EFFECT OF NATURAL FERRITE OXIDE NANOPARTICLES ON STRUCTURAL AND MAGNETO OPTIC PROPERTIES OF ERBIUM DOPED TELLURITE GLASS

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DEDICATION

To my beloved father (Alm. H. Abu Bukhori) and my mother (Almh. Icah Napisah)
To my beloved husband Mohd Fitri Bin Bachok
To my brothers and sisters (*Alm.* Denny, *Alm. Robby, Fitri, Alm. Faiz and Tita) for their enduring love, motivation and support*

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ABSTRACT

Two series of erbium doped magnesium tellurite glasses embedded with Fe_3O_4 nanoparticles (NPs) with composition (89-x) TeO_2 - 10MgO - $1Er_2O_3$ - (x) Fe_3O_4, where (x = 0, 0.2, 0.4, 0.6 and 0.8 mol%) and (89.6-y) TeO_2-10MgO-(y) Er_2O_3-$0.4Fe_3O_4$, where (y = 0.2, 0.4, 0.6, 0.8 and 1.0 mol%) were prepared using melt quenching technique. Thorough characterizations of these glasses were made using X-ray diffraction (XRD), differential thermal analyzer (DTA), ultraviolet-visible-near-infrared (UV-Vis-NIR), Fourier transform infrared (FTIR), Raman spectroscopy, photoluminescence (PL) spectroscopy, energy dispersive X-ray (EDX), high resolution transmission electron microscopy (HRTEM), vibrating sample magnetometer (VSM) and electron spin resonance (ESR) spectroscopy. The presence of a broad hump in the X-ray diffraction (XRD) pattern confirms the amorphous nature of glass. HRTEM images verified the existence of Fe NPs with average diameter of 4.8 nm (TMEF3 sample) and 4.5 nm (TMFE3 sample) corresponding to d_{311} spacing. The glass density and molar volume were found to be in the range of 4.03-5.27 gcm^{-3} and 37.20-28.54 $cm^3 mol^{-1}$, respectively. It was also found that as the amount of Fe_3O_4 NPs was increased, the glass stability increased. DTA analysis demonstrated an increase in the glass transition temperature from 321 ^{o}C to 363 ^{o}C with the increase of Fe_3O_4 NPs. The UV-Vis-NIR absorption spectra revealed seven absorption bands centered at 452 nm, 522 nm, 571 nm, 656 nm, 795 nm, 978 nm and 1528 nm which are assigned to the transition from ground state $^4I_{15/2}$ to the various excited levels $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4I_{9/2}$, $^4I_{11/2}$, $^4I_{13/2}$, respectively. Surface plasmon resonance (SPR) peaks of Fe_3O_4 NPs were observed at 408 nm. The FTIR and Raman spectra revealed modification in network structures which is evident from wavenumber of [TeO_4] and [TeO_3] structural units located around 600 cm^{-1} and 700 cm^{-1}, respectively. The luminescence of Er^{3+} ion under 375 nm excitation revealed four peaks centered at 420 nm, 460 nm, 490 nm and 516 nm which correspond to light green ($^4F_{3/2} \rightarrow {}^4I_{15/2}$), light green ($^4F_{7/2} \rightarrow {}^4I_{15/2}$), green ($^2H_{11/2} \rightarrow {}^4I_{15/2}$) and red ($^4S_{3/2} \rightarrow {}^4I_{15/2}$) emissions, respectively. The Judd-Ofelt intensity parameter, spontaneous emission probabilities, radiative lifetime and branching ratios of all transitions were calculated based on Judd-Ofelt theory. The glass magnetization in magnetic field up to 10 *kOe* at room temperature was found to be in the range of 0.012 – 0.052 emu g^{-1}. The *g*-factor values of 1.99 to 3.06 indicate that the glass samples are paramagnetic. The result of the present work revealed that the investigated glass can be a potential material for magneto-optic devices and solid-state lasers.

