPED LITHIUM BORATE GLASS

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I dedicate this work

To my dear parents

Saifeddin Said Nazzal

Intisar Mohammad Nazzal

Ina’am Yousef Dawaud

To my lovely husband

Dr. Waheeb Abdel rahman Abu-Ulbeh

To my kids

Aon and Azm

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

To my siblings

For their endless laughs and tears

To my niece and nephew

Whose presence fills my life with joy

To my friends

For their love, understanding and support through my Endeavour

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In the name of Allah, the Most Gracious, Most Merciful. Praise to Allah S.W.T, Peace and blessings of Allah upon His Messenger, Muhammad S.A.W, and all his family and companions.

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ABSTRACT

Borate glass is widely used in many scientific studies. By using melt-quenching technique ten samples of lithium sodium borate (LNB) doped with different concentrations of samarium oxide (Sm_2O_3) and dysprosium oxide (Dy_2O_3) were prepared. To investigate the influence of dopant on the optical and physical characteristics of the glass, X-ray Diffraction, DTA, FTIR, UV-vis-Spectroscopy and Photoluminescence analyses were performed. The amorphous nature was confirmed by X-ray diffraction technique. The physical parameters involved are density, molar volume, ion concentration, inter-nuclear distance and Polaron radius. The absorption transitions of Sm^{3+} starts from $^6H_{5/2}$ with hypersensitive transition at 1221 nm and the Dy^{3+} starts from $^6H_{15/2}$ with hypersensitive transition at 1256 nm. The photoluminescence emission spectra of LNB:Sm have been associated with the excitation of 544 nm, 600 nm, 613 nm, 720 nm and 747 nm, and generated at $^4I_{7/2} \rightarrow {}^6H_{5/2}$ (green color), $^4I_{7/2} \rightarrow {}^6H_{7/2}$ (orange color), $^4I_{7/2} \rightarrow {}^6H_{9/2}$ (orange color), $^4I_{7/2} \rightarrow {}^6H_{11/2}$ (red color) and $^4I_{7/2} \rightarrow {}^6H_{13/2}$ (red color) respectively. LNB:Dy was due to the transition of Dy^{3+} at $^4F_{5/2} \rightarrow {}^6H_{15/2}$ and $^4F_{5/2} \rightarrow {}^6H_{13/2}$, the photoluminescence studies showed two peaks at 479 nm (blue color) and 587 nm (green color) for all samples except the pure glass sample of lithium sodium borate. The glow curve exhibited a single peak at 164 ºC. The results show that the appropriate annealing procedure for dysprosium doped LNB is 300 °C for 30 minutes. Regarding the heating rate optimization, it was found that the appropriate heating rate of the proposed dosimeter is 6 °C. s^{-1}. A linear dose response has been observed for photon (R^2= 0.998) and electron (R^2= 0.977) irradiation at 6 MV and 6 MeV, respectively. The glass dosimeter showed higher sensitivity for electron compared to photon response. The proposed TL dosimeter with concentration of 0.7 mol% of Dy_2O_3 has been observed to be 80 times less sensitive than TLD-100.

