

OPTICAL PROPERTIES IMPROVEMENT OF DYSPROSIUM DOPED
TELLURITE GLASS VIA SILVER/TITANIUM NANOPARTICLES
COEMBEDMENT

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DEDICATION

This thesis is dedicated to my mother, who taught me that knowledge is the bridge of success. Patience and determination will distinguish us from the failure. It is also dedicated to my wife, who taught me that even a fool can be knowledgeable if you respect the educator. Actions speak louder than words.

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ABSTRACT

In this study, some dysprosium ions (Dy^{3+})-doped zinc-magnesium tellurite glasses with the co-embedding of silver nanoparticles (ANPs) and titanium nanoparticles (TNPs) were prepared using the standard melt quenching method. The compositions of the glass series I and II were $(69.5-x)\text{TeO}_2-20\text{ZnO}-10\text{MgO}-0.5\text{Dy}-(x)\text{AgCl}$ ($0 \leq x \leq 1.0$ mol%) and $(69.3-y)\text{TeO}_2-20\text{ZnO}-10\text{MgO}-0.5\text{Dy}-0.2\text{AgCl}-(y)\text{TiO}_2$ ($0 \leq y \leq 0.6$ mol%), respectively. As-quenched glass samples were thoroughly characterized using different analytical techniques. The improvement in the optical, physical and structural properties of these glasses due to the co-embedding of two types of nanoparticles was determined. The optimum glass sample from both series was chosen to determine the influence of ANPs/TNPs inclusion on the optical, physical and structural characteristics. The ultraviolet-visible-infrared (UV-Vis-NIR) optical spectral analyses of the glass series I revealed an improvement in the absorption properties due to the addition of ANPs, while the glass series II (with ANPs and TNPs) showed a decrease in the absorption. Both glass series displayed six absorption bands due to the electronic transition from the ground state to various excited states of Dy^{3+} . The Judd-Ofelt intensity parameters, Ω_λ ($\lambda=2, 4, 6$) of the glasses were evaluated from the measured absorption spectra. The X-ray diffraction patterns of the as-quenched samples verified their amorphous nature. The high resolution transmission electron microscope (HRTEM) images revealed the presence of individual ANPs, TNPs and combined ANPs/TNPs in the glass matrix. The energy dispersive X-ray (EDX) spectra and mapping of the prepared glasses exhibited their appropriate elemental compositions. The incorporation of the ANPs/TNPs into the glasses was found to enhance their Raman and Fourier transformed infrared (FTIR) spectral intensities. The up-conversion and down-conversion photoluminescence spectra of both glass series showed three prominent emission bands at 482 nm (blue: ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{15/2}$), 574 nm (yellow: ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{13/2}$), and 664 nm (weak red: ${}^4\text{F}_{9/2} \rightarrow {}^6\text{H}_{11/2}$). In addition, the values of Ω_λ displayed the trend of $\Omega_2 > \Omega_6 > \Omega_4$ for the glass containing 0.1 mol% of TNPs, while all other glasses showed the trend of $\Omega_2 > \Omega_4 > \Omega_6$. The obtained results suggested that the optical properties of the Dy^{3+} -doped zinc-magnesium tellurite glasses can be improved via the co-embedding of ANPs/TNPs compared to one type of NPs embedment. It is asserted that the proposed glass system may be useful for the development of optoelectronic and solid-state devices needed for different applications.

