Structural and Luminescence Properties of Fe³⁺ Doped Antimony Lead Borophosphate Glass

(Ciri Struktur dan Luminesen Fe³⁺ Didopkan Antimoni Plumbum Borofosfat Kaca)

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ABSTRACT

Structural and luminescence properties of borophosphate glasses with different modifier doped with transition metal ions have been investigated in this study. The glass sample from the series of xPb_3O_4 : $(0.2Sb_2O_3:0.3B_2O_3:(0.5-x)P_2O_5$ and $ySb_2O_3:0.2Pb_3O_4:(0.5-y)B_2O_3:0.3P_2O_5$ where $0 \le x \le 0.20$ mol% and $0 \le y \le 0.15$ mol%, respectively, were doped with 0.01 mol% of Fe_2O_3 system have been prepared using the melt-quenching technique. The structural properties of samples had been studied using fourier transform infrared (FT-IR) spectroscopy. The FT-IR study showed the network structure of the studied glasses based on the B_2O_3 - P_2O_5 host with the Sb_2O_3 and Pb_2O_3 modifiers. The results of FT-IR showed traces of BO_3 and BO_4 with the introduction of Sb_2O_3 and Pb_2O_3 modifiers. With the increasing content of B_2O_3 which replacing content of P_2O_5 in the glass network, the intensity of the borate band decreases and shifted to lower frequency. UV-Vis spectroscopy analysed the transition of Fe^{3+} from ground state to excited state in the ultraviolet spectral region. The photoluminescence of samples were studied using photoluminescence enhancement by doping Fe^{3+} as activator.

Keywords: Borophosphate glass; fourier transform infrared (FT-IR); photoluminescence; UV-Vis spectroscopy

ABSTRAK

Ciri struktur dan luminesen borofosfat kaca dengan pengubahsuai yang berbeza didopkan dengan ion logam peralihan telah dikaji dalam penyelidikan ini. Sampel kaca berdasarkan siri xPb₃O₄: 0.2Sb₂O₃: 0.3B₂O₃: (0.5-x) P₂O₅ dan ySb₂O₃: 0.2Pb₃O_{4:(0.5}y) B₂O₃: 0.3P₂O₅ dengan $0 \le x \le 0.20$ mol% dan $0 \le y \le 0.15$ mol% masing-masing yang didopkan dengan 0.01 mol% Fe₂O₃ sistem telah dihasilkan dengan melalui teknik sepuh lindap leburan. Ciri struktur sampel telah dikaji dengan menggunakan spektroskopi transformasi fourier inframerah (FT-IR). Pengukuran struktur menggunakan teknik FT-IR telah menunjukkan struktur rangkaian kaca yang dikaji berdasarkan hos B_2O_3 -P₂O₅ dengan pengubahsuai Sb₂O₃ dan Pb₃O₄. Keputusan FT-IR menunjukkan penambahan BO₄ daripada BO₃ dengan penambahan Sb₂O₃ dan Pb₃O₄. Tambahan pula, apabila penambahan komposisi B_2O_3 dalam siri kaca, keamatan jalur borat berkurang dan beralih kepada frekuensi yang lebih rendah. Teknik UV-Vis digunakan untuk menganalisis peralihan Fe³⁺ dari aras dasar ke aras teruja pada kawasan spektrum ultraungu. Ciri fotoluminesen telah dikaji dengan menggunakan spektroskopi fotoluminesen. Fotoluminesen spektrum menunjukkan peningkatan sifat fotoluminesen disebabkan oleh ion Fe³⁺.

Kata kunci: Borofosfat kaca; fotoluminesen; teknik UV-Vis; transformasi fourier inframerah (FT-IR)