ABSTRAK

Kaca borat digunakan secara meluas dalam banyak kajian saintifik. Dengan menggunakan teknik sepuh-lindap, 10 sampel litium natrium borat yang telah didopkan dengan kepekatan oksida samarium (Sm_2O_3) dan oksida dysprosium (Dy_2O_3) yang berbeza telah disediakan. Untuk mengkaji kesan pendopan ke atas ciri-ciri optikal dan fizikal kaca, pembelauan sinar-X, DTA, FTIR, Spektroskopi UV-vis dan analisis fotoluminesens (PL) telah dijalankan. Sifat amorfus telah disahkan dengan teknik pembelauan sinar-X. Parameter-parameter fizikal yang terlibat adalah ketumpatan, isipadu molar, kepekatan ion, jarak antara nuklear dan jejari Polaron. Peralihan penyerapan Sm^{3+} bermula dari $^6H_{5/2}$ dengan peralihan hipersensitif pada 1221 nm dan Dy^{3+} bermula dari $^6H_{15/2}$ dengan peralihan hipersensitif pada 1256 nm. Spektrum pancaran fotoluminesens daripada LNB:Sm yang berkaitan dengan pengujaan pada 544 nm, 600 nm, 613 nm, 720 nm dan 747 nm, masing-masing dijanakan pada $^4I_{7/2} \rightarrow {}^6H_{5/2}$ (warna hijau), $^4I_{7/2} \rightarrow {}^6H_{7/2}$ (warna oren), $^4I_{7/2} \rightarrow {}^6H_{9/2}$ (warna oren), $^4I_{7/2} \rightarrow {}^6H_{11/2}$ (warna merah) dan $^4I_{7/2} \rightarrow {}^6H_{13/2}$ (warna merah). LNB:Dy adalah disebabkan oleh peralihan Dy^{3+} ion pada $^4F_{5/2}$ - $^6H_{15/2}$ dan $^4F_{5/2} \rightarrow {}^6H_{13/2}$, kajian fotoluminesens menunjukkan dua puncak pada 479 nm (warna biru) dan 587 nm (warna hijau) untuk semua sampel kaca kecuali sampel tulen litium natrium borat. Lengkung berbara menunjukkan puncak tunggal pada 164°C. Dapatan menunjukkan bahawa prosedur sepuh-lindap yang sesuai untuk LNB didopkan dysprosium ialah 300 °C selama 30 minit. Untuk kadar pemanasan optimum, didapati bahawa kadar pemanasan yang bersesuaian bagi dosimeter adalah 6 $°C.s^{-1}$. Sambutan dos linear terhadap penyinaran foton (R^2 = 0.998) dan elektron (R^2 = 0.977) masing-masing telah dicerap pada 6 MV dan 6 MeV. Dosimeter kaca ini menunjukkan kepekaan yang lebih tinggi untuk elektron berbanding sambutan foton. Dosimeter Luminesens Terma (TL) yang dicadangkan pada kepekatan 0.7 mol% Dy_2O_3 telah menunjukkan kepekaan 80 kali lebih rendah berbanding TLD-100.

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LIST OF ABBREVIATIONS

BOHC	- Boron Oxygen Hole Center
B_2O_3	- Boron Oxide
BO_3	- Trigoal Borate
BO_4	- Tetrahedral Borate
DTA	- Differential Thermal Analysis
Dy_2O_3	- Dysprosium Oxide
ECC	- Elemental Correlation Coefficient
FTIR	- Fourier transform infrared spectroscopy
FWHM	- Full Width at Half Maximum
GFA	- Glass Former Ability
GeO_2	- Germanium Dioxide
IR	- Infra-Red
H_3BO_3	- Boric Acid
HC	- Hole Center
LiB_3O_5	- Lithium Triborate
$Li_2B_4O_7$	- Lithium Tetraborate
LiF	- Lithium Fluoride
Li_2CO_3	- Lithium Carbonate
$Li_2B_4O_7$:Mn	- Lithium Tetraborate Doped with Manganese
LET	- Linear energy transfer
LINAC	- Linear accelerator
LNB	- Lithium Sodium Borate
MDD	- Minimum Delectable Dose
Na	- Sodium

Na_2O	- Sodium Oxide
Na_2CO_3	- Sodium Carbonate
NBO’s	- Non-Bridge Oxygen’s
P_2O_5	- Phosphorus Pentoxide
PL	- Photoluminescence
PMT	- Photomultiplier Tube
PMMA	- Poly Methyl Metha Crylate
RCF	- Read Calibration Factor
SiO_2	- Silicon Dioxide
STD	- Standard Deviation
Sm_2O_3	- Samarium Oxide
SSD	- Source Skin Distance
SSDL	- Secondary Standard Dosimeter Lab
TL	- Thermoluminescence
TSL	- Thermally Stimulated Luminescence
TLD	- Thermoluminescence dosimetery
UMMC	- University of Malaya Medical Center
UTM	- University Technology Malaysia
UV	- Ultra Visible
XRD	- X-Ray Diffraction

