

STRUCTURAL AND LUMINESCENCE STUDIES OF MnO_2 DOPED IN GLASS FROM RICE HUSK ASH FOR GREEN AND ORANGE EMISSION MATERIAL APPLICATIONS

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Abstract

(55-x) SiO_2 : 13 B_2O_3 :1.0 Al_2O_3 : 6.3 CaO : 0.2 Sb_2O_3 : 20 Na_2O : 4.5 BaO :x Dopant (x = MnO_2) where x = 0.2, 0.4, 0.6, 0.8, and 1.0 mol% were synthesized by melt quench method. In this work the investigation of glass productions using from rice husk ash (RHA) with effect of MnO_2 was studied. The structural and optical properties were examined and discussed. The local RHA in Nakhon Pathom Province, Thailand was used as a silica source for preparation of glass. FTIR, RAMAN, Optical absorption, Photoluminescence analysis were performed for these glasses. FTIR show Si-O-Si anti-symmetric vibrations of bridging oxygens for all glasses. Raman Spectra revealed danburite structural units. Mn^{2+} : RHA luminescence were analyzed and presented corresponding Tanabe-Sugano and energy level diagrams. The Mn^{2+} : RHA glass showed green and orange emission ranging from 475 nm to 650 nm. Lifetime measurements were also performed and found that the lifetime decreases with increase in MnO_2 concentration.

Keywords: Boro-silicate glasses, FTIR, RAMAN, racah parameter, luminescence, Tanabe-Sugano diagram

Introduction

Rice growing countries (Thailand) are having ecological harms and reduces huge quantity of rice husks these are the by-product of rice paddy. Rice husks are burnt in the boilers for processing paddy. More than 20 million tons of rice husk ash are produced annually in Thailand. This is a huge ecological hazard which cause harm / spoil the land and the neighboring area in which it is discarded.

Lot of methods are being sought out for disposing rice husk ash by reprocess it into building materials.

Rice husk ash consists of 95% silica and 5% metal ions (iron, aluminum, etc.). A great fraction of it has been recycled as a raw material for cement. However, it utilizes in cement production has its restrictions because fly ash contains heavy metals as well as a silica content of more than 70 wt %. Heavy

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metals could cause secondary contamination and the extreme accumulation of a silica-rich fly ash to cement could depreciate the quality of the cement. Since RHA contains a large amount of SiO₂, which is a main glass network former and a minute contamination of transition component, it is feasible to use RHA as a raw material to develop glass matrices (Tuscharoen *et al.*, 2011; Srisittipokakun *et al.*, 2017). Among the transition metal ions, Mn²⁺ is a typical luminescent ion. A number of researchers have carried work on the role of Mn²⁺ ions in the physical and structural properties of a variety of glass systems, such as borate, phosphate, and tellurite (Samsudin *et al.*, 2016). It has been recognized that the green colored emission emitted from Mn²⁺ ions can be attributed to the spin flip transition of the d-orbital electron affiliated with Mn²⁺ ions in tetrahedral site ⁴T₁(G) - ⁶A₁(S) (An *et al.*, 2010). It has been considered in various inorganic hosts covering a wide range of emissions from blue to red. Glass is important for optical material applications due to its superior transparency in the visible region (Rajaramakrishna *et al.*, 2020, 2018a). In general, silica is a major constituent of the agricultural / food waste ash. It varies from 98-36 wt% in ash of rice husk (RHA), sugarcane leaves and corn husk (Rajaramakrishna *et al.*, 2018b), hence RHA was used as silica source in the present work. The purpose of adding Al₂O₃ and CaO modifiers are to increase stability and overcome the volatilization, while Na₂O modifier is to enhance the homogeneity and to lower the melting point of the glass matrix (Rajagukguk *et al.*, 2019). The addition of BaO in the composition acts as modifier to give more mechanical strength, chemical durability, lower thermal expansion coefficient and lower hygroscopic nature (Shoaib *et al.*, 2019). Boron oxide B₂O₃ is a basic glass former due to its higher bond length, lower cation size, smaller heat of fusion and trivalence nature of boron (Rajaramakrishna *et al.*, 2018b) which infuses well with secondary glass former such as silicate (RHA) in the present work. Luminescence mechanism usually involve both parity and forbidden transitions, therefore, the intensity of the emission is relatively weak. In this work we have focused to prepare RHA glass doped with MnO₂ to study their structural and luminescence behavior when monitored at different excitation wavelengths.