INTRODUCTION

Borophosphate glasses possess better chemical durability than phosphate glasses due to the cross-linking of phosphate chains by BO₄ groups (Koudelka et al. 2003). It was well known of borophosphate glasses having improved durability among the multi component glasses studied for various interesting applications (Shah et al. 2009). Lead oxide and antimony oxide were act as modifier in this series of glasses. Lead oxide has unique influence on the host structure and was widely used in glasses to improve the chemical durability and lower the melting temperature (Lucacel et al. 2007). Antimony was lately classified as a glass network former (NWF), especially when it is cooled rapidly (Karam Abdel-Aati & Ahmed Gamel El-Din 2003). Hence antimony oxide can participate in preparing many oxide glasses of special purposes (Mostafa et al. 2004). It is known that, phosphorous oxygen bonds are of higher covalent character than the antimony oxygen bonds. Accordingly, it is expected that antimony ions most probably occupy the NWF positions (Mostafa et al. 2004). Transition metal ions added into glass system has enhanced the chemical stability of glasses (Wong et al. 2012). Many of the recent studies indicate that the glasses containing Fe₂O₃ is widely use in electrochemical, electronic and electro-optic devices (Shiv Prakash et al. 2010) due to its unique electrical properties. However, lack of information to explain comprehensiveness on optical properties of Fe³⁺ doped glasses though many researches have been carried out. Literature has reported iron oxide exist in different valence states with different coordination in glass matrices, for example, Fe³⁺ occupy both tetrahedral and octahedral positions (Moistafa et al. 2013). The

redox and environment of iron and chemistry of the glass matrix in which it is present are closely interrelated and can substantially influence the resulting optical properties, specifically the UV–visible and IR transmission (Terczyńska-Madej et al. 2011). The Fe₂O₃ also has strong absorptivity in the visible range (Shiv Prakash et al. 2010). It is interesting to study the effect of iron oxide activated antimony lead borophosphate glasses. Photoluminescence properties of iron oxide borophosphate glasses are useful in industrial application, such as fibre optic communications, luminescent solar energy concentrators, memory module and photo-conducting properties (Kreidl 1990).

The purpose of the present paper was to characterize the antimony lead borophosphate glasses using FT-IR spectroscopy to recognize their bonding mechanism. Meanwhile, UV-Vis-NIR and photoluminescence (PL) spectroscopy were used to justify the transition of energy levels of the Fe³⁺ in the vitreous network.

EXPERIMENTAL DETAILS

A series of glasses with molar composition of $xPb_{3}O_{4}:0.2Sb_{2}O_{3}:0.3B_{2}O_{3}:(0.5-x)P_{2}O_{5}$ and $ySb_2O_3:0.2Pb_3O_4:(0.5-y)B_2O_3:0.3P_2O_5$ where $0 \le x \le 0.2$ mol% and $0 \le y \le 0.15$ mol%, respectively, were doped with 0.01 mol% of Fe₂O₃ system have been prepared using melt-quenching technique. A total of ten samples were prepared using borate acid, H₂BO₂, antimony oxide, Sb₂O₂, lead oxide, Pb₃O₄ in powder form and aqueous phosphoric acid, H₃PO₄ at 85% purity was used. We used aqueous H_3PO_4 instead of powder P_2O_5 due to the homogeneity during mixing chemicals. The chemicals were mixed in a porcelain crucible and experienced preheat in 300°C electrical furnace for an hour. After preheated, the sample was moved to 1200°C high temperature furnace for 15 min. Time of 15 min is sufficient for sample melting at temperature of 1200°C. After the sample was melted, it skips the annealing process and directly pours on a steel plate to produce a super cooled solid.

The samples produced were measured by Perkin-Elmer Spectrum 1 Fourier Transform Infrared (FT-IR) spectroscopy for structural properties studies. The samples were grounded into fine powder before undergoes FT-IR measurement. KBr technique was utilized and the powder was compressed into pellet at 7 MPa for 5 min. Meanwhile, ultraviolet-visible-Near Infrared (UV-Vis-NIR) spectrophotometer and Perkin-Elmer LS55 photoluminescence (PL) spectroscopy were employed to study luminescence properties. Table 1 shows the composition of 10 samples. Samples 1 to 9 are the samples doped with 0.01 mol% of iron oxide, Fe₂O₃.