LIST OF SYMBOLS

α	- Alpha Particle
b	- Weight of glass sample in the toluence
c	- Speed of light
D	- Distance between atomic layers in a crystal
e	- Charge of electron
E	- Activation Energy for Trapped Electron
E_g	- Optical band gap
eV	- Electron Volt
f_{exp}	- Oscillator strengths
N	- Ion Concentration
r_i	- Inter-nuclear distance
r_p	- Polaron radius
T	- Temperature
T_g	- Glass Transition
T_m	- Maximum Temperature
T_c	- Crystaline Temperature
V_m	- Molar Volume
ρ	- Density
υ	- Frequency
λ	- Wavelength
σ	- Standard deviation

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CHAPTER 1

INTRODUCTION

1.1 Background

Glass shows various colors in the visible region due to its optical absorption. In the field of communications and optical switching, glasses have been used widely. Borate and silicate glasses contain boron oxide for optical lenses. Such glasses have high indices of refraction and dispersion characteristics. Absorption and transmission in the visible region are important in optical devices. The aforementioned of three areas can be used to examine the structure of glasses. The structural relationship between density and refractive index was first determined by Fanderlik in 1991.

Addition of alkali oxide B_2O_3 shows different behavior from the corresponding silicate glasses. Cohesive structure in borate glass can be observed due to additions of alkali oxide while shattered network is responded by silicate glass. The structure of borate glass can change by the addition of oxygen. The bond structure becomes more stringent due to three dimensional networks. (Anishia *et al*., 2010; El-Fayoumi *et al*., 2009; Gaafar *et al*., 2009; Joseph *et al*., 2002 and Ratnakaram *et al*., 2004).

Characteristic of color center in glasses is associated with the rate of gamma rays irradiation. Defects are produced due to color centers. Boron E Center, silicon E Center, nonbridging oxygen holes centers are the types of color centers. These color centers are defined as HC. BOHC1 and BOHC2 includes; single non-bridging oxygen and HC2 hole trapped on two nonbridging oxygen. Other types include center defect (Pb^{3+}) and transition metal center defect (ie, Cu, Zn and Mn); (El-Alaily and Mohamed, 2003).

1.2 Lithium Borate glass

The glass preparation without glass network former like boron requires high temperature to soften it for applications of optical fiber. Several companies suggested that the release of carbon dioxide into the atmosphere requires high energy. Various methods can be followed in order to synthesize pure glasses such as silicon glass (SiO_2), boron glass (B_2O_3), phosphorus glass (P_2O_5) and germanium glass (GeO_2). Borate glass was first introduced in the field of thermolumniscence (Schulman *et al.*, 1965). Lithium carbonate (Li_2CO_3) and boric acid (H_3BO_3) were prepared by melt quenching technique for the applications of thermolumniscence. In this present study, the focus is on the glasses formation corresponding to boric acid (West, 1989).

In 1965, lithium borate was introduced by Schulman and his colleagues in the dosimetric world. Host glass was doped with manganese to enhance the TL sensitivity. The mixture was prepared based on the glassy formation at 917 °C and was cooled rapidly. The same mechanism caused formation of crystallized material by subsequent and repeated heating at 650 °C (Schulman *et al*., 1965). Numerous studies confirmed that lithium borate is cost effective, highly sensitive and more precise than lithium fluoride. In 1974, the thermally stimulated luminescence of borate compounds was used by Kazanskaya. Several attractive properties were remarked such as sensitivity, linearity and low fading (Kazanskaya *et al*., 1974).

From the literature, it can be summarized that variety of atom groups are formed due to the ability of boron atom to link with either three or four oxygen atoms. The structural change between BO_3 and BO_4 is occurred as a result of adding metal oxides (as shown in Figure 1.1). It is due to transition stage, low cost and relative stability, high transparency, easy preparation and shape, ionic conductivity (Gaafar *et al*., 2009; Joseph *et al*., 2002; Mustafa Alajerami *et al*., 2012a; Alajerami *et al*., 2012b and Alajerami *et al*., 2013b). In addition, glass is a good host of different transition metals and rare earths. Borate is considered a good source in the field of industry and medicine. Borate glasses are very helpful in the field of dosimetry due to its effective atomic number which is very close to human body's tissue. Previous studies confirmed that the effect of alkali and alkaline reduced the hygroscopic property of borate. It is known as alkali borate glasses (Anishia *et al*., 2010; Alajerami *et al*., 2013b; Rao *et al*., 2002; Alajerami *et al*., 2012a and Balaji Rao *et al*., 2008).

