# SPECTROSCOPIC STUDIES OF SAMARIUM-DOPED SODIUM TELLURITE GLASS CONTAINING SILVER NANOPARTICLES

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***Dedicated, with love,***

***To my dear parents***

***Hayati Drahman & Hj. Abdullah Dris***

***To my beloved siblings***

***Nur Aziemah, Qurratul-Ain, Qisthina Azmina and Ar'syil Ad-deen***

***And..***

***To my trustworthy best friends***

***Thank you for everything.***

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# ABSTRACT

Samples of samarium doped tellurite glass containing Ag nanoparticles with molar composition (74.5 – $x$) $TeO_2$ + 25 $Na_2O$ + 0.5 $Sm_2O_3$ + $x$ Ag where, 0.5 mol% $\leq x \leq$ 2.0 mol% have been successfully made by melt quenching technique. The amorphous nature of the glasses has been confirmed using X-ray Diffraction and the presence of Ag nanoparticles in the glass samples is verified using Transmission Electron Microscopy (TEM). It is found that the nanoparticles are almost spherical in shape with an average diameter of 31.2 nm. In this study, the effects of adding various concentrations of Ag nanoparticles into the glass samples to the thermal, structural, optical and emission properties of the glass are investigated. The thermal parameters, such as the glass transition temperature $T_g$, crystallization temperature $T_c$, melting temperature $T_m$ and thermal stability ($T_c$ -$T_g$) of the glass have been investigated using Differential Thermal Analysis (DTA) technique. It is found that the introduction of Ag nanoparticles in the glass network has increased the network rigidity and therefore increases the glass thermal stability. The vibrational spectrum of the glass has been studied using Fourier Transform Infrared (FTIR) spectroscopy. The spectra exhibit four absorption bands around 609.69 $cm^{-1}$, 736.76 $cm^{-1}$, 1636.0 $cm^{-1}$ and 3434.56 $cm^{-1}$. The presence of $Sm^{3+}$ ions and Ag nanoparticles however did not give any significant change to the infrared spectra. From the UV-Vis-NIR spectra, eight absorption peaks have been observed around 398 nm, 471 nm, 947 nm, 1085 nm, 1238 nm, 1386 nm, 1492 nm and 1546 nm, which correspond to the transitions from ground state of $^4I_{15/2}$ to the excited state of $^6F_{1/2}$, $^6F_{3/2}$, $^6F_{5/2}$, $^6F_{7/2}$, $^6F_{9/2}$, $^6F_{11/2}$, $^4I_{3/2}$, and $^6P_{5/2}$ respectively. The values of the optical band gap $E_{opt}$ of the glass systems lie between 2.806 eV to 2.878 eV and these values are slightly shifted towards higher energies as the Ag nanoparticles content is increased; probably due to the increase of non-bridging oxygen in the glass samples. The Urbach energy $E_{tail}$ is found to lie between 0.230 eV to 0.268 eV. The effect of embedding nanoparticles to the luminescence properties of the glass has been investigated through photoluminescence measurements. The photoluminescence spectra at 406 nm excitation wavelength revealed four emission bands at 562 nm, 599 nm, 645 nm and 705 nm corresponding to 4f-4f transitions; $^4G_{5/2}\rightarrow{}^6H_{5/2}$, $^4G_{5/2}\rightarrow{}^6H_{7/2}$, $^4G_{5/2}\rightarrow{}^6H_{9/2}$ and $^4G_{5/2}\rightarrow{}^6H_{11/2}$ respectively. Incorporation of Ag nanoparticles enhances the intensity of these emission bands and the enhancement increases with the increase in the content of Ag nanoparticles. The luminescence enhancement is due to energy transfer from silver plasmon band to $Sm^{3+}$ ion.

