STRUCTURAL, LUMINESCENCE AND JUDD-OFELT ANALYSIS OF RARE EARTH DOPED OF MAGNESIUM SULFOBORATE GLASSES AND CRYSTALS

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DEDICATION

Dedicated to

My mother, **Malama Hauwa Muhammad**, whose sacrifice;

My father, Malam **Dalhatu Dauda**, whose dream;

My **uncle**, Malam Sadiq G. Abubakar whose support and encouragement;

And

My wife, **Fatima Abdulwahab**, whose patience;

Lead to achieve my doctoral degree

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ABSTRACT

A series of samples of undoped magnesium sulfoborate glasses and crystals with chemical composition of xMgO+(50–x) SO_3+50B_2O_3, with $10 \leq x \leq 30$ mol% were prepared by melt quenching and solid state reaction method respectively. Then a series of glass and crystal samples doped with rare earth (RE = Dy_2O_3, Eu_2O_3 and Sm_2O_3) with the chemical compositions of 10MgO+40SO_3+(50–y) B_2O_3+yRE, with $0.1 \leq y \leq 1.0$ mol% were also prepared by melt quenching and solid state reaction method respectively. The amorphous/crystalline phases of the glass and crystal samples were characterized by X–Ray diffraction (XRD), while the structural features of the samples were measured using Fourier transform infrared (FTIR), Raman and nuclear magnetic resonance (NMR) spectroscopy. The optical properties of glass and crystal samples were characterized via UV–Vis–NIR and luminescence spectroscopy. The amorphous phase of the glass samples was confirmed by the diffused broad XRD pattern, while the crystal samples showed two crystalline phases of H_3BO_3 and $MgSO_4(H_2O)_6$. The infrared spectra show the coexistence of BO_3, BO_4, SO_4^{2-} and S–O–B (sulfoborate group) structural units in both glass and crystal samples. The Raman spectra also reveal the coexistence of BO_4, SO_4^{2-} and S–O–B (sulfoborate group) structural units in both glass and crystal samples. The NMR spectra show the existence BO_4 structural units in both glass and crystalline samples. The luminescence spectra of Dy^{3+} doped glass and doped crystal samples exhibit three emission bands at around 482 nm, 575 nm and 662 nm correspond to the $^4F_{9/2}\rightarrow{}^6H_{15/2}$, $^4F_{9/2}\rightarrow{}^6H_{13/2}$ and $^4F_{9/2}\rightarrow{}^6H_{11/2}$ transitions respectively. As for Eu^{3+} doped glass samples, the emission spectra show peaks at 592 nm, 616 nm, 658 nm and 697 nm correspond to the $^5D_0\rightarrow{}^7F_1$, $^5D_0\rightarrow{}^7F_2$, $^5D_0\rightarrow{}^7F_3$ and $^5D_0\rightarrow{}^7F_4$ transitions respectively, while for crystal samples, the emission spectra show six peaks belongs to Eu^{2+} and Eu^{3+} ions. The emission spectra of glass and crystal samples doped with Sm^{3+} ions show dominant peaks at around 565 nm, 601 nm, 646 nm and 706 nm correspond to the $^4G_{5/2}\rightarrow{}^6H_{5/2}$, $^4G_{5/2}\rightarrow{}^6H_{7/2}$, $^4G_{5/2}\rightarrow{}^6H_{9/2}$ and $^4G_{5/2}\rightarrow{}^6H_{11/2}$ transitions respectively. The refractive index and quantum efficiency were calculated for all the studied samples. The higher value of branching ratios from $^4F_{9/2}\rightarrow{}^6H_{13/2}$ and $^4G_{5/2}\rightarrow{}^6H_{7/2}$ transitions showed that Dy^{3+} and Sm^{3+} doped magnesium sulfoborate glasses and crystals are good candidates for lasing and lighting device applications.