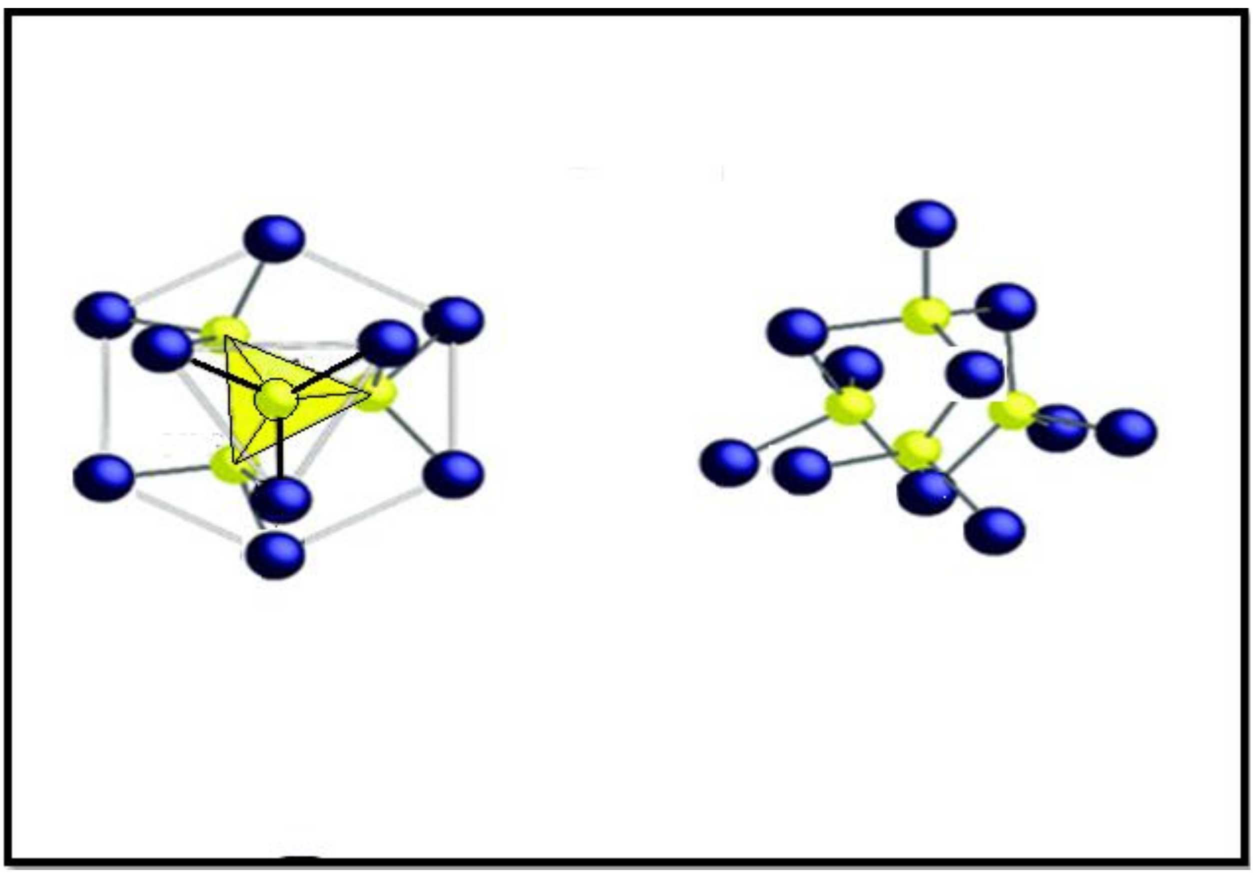

Figure 1.1: Structure of borate glass network (Bekker *et al*., 2012).

