INFLUENCE OF SILVER NANOPARTICLES ON OPTICAL PROPERTIES OF ERBIUM-SAMARIUM CO-DOPED SODIUM LITHIUM PHOSPHATE GLASS

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ABSTRACT

This thesis reported the influence of silver nanoparticles on the optical properties of erbium (Er^{3+}) and samarium (Sm^{3+}) ion co-doped sodium lithium phosphate glass. A series of glass with composition of (59-x)P_2O_5–(x)Na_2O–30Li_2O–0.5Er_2O_3–0.5Sm_2O_3 (where x = 10, 15, 20, 25 and 30 (in mol%)), (49.5-y)P_2O_5–20Na_2O–30Li_2O–(y)Er_2O_3–0.5Sm_2O_3 (where y = 0.2, 0.4, 0.6, 0.8 and 0.10 (in mol %)) and 48.5P_2O_5–20Na_2O–30Li_2O–0.5Sm_2O_3–1.0Er_2O_3–(z)AgCl (where z = 0.01, 0.03, 0.05, 0.07 and 1.00 (in gram)) were prepared using melt quenching technique. These samples were characterized using X-ray diffractometer (XRD), Transmission Electron Microscope (TEM), Differential Thermal Analysis (DTA), Fourier Transform Infrared (FTIR) spectrometer, Energy Dispersive X-ray (EDX) spectrometer, UV- Vis NIR spectrophotometer and Photoluminescence (PL) spectrometer. The XRD pattern confirms the amorphous nature of the glass and existence of silver nanoparticles with average diameter estimated to be ~0.615 nm was revealed by TEM image. Thermal stability is found to vary in the range of 143 ℃ to 197 ℃. Five major IR absorption peaks were found at 580 cm^{-1}, 763 cm^{-1}, 920 cm^{-1}, 1055 cm^{-1} and 1128 cm^{-1} which attributed to P-O-P bending vibrations, P-O-P symmetric stretching vibrations, P-O-P asymmetric stretching vibrations, $(P\text{-}O)^-$ asymmetric stretching vibrations and P=O asymmetric stretching vibrations respectively. The EDX data showed all the elements in the composition were present. The UV absorption spectra showed 12 peaks that belongs to both Er^{3+} and Sm^{3+} ions. The indirect optical energy band gap and Urbach energy were in the range of (3.43 – 3.52) eV and (0.23 – 0.40) eV respectively. Four prominent peaks are evidenced, which are assigned to various transitions among Sm^{3+} excited states to the ground state such as $^4G_{5/2} \rightarrow {}^6H_{5/2}$ (560 nm), $^4G_{5/2} \rightarrow {}^6H_{7/2}$ (596 nm), $^4G_{5/2} \rightarrow {}^6H_{9/2}$ (640 nm) and $^4G_{5/2} \rightarrow {}^6H_{11/2}$ (704 nm). Judd Ofelt analysis shows the trends in parameter which is $\Omega_2 > \Omega_4 > \Omega_6$ and spectroscopic quality factor, Q has the highest value with 4.14 for z(0.05) and the lowest for z(0.07) which is 3.48. The average electric dipole (A_{ed}), radiative lifetimes (τ_{rad}) and branching ratios (β) are estimated for Sm^{3+} ions glass matrices. It is observed that the $^6F_{3/2}$ transition have higher branching ratio values (99.93%) in all five samples. Among these five transitions, $^6H_{15/2}$ transitions in z(0.01) have the lowest values (42.17%). The photoluminescence intensity of Sm^{3+} is found to greatly enhance with the increase of Er^{3+} ion concentration together with Ag NPs in the glass matrix. Radiative properties such as stimulated emission cross section (σ_p^E), peak wavelength (λ_p), effective emission bandwidth ($\Delta\lambda_{eff}$) and gain bandwidth (ΔG) were measured. The values of σ_p^E for $^4G_{5/2}$ emission transition are in the order of $^4G_{5/2} \rightarrow {}^6H_{5/2} > {}^6H_{7/2} > {}^6H_{9/2} > {}^6H_{11/2}$ for all samples. z(0.10) has the maximum value of ΔG for all transition parameters and the highest one is at $^4G_{5/2} \rightarrow {}^6H_{11/2}$ transition (12.72×10^{-27} cm^3). Based on the result, it can be concluded that these glasses have a potential for the development of solid state laser application and photonic devices.