ABSTRAK

Satu siri sampel kaca dan kristal magnesium sulfoborate tak berdop dengan komposisi kimia xMgO+(50–x) SO_3+50B_2O_3, dengan 10 ≤ x ≤ 30 mol% telah disediakan masing–masing melalui kaedah sepuhlindap leburan dan tindak balas keadaan pepejal. Kemudian satu siri sampel kaca dan kristal berdop dengan nadir bumi (RE = Dy_2O_3, Eu_2O_3 dan Sm_2O_3) dengan komposisi kimia 10MgO+40SO_3+(50–y) B_2O_3+yRE, dengan 0.1≤ y ≤ 1.0 mol% juga telah disediakan masing–masing melalui kaedah sepuhlindap leburan dan tindak balas keadaan pepejal. Fasa amorfus/ kristal sampel kaca dan kristal telah dicirikan oleh pembelauan sinar–X (XRD), sementara ciri struktur sampel telah diukur menggunakan spektroskopi inframerah transformasi Fourier (FTIR), Raman dan resonans magnet nuklear (NMR). Sifat optik sampel kaca dan kristal dicirikan melalui spektroskopi UV–Vis–NIR dan luminesens. Fasa amorfus sampel kaca telah disahkan oleh corak belauan XRD yang melebar, sementara sampel kristal menunjukkan dua fasa kristal H_3BO_3 dan $MgSO_4(H_2O)_6$. Spektrum inframerah menunjukkan ujud sama dan struktur unit bagi BO_3, BO_4, SO_4^{2-} dan S–O–B (kumpulan sulfoborate) dalam kedua–dua sampel kaca dan kristal. Spektrum Raman juga mendedahkan ujud sama struktur unit BO_4, SO_4^{2-} dan S–O–B (kumpulan sulfoborate) dalam kedua–dua sampel kaca dan kristal. Spektrum NMR menunjukkan kewujudan struktur unit BO_4 dalam kedua–dua sampel kaca dan kristal. Spektrum luminesens sampel kaca dan kristal berdop Dy^{3+} mempamerkan tiga jalur pancaran pada sekitar 482 nm, 575 nm dan 662 nm, masing–masing berpadanan dengan peralihan $^4F_{9/2}\rightarrow{}^6H_{15/2}$, $^4F_{9/2}\rightarrow{}^6H_{13/2}$ dan $^4F_{9/2}\rightarrow{}^6H_{11/2}$. Bagi sampel kaca berdop Eu^{3+}, spektrum pancaran menunjukkan puncak pada 592 nm, 616 nm, 658 nm dan 697 nm, masing–masing berpadanan dengan peralihan $^5D_0\rightarrow{}^7F_1$, $^5D_0\rightarrow{}^7F_2$, $^5D_0\rightarrow{}^7F_3$ dan $^5D_0\rightarrow{}^7F_4$, manakala bagi sampel kristal, spektrum pancaran menunjukkan enam puncak kepunyaan ion Eu^{2+} dan Eu^{3+}. Spektrum pancaran bagi sampel kaca dan kristal berdop Sm^{3+} menunjukkan puncak dominan pada sekitar 565 nm, 601 nm, 646 nm dan 706 nm, masing–masing berpadanan dengan peralihan dari $^4G_{5/2}\rightarrow{}^6H_{5/2}$, $^4G_{5/2}\rightarrow{}^6H_{7/2}$, $^4G_{5/2}\rightarrow{}^6H_{9/2}$ dan $^4G_{5/2}\rightarrow{}^6H_{11/2}$. Indek biasan dan kecekapan kuantum telah dikira untuk semua sampel yang dikaji. Nilai nisbah cabang yang agak tinggi bagi peralihan $^4F_{9/2}\rightarrow{}^6H_{13/2}$ dan $^4G_{5/2}\rightarrow{}^6H_{7/2}$ menyarankan bahawa sampel kaca dan kristal magnesium sulfoborate berdop ion Dy^{3+} dan Sm^{3+} berpotensi untuk digunakan sebagai bahan laser dan peranti pencahayaan.

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LIST OF ABBREVIATIONS

B_2O_3	–	Borate
B	–	Boron
H_2SO_4	–	Sulfuric Acid
MgO	–	Magnesium Oxide
Dy_2O_3	–	Dysprosium Oxide
Eu_2O_3	–	Europium Oxide
Sm_2O_3	–	Samarium Oxide
Dy^{3+}	–	Dysprosium Ion
Eu^{3+}	–	Europium Ion
Sm^{3+}	–	Samarium Ion
CTB	–	Charge transfer band
f_{exp}	–	Experimental oscillator strengths
f_{cal}	–	Calculated oscillator strengths
IR	–	Infrared
S_{ed}	–	Electric dipole line strength
S_{md}	–	Magnetic dipole line strength
A_{rad}	–	Radiative transition probability
τ_{rad}	–	Radiative lifetime
β_r	–	Branching ratio
NMR	–	Nuclear Magnetic Resonance
λ_P	–	Emission band position
FTIR	–	Fourier Transform Infrared
KBr	–	Potassium bromide
XRD	–	X–Ray Diffraction
UV	–	Ultraviolet