ABSTRAK

Dua siri kaca magnesium tellurite dengan Fe_3O_4 nanopartikel (NPs) dengan komposisi (89-x)TeO_2-10MgO-1Er_2O_3-(x)Fe_3O_4, dengan (x = 0, 0.2, 0.4, 0.6 dan 0.8 mol%) dan (89.6-y)TeO_2-10MgO-(y)Er_2O_3-0.4Fe_3O_4, dengan (y = 0.2, 0.4, 0.6, 0.8 dan 1.0 mol%) disediakan menggunakan teknik lindap-kejut leburan. Pencirian kaca dilakukan melalui pembelauan sinar-X (XRD), analisis pembezaan terma (DTA), spektroskopi ultra lembayung-boleh nampak inframerah hampir (UV-Vis-NIR), spektroskopi transformasi Fourier inframerah (FTIR), spektroskopi Raman, spektroskopi fotoluminesen (PL), spektroskopi sebaran tenaga sinar-X (EDX), mikroskopi elektron penghantaran resolusi tinggi (HRTEM), magnetometer getaran sampel (VSM) dan spektroskopi resonans spin elektron (ESR). Kewujudan puncak yang lebar pada corak pembelauan sinar-X (XRD) membuktikan sifat amorfus kaca. Imej HRTEM pula membuktikan kewujudan zarah nano Fe dengan purata diameter 4.8 nm (sampel TMEF3) dan 4.5 nm (sample TMFE3) yang sepadan dengan jarak d_{311}. Ketumpatan kaca dan isipadu molar didapati masing-masing berada di dalam julat 4.03 - 5.27 g cm^{-3} dan 37.20 - 28.54 cm^3 mol^{-1}. Turut ditemui bahawa apabila jumlah NPs Fe_3O_4 meningkat, kestabilan kaca turut meningkat. Analisis DTA menunjukkan peningkatan suhu peralihan kaca daripada 321 ^oC hingga 363 ^oC dengan bertambahnya kandungan NPs Fe_3O_4. Spektrum UV-Vis-NIR menunjukkan tujuh jalur serapan yang berpusat di 452 nm, 522 nm, 571 nm, 656 nm, 795 nm, 978 nm dan 1528 nm yang mewakili transisi daripada keadaan asas $^4I_{15/2}$ kepada pelbagai keadaan teruja $^4F_{7/2}$, $^2H_{11/2}$, $^4S_{3/2}$, $^4F_{9/2}$, $^4I_{9/2}$, $^4I_{11/2}$, $^4I_{13/2}$. Puncak resonans plasmon permukaan (SPR) bagi NPs Fe_3O_4 dicerap pada 408 nm. Spektrum transformasi Fourier inframerah (FTIR) dan spektrum Raman menunjukkan pengubahsuaian dalam struktur rangkaian dan dibuktikan melalui nombor gelombang bagi struktur unit [TeO_4] dan [TeO_3] yang masing-masing terletak di sekitar 600 cm^{-1} dan 700 cm^{-1}. Luminesens bagi ion Er^{3+} di bawah pengujaan 375 nm menunjukkan empat puncak berpusat di 420 nm, 460 nm, 490 nm dan 546 nm yang masing-masing berpadanan dengan pancaran warna hijau muda ($^4F_{3/2} \rightarrow {}^4I_{15/2}$), hijau muda ($^4F_{7/2} \rightarrow {}^4I_{15/2}$), hijau ($^2H_{11/2} \rightarrow {}^4I_{15/2}$) dan merah ($^4S_{3/2} \rightarrow {}^4I_{15/2}$). Pembolehubah keamatan Judd-Ofelt, kebarangkalian pancaran spontan, jangka hayat radiatif dan nisbah cabangan bagi semua transisi telah dikira berdasarkan teori Judd-Ofelt. Kemagnetan kaca dalam medan magnet sehingga 10 *kOe* di bawah suhu bilik didapati dalam julat 0.012 – 0.052 emu g^{-1}. Nilai faktor-*g* antara 1.99 hingga 3.06 menandakan bahawa sampel kaca adalah bersifat paramagnetik. Hasil dari kajian ini membuktikan bahawa kaca yang diselidiki merupakan bahan berpotensi untuk peranti optik termagnet dan laser keadaan pepejal.