ABSTRAK

Tesis ini melaporkan kesan nanopartikel (NP) perak (Ag) terhadap sifat-sifat optik kaca sodium litium fosfat yang didopkan dengan ion erbium (Er^{3+}) dan ion samarium (Sm^{3+}). Sampel siri kaca dengan komposisi (59-*x*)P_2O_5–(*x*)Na_2O–30Li_2O–0.5Er_2O_3–0.5Sm_2O_3 (dimana x = 10, 15, 20, 25 dan 30 (dalam mol%)), (49.5-*y*)P_2O_5–20Na_2O–30Li_2O–(*y*)Er_2O_3–0.5Sm_2O_3 (dimana y = 0.2, 0.4, 0.6, 0.8 dan 0.10 (dalam mol %)) and 48.5P_2O_5-20Na_2O-30Li_2O-0.5Sm_2O_3-1.0Er_2O_3-(*z*)AgCl (dimana z = 0.01, 0.03, 0.05, 0.07 dan 0.10 (dalam gram)) telah disediakan menggunakan kaedah pelindapan peleburan. Kesemua sampel ini telah dikaji menggunakan Pembelauan Sinar-X (XRD), Mikroskopi Elektron Penghantaran (TEM), Penganalisa Terma Pembeza (DTA), Inframerah Jelmaan Fourier (FTIR), Serakan Tenaga Sinar-X (EDX), Ultralembayung Spektrofotometer (UV-Vis) dan Fotoluminesens Spektrofotometer (PL). Corak Pembelauan Sinar-X mengesahkan keadaan amorfus sebenar kaca dan kewujudan nanopartikel perak (Ag) dengan diameter purata dianggarkan bersamaan ~0.615 nm telah dipamerkan oleh imej Mikroskopi Elektron Penghantaran (TEM). Kestabilan terma telah diperoleh dari kepelbagaian kadar julat 143 ℃ sehingga 197 ℃. Lima puncak spektrum IR terdapat di 580, 763, 920, 1055 dan 1128 cm^{-1} yang masing-masing mewakili getaran membengkok P-O-P, getaran regangan simetri P-O-P, getaran regangan asimetri P-O-P, getaran regangan asimetri $(P\text{-}O)^-$ dan getaran regangan asimetri P=O. Data Serakan Tenaga Sinar-X menunjukkan kesemua unsur wujud di dalam komposisi. Spektrum penyerapan telah menunjukkan 12 puncak kepunyaan kedua-dua ion Er^{3+} dan Sm^{3+}. Tenaga jurang jalur dan tenaga Urbach masing-masing berada di dalam julat (3.43 – 3.52) eV dan (0.23 – 0.40) eV. Empat puncak yang jelas telah dibuktikan, yang mana telah di tetapkan ke pelbagai peralihan keadaan teruja Sm^{3+} ke keadaan dasar iaitu $^4G_{5/2} \rightarrow {}^6H_{5/2}$ (560 nm), $^4G_{5/2} \rightarrow {}^6H_{7/2}$ (596 nm), $^4G_{5/2} \rightarrow {}^6H_{9/2}$ (640 nm) dan $^4G_{5/2} \rightarrow {}^6H_{11/2}$ (704 nm). Analisa Judd Ofelt telah menunjukkan trend parameter $\Omega_2 > \Omega_4 > \Omega_6$ dan faktor kualiti spektroskopik, Q mempunyai nilai tertinggi 4.14 untuk *z*(0.05) dan nilai terendah bagi *z*(0.07) iaitu 3.48. Purata dwikutub elektrik (A_{ed}), hayat penyinaran (τ_{rad}) dan nisbah cabang (β) telah dianggarkan untuk matriks kaca ion Sm^{3+}. Pemerhatian yang dijalankan, mendapati peralihan $^6F_{3/2}$ mempunyai nisbah cabang yang tertinggi (99.93%) di kelima-lima sampel. Diantara lima peralihan, peralihan $^6H_{15/2}$ di *z*(0.01) mempunyai nilai terendah (42.17%). Keamatan fotoluminesens ion Sm^{3+} didapati meningkat dengan baik dengan penambahan kepekatan ion Er^{3+} bersama dengan Ag NPs di dalam matriks kaca. Ciri-ciri penyinaran seperti stimulasi keratan rentas pelepasan (σ_p^E), puncak panjang gelombang (λ_p), keberkesanan jalur lebar pelepasan ($\Delta\lambda_{eff}$) dan gandaan jalur lebar (ΔG) telah dikira. Nilai σ_p^E bagi peralihan $^4G_{5/2}$ adalah mengikut turutan $^4G_{5/2} \rightarrow {}^6H_{5/2} > {}^6H_{7/2} > {}^6H_{9/2} > {}^6H_{11/2}$ untuk kesemua sampel. *z*(0.10) mempunyai nilai ΔG maksima untuk semua parameter peralihan dan yang tertinggi berada di peralihan $^4G_{5/2} \rightarrow {}^6H_{11/2}$ (12.72×10^{-27} cm^3). Berdasarkan hasil yang diperolehi, dapat disimpulkan bahawa kaca – kaca ini mempunyai potensi untuk kemajuan aplikasi laser keadaan pepejal dan alat fotonik.