RESULTS AND DISCUSSION

The infrared spectra from 400-2000 cm⁻¹ was presented in Figure 1. The spectra divided by two parts which were $0 \le x \le 0.20 \text{ mol}\%$ and $0 \le y \le 0.15 \text{ mol}\%$ for the glass samples due to the different composition content of borate and phosphate. Vibration peak at 1384 cm1 showed the vibrations of bridging oxygen atoms between BO₂ and BO₄ groups (Mostafa et al. 2004). Srinivasulu et al. (2012) stated that the IR spectra above 1600 cm⁻¹ was assigned to the hydroxyl and water group which are O-H bond stretching mode. B-O bond stretching of tetrahedral BO₄ units and bond bending motion of B-O-B groups occurred between 670 and 750 cm⁻¹ (Almeida et al. 2001; Baykal et al. 2000; Carta et al. 2008). Carta et al. (2008) reported the P-O-P bond stretching vibration mode between 700 and 900 cm⁻¹ and PO₂ stretching mode of non-bridging atoms occurred at the range of 1400 and 1150 cm⁻¹. Almeida et al. (2001) reported three main vibrational mode of borate network at 1200-1500 cm⁻¹ for B-O bond stretching of trigonal BO₃ units, 850-1200 cm⁻¹ associated with B-O bond stretching of tetrahedral BO₄ units, 600-800 cm⁻¹ assigned to bending vibrations of various borate segments and PO₄ bending mode exists below the 600 cm⁻¹. Doweidar and Saddeek (2009) have found that a broad absorption band arises in the region 850-1100 cm⁻¹ was due to changes

 $x Pb_{3}O_{4}: 0.2Sb_{2}O_{3}: 0.3B_{2}O_{3}: (0.5-x)P_{2}O_{5} \text{ and } ySb_{2}O_{3}: 0.2Pb_{3}O_{4}: (0.5-y)B_{2}O_{3}: 0.3P_{2}O_{5}$ where $0 \le x \le 0.20 \text{ mol}\%$ and $0 \le y \le 0.15 \text{ mol}\%$

TABLE 1. Antimony lead borophosphate glass with composition of 0.01Fe₂O₃ doped

No	Composition (mol %)				
	B ₂ O ₃	P_2O_5	Pb ₃ O ₄	Sb ₂ O ₃	
Undoped	0.30	0.30	0.20	0.20	
1	0.30	0.30	0.20	0.20	
2	0.30	0.35	015	0.20	
3	0.30	0.40	0.10	0.20	
4	0.30	0.45	0.05	0.20	
5	0.30	0.50	0.00	0.20	
6	0.35	0.30	0.20	0.15	
7	0.40	030	0.20	0.10	
8	0.45	0.30	0.20	0.05	
9	0.50	0.30	0.20	0.00	

in borate structure where conversion of BO_4 unit at the expense of BO_3 structural unit while increasing content of Sb_2O_3 and Pb_2O_3 .

The infrared spectrum of glass sample in this study shows three prominent broad band at around 550, 1000 and 1300 cm⁻¹. Modifiers were added into the network to change the host bonding at these three broad bands. Chemical bond at the range of 983-1047 cm⁻¹, which assigned to the vibrations of boron atoms attached to nonbridging oxygen in the form of tetrahedral BO₄ vibrations, was getting shift to the higher frequency followed by Pb₃O₄ content decreasing.

Absorption band at around 539 cm⁻¹ was assigned to asymmetric bending vibration of P-O bond and bending motion of O-P-O bond. Stronger absorption band might be due to the host glass network and addition of modifier in the network. While the glass network is dominated by borate or phosphate, the asymmetric stretching of O-P-O bond and P=O stretching vibration of BO3 groups became weaken. Modifier content getting lesser in the network causes the intensity of absorption band reduces to almost unidentifiable. The band which appears in the region of 450-550 cm⁻¹ was assigned to the deformation vibrations of the phosphate groups. Bending mode of PO, possesses the band centred at around 484 cm⁻¹. This spectrum might be associated with the Sb-O bond and Pb-O bond since the modifiers content is reducing with deformation vibration of phosphate groups. Absorption band intensity was reducing as decreasing Sb_2O_3 and Pb_3O_4 content. Asymmetric B-O bond stretching vibration occurred at around 1340 cm⁻¹. Certain overlapping of vibration peaks might occur, causing difficulties in identifying the existing of peaks and exact vibration range of peaks (Table 2).

