

Elastic and Structural Properties of Potassium and Calcium – Doped Borate Lithium Glasses

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ABSTRACT

Glass sample of the system, B_2O_3 -Li₂O containing different concentration of potassium oxide and calcium oxide ranging from 0 to 10 mol% were prepared by the melt quenching method. Structure that was investigated using X-ray diffraction has indicated the amorphous state of the prepared glasses. Elastic properties of the glasses were studied by measuring the ultrasonic velocities (both longitudinal and shear velocities) and attenuation using the pulse-echo method. Measurements were carried out 2MHZ frequency at room temperature. The density of all the prepared glasses was measured using Archimedes principle. A decrease in density and increase in molar volume with increasing of K_2O and CaO content reveals that the formation of non-bridging oxygen (NBO) in the structural network. Elastic moduli, Poisson's ratio, acoustic impedance, Debye temperature, internal friction, microhardness and thermal expansion coefficient have been calculated using ultrasonic velocities, attenuation and density measurements. Furthermore, these results are interpreted in terms of the change in the topology of these glass structures. The elastic moduli are observed to decrease with the increase of K_2O and CaO content due to the decreased fractal bond connectivity

Key Words: Ultrasonic velocities, Elastic moduli, Poisson's ratio, X-ray diffraction and Debye temperature

INTRODUCTION

Glasses are amorphous materials and are transparent in the visible region. The structure of oxide glasses reveal their ionic conductivity and potential usage as solid electrolytes in a variety of electrochemical devices like solid state batteries, fuel cells, chemical sensor and smart window.⁽¹⁾ B₂O₂ glasses are found to be very interesting amorphous materials whenever the specific structure and physical properties of which taken into consideration. Borate glass is the important glass former because it has high bond strength, low cationic size and mostly used as a dielectric material.⁽²⁾ Alkali borate glasses are well known due to their high transparency, low melting point, high thermal stability, and good rare earth ion solubility.⁽³⁾ Alkali borate glasses are an important class of solid materials for various technological applications such as solid electrolytes, insulation materials, textile fibre glass, etc. These materials have been under intense research to understand the structural and physical properties through various experimental techniques. Studies of the structure of alkali

borate glasses have been reported by many investigators,^(4,5) and have provided very informative data on the nature of the network, the type concentration of a variety of boron-oxygen arrangements. Addition of various alkali modifiers, to the borate glasses brings drastic changes in the structural units. Lithium borate glasses are great interest because of their good ionic conductance properties.⁽⁶⁾ Since the addition of Li₂O to the borate glass adds extra oxygen atoms, which gets accommodated in the network, a transfer of some boron atoms from triangle BO₃ to tetrahedral BO₄ occurs. Lithium conducting glasses have large endeavour for utilization in electrochemical device, phosphors, and solar energy converters. In oxide glasses K₂O and CaO are classified as glass network modifier. This means that, in borate glasses, they modify the borate network by forming either BO, units or non-bridging oxygen ions (NBO), but they are not able to build any own structural units. The lithium borate glass containing K₂O as network modifier was seen as bubble free highly stable and moisture resistant, suitable for a systematic analysis. The calcium oxide has also been extensively used as a component ensuring

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an improvement of a number of physiochemical properties of glasses. Borate lithium glasses mixed with CaO (alkaline earth oxide) are considered as good materials for dosimetry applications since they are relatively moisture resistant when compared to the pure borate glasses.

Ultrasonic tools are very important for characterizing materials because they have many applications in chemistry, physics, engineering, biology, food industry, and medicine, etc.⁽⁷⁾ Ultrasonic technique similar to other techniques plays a significant role in understanding the structural characteristics of glass network. Ultrasonic characterization of materials is a versatile tool for the inspection of their microstructure and their mechanical properties.⁽⁸⁾ The measurement of ultrasonic parameters such as ultrasonic velocity and attenuation as a function of composition, temperature and frequency is of great interest in glass. The ultrasonic parameters besides density and molar volume as sensitive and informative about the changes occurred in the structure of glass network.⁽⁹⁾ The ultrasonic velocity, and hence, elastic properties are particularly suitable for describing glasses because they give some information about the microstructure and the dynamics of glasses. So, the ultrasonic study of the borate lithium glasses is very important as they can provide us with some idea about the glass structure.⁽¹⁰⁾ For glasses, ultrasonic investigation is very useful as besides providing information on rigidity it also can indicate a structural modification of the glass network. The measurement of elastic properties of glasses by pulse-echo method becomes a more interesting subject, due to the non-destructive nature and the high precision of the technique. The measurement yields valuable information regarding the forces operating between the atoms or ions in a solid. Since the elastic properties describe the mechanical behaviour of the materials, so the study of these properties is of fundamental importance in interpreting and understanding the nature of bonding in the solid state.⁽¹¹⁾ Hence, the elastic properties are suitable for describing the compactness of glass structure.⁽¹²⁾ To the best of our knowledge, a concurrent study on the effects of K₂O and CaO addition on elastic properties and structure of ternary 80B₂O₂-(20-x)Li₂O-xK₂O and 80B₂O₃-(20-x)Li₂O-xCaO glasses have not been previously reported.