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LIST OF SYMBOLS

A	-	The cross-sectional area of material with area parallel to the applied force vector
A_{ed}	-	Electric-dipole
A_{md}	-	Magnetic-dipole
α	-	Absorption coefficient
aJ	-	Ground state
B	-	Constant
β	-	Branching ratio
bJ	-	Excited state
C	-	Speed of light
C_{RE}	-	Concentration of the rare-earth
C	-	Light velocity
d	-	Interplanar separation
D	-	Density of air
E_u	-	Urbach energy
E_f	-	Energy of electron of final state
Ei	-	Eenergy of an electron at lower band
E_g	-	Optical energy bandgap
$\varepsilon_a(v)$	-	Molar extinction coefficient
e	-	Electron charge
F	-	Applied force
f_{exp}	-	Experimental oscillator strength
f_{cal}	-	Oscillator strength

ΔG	-	Gain bandwidth
$h\nu$	-	Photon energy
H_R	-	Hruby parameter
Λ	-	Wavelength
Ω_λ	-	Judd-Ofelt parameters
M	-	Molecular weight
M	-	Electron mass
m_r	-	Atomic weights in kg of cation
m_o	-	Atomic weights in kg of anion
η	-	Viscosity
n	-	Order of diffraction
N	-	Integer
θ	-	Angle
ρ	-	Density
ρ_o	-	Density of distilled water
P	-	Number of transitions
Q	-	Quality factor
rms	-	Root-mean-square
S_{ed}	-	Line-strength for electric
T_c	-	Glass crystallization temperature
T_m	-	Glass melting temperature
T_g	-	Glass transition temperature
T_{rad}	-	Radiative lifetime
ΔT	-	Glass thermal stability
T	-	Thickness of the sample
τ	-	Shear stress

U_{κ}	-	Values of reduced matrix elements
μ	-	Reduced mass
V_m	-	Molar volume
w_a	-	Weight of sample in air
w_1	-	Weight in distilled water
Ω	-	Frequency dependence
λ	-	Wavelength
λ_p	-	Peak wavelength
$\Delta\lambda_{eff}$	-	Effective emission bandwidth
σ_p^E	-	Stimulated emission cross section