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LIST OF ABBREVIATIONS

Al_2O_3	-	Alumunium Oxide
BO	-	Bridging Oxygen
DC	-	Down-Conversion
Er^{3+}	-	Erbium
ET	-	Energy Transfer
fcc	-	Face Centered Cubic
FeO	-	Wustite
Fe_3O_4	-	Ferrite Oxide or Magnetite
α-Fe_2O_3	-	Hematite
γ-Fe_2O_3	-	Maghemite
FWHM	-	Full Length at Half Maximum
HR-	-	High-Resolution
HRTEM	-	High Resolution Transmission Electron Microscope
JCPDS	-	Joint Committee for Powder Diffraction Standards
JO	-	Judd-Ofelt
MgO	-	Magnesium Oxide
NBO		Non-Bridging Oxygen
NPs	-	Nanoparticle
NR	-	Non-Radiative
Oe	-	Oersted
TeO_2	-	Tellurite Oxide
P	-	Phosphorous atom
PL	-	Photo-Luminescence
RE	-	Rare Earth
TEM	-	Transmission Electron Microscope
UTM	-	Universiti Teknologi Malaysia
UV	-	Ultraviolet
VBM	-	Valence Band Maxima
VIS	-	Visible
VSM	-	Vibrating Sample Magnetometer

XRD	-	X-Ray Diffraction
ZnO	-	Zinc Oxide

LIST OF SYMBOLS

A	-	Absorption Coefficient
2θ	-	Angle of Diffraction
B	-	Magnetic Induction
d	-	Size of Nanoparticle, Thickness of the Sample
e	-	Charge of Electron
E	-	Electric Field
E_{dir}	-	Direct Optical Band Gap
E_{ind}	-	Indirect Optical Band Gap
ΔE, E_U	-	Urbach Energy
f	-	Oscillator Strength
H	-	Magnetic Field
I	-	Intensity
l	-	Length
J	-	Orbit Angular Momentum
m	-	Mass of Electron
n_2	-	Non-linear Refractive Index
M	-	Average Molecular Weight
N	-	Concentration
N_A	-	Avogadro’s number
R	-	Glass Constant
S	-	Stability Factor
T'	-	Transmission
T	-	Temperature
T_c	-	Crystallization Temperature
T_g	-	Glass Transition Temperature
T_m	-	Melting Temperature
T	-	Time
V	-	Molar Volume
W	-	Weight
α_m	-	Polarizability

β	-	Branching Ratio
E	-	Dielectric Function
X	-	Susceptibility
P	-	Density
λ	-	Wavelength
T	-	Lifetime
S	-	Spin
L	-	Orbit
ρ	-	Density
w_a	-	Weight of the glass sample in air
w_b	-	Weight of the glass sample in liquid
ρ_b	-	Density of Tolluene
E_f	-	Energy of the final state
E_i	-	Energy of electron in lower band
B	-	Band tailing
$h\upsilon$	-	Photon energy
τ	-	Radiative lifetime
η_R	-	Luminecent efficiency
τ_{nr}	-	Non-radiative lifetime
M	-	Magnetic moment
H_c	-	Coercivity
Mr	-	Remanent magnetization
Ms	-	Saturation magnetization
Mr/Ms	-	Squarness

