UNIVERSITI PUTRA MALAYSIA

STRUCTURAL AND OPTICAL PROPERTIES OF NEODYMIUM-DOPED ZINC SILICATE-BASED GLASS CERAMIC

ZAMRATUL MAISARAH BINTI MOHD ISMAIL

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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Chair: Khamirul Amin Matori, PhD
Faculty: Science

The need for fabricating low cost luminous material have gained considerable attention in optoelectronic field. Therefore, in this research, $Zn_2SiO_4:Nd^{3+}$ based glass ceramic were prepared from recyclable waste soda lime silica (SLS) as silica source and ZnO using solid state method. The effect of Nd^{3+} ions (x = 0, 1, 2, 3, 4 and 5 wt.%) and the effect of sintering temperatures, ranging from 600 to 1000 °C on the thermal, structural, morphological and optical properties of the phosphors were also investigated using Differential Scanning Calorimetry (DSC), X-ray diffraction (XRD), Field emission scanning electron microscopy (FESEM), Fourier transform infrared (FTIR) spectroscopy, Ultraviolet-visible near infrared (UV-Vis-NIR) spectroscopy and Photoluminescence (PL) spectroscopy. Thermal analysis were carried out to determine the thermal stability and glass transition temperature of the sample. Structural investigation using XRD revealed the presence of α-zinc silicate phase at 800 °C onwards. The effect of sintering temperature showed phases changes and enhancement in crystallinity. With respect to Nd^{3+} doping, the diffraction peaks shifted due to lattice distortion. The morphologies from FESEM analysis showed the transformation in particles and grain boundaries formation. With increment in temperature and doping, the microstructure become densely packed grains. FTIR spectra showed that the progression of sintering temperature and doping revealed the existence of SiO_4 and ZnO_4 bonding which indicate the formation of Zn_2SiO_4 network. Furthermore, the optical properties of the $Zn_2SiO_4:Nd^{3+}$ based glass ceramic were analysed for its UV absoprtion to determine the optical band gap. The optical band gap obtained were in the range 2.56 eV – 3.74 eV which is in agreement with WLEDs optical band gap. The increment and decrement of optical band gap (E_{gap}) were speculated due to structural changes of host material and Burstein Moss effect. The photoluminescence spectra of Nd^{3+} ions exhibit blue to red emission at ~484 nm (blue), ~529–570 nm (green) and ~600-676 nm (orange-red) corresponding to transitions $^2P_{1/2} \rightarrow {^4I_{11/2}}$, $^4G_{7/2} \rightarrow {^4I_{9/2}}$, ($^4G_{7/2} \rightarrow {^4I_{11/2}}$, $^4G_{5/2} \rightarrow {^4I_{9/2}}$), ($^4G_{5/2} \rightarrow {^4I_{11/2}}$) and ($^4G_{7/2} \rightarrow {^4I_{13/2}}$, $^4G_{5/2} \rightarrow {^4I_{11/2}}$) respectively. These spectra proved the incorporation of Nd^{3+} into Zn_2SiO_4 lattice. The emission intensity changes with respect to dopant percentage and sintering temperatures. The

intensity of PL emission were affected by enhancement the crystallite of zinc silicate and Nd^{3+} ions into the crystals. Overall, from the obtained results this study concluded that $Zn_2SiO_4:Nd^{3+}$ based glass ceramic have promising parameters for WLEDs application that exhibit blue to red region under infrared excitation.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SIFAT STRUKTUR DAN OPTIK KE ATAS NEODYMIUM-DOP ZINK SILIKAT-BERASASKAN KACA SERAMIK