LIST OF ABBREVIATIONS

P_2O_5	-	Phosphate
Ag	-	Silver
BO	-	Bridging oxygen
CB	-	Conduction band
DTA	-	Differential thermal analysis
DBO	-	Double bonded oxygen
EDX	-	Energy dispersive X-ray
Er	-	Erbium
FWHM	-	Full width at half maximum
FTIR	-	Fourier transform infrared
H	-	Hydrogen
IR	-	Infrared
J-O	-	Judd-Ofelt
NBOs	-	Non-bridging oxygens
NPs	-	Nanoparticles
O	-	Oxygen
PL	-	Photoluminescence
RE	-	Rare earth
SPR	-	Surface plasmon resonance
Sm	-	Samarium
TEM	-	Transmission electron microscopy
UV-Vis	-	Ultraviolet visible
VB	-	Valence band

XRD	-	X-ray diffraction
Li_2O	-	Lithium oxide
K_2O	-	Potassium oxide
Na_2O	-	Sodium oxide

LIST OF APPENDIX

CHAPTER 1

INTRODUCTION

1.1 Introduction

This chapter briefly discuss about the background of the research, problem statement, objectives, scope of study and research significance.

1.2 Background

Glasses incorporated with various rare-earth ions (RE) find number of applications in the development of optoelectronic devices such as visible lasers, optical amplifiers, optical detectors, fluorescent display devices, , optical fibers, planar waveguides, hole burning high density memories and compact microchip lasers (Venkataiah *et al.*, 2015). Among oxide glasses, phosphate glasses have several advantages over the conventional silicate and borate glasses due to their unique characteristics that includes high transparency, low melting point, high thermal stability, high gain density that is mainly due to high solubility of RE^{3+} ions besides low refractive index and dispersion. (Amjad *et al*., 2012; Sahar *et al*., 2012; Lim *et al*., 2013; Basavapoornima and Jayasankar, 2014; Ramteke *et al*., 2017; Sangwaranatee *et al*., 2017) The RE^{3+} ions doped in various host matrices such as, tellurite-tungsten zirconium (Venkataiah *et al*., 2015), boro- telluro-phosphate (Selvi *et al*., 2015) sulfophosphate (Ahmadi *et al*., 2017), sodium tellurite (Mawlud *et al*., 2017), bismuth borate (Agarwal *et al*., 2009), fluorosilicate (Linganna *et al*., 2015) and lead bismosilicate (Bhardwaj *et al*., 2014) glasses are receiving considerable attention.

The properties of the phosphate glasses are strongly related to the peculiar structure of their glass network and the role of network modifiers and intermediates. The glass forming component in phosphate glasses is P_2O_5 and the basic unit is based on $[PO_4]^{3-}$ tetrahedrons. It contains a P=O double bond, which is an asymmetric center within the glass structure that accounts for the relatively poor mechanical properties and chemical durability, if compared to silicate glasses. (Moustafa and El-Egili, 1998; Tashtoush and El-Desoky, 2007; Brauer, 2012; Mura *et al*., 2013; Andronache and Racolta, 2014; Basavapoornima and Jayasankar, 2014) To overcome the problem, many constituents can be added to the composition to improve physical properties and chemical stability of the glass. Specifically, alkali metal oxides (R_2O with R=Li, Na, K, Rb and Cs) were added to the glass matrix to improve strength, chemical durability and glass forming ability (Mura *et al*., 2013; Basavapoornima and Jayasankar, 2014; Oueslati *et al*., 2014; Chanthima *et al*., 2018).

The glass composition significantly effects on optical properties of RE ions, hence selection of suitable network former and network modifier is essential for the enhancement of the optical performance. (Bhardwaj *et al*., 2014; Kumar *et al*., 2017; Srihari and Jayasankar, 2017) Alkali metal ions such as lithium oxide and sodium oxide are well-known modifiers widely used in phosphate glasses to increase the chemical durability and also play an important role to improve the luminescent properties (Mawlud *et al*., 2017; Sangwaranatee *et al*., 2017; Ravangvong *et al*., 2017). Strength, chemical durability and luminescence properties are improved with incorporation of lithium oxide and sodium oxide as a modifier in RE doped phosphate glass.