ABSTRAK

Dalam kajian ini, beberapa kaca tellurit zink-magnesium didop ion dysprosium Dy^{3+} dengan penanaman bersama nanozarah perak (ANPs) dan nanozarah titanium (TNPs) telah disediakan melalui teknik piawai lindap-kejut leburan. Komposisi untuk siri kaca I dan II masing-masing ialah $(69.5-x)TeO_2-20ZnO-10MgO-0.5Dy-(x)AgCl$ ($0 \leq x \leq 1.0$ mol%) dan $(69.3-y)TeO_2-20ZnO-10MgO-0.5Dy-0.2AgCl-(y)TiO_2$ ($0 \leq y \leq 0.6$ mol%). Sampel kaca dicirikan dengan teliti menggunakan teknik analisis yang berbeza. Penambahbaikan dalam sifat optik, fizikal dan struktur kaca ini disebabkan oleh penanaman bersama dua jenis nanozarah telah ditentukan. Sampel kaca yang optimum dari kedua-dua siri telah dipilih untuk menentukan pengaruh kemasukan ANPs/TNPs terhadap sifat optik, fizikal dan struktur. Analisis spectrum optik ultralembayung-cahaya nampak-inframerah (UV-VIS-NIR) bagi siri kaca I menunjukkan penambahbaikan dalam sifat penyerapan disebabkan oleh penambahan ANPs, sementara siri kaca II (dengan ANPs dan TNPs) menunjukkan penurunan dalam penyerapan. Kedua-dua siri kaca ini memaparkan enam jalur penyerapan disebabkan oleh peralihan elektronik dari keadaan dasar ke pelbagai keadaan teruja Dy^{3+} . Parameter keamatan Judd-Ofelt Ω_λ ($\lambda=2, 4, 6$) bagi kaca telah dinilai daripada spectrum penyerapan yang dicerap. Corak pembelauan sinar-X sampel mengesahkan sifat amorfus kaca. Imej mikroskopi pancaran elektron beresolusi tinggi (HRTEM) mendedahkan kewujudan ANPs, TNPs tunggal dan gabungan ANPs/TNPs di dalam matrik kaca. Spektrum penyebaran tenaga sinar-X (EDX) dan pemetaan bagi kaca yang disediakan menunjukkan komposisi unsur yang sesuai. Pengabungan ANPs/TNPs didalam kaca didapati meningkatkan keamatan spektrum Raman dan Inframerah Fourier (FTIR). Spektrum fotoluminesen penukaran-atas dan penukaran-bawah dari kedua-dua siri kaca menunjukkan tiga jalur pancaran utama pada 482 nm (biru: $^4F_{9/2} \rightarrow ^6H_{15/2}$), 574 nm (kuning: $^4F_{9/2} \rightarrow ^6H_{13/2}$) dan 664 nm (merah lemah: $^4F_{9/2} \rightarrow ^6H_{11/2}$). Disamping itu, nilai Ω_λ menunjukkan tren $\Omega_2 > \Omega_6 > \Omega_4$ bagi kaca yang mengandungi 0.1 mol% TNPs, manakala kesemua kaca yang lain menunjukkan tren $\Omega_2 > \Omega_4 > \Omega_6$. Keputusan yang diperoleh mencadangkan bahawa sifat optik bagi kaca tellurit zink-magnesium didop Dy^{3+} mampu ditambahbaik melalui penanaman bersama ANPs/TNPs berbanding dengan penanaman sejenis nanozarah. Ia ditegaskan bahawa sistem kaca yang dicadangkan mungkin berguna untuk pembangunan peranti optoelektronik dan peranti keadaan pepejal bagi aplikasi yang berbeza.

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LIST OF ABBREVIATIONS

ANPs	-	Silver Nanoparticles
B	-	Blue emission
BDP	-	Bonding Dimer Plasmon
BO	-	Bridging Oxygen
CCT	-	Colour Correlated Temperature
CIE	-	Commission International de l'Eclairage
CR	-	Cross Relaxation
DC	-	Down-conversion
DDA	-	Discrete Dipole Approximation
EDX	-	Energy Dispersive X-ray
ESA	-	Excited State Absorption
ET	-	Energy Transfer
ETU	-	Excited Transfer Upconversion
FFT	-	Fourier Fast Transform
FTIR	-	Fourier Transform Infrared
GSA	-	Ground State Absorption
HRTEM	-	High Resolution Transmission Electron Microscopy
JCPDS	-	Joint Committee On Powder Diffraction Standard
LSPR	-	Localized Surface Plasmon Resonance
NBO	-	Non Bridging Oxygen
NR	-	Non Radiative
PL	-	Photoluminescence
RET	-	Resonance Energy Transfer
SAED	-	Small Angle Electron Diffraction
SEC	-	Stimulated Emission Cross section
SP	-	Surface Plasmon
TNPs	-	Titanium Nanoparticles
UC	-	Up conversion
UV-VIS-NIR	-	Ultra Violet visible near infrared
X-RD	-	X-ray diffraction