# ABSTRAK

Sampel kaca tellurit berdop samarium yang mengandungi nanopartikel Ag dengan komposisi molar (74.5 – *x*) $TeO_2$ + 25 $Na_2O$ + 0.5 $Sm_2O_3$ + *x* Ag untuk, 0.5 mol% $\leq x \leq$ 2.0 mol% telah berjaya dihasilkan menggunakan teknik pelindapan leburan. Sifat amorfus kaca telah dibuktikan dengan menggunakan Pembelauan Sinar-X dan kehadiran nanopartikel Ag dalam sampel kaca disahkan menggunakan Mikroskop Transmisi Elektron. Didapati bahawa nanopartikel tersebut berbentuk hampir sfera dengan diameter purata 31.2 nm. Dalam kajian ini, kesan penambahan nanopartikel Ag dengan kepekatan berbeza kepada sampel kaca terhadap sifat terma, struktur, optik dan pancaran kaca tersebut akan dikaji. Parameter terma seperti suhu transisi kaca $T_g$ , suhu penghabluran $T_c$ , suhu lebur $T_m$ dan kestabilan terma ($T_c$-$T_g$) kaca telah ditentukan dengan menggunakan teknik Analisis Terma Pembezaan. Didapati bahawa kehadiran nanopartikel Ag dalam rangkaian kaca meningkatkan ketegaran rangkaian kaca dan seterusnya meningkatkan kestabilan terma kaca tersebut. Spektrum getaran kaca telah dikaji menggunakan spektroskopi Inframerah Transformasi Fourier. Spektrum sampel tersebut mempamerkan empat jalur penyerapan sekitar 609.69 $cm^{-1}$, 736.76 $cm^{-1}$, 1636.0 $cm^{-1}$ dan 3434.56 $cm^{-1}$. Walaubagaimanapun, kehadiran ion $Sm^{3+}$ dan nanopartikel Ag tidak memberi apa-apa perubahan ketara kepada spektrum inframerah. Daripada spektrum UV-Vis-NIR, lapan puncak penyerapan telah dikenalpasti sekitar 398 nm, 471 nm, 947 nm, 1085 nm, 1238 nm, 1386 nm, 1492 nm dan 1546 nm yang masing-masing merujuk kepada transisi daripada keadaan asas, $^4I_{15/2}$ kepada keadaan teruja $^6F_{1/2}$, $^6F_{3/2}$, $^6F_{5/2}$, $^6F_{7/2}$, $^6F_{9/2}$, $^6F_{11/2}$, $^4I_{3/2}$, dan $^6P_{5/2}$. Nilai jurang jalur optik kaca $E_{opt}$ bagi sistem kaca terletak antara 2.806 eV hingga 2.878 eV dan nilai ini teranjak sedikit ke arah tenaga yang lebih tinggi apabila kandungan nanopartikel Ag bertambah; berkemungkinan disebabkan oleh peningkatan oksigen tidak bertautan dalam sampel kaca. Tenaga Urbach $E_{tail}$ didapati terletak antara 0.230 eV hingga 0.268 eV. Kesan penambahan nanopartikel ke atas sifat luminesen kaca telah dikaji menerusi pengukuran spektrum fotoluminesen. Pada panjang gelombang pengujaan 406 nm, terdapat empat jalur pancaran dalam spektrum fotoluminesen iaitu di sekitar 562 nm, 599 nm, 645 nm dan 705 nm yang merujuk kepada transisi 4f-4f; yang mana masing-masing adalah $^4G_{5/2}\rightarrow{}^6H_{5/2}$, $^4G_{5/2}\rightarrow{}^6H_{7/2}$, $^4G_{5/2}\rightarrow{}^6H_{9/2}$ dan $^4G_{5/2}\rightarrow{}^6H_{11/2}$. Penambahan nanopartikel Ag meningkatkan keamatan jalur pancaran dan darjah keamatannya. Peningkatan keamatan pancaran ini adalah disebabkan oleh perpindahan tenaga daripada jalur plasmon perak kepada ion $Sm^{3+}$.