Figure 1.1 displays the structure of BO_4 with different shape, the structural change between BO_3 and BO_4 is occurred as a result of adding metal oxides. In this study the addition of lithium carbonate affects to change the structure of BO_3.

The increment of alkali oxide as a glass modifier causes an obvious change in the characterizations and luminescence properties of borate glass. Balaji Rao *et al.*, 2008 show two considerable states are already identifies as a results of incorporating alkali and alkaline oxide to the boron host. These states are the trigonal and the tetrahedral states which are greatly attributed to the creation of ionic bond with oxygen atom. The modified borate glasses have an amorphous structure close to the binary borate glasses, and all states have large phonon energy.

1.3 Modifiers and Activators

The efficiency of borate glass needs to examine by using the modifiers and activators, the modifiers as lithium carbonate and sodium carbonate to improvement the luminescence intensity and increase the strengthen glass network. The activators as samarium oxide and dysprosium oxide used to increment the luminescence properties.

1.3.1 Lithium Carbonate and Sodium Carbonate Modifiers

Lithium is one of the most suitable modifiers that used to increase/improve the host strengths (mechanical stability), but the energy level of lithium ion is recorded 10 eV. But energy that more than 10 eV is required and it does not show any effect directly in the luminescence. Although it was an activator during the exposing process.

The addition of lithium ions to the glass mixture creates number of non-bridge oxygen's (NBO's). These ionic bonds enhance the network, and strengthen the formation of color-center during the excitation process. Furthermore, the addition of Li^{+} ions leads to support the formation of borate rings by replacing the two BO_4 units with one BO_4 unit (Ratnakaram *et al*., 2006; Alajerami *et al*., 2012a and Alajerami *et al*., 2013b).

Sodium with symbol Na [$3s^1$] has a free electron in the outer L-shell. This sequence describes the stability of sodium. The addition of sodium on the borate system leads to enhance the luminescence intensity and strengthen its network after the excitation process (Farouk *et al*., 1995 and Alajerami *et al*., 2013b).

1.3.2 Samarium Oxide and Dysprosium Oxide Activators

Samarium ($4f^6$) is one of the transition elements that includes in the lanthanides group. Samarium ions (Sm^{3+}) usually found in triply ionized Sm^{3+}. It has specific advantages. The uses of Samarium in various optical devices such as storage of high optical density, sea contacts and screens, as well as lasers in the solid state devices. Samarium exhibited relatively high quantum efficiency, and showed different emission cooling where they appear, including emissions shining in the orange/ red, emitting $^4G_{5/2}$ level (Ratnakaram *et al*., 2005; Lakshminarayana and Qiu, 2009; Venkateswarlu *et al*., 2011; Ratnakaram *et al*., 2012 and Vijaya *et al*., 2013). The Sm^{3+} ions is important activator for inorganic lattices such as the emission of reddish orange light to electronic transitions $^4G_{5/2}$, $^4G_{3/2}$, $^4I_{7/2}$ and $^4I_{5/2}$.

Sm^{3+} ions as local structure are utilized in condensed matter due to electric dipole $^4G_{5/2}$ and $^6H_{5/2}$ transition. Hypersensitivity of Sm^{3+} ions increases due to reduction in the intensity of luminescence. Although previous researcher have been paid less attention on samarium glasses from other lanthanides.

Dy^{3+} ($4f^9$) ion is one of the best ions in lanthanides group. It plays a great role in the luminescence behaviour and glass configuration. In the visible and NIR regions, the analysis of luminescence of Dy^{3+} ions from the $^4F_{9/2}$ level is very interesting. Previous studies showed difficulties to interpret the spectra of dysprosium (Karunakaran *et al*., 2010 and Venkateswarlu *et al*., 2011). These barriers attributed to the approach of a large number of energy levels laying to each other, and the electronic structure of complex configuration of $4f^9$ that has limited attention paid to the emission of visible source state $^4F_{9/2}$ (Jayasankar *et al*., 2004 and Lakshminarayana *et al*., 2008).