Among RE ions, samarium (Sm^{3+}) ion is one of the interesting Ln^{3+} ions because of the excellent luminescence properties (Venkatarao *et al*., 2009; Lim *et al*., 2013; Brahmachary *et al*., 2015). Glasses doped with Sm^{3+} ($4f^5$) ions exhibit relatively high quantum efficiency because of the large energy gap (less non-radiative decay) between the $^4G_{5/2}$ level and the next lower lying energy level $^6F_{11/2}$, which is approximately 7200 cm^{-1}. These glasses show various populating as well as different channels that lead to luminescence quenching. It possesses very strong fluorescence intensity, rich energy levels, high luminescence efficiency and large stimulated emission cross-section. The host glass containing Sm^{3+} ions possess most attractive qualities due to their potential applications in solid state lasers, under sea communication, color displays, temperature sensors, medical diagnostics and high density optical storage materials (Kumar *et al*., 2003; Amjad *et al*., 2012; Do *et al*., 2012; Reddy *et al*., 2014; Elisa *et al*, 2013; Selvi *et al*., 2015; Yamsuk *et al*., 2018). Based on these potentialities, sodium lithium phosphate glass system with Sm^{3+} are prepared to achieve the lasing glass material.

Another RE ions used in this research is erbium ion (Er^{3+}). A large number of researches have been done on Er^{3+} ions due to its potentiality of blue, green, and red emissions, in addition to an IR broadband line around 1.5 μm for applications in solid state lasers and optical amplifiers. Among all the rare-earth ions, Er^{3+} is frequently employed as an upconversion luminescence center due to its more homogeneous energy level array, enabling longer lifetimes of metastable energy levels. In the short-wavelength region, Er^{3+} can emit blue ($^4F_{7/2} \rightarrow {}^4I_{15/2}$, 488 nm), green ($^2H_{11/2}/{}^4S_{3/2} \rightarrow {}^4I_{15/2}$, 520/550 nm) and red ($^4F_{9/2} \rightarrow {}^4I_{15/2}$, 660 nm), and the upconversion emissions of all these wavelengths have been observed when pumped with various wavelengths matching $^2H_{11/2}/{}^4S_{3/2}$, $^4F_{9/2}$, $^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{13/2}$ levels. (Fu *et al*, 2013; Soltani et al., 2015)

Interest in glass containing rare-earth (RE) ions and metallic nanoparticles (NPs) including gold and silver are expected for the intensification of luminescence. Metallic nanoparticles exhibit unique optical response, enhanced electromagnetic field and constitute the area of plasmonics (Sahar *et al*, 2012). In particular, embedding materials such as metal nanoparticles in glasses are expected to produce promising materials for functional optical devices due to a large third-order nonlinear susceptibility and an ultra-fast non-linear response (Sahar et al., 2012; Rahman et al., 2016). Enhancement in the upconversion (UC) luminescence and the nonlinear properties makes rare-earth doped glasses containing metallic nanoparticles (NPs) are attractive because of the presence of NPs. It makes the glass suitable for optical device applications. One of the most important properties of Ag NPs is related to the excitation of surface plasmon resonance (SPR) within the optical spectral band. The luminescence emission can be enhanced by the resonance of the surface plasmon frequency (SPR) of the NPs with the frequency of excitation beam together with the materials luminescence frequency (Amjad *et al*, 2012; Vodnik *et al*., 2012; Dousti, 2014; Solatani *et al*., 2015). The preparation and characterization of RE doped glasses embedded with metallic nanoparticles have been studied by many researchers (Dousti et al., 2013; Adnan *et al*., 2015; Anigrahawati *et al*., 2015; Ahmadi *et al*., 2017). Specifically, there is no report on metallic nanoparticles embedded inside the sodium lithium phosphate glass matrix with RE ions. Regards to this matter, deeper study of the effect of nanoparticles on luminescence enhancement and energy transfer processes in the sodium lithium phosphate glass matrix is necessary.