Y - Yellow emission

LIST OF SYMBOLS

f_{exp}	-	Experimental oscillator strength
f_{cal}	-	Calculated oscillator strength
$\varepsilon(\nu)$	-	Molar absorptivity at a frequency (in cm^{-1})
$ (S, L)J\rangle$	-	Absorption transition from initial state
$ (S', L')J'\rangle$	-	Excited state
m	-	Electron mass
c	-	Velocity of light in vacuum
n	-	Refractive index of glass sample
J	-	Total angular momentum
S_{ed}	-	Electric line strength
S_{md}	-	Magnetic line strength
A_{ed}	-	Electric dipole contribution of the total spontaneous emission probability
A_R	-	Total radiative probability
τ_R	-	Radiative lifetime
β_R	-	Branching ratio
$\sigma(\lambda_p)$	-	Stimulated emission cross-section
λ_p	-	Wavelength of the emission peak
$\Delta\lambda_{eff}$	-	Effective line width
η	-	Quantum efficiency
ε_∞	-	Optical dielectric function of the metal
N	-	Free electron concentration
ρ	-	Density
V_m	-	Molar volume
R_m	-	Molar refraction
α_e	-	Polarizabilities
Ω_x	-	Judd-Ofelt parameter
χ	-	Spectroscopic quality factors
β	-	Nephelauxetic ratio
δ	-	Bonding parameter

A_1, A_2	-	Weight factors
τ_1, τ_2	-	Fast and slow decay time
τ_{Exp}	-	Experimental lifetime
χ_c	-	Raman peak position
α	-	Amplitude
E_{opt}	-	Optical band gap
θ	-	Angle of incident
W_a	-	Weight of sample in air
W_b	-	Weight of sample in immersion liquid
ρ_x	-	Density of immersion liquid

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Among conventional glasses, tellurite has a good reputation for lanthanide (rare earth) and plasmonic equation. It was realised that having rare earth doped as an activator could improve their properties toward potential applications, especially in photonic device since their lower phonon energy is resulting in a weaker non radiative (NR) transition. By having low phonon energy, obtaining high quantum efficiency were expected thus finding optimum composition can be achieved. However, achieving optimised properties could reach certain limits where the quenching effect occurs. Here, researcher found a way to enhance the optical properties by embedding metallic nanoparticles. Literature shows the enhancement towards radiative emission by using metallic nanoparticles particularly noble metal as the sensitizer. Enhancements occur due to the absorption of light by metallic nanoparticles which is the coherent oscillation of the conduction band electron, formed directly into the incident electromagnetic radiation. Sensitizer absorbs the energy thereby transfer it to the activator while producing enhanced field within the glass matrix (Som and Karmakar, 2010b). Some researchers reported that by addition of more than one type nanoparticles, the intensity of surface plasmon resonance can be enhanced thus enhancing the glass properties especially optical (radiative emission). However, finding the optimum composition of more than one nanoparticles in host glass matrix for improving optical properties are needed to be study and analyse in order to understand the effect of co-embedding nanoparticles.

In order to observed the effect of co-embeddment nanoparticles towards the glass composition, the mol % of network modifier and dopant must be fixed. Any changes in optical, physical and structural will be due to the participation of sensitizer (single and co-embedded nanoparticles). From the literature it was found

that zinc oxide and magnesium oxide provide good physical properties and stability for different host glass system. It is also recorded that by using zinc oxide as modifier, positive sign are showed for optical application such as low laser threshold and strong visible emission spectrum. To conclude, mol % for ZnO, MgO and Dy₂O₃ were fixed for Series I and Series II to observed the effect of sensitizer (single and co-embedded nanoparticles) can be shown mainly on the optical (emission), physical and structural properties. Since achieving upconversion emission are possible for the Dy³⁺ ions, the participation of co-embedding nanoparticles can provide current work with significant knowledge on tellurite glass properties particularly.

From previous literature of Judd-Ofelt theory, it was found that the addition of nanoparticles towards host glass matrix doped with rare earth ions shows modification towards the optical properties especially the radiative emission. However, up to our knowledge the co-embedding nanoparticles towards Judd- Ofelt analysis were not recorded well. We believe that our current work can determine the influence of mediated surface plasmon resonance in optical properties for validation of Judd- Ofelt theoretical analysis. Not to mention that Judd- Ofelt intensity parameter of Dy³⁺ was shown to be predominant where Ω_2 are sensitive to the local symmetry and glass composition. In fact, radiative properties and stimulated emission cross section also can be observed as the participation of co-embedding nanoparticles take place in the tellurite glass composition.