Experiment

The local RHA (Rice Husk Ash) in Nakhon Pathom Province, Thailand was used and heat treated at 1,050°C as a silica source to prepare glasses. The

glass samples were prepared by using high purity Na₂CO₃, Al₂O₃, B₂O₃, CaO, Sb₂O₃, BaCO₃, SiO₂ (use RHA as SiO₂) in formula. The glasses were prepared using (55-x)SiO₂: 13B₂O₃:1.0Al₂O₃: 6.3CaO: 0.2Sb₂O₃: 20Na₂O: 4.5BaO:x Dopant (x = MnO₂) where x = 0.2, 0.4, 0.6, 0.8, and 1.0 mol% respectively for MnO₂. The thorough experimental procedure for glass making process is given by (Srisittipokakun *et al.*, 2017). The prepared glasses are shown in Figure 1(a).

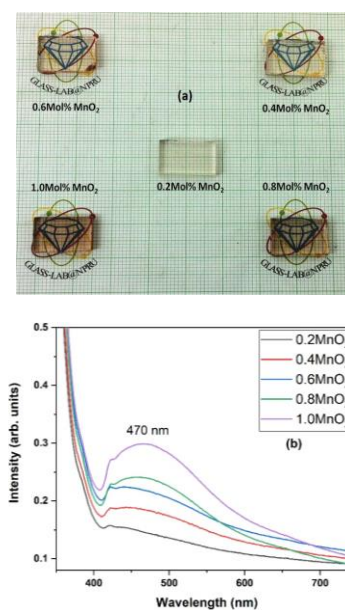


Figure 1. (a) Picture of RHA glass doped with MnO₂ (b) Absorption spectra of RHA glass doped with MnO₂

Results and Discussions

Absorption Studies

The study of optical absorption in the UV-Vis region is advantageous in order to understand the electronic band structures and transitions of glasses as presented in Figure 1(b). The broad absorption band ranging from 410-580 nm (centered at 470 nm) is clearly visible that the intensity increases as the Mn-doped content increases also matching well with the near-UV emission band of Mn²⁺, attributed to ⁴T₁(G) - ⁶A₁(S) transition which will be discussed in photoluminescence studies. The absorption edge of these glasses is not as sharp as in crystalline compounds due to the disorder in the matrix. The wide band range of absorption provides certain coloring in the glass samples for higher concentration of MnO₂ content as shown in Figure 1(a).

FTIR Analysis

The observed FTIR absorption spectra are shown in Figure 2 which are recognizable, palpable and distinctive to the silicate chains which are active in the mid region of the spectrum ($600\text{--}1,800\text{ cm}^{-1}$). Figure 2 represents the FTIR spectra of metal transition (MnO_2) doped in RHA glass. From Figure 2 it can be realized and interpreted as follows: (a) A small band at $745\text{--}800\text{ cm}^{-1}$ is ascribed to symmetry stretching vibration of O-Si-O bonds (Wong and Angell, 1976), (Khalifa *et al.*, 1997; 1998). (b) A shoulder at about $920\text{--}970\text{ cm}^{-1}$ can be attributed to vibrations of non-bridging oxygens (Khalifa *et al.*, 1998), (Cornejo *et al.*, 2014), (Bondioli *et al.*, 2010), (Yalcin and Sevinc, 2001). (c) A shoulder at 1100 cm^{-1} to $1,270\text{ cm}^{-1}$ is recognized and related to Si-O-Si anti-symmetric vibrations of bridging oxygens (Wong and Angell, 1976). (d) A small band at about $1,520\text{--}1,650\text{ cm}^{-1}$ is related to molecular water (Wong and Angell, 1976).

RAMAN Analysis

Raman intensity peaks at lower wavenumber attributed to the deformation modes of the metal-oxygen chain of Mn-O-Mn in the MnO_2 octahedral lattice. In this structure, the oxygen ions bridge three Mn ions and lie in a site with C_{2v} symmetry. Hence, three modes are expected with A_{1g} , B_{1g} , and B_{2g} symmetry (Julien *et al.*, 2002). The Raman spectrum of MnO_2 with various concentration is shown in Figure 3 below which displays the overlapping of main contributions at 520 , 580 , and 630 cm^{-1} due to