The proper selection of the host to facilitate the extraction of the primary colors of yellow transition $^4F_{9/2}$ - $^6H_{13/2}$ and blue transmission of $^4F_{9/2}$ - $^6H_{15/2}$ from Dy^{3+} ions. Although a number of spectral studies on Dy^{3+} ion doped materials is performed. A systematic analysis of the thermal behaviour of the optical and structural Dy^{3+} based anaesthetic borate glasses by changing the alkali element has not been published.

The attracted optical properties of Dy^{3+} ions in solid-state laser increased the optical up transformation emission. Several studies have been reported to identify the emission of Dy^{3+} doped crystals and glasses (Lakshminarayana *et al*., 2008 and Rajesh *et al*., 2012). All these studies recommended the performing of dysprosium oxide as activator to enhance the luminescence.

1.4 Problem Statement

The first TL material based on lithium borate which was introduced in dosimetry was $Li_2B_4O_7$:Mn. $Li_2B_4O_7$:Mn phosphor with low TL sensitivity caused partly by the emission in the 600 nm region. The borate glass has many advantages: low cost, high ionic conductivity, low melting point, high transparency, high thermal stability, however it has some disadvantages such as formation of crystal.

Generally, there are some specific features that should fulfill with the proposed composition to be used as dosimeter i.e. shapes of the glow curve, effective atomic number, linearity, sensitivity. Some of drawbacks are appeared by using the conventional dosimeter TLD-100 (LiF:Mg,Ti). It can be summarize that the disadvantages for TLD-100 (LiF:Mg,Ti) are due to annealing complexity, dose response linearity, and the energy response.

Several studies have been carried to examine the efficiency of lithium borate with different activators (Rare earths or transition metals) to improve the dosimetric properties (Furetta *et al*., 2000; Prokic, 2002; Sangeeta and Sabharwal, 2004) and glass preparation (Pontuschka *et al*., 2001; Venkateswara *et al*., 2002; Rojas *et al*., 2006).

In the present study we attempt to prepare new dosimeter by using borate glass modified with lithium carbonate and sodium carbonate and activated by samarium and dysprosium oxides.

1.5 Objectives

The objectives of this study are:

I. To fabricate new glass composition of lithium sodium borate glass undoped and doped with different concentration of samarium and dysprosium.

II. To determine the optical and the thermoluminescence properties of the doped and un-doped lithium sodium borate glass with different concentration of samarium and dysprosium samples.

III. To investigate relationship between the optical and thermoluminescence properties of the doped and undoped lithium sodium borate glass with different concentration of samarium and dysprosium samples.

1.6 Scope of Study

In order to achieve the above mentioned objectives the study will be focused on the following scopes as being summarized in Figure 1.2:

i. Preparation of glass samples from lithium sodium borate doped different concentration of samarium and dysprosium by using melt quenching technique.

ii. The used of X-ray diffraction to identify the amorphous phase.

iii. The used of UV-vis-NIR spectroscopy to investigate the absorption properties of the obtained glass.

iv. The used of Photoluminescence spectroscopy to determine the excitation and emission spectra of the samples.

v. Irradiation of TL materials in order to investigate the thermoluminescence characteristics