Composition of glass containing lanthanide and metallic nanoparticles can be classified as photoluminescence (PL) intensity enhancement, stimulated emission cross section and quality factors. Intense local electromagnetic field generated from the localized surface plasmon resonance (LSPR) has proven to amplify the emission which can be explained in terms of charge density fluctuation at the nanoparticle (NP) surfaces. Other than that, the strengthen of Raman signal with addition of NPs indicate that plasmonic reflected surface enhanced Raman scattering (SERS)(Lee et al, 2007). When the electric dipole of nanoparticle is similar to the excitonic mode, the signal can be detected by molecular spectroscopy. Tuning of plasmonic band through shape, size and concentration of nanoparticles can be achieved in order to fulfil the application objective (Lee and El-Sayed, 2006). For example, in vivo

imaging and therapeutic, the SPR band must be in near-infrared region of the biological window, where the tissue transmissivity is at the highest (Jain *et al.*, 2006a).

Characterization of glass composition containing lanthanide with two type metallic nanoparticles are prepared in this project. Tellurite has been chosen for the host glass matrix due to its excellent properties, such as low multi phonon relaxation, large refractive index, great solubility towards rare earth ions (REI) and low melting point. Meanwhile, among REI candidate, we chose Dy³⁺ as the activator due to the multiple emissions near infrared region (0.9, 1.1, 1.3, 1.7 and 3 μm) which are valuable towards enhancing optical signal, telecommunication and visible light Upconversion (UC) solid state laser. From literature, it is revealed that Dy³⁺ displays two intense visible emission which are blue (482 nm) and yellow (574 nm) together with feeble red emission at (664 nm). By tuning the ratio between yellow and blue emission, we can generate white colour especially for LED according to Commission International de l'Eclairage (CIE) colour chromatic diagram. For nanoparticles candidate, we use silver (ANPs) and titanium (TNPs) nanoparticles for the co-embedding. Apart from the low production cost, silver nanoparticles also represent sharp features which particularly reflected in high refractive index (Martinsson *et al.*, 2013). Besides that, this sharp edged act as a light-harvesting nano-optical antennas converting visible light into a large localised electric field. Meanwhile TNPs are the pairs for co-embedded due to its excellent properties such as hydrophilic and photocatalytic characteristics. High surface volume ratio provides an increase in the surface area ratio at which photo induced reaction may occur, while light absorption rate enhance and surface photo induce carrier density (Lan *et al.*, 2013).

Table 1.0 shows the numerous researches on the development using mono and two types of nanoparticles in enhancing optical, structural and photocatalytic activity characteristics. The selection of noble metal especially silver and gold nanoparticles is due to the unusual catalytic, electric and optical properties, which also possess a strong SPR band in the UV and infrared region. In particular, the presence of metallic nanoparticles in enhancing luminescence has already been recorded by the previous research due to the energy transfer between NPs and the

lanthanide ions. Thus, co-embedding nanoparticles between silver and titanium can be one of the approach to enhance the optical and structural properties of tellurite glass.

Table 1.1 Previous literature review on using single and co-embedded nanoparticles

Year	Researcher	Scope of Research	Nanoparticles
2005	(Stranik <i>et al.</i> ,)	Plasmonic enhancement of fluorescence for sensor applications	Polystyrene bead (fluorophore Cy5)
2009	(Sepúlveda <i>et al.</i>)	LSPR based nanobiosensors	Gold, Au NPs
2007	(Lee <i>et al.</i>)	Origin of Surface Enhanced Raman Scattering	Silver nanowire, AgNWs
2009	(Tirtha and Basudeb)	Enhancing upconversion spectra of Nd ³⁺ doped antimony glass	Gold, Au NPs
2006	(Kuhn <i>et al.</i>)	Enhancing fluorescence spectra	Gold, Au NPs
2008	(Awazu <i>et al.</i>)	Enhancing photocatalytic behaviour	Silver, Ag NPs
2005	(Hirakawa and Kamat)	Charge separation and catalytic activity	Core shell Ag@TiO ₂
2007	(Cortie <i>et al.</i>)	Self-regulating plasmonic	Core shell Au@VO ₂