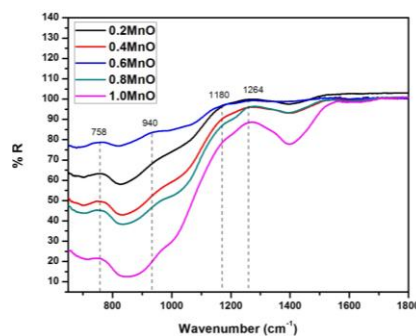


Figure 2. FTIR Spectra of d-transition metal MnO_2 doped RHA glass

amorphous solid and also intense peak recorded at 340 cm^{-1} . The broad peak indicative of a well-developed orthorhombic structure with an interstitial space. There are assigned to the A_{1g} spectroscopic species. It is worth mentioning that, in MnO_2 , Raman absorption comes from the collective vibration mode of MnO_6 octahedron. The two main Raman peaks correspond to the stretching mode of the MnO_6 octahedra and the peaks at lower wavenumber are attributed to the deformation modes of the metal-oxygen chain of Mn-O-Mn in the MnO_2 octahedral lattice (Jana *et al.*, 2007; Jana *et al.*, 2009). The low-wavenumber peak around 340 cm^{-1} is assigned to an external mode that derives from the translational motion of MnO_6 units (Julien *et al.*, 2002; Jana *et al.*, 2009). According to the above results, the stretching vibrations of MnO_6 octahedra (corner, edge and face sharing) lie in the

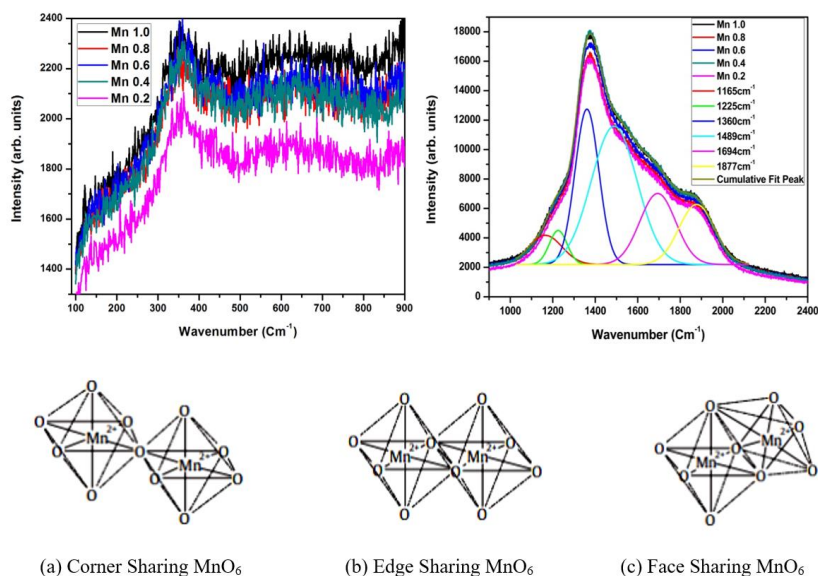


Figure 3. RAMAN Spectra of MnO_2 doped RHA glass. (i) Lower frequency range (ii) Higher frequency range (iii) Structural configurations of MnO_6

region of 500-700 cm⁻¹ (Julien *et al.*, 2002). The distinct peak around ~630 cm⁻¹ is frequently attributed to danburite-like units, four-member rings having two adjacent SiO₄ and two BO₄ tetrahedra (Manara *et al.*, 2009; Malchow *et al.*, 2015; Kilymis *et al.*, 2017). The strong bands at region of 580-632 cm⁻¹ are due to the symmetrical stretching vibrations of Mn-O (Cheng *et al.*, 2014; Kumar *et al.*, 2017). The Raman absorption peak at 820-850 cm⁻¹ which may have been due to stretching vibrations of the B-O-Si linkage (Gautam, 2014). While the wide bands in the high wave numbers region (1,200-1,500 cm⁻¹). Intensities were interpreted after deconvolution around wide band range and obtained 1,165 cm⁻¹, 1,225 cm⁻¹, 1,360 cm⁻¹, 1,489 cm⁻¹, 1,694 cm⁻¹, and 1,877 cm⁻¹ of the bands in the 1,200-1,500 cm⁻¹ range in the spectra are attributed to the terminal vibration of oxygen atoms in Si-O⁻ in Q³ Si units (structural units Qⁿ Si, where n means the number of bridging oxygens) (Łagowska *et al.*, 2018) and stretching vibrations of B-O⁻ bonds and stretching vibrations of O₃B-O-BO₄ bonds in BO₃ pyramidal units, Stretching vibrations of B-O-B bonds in pyramidal BO₃ units (Ardelean *et al.*, 2008; He *et al.*, 2014). In Raman spectra, the appearance of unexpected bands located at Raman shifts higher than 1,500 cm⁻¹ led to interpretations of excitation/emission wavelengths.