RE	–	Rare Earth
PL	–	Photoluminescence
IR	–	Infrared
LED	–	Lead Emitting Diode

LIST OF SYMBOLS

τ	–	Decay time
h	–	Planck's constant
^{0}C	–	Degree Celsius
ν	–	Frequency
c	–	Speed of light
t	–	time
$\alpha(\nu)$	–	Absorption coefficient
β	–	Nepheleuxetic ratios
Ω_2	–	Judd–Ofelt parameter
Ω_4	–	Judd–Ofelt parameter
Ω_6	–	Judd–Ofelt parameter
I_t	–	Actual luminescence intensity
J	–	Total angular momentum
θ	–	Diffracted angle of the X–Ray beam
λ	–	Wavelength
n	–	Refractive Index

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CHAPTER 1

INTRODUCTION

1.1 Background of the Research

A Glass is solid that has an amorphous structure, short range order of atomic arrangement, lack of uniformity, and have no long range periodically which yielded fairly random structure unlike crystal with a well–defined structure and atoms arranged in three dimensional periodic and long range order. Therefore, instead of crystalline sharp peaks, a glass has broad hump is seen in the X–ray diffraction pattern of a glass. A glass has significance role both scientifically and technologically due to its good transparency, chemical durability, electrical and thermal features (Alajerami et al., 2012). Hence, glass has wide range of application, such as television screen, containers, chemical laboratory equipment, fiber optics, lasers (Mhareb et al., 2014a). Therefore, the difference between glass and crystal are the presence of long range arrangement, symmetric and uniformity in the crystal structure (Sahar, 1998). Crystal is playing significant role, due to their potential applications in various field, such as phosphor for plasma display panels, nonlinear optical (NLO), luminescent materials and optical communication components (Pavani et al., 2011).

The glass composition is very significant for the formation of glass. The basic condition for glass formation is the existence of strongly bonded large networks or long chains of atoms in the liquid, and showed that a good glass must contain many bonds or linkages of the types that have high bond strengths such as B–O–B, Si–O, Ge–O and P–O as glass formers. However, some oxides are defined as glass formers such as borate (B_2O_3), Phosphate (P_2O_5), silicate (SiO_2) and Germinate (GeO_2), because they have glass–forming ability under normal quenching conditions by themselves, but act like glass formers when combined with others such as ZnO, PbO, MgO CaO, BaO (Gautam *et al.*, 2012).

For the formation of crystal, generally, the glass compositions are decided for crystal. The formation of $Bi_3B_5O_{12}$ and $Bi_4B_2O_9$ crystalline phase by heat treatment from the composition of glass $3Bi_2O_3$–$5B_2O_3$ (Bajaj et al., 2009; Burianek et al., 2006; Muehlberg et al., 2002). $Bi_3B_5O_{12}$ and $Bi_4B_2O_9$ crystals also could be formed in the glasses with composition of xBi_2O_3–$(100–x)B_2O_3$ (x = 20 to 66 mol%) (Bajaj et al., 2009). According to Lin et al., 2007 the composition La_2O_3–3B2O3–$0.06Eu_2O_3$ formed both.

Currently, much more attention has been paid to borate glass and crystal due their applications in technological such as solid state lasers, nonlinear optics and solar (Alajerami et al., 2012). Borate glass are known to have important properties which include low melting point, good thermal stability, good solubility of rare–earth ions (Guana et al., 2013). Borate acts as the glass former, because of its high bond strength, lower cation size and smaller heat of fusion and is incorporated into various glass systems as a flux material to attain materials of high technological application (Sumalatha et al., 2011). Borate constitute an interesting system, which the network building unit can be either borate triangles (BO_3) with non–bridging atoms or borate tetrahedral (BO_4) with all bridging oxygen atoms. Borate glass can easily be melted, owning smaller mass compare to other glass network former, thermal stable and chemical durable. In addition, they are high transparency and acted as a good host for transition metal ions and rare earth ions making them suitable for optical materials. Therefore, hydroscopic properties and the high phonon energy of B_2O_3 are considered as a drawback to the glass industry (Vijayakumar et al., 2015).