The UV-Vis transmittance spectra and absorption spectra are mirror image in theory. The spectra has generally been divided into two parts which are sample of $0 \le x \le 0.20$ mol% and $0 \le y \le 0.15$ mol% due to the different composition configuration. ElBatal et al. (2008) and Moistafa et al. (2013) have reported that the strong UV absorption at around 235 and 280 nm observed in the spectra might be related to the small amount of transition metal which means impurities or traces of iron containing inside the raw material. Thus, iron oxide was chosen to be used as activator in this study. The UV-Vis spectra showed the transmittance peak occurred at UV band region at 230 nm and a broad absorption band extending from 270 to around 480 nm at ultraviolet-visible band for all the activated samples due to the presence activator, iron oxide, inside the samples. Overlapping of absorption band might occurred due to the impurities absorption which also present at somewhere nearby the visible band region.

The colour of the as prepared glass changes gradually as Pb_3O_4 and Sb_2O_3 concentrations decreasing in these glasses. The prepared glasses appeared in deep black colour, but it becomes yellow to light yellow and again deep yellow. The undoped sample displayed in white colour and opaque. In this case, transparency of glasses increases and then decreases as modifiers content reducing.

As seen from Figures 2 and 3, iron oxide doped sample x = 0.20 mol% shows as a flat band. This was due to excess



FIGURE 1. Infrared Spectrum of iron oxide doped antimony lead borophosphate glass

FT-IR Spectral assignment	PO_4 bending motion	O-P-O bending motion	B–O stretching vibration, B–O–B bending motion and P–O–P stretching vibration	O-B-O (NBO)	$(O-P-O)_{as}$, $(P=O)_{as}$ of BO ₃	$(B-O)_{as}$	Vibration BO atoms between BO_3 and BO_4 groups
y=0	480	544, 566	708	1047	1271		1384
y= 0.05	480	535	669	1025	1271		1384
y= 0.10	482	539	692	1015	1251	1333	1384
y= 0.15	479	551	697	1002	1258	1324	1384
x= 0	478	537,587	694	1047	1279		1384
x= 0.05	478	534	703	1032	1275		1384
x=0.10	473	535	669	1019	1268	1348	1384
x= 0.15	479	551	693	666	1250	1340	1384
x= 0.20	479	539	702	983	1257	1325	1384
Undoped		539	696	1015	1275		1384
Wavenumber cm ⁻¹	484 [14]	530-588 [14]	700 [12-14]	1033 [5]	1280 [16]	1340 [11]	1384 [10]

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FIGURE 2. UV-Vis absorption spectra of iron oxide doped antimony lead borophosphate glass



FIGURE 3. UV-Vis transmittance spectra of iron oxide doped antimony lead borophosphate glass

addition of Sb₂O₃ and Pb₃O₄ modifiers into the host glass network which were ~ 0.40 mol% of the composition. Activator, iron oxide, doped into the glasses led to a wider and broader the absorption. The UV absorption band of undoped sample extending from 270 to 320 nm with no visible band could be identified. Regarding the doped samples, addition of Fe³⁺ ion has broaden the UV absorption band extending from 270 to 470 nm become UV-blue broad band. This extend occurred because of the energy required to excite Fe³⁺ from ground state to excited state is in the ultraviolet-blue region.

The replacement modifier content with host content has significantly contributed effort to the enhancement of absorption spectra at UV-blue band extending from 270 to 480 nm. The transmittance spectra at around 250 nm were improved by substituting the Pb_3O_4 content with P_2O_5 content. Composition of phosphate which increased to 0.50 mol% without lead oxide produced a strong absorption spectra as detected in the sample x = 0 mol%. However, the complete replacement of Sb_2O_3 by B_2O_3 leads to the disappearance of the transmittance peak at around 250 nm.

The UV-Vis spectra are similar to that reported by ElBatal et al. (2012) in which strong UV absorptions were around 235 and 280 nm observed in the spectra related to the small amount of traces of iron containing inside the raw material. In other words, these spectra are in good agreement with the previous researchers and also proved ElBatal et al. (2012) presumption on the strong UV absorption at around 235 and 280 nm. The absorption band which arises represents the energy band that able to excite the Fe³⁺ ion from ground state to higher energy state. Broad absorption band might be related to the overlap of many different excitation states. These different excitation states are due to the free electrons in the ground state which are excited to the localized states within the forbidden band.