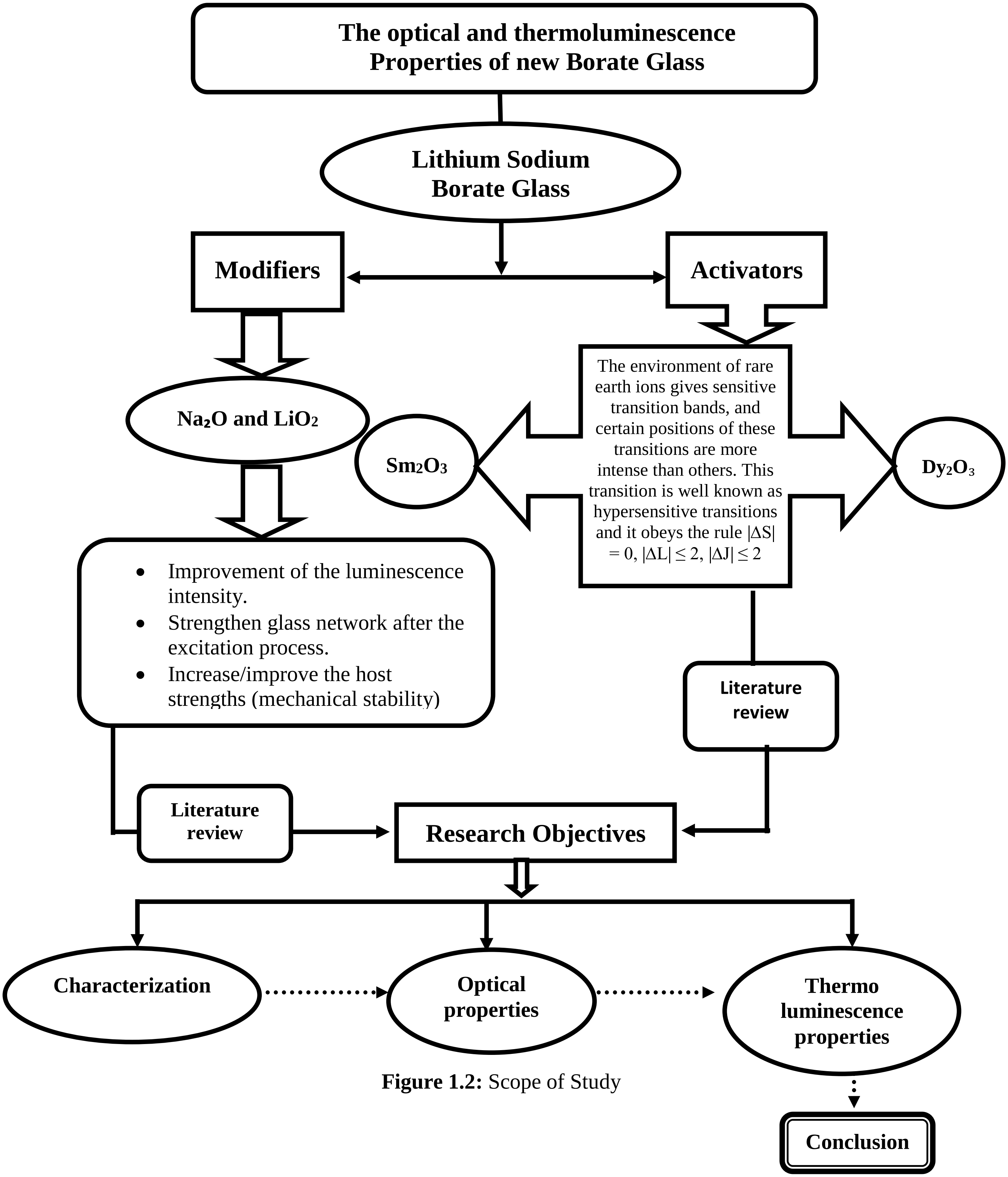

Figure 1.2: Scope of Study

1.7 Organization of study

The thesis is organised to five chapters. Chapter one has the background of borate glass, describes the significant of the proposed samples in term of its optical and thermoluminescence properties. Lithium sodium borate glass doped with samarium and sodium is to introduce a new thermoluminescence material. Three main objectives are listed in this chapter to be accomplished.

In chapter two, general descriptions will be reported regarding the glass structure and lithium sodium borate by using dysprosium and samarium oxide as rare earth activators. The optical and thermoluminescence properties also will be discussed.

In chapter three, the glass preparation process and the methodology used to investigate the optical properties (UV and PL) and physical properties are described in details. The techniques that are being used for the characterization are XRD, FTIR and DTA. The thermoluminescence studies will be carried out using ionizing radiation.

In chapter four, a full analysis and discussion of the optical, physical and TL results are presented.

Finally, chapter five conclude the obtained results and suggests some recommendations for the future studies.

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