The use of sulfate as a intermediate in to the borate network influence the structure of the borate units and the boron in the system retain of four coordinate from interaction between sulfate and borate units, as observed from Raman, IR and NMR spectroscopy (Ganguli and Rao, 1999). The sulfate have lower operating temperature of 700–1000 oC (Pitha et al., 1947). Sulfate have studied because of good properties such as good transparency and low melting temperature which are material for good UV and IR transmission. However, sulfate is an attractive compound and important for a range of many applications (Gedam et al., 2006). Unfortunately, poor chemical durability and hygroscopic nature of sulfate discourage their limit practical applications. Therefore, addition of alkali earth metals has proven to enhance their chemical stability (Vyatchina et al., 2005).

However, to overcome the individual limitations of borate and sulfate, the two are combined to form a new material called “Sulfoborate” which offers greater advantage as they show different properties (Vyatchina et al., 2005). The presence of SO_3 in the borate glass can enhance the glass quality when modified with alkali earth metals (Vyatchina et al., 2009). Vyatchina et al. (2005) reported that sulfoborate glass have acceptable chemical durability compared to pure borate and has drawn attention of researchers because of their good stability.

Meanwhile, according to Mansour, (2012) addition of network modifier (magnesium oxide) into borate glass could create the conversion of the triangular BO_3 structural units to BO_4 tetrahedral, and also alter the structure and improve the glass and crystal properties (Reduan et al., 2014). Alkali or alkaline oxides were frequently applied as modifiers; Therefore, this modifier shift up the boroxyl rings, and the active groups in the mixture, to form tri–and tetra–bond on the host (Alajerami et al., 2012). The alkaline earth ions based borates have been used in various applications such as vacuum ultraviolet (VUV) optics, radiation dosimetry and solar energy converters (Lim et al., 2014a). Addition of magnesium as modifier into sulfoborate can enhance the release of electrons and to reduce the hygroscopic nature of sulfoborate (Mhareb et al., 2014a).

Furthermore, glass and crystal activated with activator. Such activator is either rare earth or transition metals ion which have been identified as a good luminescence host material which convert an incident energy input into emission of electromagnetic waves in the ultraviolet (UV), visible (Vis), or infrared (IR) regions of the spectrum (You et al., 2011). Rare earth (RE) doped glasses and crystal materials have potential application in the fields such as laser material, fiber, information display, optoelectronic (Rajesh et al., 2012a). Rare earth (RE) doped materials correspond to the 4f–4f and 4f–5d electronic transitions which is due to the shielding effect from the outer orbital (5s and 5p) on the 4f electrons. The rare earth doped materials have potential applications for instance in lasers, security, decoration, semiconductor and medication. Some of the products for example fluorescence lamp, escape routes, television monitor, warning signs, light emitting diodes, laser detection, luminous paints and so on. The sulfoborate glass host doped with rare earth ion are known to have important properties such as lower melting point, good solubility of rare earth ion and good thermal stability (Lim et al., 2014).

In addition, intensity trivalent rare earth ions, some host media were describing and estimated quantitatively via Judd–Ofelt theory (Agarwal et al., 2009). In the Judd–Ofelt theory the transition probability between any pair of stark sublevels of the rare earth (RE) ion activator in $4f^N$ configuration can be written in terms of three phenomenological parameters called Ω_λ (λ = 2, 4, 6), which are called Judd–Ofelt parameters. These parameters are determined experimentally by means of an adjustment intensities of the lines with corresponding theoretical and experimental lines registered in the absorption spectrum. Most of the study have conducted to describe the behaviour of these parameters, for instance, according to Kindrat et al., (2015b) the intensity parameter Ω_2 shows the dependence on the covalence between rare earth ions (RE) and ligands an ions, since the parameter Ω_2 reflects the asymmetry of the local environment at the rare earth ion (RE) site, and therefore Ω_2 is very small for ionic materials, and quite large for covalent materials, while the parameters Ω4 and Ω_6 are related to the rigidity of the matrix. These parameters are used to evaluate the radiative properties such as radiative transition probability (A_{rad}, s^{-1}), radiative lifetime (τ_{rad}, sec) and an important parameter called fluorescence branching ratio (β_r) that characterizes the lasing power transition (Agarwal et al., 2009).

Over the past few decades, much attention has been focused towards dysprosium, europium and samarium ions doped glass or crystal materials for the development of optical devices such as lighting devices and solid state laser (Li et al., 2010). To date, these material become an interesting topic in the field of material science and hence need to be further investigate.