Oleh

ZAMRATUL MAISARAH BINTI MOHD ISMAIL

Mei 2108

Pengerusi: Khamirul Amin Matori, PhD
Fakulti: Sains

Keperluan dalam menghasilkan bahan kependarkilauan berkos rendah telah menjadi perhatian di bidang optoelektronik. Oleh itu, dalam kajian ini, $Zn_2SiO_4:Nd^{3+}$ berasaskan kaca seramik telah dihasilkan dari kaca soda silika (SLS) yang dikitar semula sebagai sumber silika dan ZnO dengan menggunakan teknik lindapan leburan. Kesan dopan Nd^{3+} ion (x = 0, 1, 2, 3, 4 and 5 wt.%) dan kesan suhu rawatan haba dalam jurang 600 hingga 1000 °C ke atas sifat haba, struktur, morfologi dan optik ke atas sampel telah dijalankan menggunakan DSC, XRD, FESEM, FTIR spectroskopi, UV-Vis-NIR spectroskopi dan PL spetroskopi. Analisis haba telah dijalankan untuk mengenalpasti kestabilan haba kaca dan suhu transisi kaca pada sampel. Analisis struktur dengan menggunakan XRD telah mendapati penghasilan fasa α-zink silikat pada suhu 800 °C dan ke atas. Kesan suhu rawatan haba menghasilkan perubahan fasa dan peningkatan pada pengkristalan. Kesan dopan Nd^{3+} ke atas sampel menghasilkan darjah penganjakan kesan dari perubahan pada struktur. Analisis FESEM ke atas morfologi sampel menunjukkan transformasi dalam partikel dan pembentukan butiran. Dengan peningkatan suhu rawatan haba dan kepekatan dopan, morfologi menjadi butiran yang berketumpatan. Analisis FTIR menunjukkan bahawa perubahan dari suhu rawatan haba dan dopan telah menunjukkan dengan kehadiran ikatan SiO_4 dan ZnO_4 sekaligus membuktikan pembentukkan jaringan Zn_2SiO_4. Selain itu, sifat optik $Zn_2SiO_4:Nd^{3+}$ berasaskan kaca seramik telah dianalisa dari penyerapan UV-Vis untuk mendapatkan nilai jurang jalur optik. Nilai jurang jalur optik yang diperolehi berada dalam julat 2.56 eV – 3.74 eV adalah selari dengan julat yang diperlukan untuk WLEDs. Peningakatan dan penurunan nilai jurang jalur optik (E_{gap}) berkemungkinan hasil dari perubahan struktur bahan dan efek Burstein-Moss. Spektra kefotopendarcahayaan dari ion Nd^{3+} menunjukkan kependarkilauan biru ke merah pada ~484 nm (biru), ~529–570 nm (hijau) dan ~600-676 nm (jingga-merah) yang diwakili daripada transisi

$^2P_{1/2} \rightarrow {}^4I_{11/2}$, $^4G_{7/2} \rightarrow {}^4I_{9/2}$, ($^4G_{7/2} \rightarrow {}^4I_{11/2}$, $^4G_{5/2} \rightarrow {}^4I_{9/2}$), ($^4G_{5/2} \rightarrow {}^4I_{11/2}$) dan ($^4G_{7/2} \rightarrow {}^4I_{13/2}$, $^4G_{5/2} \rightarrow {}^4I_{11/2}$). Spektra ini membuktikan bahawa Nd^{3+} ion menyerap ke dalam kekisi Zn_2SiO_4. Pancaran juga menunjukkan perubahan berdasarkan kepekatan dopan dan suhu rawatan haba. Peningkatan intensiti pancaran ini adalah disebabkan pembentukan kristalit zink silikat dan penyerapan ion Nd^{3+} ke dalam kristal. Secara keseluruhan, dapatan dari kajian ini merumuskan bahawa $Zn_2SiO_4:Nd^{3+}$ berasaskan kaca seramik memiliki parameter yang berpotensi sebagai aplikasi WLEDs yang memancarkan sinaran dari ruang biru sehingga merah dengan menggunakan penganjakan inframerah.
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May Allah SWT bless and grant happiness to all of you.