Photoluminescence Studies

The emission and excitation spectra of RHA glass doped with MnO₂ show Mn²⁺ excitation/emission which when monitored at λ_{emi} = 528 nm (2.34 eV) and λ_{exi} = 422 nm (2.93 eV), respectively as shown in Figures 4-5. Photoluminescence excitation spectra show peaks at 359, 375, 422, 432, 465, 483, and 489 nm (Figure 4). The photoluminescence emission spectra show a wide peak at 528 and 620 nm (Figure 5). The peak at 528 nm is attributed due to spin-forbidden of Mn²⁺ (⁴T_{1g}(⁴G)→⁶A_{1g}) (Tanabe and Sugano, 1954; Henderson and Bartram, 2000; Lakshminarayana and Buddhudu, 2006; Thulasiramudu and Buddhudu, 2006; Onufrieva *et al.*, 2018; Shcherbakov and Nizamutdinov 2019). In addition, the low intensity broad peak at 620 nm corresponds to luminescence characteristic of Mn³⁺ ions. This reveal that the MnO₂ in the present glass has reduced to form Mn²⁺ and partly to Mn³⁺, while the former being dominated. The excitation spectra show signature Mn²⁺ corresponding from ground state ⁶A₁ to excited states of ⁴T_{2g} (⁴G), ⁴E (⁴G), ⁴A_{1g} (⁴G), ⁴T_{2g} (D), ⁴E (D) (Tanabe and Sugano, 1954; Xu *et al.*, 2017). The high intensity peak at 422nm corresponds to ground level of ⁶A₁ (S)→⁴E (⁴G), ⁴A_{1g} (⁴G) of excited levels which has a 3d⁵-electron configuration (Lakshminarayana and Buddhudu,

2006; Thulasiramudu and Buddhudu, 2006), according to Tanabe-Sugano (T-S) diagram (Figure 6), (Tanabe and Sugano, 1954). All spectra contain a narrow band at 422 nm excitation. In this case, all transitions are spin forbidden and therefore the intensity is much less than for spin allowed transitions. The detailed transition of energy level

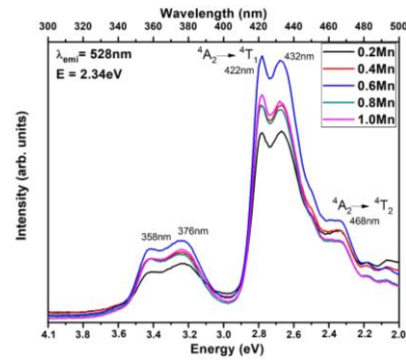


Figure 4. Photoluminescence excitation spectra of RHA: Mn²⁺ glass monitored at λ_{emi} = 528nm

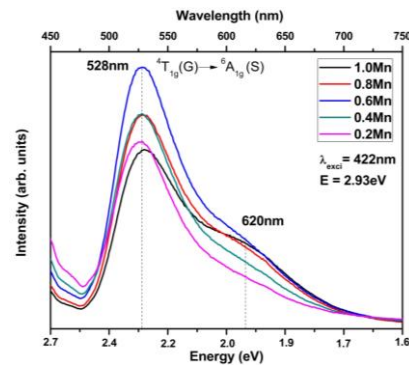


Figure 5. Photoluminescence emission spectra of MnO₂ doped RHA glass

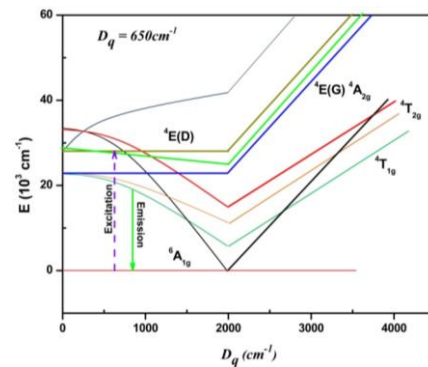


Figure 6. Tanabe-Sugano diagram and corresponding optical absorption spectrum for RHA glass doped with 1 mol% MnO₂