Figures 4 and 5 show the photoluminescence emission profile of antimony lead borophosphate glass doped with iron oxide for sample $0 \le x \le 0.20 \text{ mol}\%$ and $0 \le y \le 0.15$ mol%, respectively. Fe³⁺ containing glasses emit violet light at around 419 nm under UV excitation, 300 nm. Fe³⁺ doped glasses caused the shifting of emission peak to higher energy level. The undoped glass PL spectra expected to be smooth as the host emission spectra which shows no peak in the spectra. As seen from Figure 4, the undoped sample showed three emission peaks at 400, 420 and 482 nm. This might be due to the impurities inside the sample. Emission peaks around 303, 379 and 419 nm were observed for glasses doped with 0.01 mol% of iron oxide. As seen from Table 3, three main energy transition of Fe3+ doped glasses from higher energy level $z^6 F^\circ$ to lower energy level $a^4 D$, $a^2 G$ and $a^2 P$ for 303, 379 and 419 nm, respectively.

Excitation wavelength 300 nm was employed to excite Fe³⁺ doped glasses. However, the undoped glasses was excited by 290 nm was unable to supply sufficient energy

to release free electrons in the ground state. The 290 nm excitation energy is higher than the threshold energy of host therefore host emission occurred. Introduction of Fe^{3+} to the glasses, the minimum excitation energy required became lower than excitation energy of undoped sample. The 300 nm wavelength energy is capable of releasing free electrons inside iron atom to higher energy levels. Refer to the UV-Vis spectra, it was discovered that undoped sample has short absorption band relative to the Fe^{3+} doped samples. This is reasonable because the Fe^{3+} doped samples have higher excitation wavelength than the undoped sample.

The addition of activator, iron oxide, into antimony lead borophosphate glasses led to the observation of emission bands at region nearer to UV emission band and violet emission band. Transition of energy level $z^6F^\circ \rightarrow a^4D$ occurred at around 303 nm and $z^6F^\circ \rightarrow a^2G$ at around 379 nm in UV emission band region and $z^6F^\circ \rightarrow a^2P$ at around 419 nm which is violet band region for all the Fe³⁺ doped samples in this study. Content of modifiers was varied to optimize the intensity of emission. The sample with composition of y = 0.10 mol% has the most intense UV emission while sample x = 0.05 mol% has the highest intensity of visible (violet) emission line.

In fact, glasses activated by Fe^{3+} raise the probability of splitting of energy levels hence an increase in the emission peaks. This might be due to the factor of hyperfine glasses structure (Wong et al. 2012) due to the activator, iron oxide. Hyperfine of structure depended on the d-d transition energy level of Fe^{3+} .



FIGURE 4. Emission spectra of undoped sample and Fe³⁺ doped sample with $0 \le x \le 0.20$ mol%



FIGURE 5. Emission spectra of undoped sample and Fe³⁺ doped sample with $0 \le y \le 0.15 \text{ mol}\%$