I certify that a Thesis Examination Committee has met on 3 May 2018 to conduct the final examination of Zamratul Maisarah Binti Mohd Ismail on her thesis entitled "Structural and Optical Properties of Neodymium-Doped Zinc Silicate-Based Glass Ceramic" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

Members of the Thesis Examination Committee were as follows:

Chen Soo Kien, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Halimah binti Mohamed Kamari, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Sidek bin Hj. Ab Aziz, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Internal Examiner)

Yasser Bakr Saddeek Muhammed, PhD
Professor
Al Azhar University
Egypt
(External Examiner)

RUSLI HAJI ABDULLAH,PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date: 30 July 2018

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

Khamirul Amin Matori, PhD
Associate Professor
Faculty of Science
Universiti Putra Malaysia
(Chairman)

Abdul Halim Shaari, PhD
Professor
Faculty of Science
Universiti Putra Malaysia
(Member)

Raba'ah Syahidah Azis, PhD
Senior Lecturer
Faculty of Science
Universiti Putra Malaysia
(Member)

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ROBIAH BINTI YUNUS, PhD
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Name and Matric No.: Zamratul Maisarah Binti Mohd Ismail (GS40975)

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Signature: ______________________________

Name of Chairman of Supervisory Committee: Khamirul Amin Matori

Signature: ______________________________

Name of Member of Supervisory Committee: Abdul Halim Shaari

Signature: ______________________________

Name of Member of Supervisory Committee: Raba'ah Syahidah Azis

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

SLS	Soda lime silica
T_g	Glass transition temperature
T_c	Crystallization temperature
T_x	Onset temperature
WLEDs	White light emitting diodes
E_{opt}	Optical band gap
IR	Infrared
DSC	Differential scanning calorimetry
XRD	X-ray diffraction
FESEM	Field emission scanning electron microscopy
EDX	Electron dispersive X-ray
FTIR	Fourier transform infrared
UV-Vis	UV-visible
PL	Photoluminescence
ICDD	International Centre for Diffraction Data

CHAPTER 1

INTRODUCTION

1.1 Research background

Research innovation has recently committed to environmental concerns and energy consumption which have become technological importance for most industrial field in particular manufacturing sector. Emphasizing in industrialisation, the need for green alternative is critical to balance resource productivity and reduce pollution. In realizing this goal, various alternative have been done. Among this concern, initiative in research has been intensified on waste management. Solid waste recycling have been chosen as ideal ways to address the abovementioned context.

Recycling of waste via research technologies represents an effective way to safely use the discarded materials. In fact, recycling of waste also helps in reducing dependency on natural resources as raw materials (Chinnam *et al*., 2013). In addition, manufacturing process for instance benefits from this routes by energy saving and minimized waste disposal. Recent research has reported that utilizing secondary materials can reduce energy and pollution compared to production by using natural raw materials (Eckelman *et al*., 2009). In mass production, these advantages are essential because reducing energy and recycled sources interrelated to production cost (Mymrin *et al*., 2016; Wiemes *et al*., 2017).

In general, the sources of generated waste are classified into household, industrial, commercial and institutional waste (Chinnam *et al*., 2013). In fact, the curent trend in manufacturing has placed major emphasis on the use of post-consumer wastes and industrial by-product in the production process (Shakir and Mohammed, 2013). Among the generated wastes, glass is known as waste that amenable to recycling and reprocessing into other products. According to Glass Packaging Institute (2015), production of recycled glass into new product can save enormous amount of raw material consumption. In view of manufacturing side, processing glass waste means energy saving and thus reducing CO_2 emission. In fact, incorporated waste glass can reduce glass melting time which eventually offers possiblity in cost reduction.

Furthermore, most of glass waste made of soda lime silicate (SLS) glass coming from botttles. Glass from bottles is considered as a major waste product. A routine way to utilize these glass bottles is by recycling. SLS glass has been nominated as vitreous waste that worth attention as raw material. The main component of SLS glass is consisted of silicon dioxide (SiO_2), sodium oxide (Na_2O) and calcium oxide (CaO) (Chimalawong *et al*., 2012). Owing to its good glass-forming nature, SLS glass have fine optical and mechanical properties, such as good chemical stability, good durability, low melting point, high ultraviolet transparency, and good rare earth (RE) ion solubility as reviewed in previous studies (Xu *et al*., 2004; Qiao *et al*., 2006; Wang *et al*., 2011; Chimalawong *et al*., 2012). In addition, SLS glass has been explored previously for its

potential as glass precursor. Research innovation have evolved where glass system containing ZnO is of interest due to its ability as host material. In a glass system, ZnO have ability as glassformer and modifier (Centikaya *et al*., 2013).
This glass system known for its ability widely used for lowering the sintering temperature and optimizing coefficient thermal expansion in the field of electric devices such as region visible to infrared region transparency and allows doping ions to stimulated luminescence efficiency that suits for lasers, sensors and optical signal amplifiers (Karthikeyan *et al*., 2003; Pascutta *et al*., 2009; Sontakke and Annapurna, 2013; Pandey *et al*., 2015; Sadat *et al*., 2015).