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Glass is a non-crystalline solid material that exhibits a glass transition and possess several incredible properties including thermal stability, luminescence intensity (Linda et al., 2013, Nedelcheva et al., 2014, Savikin et al., 2013), electrical properties (Jalajerdi et al., 2012) and magnetic properties (Kim et al., 2007, Song et al., 2013). Typical glasses based on different hosts such as tellurite (TeO_2), borate (B_2O_3), silicate (SiO_2) and phosphate (P_2O_5) are usually made transparent in visible spectrum to be used as optical materials (Hussin, 2011). Amongst the present interest materials, the TeO_2 based glasses have gained much attention because of different applications at optical data transmission, laser technologies and sensing. The characteristics of tellurite glass includes lower maximum vibration energy (equal to 750 cm^{-1}) that lower the possibility of multiphonon decay, better thermal and chemical stability, low melting temperature, higher thermal expansion, higher refractive index and better infrared transmission (Barney et al., 2015, Fares et al., 2014). The rare earth (RE) doped glass, has been broadly explored in orders to characterize noble source of optical fiber, amplifier, solids state laser, color display, energy convertor, solar cell, army tool and sensor (Figueiredo et al., 2015, Zhang et al., 2015).

Rare-earth (RE)-doped glass has been widely studied because of their photonic applications, and nonradiative energy transfer (ET) processes, along with their applications of extending from light energy conversion (up and down conversions). In main ET process can favour specific application (for example an operation of anti-Stoke emitter) but it can be dangerous as in place of RE based laser because interaction amongst active ions participate for the augmentation of laser threshold. Particularly the research of ET process in glass having frequency gaps in visible region deserve huge attention since when mixed with RE ion few glasses can be exist as effective visible luminescence (Stambouli et al., 2013, Rivera et al., 2014).

Magnesium tellurite doped with rare earth (RE) ions has attracted research interests in the field of photoluminescence since they are suitable hosts with high chemical stability, offers better homogeneity and lowers sintering temperature and also can produce plenty of crystal field environments imposed on emission centers (Hussin et al., 2011). Basically, the rare-earth ions doped glasses have extensive applications in the field of developing up-conversion (UC) lasers (Taherunnisa et al., 2019). The introduction of erbium oxide (Er_2O_3) can help to improve the optical properties of tellurite glasses. Er^{3+} can show up-conversion (UC) luminescence which has possibility obtaining efficient solid-state laser (Singh B. P et al., 2013).

A glass of Er^{3+} has been shown to possess larger magnetic contributions to low temperature heats well-known at oxide glass. Natural iron oxide, which is majorly in a form of hematite (α-Fe_2O_3), maghemite (γ-Fe_2O_3) and magnetite (Fe_3O_4) may be normally detected in iron sands. A α-Fe_2O_3 shows the similar spinel structures like magnetite but have no divalent ion. Magnetic Fe^{3+} ion is located in two sublattice with dissimilar oxygen coordination. Ferrimagnetism originates from unequal dispersion of such ions in B and A sites. Presently, a lower temperature spin glasses transition was observed at T = 42 K. This γ-Fe_2O_3 possess one corundum crystal structure, possess an antiferromagnetic feature below 950 Kelvin, whereas above Morin point (260 Kelvin) it displays weak ferromagnetism behaviour. Below melting temperature these two magnetized sublattices are produced along rhombohedral axis [111]. The spin canting shows weak ferromagnetism in this plane. Hematite is one of the most stable forms of iron (iron oxide) (Liang et al., 2011).

The magnetic properties of oxide glasess with transition metal ions were mainly determined by morphology that is size and shapes. Iron ions have strong bearing electrical, optical and magnetic properties of glasses. Electron Spin Resonance (EPR) of Fe^{3+} ions in vitreous matrices may provide useful information about the short-range ordering of the paramagnetic ions. This is because the EPR absorption spectra show distinct resonance lines for the ions involved in structural units of well-defined symmetry and those connected in clusters. In a resonance experiment, the particles are exposed to local magnetic field, which besides the DC magnetic field of the spectrometer includes a magnetic anisotropy field, depending on the physical nature of the particles, as well as a demagnetizing field depending on the particle shape. In very fine particles, the two latter contributions are averaged because of the thermal fluctuations of the magnetic moments. This results in either narrowing of the resonance spectra or a characteristic "two-line pattern (a broader line super posed with a narrower one)". The resonance in such system is often called superparamagnetic resonance.