1.2 Problem Statement

Currently, a great deal of research has been focused on rare earth doped magnesium borate glasses due to their potential applications (Reduan et al., 2014b; Alajerami et al., 2012). But, the investigation on the luminescence properties of rare earth doped sulfoborate glass and crystal is not many. However, there was limited structural information regarding effect in the sulfo–borate as the host that can be reasoned to find a good luminescence material. Meanwhile, the study on the luminescence properties of rare earth doped sulfo–borate glass and crystal are not fully understood. In addition, the Judd–Ofelt analysis on the Dy^{3+}, Eu^{3+} and Sm^{3+} ions doped in sulfoborate glass and crystal is very less reported. Therefore, in this study, magnesium sulfoborate doped Dy^{3+}, Eu^{3+} and Sm^{3+} ions present to synthesis the glass and crystal materials by using melt quenching and solid state reaction method respectively. The investigation of structural features was important in order to study the structures changes in the doped and un–doped samples. Also, to investigate the influence of vary concentration of Dy^{3+}, Eu^{3+} and Sm^{3+} ions on the optical properties.

1.3 Objectives of the Study

The following are the objectives of this study

i. To determine the influence of doped and undoped magnesium sulfoborate glasses in terms of structure features and to compare with the similar composition of the crystalline.

ii. To determine the impact of concentration and types of dopants such as Dy^{3+}, Eu^{3+} and Sm^{3+} ions in terms of enhancement of luminescence characteristic between glass and crystal.

iii. To analyse and compare the absorption and emission data of sulfoborate glass and crystal doped Dy^{3+}, Eu^{3+} and Sm^{3+} ions in terms of radiative properties by using Judd–Ofelt analysis.

1.4 Scope of the Research

In this study, the samples undoped magnesium sulfoborate glasses and crystals with chemical composition $xMgO+(50–x) SO_3+50B_2O_3$ with $10 \leq x \leq 30$ mol % were prepared by conventional melt quenching and solid state reaction method respectively. The series of glass and crystal samples doped with rare earth (RE= Dy_2O_3, Eu_2O_3 and Sm_2O_3) with the chemical compositions of $10MgO+40SO_3+(50–y) B_2O_3+yRE$ with $0.1\leq y \leq 1.0$ mol% were also been prepared by conventional melt quenching and solid state reaction method respectively. Sulpur oxide was incorporate into borate as intermediate to enhance the host network whereas magnesium oxide was used as modifier to reduce the hygroscopic properties. Dy^{3+}, Eu^{3+} and Sm^{3+} ions were chosen to be dopant ions in order to investigate the impact of the dopant on the structural, luminescence properties and Judd–Ofelt analysis. Different types of measurements were used. The phase of the prepared samples was determined by the X–Ray Diffraction measurement. The structural features for doped and un–doped samples were determined by Infrared, Raman and Nuclear magnetic resonance spectrometer. As for luminescence properties was determined by photoluminescence spectrometer. The optical properties in the glass and crystal samples was determined using UV–Visible–NIR spectrometer, meanwhile, band gap, refractive index, Judd–Ofelt parameters was calculated from UV–Visible–NIR spectra and radiative properties was calculated from emission spectra.

1.5 Significance of the study

This study has been done to understand more on the structural features, luminescence properties and Judd–Ofelt analysis of glass and crystal. However, doping the samples with Dy^{3+}, Eu^{3+} and Sm^{3+} may develop new luminescence materials. In addition, the study on optical and luminescence properties in this work is important in providing a baseline data that can be used for further research and development of luminescence host material for solid state lighting devices.

1.6 Outline of Study

There are six chapters in this study. The background, problem statement, objectives, Scope, Significance and outline of study are described related to magnesium sulfoborate glass and crystal, and magnesium sulfoborate doped with Dy^{3+}, Eu^{3+} and Sm^{3+} ions are presented in chapter 1. Chapter 2 covered the general review on magnesium sulfoborate glass and crystal with more emphasis on its structural features, luminescence properties and Judd–Ofelt analysis. Chapter 3 presents the experimental procedures which including their method of preparation, types of spectroscopy being used and the principles of X–Ray Diffraction (XRD), FTIR, Raman and NMR spectroscopy, luminescence and UV–Visible–NIR spectrometer. The results and discussion of glass along with the Tables and figures are stated in chapter 4. Chapter 5 covered the crystal results and discussion together with tables and figures. Lastly, chapter 6 presents conclusion, and recommendation for future work.

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