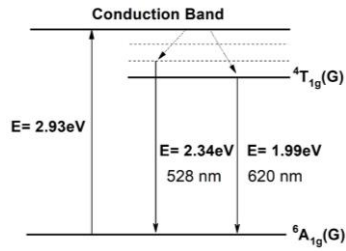


Figure 7. Energy level diagram of Mn²⁺ in RHA glass

diagram is presented in Figure 7. The Racah parameter has been determined using the formula,

$$\begin{aligned} v_1 &= E [{}^4E ({}^4G), {}^4A_{1g} ({}^4G)] - E [{}^6A_1] \\ &= 10B + 5C \end{aligned} \quad (1)$$

$$\begin{aligned} v_2 &= E [{}^4E (D)] - E [{}^6A_1] \\ &= 17B + 5C \end{aligned} \quad (2)$$

By subtracting (1) by (2), we have

$$v_2 - v_1 = 7B \quad (3)$$

The data obtained from Figure 4 we have $v_2 = 27,855 \text{ cm}^{-1}$ and $v_1 = 23,696 \text{ cm}^{-1}$ which is also shown in the Figure 6 as by drawing a vertical line from ground state 6A_1 to excited state ${}^4T_{2g}({}^4G)$ in T-S diagram. Substituting these values, we obtain the values for Racah parameters, $B = 594 \text{ cm}^{-1}$ and $C = 4,383 \text{ cm}^{-1}$. The nephelauxetic effect β is calculated by the formula using (Barteccki and Kurzak, 1981) and (Liem and Tran, 2018) $\beta = B/B_0$, where $B_0 = 785 \text{ cm}^{-1}$ Racah parameter for free Mn²⁺ ion. By substituting the B and B_0 (as displayed above), we obtain $\beta = 0.7566$. T-S diagram has been explained in the Figure 6, which determines splitting parameter D_q we draw a parallel line to the vertical axis mentioned as emission which intersects excited state ${}^4T_{1g}$ showing emission at 528 nm (2.34 eV) to ground state ${}^6A_{1g}$ as represented in Figure 6 and energy level diagram in Figure 7. Hence, $D_q = 650 \text{ cm}^{-1}$ or the crystal field of Mn²⁺ doped RHA glass is $\Delta = 6,500 \text{ cm}^{-1}$ ($\Delta = 10 D_q$). One would have expected higher covalence bonding relative to the Mn²⁺-O²⁻ in RHA glass.

Lifetime Analysis:

The lifetime time characteristics of ${}^4A_2 \rightarrow {}^4T_1$ level for Mn²⁺: RHA glasses with different MnO₂ concentration were obtained by exciting the glass 422 nm and monitoring the emission at 528 nm (${}^4T_{1g}({}^4G) \rightarrow {}^6A_{1g}$) and are shown in Figure 8. All the decay profile is fitted to single exponential function $I_t = I_0 e^{-t/\tau}$ whereas I_0 is the decay intensity when $t=0$ and τ represent the decay time of the excited state. The measured decay time (τ) showed 7.18, 6.64,

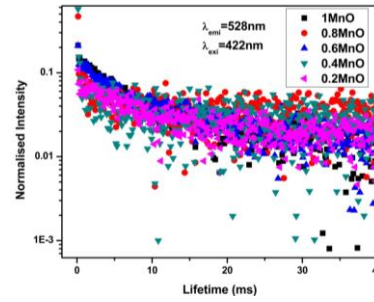


Figure 8. Lifetime of Mn²⁺: RHA glass

6.34, 5.98 and 5.60 ms for 0.2mol%, 0.4mol%, 0.6mol%, 0.8mol% and 1.0mol% MnO₂ content respectively. The decrease in the decay time with increase in concentration is due to increase in the radiative transition probability.

Conclusions

To concise present research, the work signifies to bring out the importance of the MnO₂ doped in RHA based glasses. The unique in its kind where the rice husk ash was used as silica source to synthesis glasses. The glasses showed intense greenish yellow emission (528 nm) (${}^4T_{1g}({}^4G) \rightarrow {}^6A_{1g}$) and orange red emission (620 nm) when monitored at 422 nm. Such emissions with no rare-earth ions and rice husk ash as a source could prove their cost effective, reliable to prepare and potential use in optical device industries. These glasses are new option for recycling waste obtained from rice industry as rice husk ash especially from Thailand and one can control air pollution.

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