Two main reasons are typically considered to describe photoluminescence (PL) improvement of rare earth ions presented in glasses having metal NPs. One cause is the detention of electromagnetic fields that originate increased local field at the area of NPs where rare earth ion may be situated. Another cause is a strong light absorption by NPs and energy transfer at the rare earth ions. This field amplitude is affected by the superficial plasmons in NPs and can reach big values which are reliant on the distance amongst the rare earth ions and NPs. When incident light and the PL wavelength is close to NP surface plasmons resonance (SPR) wavelength λsp, the absorption rate and amplitude of the radiative transition of RE ions can be amplified. The magnitude of λsp depend on dielectric function of NPs and host, besides on the shape and size of the NPs. It is well known that there found optimum gap between the NP and RE ion so as to happen related PL improvement. When the gaps between the NP and the RE are very minor, PL quenching happens as established in experiment that detect emission from one single molecule or atom situated nearby one metallic NPs or at proximity of any metallic films. In the last few years, characterization and synthesis of heavy-metals oxide (HMO) glasses incorporated with RE ion and comprising metallic NP was stated for large varieties of glasses (Singh, 2013).

HMO glass doped with trivalent RE ion is interesting materials and with important technical applications. They display powerful luminescence because of their minor cut-off phonon frequency. However, for the purpose to make device with increased optical characteristic, the concentrations of rare earth ions have to be lowered adequately so that luminescence quench is lessened. This is also possible to avoid such quenching effects by adjusting the environment perceived by luminescent ions. Consequently, glasses comprising metallic NPs incorporated with lower concentration of rare earth ions are of specific interest since the larger local field working on the ion located near NPs can increase luminescence efficiencies when optical frequencies of excitation beam or/and the luminescence frequencies are near the resonance with surface plasmon incidence of the nanoparticle (Singh, 2013, Rai et al., 2008).

1.2 Problem Statement

Magnetic properties of glasses containing iron oxide depend on the concentration of the 3d element and the ratio (Fe^{3+}/Fe^{2+}) of valence states as well as the structure of the vitreous matrix. It is well known that the multiple valence states of transition metal ions and their distribution in glasses influence the magnetic properties of the materials (Singh et al., 2008). Among all iron oxides, magnetite Fe_3O_4 possess the most interesting properties because of the presence of iron cations in two valences states, Fe^{2+} and Fe^{3+}, in the inverse spinel structure. Fe_3O_4 nanoparticles are common ferrite oxides with an inverse spinel structure (Vijaya et al., 2005). These classes of compound exhibit exceptional magnetic and electrical properties because the electrons transfer between Fe^{2+} and Fe^{3+} in octahedral and tetrahedral sites. Moreover, these transition metal ions shall contribute to a multivalence state in the glass which influence the properties (Ensanya et al., 2009).

The well organised research involving ferrite glass as modifier has been started earlier (Raghaviah et al, 2004) followed then by Battisha et al (Battisha et al, 2006). Both group has since been successfully focused on the study of magnetic part of glass containing ferrite. By using multicomponents of glass composition, they have found better ways to get the better magnetic properties of the glass. The effect of ferrite on magnetic properties has been then continually done by other group of researchers including by Singh (Singh et al, 2010) and Ghandoor (Ghandoor et al, 2013). They have claim to get a better magnetic properties such as magnetic moment up to 46.7 emu/g compared to 5×10^{-3} emu/g, which is much better than the previous research. Later, this research on magnetic properties has successfully been organised by Pelluri