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| CCD | - | Charge-Coupled Device |
| DL | - | Deuterium Lamp |
| DTA | - | Differential Thermal Analysis |
| FRET | - | Förster Resonance Energy Transfer |
| FTIR | - | Fourier Transform Infrared |
| HL | - | Halogen Lamp |
| IR | - | Infrared |
| LSPR | - | Localized Surface Plasmon Resonance |
| NPs | - | Nanoparticles |
| PL | - | Photoluminescence |
| RE | - | Rare-Earth |
| SPR | - | Surface Plasmon Resonance |
| TEM | - | Transmission Electron Microscopy |
| UV-Vis-NIR | - | Ultra-Violate Visible Near Infrared |
| XRD | - | X-Ray Diffraction |
| tbp | - | Tetrahedral bipyramid |
| NBO | - | Non-bridging Oxygen |
| BO | - | Bridging Oxygen |

## LIST OF SYMBOLS

| | | |
|---|---|---|
| $A$ | - | absorbance |
| $\alpha$ | - | Absorption coefficient |
| $c$ | - | sample concentration |
| $d$ | - | sample thickness |
| $E_{opt}$ | - | optical energy band gap |
| $E_{tail}$ | - | Urbach energy |
| $\hbar$ | - | Planck constant |
| $I$ | - | transmitted light intensity |
| $I_o$ | - | incident light intensity |
| $\lambda$ | - | wavelength |
| $n$ | - | refractive index |
| $\omega$ | - | frequency’ |
| $\rho$ | - | density |
| $T_c$ | - | crystallization temperature |
| $T_g$ | - | glass transition temperature |
| $T_m$ | - | melting temperature |
| $\Delta T$ | - | thermal stability |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

Today, materials play a decisive role in all technological developments. Whatever his specialty, the engineer cannot build new objects without taking into account the properties of the materials used. It is generally the behaviour of materials which limits the performance of machines and equipments (Mercier *et al.*, 2002). Principal materials such as metals, organic polymers, ceramics, crystals and glasses are of great research interest because of their importance in various applications.

Glass manufacturing is believed to have started long time ago but only found documented in the year 1673 – 1676 by Ravenscroft. However, the glass industry faced a big problem in that era because fundamental knowledge on its properties such as chemical durability is almost none (Sahar, 1998). Because of its noble properties and importance, continuous and vast researches on glass were carried out to produce a high quality glass to meet the demand in various industries; from the manufacture of window glass to the application in photonic and optoelectronic devices.

From the mid of this century, inorganic glasses doped with rare-earth (RE) metal ions have become an important solid state laser material, specially used in high power lasers. Hence, study of spectroscopic properties of RE metal ions in glasses has been increasing (Fuxi, 1992). Among all of the oxide glass formers, tellurite-based glasses are of scientific and technological interest because of their high refractive index ($n > 1.80$), high density ($\rho > 4.5$ g/cm$^3$), wide transmission region (0.35-5 μm), good glass stability and corrosion resistance, a relatively low phonon energy, high rare earth solubility, as well as their good ultraviolet and infrared transmissions (Hussain *et al.*, 2008; Wang *et al.*, 1994).

Since various properties of glass can be varied by modifying their compositions, tellurite glass containing RE ions and metallic nanoparticles (NPs) are attracting large interest recently because the presence of NPs may lead to intensification of luminescence and enhancement of third-order non-linear properties (Almeida *et al.*, 2007; Kassab *et al.*, 2009). Thus, in this work the non-linear optical properties of RE-doped metal nanoparticles-embedded tellurite glass and the phenomena responsible of the modifications will be studied.