2012	(Angkaew and Limsuwan)	Optical characteristic behaviour	Core shell Ag@TiO ₂
2011	(Cortie and Mcdonagh)	Optical properties of hybrid and alloy plasmonic nanoparticles	Au and Ag NPs Hybrid/Alloy
2005	(Hubenthal <i>et al.</i>)	Tuning SPR band by manipulating thickness of core and shell	Au and Ag core shell/Alloy
2005	(Rodrı <i>et al.</i>)	Synthesis bimetallic and investigating on structural and optical properties	Au Ag NPs
1997	(Mizukoshi <i>et al.</i>)	Synthesized bimetallic nanoparticles	Au and Pd bimetallic
2009	(Seifert <i>et al.</i>)	Synthesized bimetallic nanoparticles using ion implantation	Ag and Au bimetallic
2005	(Aizpurua <i>et al.</i>)	Coupled metallic nanorod towards field enhanced spectroscopy on optical properties	Au nanorod pair
2012	(Rivera <i>et al.</i>)	Effect of photoluminescence towards plasmonic coupling	Ag NPs and Au NPs
2014	(Hooshmand <i>et al.</i>)	Hot spot investigation between two nanocube using Discrete Dipole Approximation (DDA) simulation	Au and Ag nanocubes
2016	(Oh <i>et al.</i>)	Hot spot engineering on nanopillar	ZnO, TiO ₂ and NiO

		array	
2010	(Camargo <i>et al.</i>)	Measuring SERS enhancement from hot spot of different structure nanocubes	Silver nanocubes

1.2 Problem Statement

In recent times, different rare earth ions (REIs)-doped various binary and ternary oxide glasses and glass-ceramics (tellurite, phosphate, borate, silicate, etc) have widely been studied due to their potential for diverse applications in the ultraviolet to infrared region such as the optical amplifier, eye-safe solid state lasers, fibers and other optical devices. Several optical transitions in the 4f level of the REIs make them very useful when doped inside crystals, glasses and glass-ceramics. However, the low absorption and emission of cross-section of the REIs needs improvement for practical applications. To improve the optical absorption and emission characteristics of these REIs-doped glasses different researchers have embedded or incorporated various metal nanoparticles (for example Ag, Au, Cu, TiO₂, Fe, Mn, etc.) into these hosts. It is known that the surface plasmon resonance (SPR) of different metal nanoparticles can enhance the optical properties of the REIs when doped inside the glasses. Most of the earlier researches used one type of nanoparticles to improve the optical properties of different REIs mostly in phosphate and tellurite glasses. Among all the REIs (Er³⁺, Sm³⁺, Nd³⁺, Dy³⁺, Eu³⁺, etc), due to intense yellow and blue emission shown by Dy³⁺ it became interesting as activator in tellurite and phosphate based glasses. So far, not many detail studies have been performed on the two types (silver and titanium) of nanoparticles co-sensitization or co-embedding on the optical properties improvement of Dy³⁺-doped zinc-magnesium tellurite glasses. Thus, to improve the optical properties of the Dy³⁺-doped zinc-magnesium tellurite glasses co-embedded with Ag and TiO₂ nanoparticles, careful preparation of such glasses and composition optimization through diverse characterizations are still necessary. Detail characterization of

prepared glasses for their structural, physical and other properties are also required to select the optimum glass sample that produce the best optical properties. Therefore, one of the objectives this study is to optimize the composition of the dysprosium ions doped zinc-magnesium tellurite glasses co-embedded with silver/titanium nanoparticles via preparation and detailed characterizations.