1.3 Problem Statement

The energy transfer between two rare earths codoped in glasses has been studied in many systems because the sensitized luminescence is not only of interest for applications but also for understanding basic mechanisms. Many researchers have discussed the phenomena of radiative and non radiative transitions in the rare earth ions and succeeded in realizing energy transfer in codoped glasses, as indicated by enhancement in the intensity of fluorescence or variation in the lifetime of the emitting levels. (Tripathi *et al*, 2008)

Although there are researches on codoped between two rate earth ions but not so much about energy transfer between erbium ion and samarium ion. Based on the searching of the previous study focusing on these rare earths, Tripathi *et al*. (2008) has studied about them in lithium tellurite glass, TeO_2-Li_2O. Then, Cosmo *et al*. (2014) has studied on novel samarium-erbium and samarium-terbium codoped glass phosphor for application in warm white light-emitting diode. So, the research of erbium oxide and samarium oxide in phosphate glass as co-dopant are needed to be explored.

Besides that, researches on silver NPs with combination of these rare earths are not that much compared to single doped. Erbium oxide doped sodium lead tellurite glass and samarium oxide doped sodium borosilicate glass containing silver NPs has been studied by Dousti (2012 & 2014). Moreover, erbium oxide in phosphate glass and zinc tellurite glass with embedment of silver NPs has been studied by Amjad *et al*. (2012 & 2013).

Although erbium-samarium codoped glass has been studied by other researchers, but rarely in phosphate glass. Therefore, the study of erbium oxide and samarium oxide as a co-dopant with a combination of sodium oxide and lithium oxide as a modifier in a phosphate glass is needed. The influence of embedment of silver nanoparticles in the phosphate glass co-doped with erbium oxide and samarium oxide on structural, thermal and optical properties should be explored. More importantly, the lasing glass properties are needed to be studied in terms of analysis of Judd-Ofelt theory and its parameter together with radiative properties.

1.4 Objectives

In this research, a stable with wide formation ranges of phosphate glasses are prepared to full fill these objectives which are:

i. To optimize the concentration of sodium oxide, erbium oxide and silver nanoparticles via absorption spectra analysis and photoluminescence spectra analysis.

ii. To determine effect of silver nanoparticles on optical properties of erbium-samarium codoped sodium lithium phosphate glass.

iii. To analyse Judd-Ofelt analysis and radiative properties towards lasing properties of erbium-samarium codoped sodium lithium phosphate glass with silver nanoparticles.

1.5 Scope of Study

To achieve this research, three series of glass with the composition of $P_2O_5 - Na_2O - Li_2O - Er_2O_3 - Sm_2O_3$ with and without silver nanoparticles was prepared by melt quenching technique. Differential Thermal Analysis (DTA) was used to determine the thermal properties of glass. Fourier Transform Infrared Spectroscopy (FTIR) was responsible to determine the structural properties. Energy Dispersive X-ray spectroscopy (EDX) was used to characterize the elements present in the glass. Transmission Electron Microscopy (TEM) was used to investigate the presence, size and shape of silver nanoparticles in glass. UV-Vis and Photoluminescence (PL) spectroscopy were used for the optical characterization.

1.6 Research Significance

The important of this research on erbium-samarium co-doped sodium lithium phosphate glass containing silver nanoparticles is to achieve high optical properties. The enhancement of optical properties of glass can be seen through UV-Vis and Photoluminescence analysis. From the characterization and analysis, the study outcome contributes better understanding towards properties of RE ions (Er^{3+} and Sm^{3+}) and silver nanoparticles (Ag) on sodium lithium phosphate glass. Importantly, through Judd-Ofelt analysis and radiative properties, the glasses can be promising materials with enhanced optical properties for photonic devices such as solid state laser and sensor.

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