Accordingly, the abovementioned host glass featuring ZnO can be transformed into glass–ceramics. Glass ceramics are inorganic silicate materials that have crystalline characteristics which can exist in one or more phases. In addition, crystals can be found embedded in a glass (amorphous) matrix prepared by the controlled crystallization of suitable glass compositions (Holland, 2002). Typically, the formation of glass ceramic can be carried out by heat treatment for certain amount of time focusing of nucleation and crystal growth. Furthermore, in order to synthesize waste derived glass and glass ceramic, vitrification is noted as the best approach as reported by Colombo *et al*. (2003) and Rawlings *et al*. (2006). Pisciella *et al*. (2000) stated that this technique is a promising solution to overcome environmental effect that comes from industrial practices and have advantage in adding value to waste. According to Erol *et al*. (2007), the complex composition of waste need control by tuning the initial composition and heat tretament conditions. These crucial parameters may effect the kind of crystalline phase developed in the glass ceramic and final properties of the materials. Furthermore, controlled cooling of a molten glass or by sintering and crystallisation of glass powders glass–ceramic have been reported as method to produce glass ceramic (Hing *et al*., 1997; Chinnam *et al*., 2013).

Zinc silicate (Zn_2SiO_4) often times introduced as willemite, is one of silicates based phosphor (Takesue *et al*., 2009; Tarafder *et al*., 2014). Previous studies have been reported that tailoring Zn_2SiO_4 have been explored suitable for hosting rare earth (RE) (Tarafder *et al*., 2014; Rivera-Enríquez *et al*., 2016). Due to its tetrahedral rigid with three different fourfold crystallographic sites, trivalent RE doping with Zn_2SiO_4 ensure in emitting strong emission in visible region that promising in fabricating solid state laser and white LEDs (WLEDs) (Takesue *et al*., 2009; Krsmanović *et al*., 2009; Tarafder *et al*., 2014). As a function of temperature, Zn_2SiO_4 can be formed in α, β and γ-phases where α phase is noted as the most stable according to phase equilibrium by Takesue *et al*. (2009).

In this study, a series of zinc soda lime silica (ZnO-SLS) doped Nd_2O_3 glasses are prepared from conventional melt-quenching technique and zinc silicate based glass-ceramics are derived from these precursor glasses by a sintering process. In order to study the effect of sintering temperature and Nd_2O_3 doping, the properties of the glass and zinc silicate glass-ceramics have been characterized for its thermal, physical, structural, morphological and optical behaviour. Throughout the study, the structural and optical properties of precursor glass and Nd_2O_3 doped zinc silicate based glass-ceramics have been evaluated by differential scanning calorimetry (DSC), X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), Fourier transform infrared (FTIR), UV-Visible (UV-Vis) and photoluminescence (PL) spectroscopy.To address the abovementioned scope, the aim of this work is to fabricate and characterize Nd_2O_3-doped zinc silicate based glass-ceramics derived from ZnO-SLS glass system as a potential materials in the form of phosphor.