Previous studies on different REIs-doped tellurite glasses with one type of nanoparticles embedment showed that the structural, physical, and optical properties of the glasses can be improve via nanoparticles sensitization. The observed improvements in the structural, physical and optical properties of such glass system were attributed to the localised surface plasmon resonance (LSPR)-mediated strong local field effect. It is believed that the strong local electric field and the energy transfer between nanoparticles and REIs play a significant role for the modification of overall properties of the glass system. However, no investigations have been made so far to evaluate the impact of the Ag/TiO₂ nanoparticles co-embedment on the structural, physical and optical properties (especially up-conversion and down-conversion emission traits) of the dysprosium ions-doped zinc-magnesium tellurite glass system. It is therefore important to determine the effect of Ag/TiO₂ nanoparticles co-embedment on the network structures and bonding, absorption and emission properties, and physical characteristics of the dysprosium-doped zinc-magnesium tellurite glasses. The detail understanding and knowledge of the structural of physical properties are essential to get the best optical properties and further validation of the experimental optical data through Judd-Ofelt theoretical calculations. Different structural parameters are also required for the Judd-Ofelt evaluation to support the experimentally measured optical properties. Detail literature review revealed that the emission mechanisms from the dysprosium ions with nanoparticles co-embedment into the magnesium-tellurite glasses are not fully understood. In this view, structural and physical properties are useful to predict the effects of nanoparticles co-embedment on the improved absorption and emission traits of the dysprosium-doped zinc-magnesium tellurite glasses.

Various studies revealed that the presence of the nanoparticles in the vicinity of the REIs inside glass matrix can modify the symmetry/asymmetry of the network bonding structure of the host wherein the number of non-bridging and bridging

oxygen bonds gets altered. In addition, due to the presence of the strong signal with respect to the hot spot junction between silver and titanium nanoparticles, it is possible to apply the surface enhance Raman scattering (SERS) inside the prepared zinc-magnesium tellurite glasses with co-embedded nanoparticles. Therefore, the mechanism behind the optical properties improvement due to the incorporation of two types of nanoparticles is required for further application of these glasses in different photonic devices. To the best of our knowledge, the emission from the dysprosium ions in zinc-magnesium glasses with co-embeded nanoparticles are not well reported. The correlation between the LSPR and the emission especially the upconversion (UC) is far from being understood. To achieve this goal, three series of these glasses were prepared using the melt quenching technique and characterized. In order confirm the improvement in the absorption and emission properties of the optimum glass system useful for lasing, the Judd-Oflet intensity parameters and radiative properties were calculated. It is believed that this work might provide some new knowledge and information on the improved optical properties of the dysprosium-doped zinc-magnesium tellurite glasses containing single (Ag) and co-embedded (Ag/TiO₂) nanoparticles. The obtained results indicated that the modification in the fluorescence and structural properties of the dysprosium doped tellurite glasses due to the embedment of single (Ag) and two types (Ag/TiO₂) of nanoparticles. It provided some significant data especially on the radiative properties useful for the solid state laser and optoelectronic applications. In addition, this research was crucial in understanding the physics behind the nanoparticles co-embedding, particularly regarding the co-embedded nanoparticles SPR-mediated emission properties enhancement of the Dy³⁺-doped zinc-magnesium tellurite glasses.

1.3 Research Objectives

The main aim of this study was to improve the optical absorption and PL emission (up-conversion and down-conversion) properties of the Dy³⁺-doped zinc-magnesium tellurite glass by embedding single type of nanoparticles (Ag) and two

types of nanoparticles (Ag/ TiO₂) via the preparation using melt quenching technique, composition optimization, characterizations and validation using Judd-Ofelt calculation. Based on above problem statement, the following objectives are proposed:

- I. To optimize the composition of a series Dy³⁺-doped zinc-magnesium tellurite glass included with single nanoparticles (Ag) and co-embedded nanoparticles (Ag/TiO₂) for improving the optical properties.
- II. To determine the effect of silver nanoparticles (Ag) and co-embedded nanoparticles (Ag/TiO₂) on the physical and structural properties of the obtained glass system for getting the optimum glass sample.
- III. To determine the influence of the embedded (Ag) and co-embedded (Ag/TiO₂) nanoparticles -mediated surface plasmon resonance field on the optical properties of the Dy³⁺-doped zinc-magnesium tellurite glasses for the validation using Judd-Ofelt theoretical analysis.