## 1.2 Statement of Problem

Despite of the intensive research on rare-earth tellurite glasses, the understanding on unusual optical and electronic properties is still lacking. The absorption cross-section of most of the rare earth ions is small and need ways to enhance it for applications (Singh *et al.*, 2010). Since the use of metal nanoparticles has been found to enhance the absorption cross section of rare earth ions, many recent studies have been devoted to the contribution of NPs to the material's luminescence efficiency and the enhanced emission. However, in terms of material selection, samarium has gained less attention compared to other rare earth ions such as $Er^{3+}$, $Nd^{3+}$, $Pr^{3+}$ and $Eu^{3+}$. Most of the research for laser applications focuses more on $Er^{3+}$-doped glasses. A few studies have proposed that samarium shows better optical absorption in borate glassess (Lin *et al.*, 2005; Agarwal *et al.*, 2009), a better combination of lifetime-emission cross section product in fluorine-modified silicate glass hosts (Huang *et al.*, 2008) and an extraordinary infrared-to-red upconversion luminescence in antimony glasses (Som *et al.*, 2008) than in tellurite glass. However, no study has yet measure these properties in samarium-doped tellurite glass with incorporation of silver nanoparticles. This study would be very important to investigate the enhanced luminescence due to the incorporation of nanoparticles into glass and to understand the phenomena responsible for the enhancement.

## 1.3 Choice of Glass System

In order to achieve the objectives of this study, a series of glass sample has been prepared with glass former Tellurium dioxide, $TeO_2$ with one modifier which is Sodium Oxide, $Na_2O$. The glass system is doped with rare earth element Samarium Oxide, $Sm_2O_3$ and embedded with metallic NP which is silver, Ag.

The composition of glass sample series prepared is:

1. $(74.5 - x)$ $TeO_2$ + 25 $Na_2O$ + 0.5 $Sm_2O_3$ + $x$ Ag where $x$ = (0.0, 0.5, 1.0, 1.5, 2.0) mol%.

## 1.4 Research Objectives

The objectives of this study are:

1. To determine the effects of adding Ag NPs into the $Sm^{3+}$ glass samples to the thermal and structural properties of the glass.
2. To characterize the optical absorption of the glass.
3. To identify and determine the effects of adding various concentration of Ag NPs to the emission properties of the $Sm^{3+}$ ions.

## 1.5 Scope of Study

In order to achieve the objectives above, this research has been carried out within the stated scope as follows:

1. The preparation of glass samples with composition $(74.5 - x)$ $TeO_2$ + 25 $Na_2O$ + 0.5 $Sm_2O_3$ + $x$ Ag where, $x$ = (0.0, 0.5, 1.0, 1.5, 2.0) mol% by melt-quenching method.
2. The determination of the presence of NPs.
3. The determination of thermal parameters.
4. The characterization of optical absorption.

5. The characterization the vibrational spectrum
6. The identification and determination of emission behaviour of the glass.

## 1.6 Significance of Study

The phenomena related to the increase of the luminescence and the enhancement of non-linear optical properties enables several applications such as photonic devices, optical amplifiers, coloured displays, compact fibre laser for medical application, optical limiters and etc. (Kassab *et al.*, 1994). For devices that use rare earth glass in its fabrication, deep understanding on the fundamental properties of the glass will help during material selection and device improvement. Furthermore, the findings of this research will give some significant contribution to other researchers who are working on rare earth-doped glasses development and its applications. Thus, detailed study of the optical properties and luminescence enhancement of samarium-doped tellurite glass containing silver NPs becomes essential to understand the phenomena and thus contributes to further material development.

## 1.7 Thesis Outline

This thesis describes the preparation and characterization of samarium-doped tellurite glass prepared by melt quenching technique. This thesis is divided into five chapters. Chapter 1 is the introduction of the study, which explains why this research study is being done and its importance, and the justification of material selection. Chapter 2 will describe some background knowledge on glass and the lanthanides, review on previous works done on incorporation of metallic NPs to glass matrix, and basic principles of spectroscopic techniques used in this study. Details of the sample preparation, design of the experimental and the measurement techniques employed are outlined in Chapter 3. In Chapter 4, all the experimental results and discussion will be presented. Finally, Chapter 5 presents major conclusions of the research and future outlook of the study.

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