(Pelluri et al, 2016) but the result on magnetic moment is not as good as the previous research. And again, it must be noted that all of them are using ferrite that is obtained from the chemical lab scale of production. The idea of using ferrite nanoparticles perhaps has been well proposed by a group of researchers including the one lead by Widanarto (Widanarto, 2013). These wonderful works have been further done by using a natural ferrite Fe_3O_4 nanoparticles (NPs) as the main components of the glass (Widanarto et al, 2013). However, these works have once again been focussed on the optical properties especially in the enhancement of emission intensity and the electronic properties such as optical energy band gap and electronic structure. As can be seen, there are still very limited study on the optical properties especially regarding the JO parameters and the interconnections between physical properties, optical properties in the presence of metallic NPs and magnetic properties of glass containing natural Fe_3O_4 NPs. The problem in the interconnection between properties are still far behind. The question on how the physical properties may affected on the magnetic parts and vice versa are still in the grey area. Further, the question on the relation between magnetic properties and the optical parts and vice versa are still remain unanswered. Thus, this study will provide a comprehensive works, aims to focus specifically on the structural, optical and magnetic properties and to stimulate the relationship between these behaviours. This study will also hopefully, get to connect a bridge between those properties, which seems to have some gap in between. Additionally, since there are only few studies on these glasses, there is lack of information on the influence of Fe_3O_4 NPs and Er^{3+} ion on the tellurite glass. Therefore, this research aims to provide more information on the structural, optical and magnetic analysis of erbium doped tellurite glass embedded with natural Fe_3O_4 NPs. It is expected that this study can provide more information on the basic knowledge of the glass system.

1.3 Objective of the research

The following are the objectives of this research:

i. To prepare Fe_3O_4 NPs embedded into erbium doped magnesium tellurite glass at varying concentrations by melt quenching technique.

ii. To determine the effect of Fe_3O_4 NPs on structural, optical and magnetic properties of erbium doped tellurite glass.

iii. To determine the J-O intensity parameters and analyse the relation between absorption and emission spectra to complement the experimental data.

1.4 Scope of Study

For the achievement of the above-mentioned objectives, the study has been emphasized on given scopes:

i. Preparations of two glass series by melt-quenching technique. The composition of glass is chosen due to the high stability and high performing ability of glass.

   (a) Glass based with different NPs concentration of composition (89-x) TeO_2-10MgO-1Er_2O_3-(x) Fe_3O_4, system (x = 0, 0.2, 0.4, 0.6 and 0.8 mol%).
   (b) Glass with different rare earth concentration of composition (89.6-y) TeO_2-10MgO-(y) Er_2O_3-0.4Fe_3O_4, system (y = 0.2, 0.4, 0.6, 0.8 and 1 mol%).
   (c)

ii. Determination of the effect of Fe_3O_4 NPs on the structural, optical and magnetic properties in terms of XRD, UV-Vis, FTIR, RAMAN, PL, EDAX, HRTEM, EPR and VSM.

iii. Calculating the JO intensity parameters by using the JO theory and analysing the correlation between absorption and emission cross section area data.

1.5 Significance of the study

Glass doped with rare earth ions are being investigated effectively. Their fabricating application e.g., laser in visible regions of 400-700 nm is of attention at the current time at optical material science. A rare earth ion like erbium (Er) has successfully been used in the optical fiber as dopant to increase signal in the optical communication system because of their high refractive indexes, big resistance against corrosions, more solubility of rare earth and better transparency in region from the visible to the infrared (0.35 to 6 μm). Erbium doped fibre amplifier (EDFA) amplifies signal in C-band (from 1535 nm to 1565 nm) in optical domain. Various different glass has been explored for the erbium doping with an aim to achieve large bandwidth and efficient fluorescence. This study can provide great knowledge on the structural, optical and magnetic behavior of Er^{3+} ions embedded in tellurite glass. Furthermore, the next step will be to adding the magnetic NPs in proposed glass to examine its influences on the structural, optical and magnetic properties of the glass. The ideal system can be used largely in wide range of application from color displays, optical fiber, magneto optic device and amplifiers to medical and army device.

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