1.4 Scope of Study

The prepared samples for this study involved two series of glasses which are (69.5-x)TeO₂-20ZnO-10MgO-0.5Dy-(x)AgCl, where (x= 0.2, 0.4, 0.6, 0.8 and 1.0) for series I. Meanwhile for series II will be (69.3-y)TeO₂-20ZnO-10MgO-0.5Dy-0.2AgCl -(y)TiO₂, where (y= 0.1, 0.2, 0.3, 0.4, and 0.5 and 0.6). Both series were sensitized using the conventional melt quenching technique. The usage of modifier for both compositions remain the same. The reason for the constant modifier is to observe that various nanoparticles affect the properties of prepared sample, rather than changing the network modifier. The selection of zinc and magnesium oxide as the modifier is due to the extensive studies regarding optical application especially ZnO-TeO₂ for various tellurite glass composition. The establishment of amorphous nature were proved by the diffraction pattern of XRD. Optical properties including absorption and emission radiation were analysed by

using absorption spectroscopy, UV-VIS-NIR and photoluminescence spectroscopy. From the absorption band, we can calculate the Judd-Ofelt theory therefore determine the parameter and trend for each samples. Meanwhile, radiative emissions from the prepared samples can be evaluated to determine stimulated emission cross section until radiative decay.

Furthermore, structural analysis were done by using Raman and FTIR spectroscopy which demonstrate the bond and vibration intensity towards the influence of single and co-embedded nanoparticles to the host glass matrix. The existence and size distribution of both nanoparticles were also verified using HRTEM imaging technique. Elemental analysis regarding glass composition was done using EDX analysis and EDX mapping. Additionally, physical properties of the prepared samples were calculated by determining the density of each series. Archimedes method was applied to calculate the density, while Brewster angle system was implemented to determine the refractive index for each sample. By determine the refractive index from each glass composition, molar refraction and polarizability can be obtained and compared with previous literature results.

1.5 Significant of Study

Co-embedded of nanoparticles can be one of the methods in obtaining high emission intensity, particularly regarding lanthanide ions. Enhancing emission especially upconversion (UC) can be utilised through the small distant between silver (ANPs) and titanium nanoparticles (TNPs), which boost up the SPR signal. Current data shows that the enhancement from the prepared samples can be a good candidate for optoelectronic devices and optical fibre amplifier. In addition, improvement of UC intensity towards co-doped shows that the prepared sample can be used as a solid state laser by using IR diode laser (low energy) to produce high intense luminescence. Apart from that, upconversion also recently caught the attention in bio imaging and biosensors. The feature from upconversion that attracted bioimaging and biosensors is due to the minimum photo damage to the biomolecules, and enhancement of signal-to-noise ratio, thus improve the result of the detection. This

feature of upconversion is contrary compared to down-conversion, which has the tendency to be phototoxic and damaging the samples. In fact, upconversion also proved to be highly sensitive towards biological tissue, resulting in a large penetration depth of samples. Furthermore, upconversion can also be utilised in ink security application for example, with codes embedded into microparticles towards bank note. By illuminating upconversion emission, the embedded code will be revealing the counterfeit/authentic tags.

In addition, co-embedding nanoparticles towards dysprosium ion also shows an enhancement towards vibrational analysis. This observe phenomenon might favour many applications particularly surface enhance Raman scattering (SERS). The strong improvement towards luminescence spectra suggests that the local field enhancements increased due to the co-embedding of nanoparticles towards dysprosium ion. The inelastic scattering of photon provides a crucial information at molecule level, thus by enhancing Raman signal, it enables the detection of biomolecules at low concentration and detection at single molecule might be achieved. By understanding the vibrational bond of co-embedded nanoparticles in host glass matrix, we believe that the significant finding will lead to the extent of knowledge where application involving glassy state will be well developed. As a matter of fact, co-embedding nanoparticles can be established as one of the approach to enhance the properties for related application. Current result of the co-embedding of nanoparticles towards dysprosium ion will possibly provide useful information regarding the enhancement mechanism of SERS especially on electromagnetic effect.

1.6 Thesis Outline

In this thesis, there are five chapters presenting the optical and structural characterizations of co-embedded silver (ANPs) and titanium (TNPs) nanoparticles into doped dysprosium zinc-magnesium tellurite glass. This work provides an alternative way to enhance the glass properties by using the melt quenching

technique, thus contributes to the extent of knowledge regarding the correlation between LSPR effects and the glass composition.

Chapter 1 briefly explains the introduction to the motivation of this research. The research gap in this current project can be explained through problem statement and research objectives. The benefits and outline of this research work can also be found in this chapter.

Chapter 2 compresses literature review regarding host glass, network modifier, lanthanide ions, nanoparticles, optical and structural characterization properties. Detailed mechanisms regarding optical and structural properties related to co-embedded are explained briefly. This chapter includes a broad overview.