Sample	Energy levels	Experimental	Calculated
1	$z {}^{6}\mathrm{F}^{\circ}{}_{11/2} \rightarrow a {}^{4}\mathrm{D}{}_{1/2}$	303	302
	$z {}^{6}\mathrm{F}^{\circ}_{7/2} \rightarrow a {}^{2}\mathrm{G}_{9/2}$	379	379
	$z {}^{6}\mathrm{F}^{\circ}_{7/2} \rightarrow a {}^{2}\mathrm{P}^{3/2}_{3/2}$	419	419
2	$z {}^{6}\mathrm{F}^{\circ}{}_{1/2} \rightarrow a {}^{4}\mathrm{D}_{7/2}$	287	290
	$z {}^{6}\mathrm{F}^{\circ}_{9/2} \rightarrow a {}^{2}\mathrm{G}_{7/2}$	388	388
	$z {}^{6}\mathrm{F}^{\circ}_{5/2} \rightarrow a {}^{2}\mathrm{P}_{3/2}$	417	417
3	$z {}^{6}\mathrm{F}^{\circ}{}_{1/2} \rightarrow a {}^{4}\mathrm{D}_{7/2}$	289	290
	$z {}^{6}\mathrm{F}^{\circ}{}^{n_{2}} \rightarrow a {}^{2}\mathrm{G}^{n_{2}}$	389	388
	$z {}^{6}\mathrm{F}^{\circ}_{7/2} \rightarrow a {}^{2}\mathrm{P}_{3/2}$	419	419
4	$z {}^{6}\mathrm{F}^{\circ}{}_{7/2} \rightarrow a {}^{4}\mathrm{D}_{2/2}$	298	298
	$z {}^{6}\mathrm{F}^{\circ}_{7/2} \rightarrow a {}^{2}\mathrm{G}^{5/2}_{0/2}$	379	379
	$z {}^{6}\mathrm{F}^{\circ}{}^{1/2}_{1/2} \rightarrow a {}^{2}\mathrm{P}^{9/2}_{3/2}$	414	415
5	$z {}^{6}\mathrm{F}^{\circ}{}_{9/2} \rightarrow a {}^{4}\mathrm{D}_{7/2}$	293	293
	$z {}^{6}\mathrm{F}^{\circ}{}_{7/2} \rightarrow a {}^{2}\mathrm{G}{}_{9/2}$	386	387
	$z {}^{6}\mathrm{F}^{\circ}_{3/2} \rightarrow a {}^{2}\mathrm{P}_{3/2}$	416	416
6	$z {}^{6}\mathrm{F}^{\circ}{}_{1/2} \rightarrow a {}^{4}\mathrm{D}_{7/2}$	289	290
	$z {}^{6}\mathrm{F^{o}}_{11/2} \rightarrow a {}^{2}\mathrm{G}_{9/2}$	383	383
	$z {}^{6}\mathrm{F}^{\circ}{}_{9/2} \rightarrow a {}^{2}\mathrm{P}_{3/2}$	420	421
7	$z {}^{6}\mathrm{F}^{\circ}{}_{1/2} \rightarrow a {}^{4}\mathrm{D}_{7/2}$	289	290
	$z {}^{6}\mathrm{F}^{\circ}{}_{7/2} \rightarrow a {}^{2}\mathrm{G}{}_{9/2}$	387	387
	$z {}^{6}\mathrm{F}^{\circ}_{3/2} \rightarrow a {}^{2}\mathrm{P}_{3/2}$	416	416
8	$z {}^{6}\mathrm{F}^{\circ}{}_{9/2} \rightarrow a {}^{4}\mathrm{D}_{1/2}$	301	301
	$z {}^{6}\mathrm{F}^{\circ}_{9/2} \rightarrow a {}^{2}\mathrm{G}_{7/2}$	388	388
	$z {}^{6}\mathrm{F}^{\circ}{}_{9/2} \rightarrow a {}^{2}\mathrm{P}_{3/2}$	420	421
9	$z {}^{6}\mathrm{F}^{\circ}{}_{1/2} \rightarrow a {}^{4}\mathrm{D}_{3/2}$	296	296
	$z {}^{6}\mathrm{F}^{\circ}_{3/2} \rightarrow a {}^{2}\mathrm{G}_{7/2}$	384	384
	$z {}^{6}\mathrm{F}^{\circ}_{11/2} \rightarrow a {}^{2}\mathrm{P}_{3/2}$	423	424
	11.2 5.2		

TABLE 3. Transition energy level of iron oxide doped antimony lead borophosphate glass

CONCLUSION

A series of antimony lead borophosphate glasses doped with Fe³⁺ ions have been prepared. In structural measurement, FT-IR spectra showed that substitution of host and modifier content has affected the bond strength and cross linking of the network as well as a shift in the frequency. The IR spectra are well related to the structure. UV-Vis spectra showed the enhancement of absorption band in UV-blue band region by an addition of Fe³⁺. Photoluminescence results exhibited that Fe³⁺ doped antimony lead borophosphate glasses have produced emission line at UV and violet band region. The prominant energy level transitions are $z^6F^\circ \rightarrow a^4D$, $z^6F^\circ \rightarrow a^2P$, and $z^6F^\circ \rightarrow a^2G$. Splitting of energy levels occurred due to the activator, Fe³⁺.

Future aim is to observe the Raman scattering and to determine the chemical bonds since Raman spectroscopy is less sensitive to the O-H bond. Moreover, the concentration of activator could be varied or the co-activate with other dopant as to optimized the luminescence intensity and characterize the optical absorption spectra. Structural solid state or crystal phase can be identified using the X-ray diffraction (XRD) technique.

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