1.2 Problem statement

The development of inorganic luminous material based glass and glass ceramic has been strongly pursued due to their high thermal stability, low melting point and well solubilty with rare earth (Lahoz *et al*., 2005; Jyothi *et al*., 2011; Reben *et al*., 2012; Xiangyu *et al*., 2013). Among the materials, Zn_2SiO_4 has gained interest due to its ability to display emission from UV to IR regions and it is suitable as a host for transition metal and rare earth doping (Zhang *et al*., 2003; El-Ghoul *et al*., 2013; Babu and Buddhudu, 2013; Tarafder *et al*., 2013). Benefiting from these characteristics, Zn_2SiO_4 is widely nominated for solid state laser, optical communication and light emitting diode (LED) application. Various approaches have been reviewed to synthesize Zn_2SiO_4 including via hydrothermal method (Yu *et al*., 2010), sol gel method (Demsbski *et al*., 2011; Babu and Buddhudu, 2013; Tung *et al*., 2016) and pyrolysis (Sivakumar et al., 2014). However, these methods suffer from high cost and high temperature arises from the usage of SiO_2 as raw material (Bunting *et al*.,1930; Patrascu *et al*., 2009). Therefore, the utilization of recyclable vitreous SLS glass waste is expected to outcost the used of SiO_2 as source and ease the fabrication at lower temperature. In addition, when targeting components to produce Zn_2SiO_4, ZnO is often introduced as wide band gap semiconductor material that suitable for RE doping where placing f electron elements into ZnO based material promising for solid state photonic device applications (Lakshiminarayana *et al*., 2008; Singh *et al*., 2008; Abo-Mosallam *et al*., 2010); Kaur *et al*., 2011; Xian and Li, 2013; Jayanthi *et al*., 2013). The contribution of these components sums up this work by presenting a simple, cost effective and eco-friendly route.

Till date no research have been reported on Nd^{3+} doped Zn_2SiO_4 by using SLS glass waste as silica source yet. In this work, the behaviour of Nd^{3+} doped Zn_2SiO_4 containing ZnO-SLS system have been investigated via low cost route solid state reaction. This study is the first study in literature to report on Nd^{3+} doped on Zn_2SiO_4 that produce visible emission in blue to red region.

1.3 Objectives of the Study

The main objective of this research is to fabricate and characterize zinc silicate based glass and glass ceramic containing SLS-ZnO glasses. This research have been experimentally design accordingly starting from mixing the glass composition, undergo melt-and quenching process, progresssion of doping process and heat treatment and followed by various steps of analysis. The objectives of this research are summarized as follows:

1. To synthesize $Zn_2SiO_4:Nd^{3+}$ from waste SLS glass, ZnO and Nd_2O_3 using solid state method.
2. To determine the thermal and physical analysis of $Zn_2SiO_4:Nd^{3+}$.
3. To study the effect of sintering temperature on structural and optical properties of $Zn_2SiO_4:Nd^{3+}$.
4. To evaluate the effect of Nd_2O_3 doping on structural and optical properties of $Zn_2SiO_4:Nd^{3+}$.

1.4 Hypotheses

Based on objectives, the hypotheses for this study are:

1. The melt-quenching process of SLS, ZnO and Nd_2O_3 is expected to form blueish glass frits.
2. The thermal analysis of ZnO-SLS doped Nd_2O_3 glasses by DSC analysis is expected to show changes as Nd^{3+} concentration increased. The changes is due to formation of non-bridging oxygen that caused by structure rearrangement after doping.
3. Density of $Zn_2SiO_4:Nd^{3+}$ is expected to show non linearity trend. The density is expected to increase as Nd^{3+} doping increases due to large molecular weight between Nd_2O_3 compared to ZnO and SiO_2 and crystallization process. In addition, the density might also decreased due to formation of non bridging oxygens due to doping.
4. XRD results for ZnO-SLS doped Nd_2O_3 glasses are expected to show broad hump and the sintered Zn_2SiO_4 glass ceramic are expected to show crystalline pattern. Based on ZnO-SiO_2 phase diagram, the samples are expected to form β-Zn_2SiO_4 and α-Zn_2SiO_4 as sintering temperature increases. The Nd^{3+} doping is expected to assist in nucleation during crystallization.
5. FTIR bands are expected to detect the SiO_4, ZnO_4 and Si-O-Zn bands which confirmed the formation of Zn_2SiO_4.
6. FESEM micrograph $Zn_2SiO_4:Nd^{3+}$ glass and glass ceramic are expected to show evolution of microstructure from irregulars particles to more refined grains boundaries.
7. UV-Vis absorption is expected to shows Nd^{3+} related absorption peaks which confirmed the incorporation of Nd^{3+} into Zn_2SiO_4 lattice.
8. Photoluminescence results is expected to shows emission in blue, green and red visible range upon excitation at 800 nm. The emission is assigned to Nd^{3+} related transition.