Chapter 3 explains the methodology in preparing glass sample using the melt quenching technique. In fact, analytical techniques regarding optical, structural and physical properties were highlighted. The theoretical and schematic diagram for all analytical technique were presented.

Chapter 4 presents the results of all analytical techniques from UV-VIS-NIR, PL, XRD, Raman, FTIR, HRTEM and EDX. For optical characterization, the Judd-Ofelt calculations and parameters have been shown up to a radiative lifetime and stimulated emission cross-section. For radiative emission, UC and DC were recorded for all prepared sample and optimized indicators were established. Co-embedded between ANPs and TNPs also has an impact on bonding and vibration intensity. The presence of both nanoparticles were confirmed by imaging and elemental analysis. A full discussion of each analysis was prepared.

Chapter 5 concludes the entire results for all analyses to ensure objectives are achieved. There are some topics related to the current project that are not included. However, future recommendations for further understanding regarding co-embedded nanoparticles were listed for future research. Certain important data such as calculations for sample composition were added in appendices. A list of publications and conferences attended were also attached for references.

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Appendix I

List of Publications

Journal with impact factors

1. **Mohd Saidi, M. S. A.**, Ghoshal, S. K, Arifin. R, Roslan. M. K, Muhammad. R, Shamsuri. W. N. W, M. Abdullah, Shaharin. M. S. (2018). Spectroscopic properties of Dy³⁺ doped tellurite glass with Ag/TiO₂ nanoparticles inclusion : Judd-Ofelt analysis. *Journal of Alloys and Compounds*, 754, 171–183. <http://doi.org/10.1016/j.jallcom.2018.04.280>. (Q1, IF:4.12)
2. **Mohd Saidi, M. S. A.**, Ghoshal, S. K., Hamzah, K., Arifin, R., Omar, M. F., Roslan, M. K., & Sazali, E. S. (2018). Visible light emission from Dy³⁺doped tellurite glass: Role of silver and titania nanoparticles co-embedment. *Journal of Non-Crystalline Solids*, 502(July), 198–209. <http://doi.org/10.1016/j.jnoncrysol.2018.09.012>. (Q1, IF:2.488)
3. **Mohd Saidi., M.S.A.** Ghoshal. S.K, R. Arifin, M.K. Roslan. (2017). Absorption and raman spectra of Dy³⁺ doped tellurite glass: Combined effects of silver and titanium nanoparticles. *Solid state phenomenon*, 268 (111–116). <http://doi.org/10.4028/www.scientific.net/ssp.268.111>. (Q4, IF:0.16)

Indexed Journal

1. **Mohd Saidi., Mohd Syamsul Affendy**, S.K.Ghoshal, R.A. and A.Awang., 2016. Improved spectral features of silver nanoparticles sensitized samarium doped zinc-sodium-tellurite glass. *Solid State Science and Technology*, 24(1), pp.163–170. (Indexed by SCOPUS)

Appendix J

Calculation of density and uncertainty

Example Series II TZMDyAg0.2TiO₂0.4

$$\text{Density} = \rho_{\text{glass}} = \rho_L \left(\frac{W_{\text{air}}}{W_{\text{air}} - W_{\text{liquid}}} \right)$$

- $\rho_{\text{glass}} = 0.999 \left(\frac{4.2541}{4.2541 - 3.5768} \right)$
- $\rho_{\text{glass}} = 6.2747$

$$\text{Uncertainty} = \Delta\rho_{\text{glass}} = \left(\frac{\Delta\rho_L}{\rho_L} + \frac{\Delta W_{\text{air}}}{W_{\text{air}}} + \frac{\Delta(W_{\text{air}} - W_{\text{liquid}})}{W_{\text{air}} - W_{\text{liquid}}} \right) \times \rho_{\text{glass}}$$

- *Since the ρ_L = standard value, $\Delta\rho_L = 0$*
- $\Delta\rho_{\text{glass}} = \left(\frac{0.0003}{4.541} + \frac{\Delta(0.0003 - 0.0003)}{4.2541 - 3.5768} \right) \times 6.2747$
- $\Delta\rho_L = 0.0004$

Therefore for sample Series II TZMDyAg0.2Ti0.4 the density is $(6.2747 \pm 0.0004) \text{ gcm}^{-3}$