1.5 Scopes of the Study

The scopes of the study are stated as follows:

1. Zinc silicate doped neodymium ions, $Zn_2SiO_4:Nd^{3+}$ was prepared from waste SLS glasses, ZnO and Nd_2O_3 powders according to stoichiometric equation, $(ZnO_{0.5}SLS_{0.5})_{1-x}(Nd_2O_3)_x$ where x = 0, 0.01, 0.02, 0.03, 0.04 and 0.05 by using conventional solid state method.

2. $Zn_2SiO_4:Nd^{3+}$ based glass ceramic samples were prepared at varied sintering temperature from 600 °C to 1000 °C.
3. The physical properties of $Zn_2SiO_4:Nd^{3+}$ were analyzed by using densimeter to determine the density of the sample.
4. The thermal properties of $[(SLS)_{0.5}(ZnO)_{0.5}]_{1-x}(Nd_2O_3)_x$ were analyzed by DSC measurement to determine the glass transition temperature (T_g) and crystallization temperature (T_c) of the sample.
5. The structural properties of $Zn_2SiO_4:Nd^{3+}$ were analyzed by XRD, FTIR, FESEM and EDX to determine the phase formation, chemical bonding, microstructure and elemental composition of the sample.
6. The optical properties of $Zn_2SiO_4:Nd^{3+}$ were analyzed by UV-Vis and PL spectroscopy to evaluate the absorption, optical band gap and luminescence of the sample.

1.6 Importance of study

The need for novel luminous material has intensified research into glass and glass ceramic derived from waste. Considerable research has proven that these glasses have potential in emerging as solid state technology in the form of phosphor. Recently, great effort have been devoted to achieve white light emitting diodes sources (WLEDs) for many application purposes such as high luminous efficiency, low energy consumption, long working lifetime, and convenient manufacturing method (Chen *et al*., 2015).

Due to its proven transparency in visible light region and efficient light emitting of the luminescent glasses, Zn_2SiO_4 have been a kind of desirable optical materials and also a potential candidates to replace the phosphors for WLEDs concerning their benefits such as lower fabrication cost, easy to shape, and superior thermal stability. Currently, Zn_2SiO_4 have been attempted as sources for red and green phosphor for WLEDs by UV and blue excitation (El-Ghoul *et al*., 2013; Babu and Buddhudu, 2013; Tarafder *et al*., 2013). However, no attempts have been reported yet for Zn_2SiO_4 that excited by near infrared wavelength.

To the best of our knowledge, no study have been reported for Nd^{3+} doped Zn_2SiO_4 prepared from SLS glass waste as silica source yet. In the present work, investigation are focused on the behaviour of Nd^{3+} doped Zn_2SiO_4 containing ZnO-SLS system fabricated via inexpensive solid state method. In fact, this study is the first study in literature to report on Nd^{3+} doped Zn_2SiO_4 with visible emission that covers blue to red region. In fact, based on the previous report on Nd^{3+} doped phosphor, luminescence studies are frequently done on the infrared region, however there have been very few reports on the emission behavior of Nd^{3+} in visible region. The research gap has thus motivated this work to explored the potential of Zn_2SiO_4 doped Nd^{3+} as a potential candidate for WLEDs based glass ceramic in visible region via excitation using an infrared diode. Furthermore, details of the upconverting process in this work are also worth attention.

1.7 Outline of the Thesis

This thesis is outlined as follows. Chapter 1 gives an overview of research background, problem statements, objectives, scopes of study and importance of study. The pioneering work of glass, glass ceramic and Zn_2SiO_4 doped rare earth and their properties are covered in Chapter 2. Overall research methodology including sample preparation and characterization of Zn_2SiO_4 doped neodymium based glass ceramic are explained in Chapter 3. The results and discussion of Zn_2SiO doped neodymium based glass ceramic in terms of effect of sintering temperature and effect of Nd^{3+} doping are discussed thoroughly for their physical, thermal, structural and optical properties in Chapter 4. The conclusions and recommendation for future work are explained in Chapter 5.

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