The aim of this present work is to investigate the effect of K_2O and CaO as s partial replacement of Li_2O in the $80B_2O_3$ - $20Li_2O$, $80B_2O_3$ - $(20-x)Li_2O$ - xK_2O and $80B_2O_3$ - $(20-x)Li_2O$ -xCaO (where x = 0 to 10 in steps of 2 mol %) glass systems on its elastic and structural properties by density, ultrasonic velocity and attenuation measurements.

EXPERIMENTAL SECTION

The glass samples of the formula $80B_2O_3-20Li_2O(BL)$, $80B_2O_3-(20-x)Li_2O-xK_2O(BLK)$ and $80B_2O_3-(20-x)Li_2O-xK_2O(BLK)$

xCaO(BLC) where x = 0 to 10 in steps of 2 mol% have been prepared by the conventional melt quenching technique. Required quantities of analytical grade of B₂O₂ Li₂CO₂, K₂CO₂ and CaCO₃ are obtained from E-Merck, Germany and sd fine chemicals, India. The proper compositions were mixed together by grinding the mixture repeatedly to obtain a fine powder. The mixture is melted in alumina crucible at about 1373K for about 1 hour to homogenise the melt. The melt was quickly quenched by pouring on to a copper mould and covering with another plate and the random pieces of samples thus formed were collected. Then the glass samples were annealed at 473K for two hours to avoid the mechanical strains developed during the quenching process. The samples prepared were chemically stable and non-hygroscopic. The prepared glass samples were polished and the surfaces are made perfectly plane and smoothened by diamond disc and diamond powder. Thickness of the glass samples are measured using digital Vernier calliper (MITUTOYO DIGI-MATIC CALIPER) with an accuracy of 0.0001mm.

The amorphous nature of glass samples was confirmed by X-ray diffraction technique using an X-ray diffractometer (Model: Diffractometers de rayons X- Inel- EQUNIOX 1000) at a range of $2\theta = (10-100^\circ)$ utilizing Cu radiation with an applied voltage of 40Kv and 30mA anode current.

The density of the glass samples at room temperature was measured using Archimedes' principle. The distilled water was used as an immersion liquid. The density was calculated using the formula

$$\rho = [a / (a-b)] \rho_x$$

where, a and b are the weight of the sample in air and in distilled water. ρ_{v} is a density of the distilled water at 303K.

The molar volume V_m was calculated by using the formula

$$V_m = M_{eff} / \rho$$

where, M_{eff} is the effective molecular weight.

$$M_{eff} = \Sigma x_i M_i$$

where, x_i and M_i are the mole percentage and molecular weight of the individual component in the mixtures. Ultrasonic velocities are measured using an Ultrasonic Pulse Meter having the frequency of 2MHz. Ultrasonic velocity was also calculated using the relation

$$U = 2d/t$$

where, U is the ultrasonic velocity of the specimen (ms⁻¹), d is the thickness of the specimen (mm) and t is the transit time in micro seconds.

A technique based on the idea of pulse-echo method is used to calculate the attenuation of the ultrasonic waves propagated in the tested glasses. The ultrasonic attenuation is obtained from the measurement of amplitude decay of successive (2)

(5)

(6)

(7)

echoes observed on the screen. The attenuation coefficient α of the sample in neper per unit length is obtained from the relation

 $I = I_0 e^{-\alpha d}$

where, I_0 and I are the ratios of amplitude of the two successive echoes. The estimated accuracy in the velocity measurement is about 0.05% and that of the attenuation measurement is about 5%.

THEORY AND CALCULATION

The elastic and thermal properties of the glass specimens were investigated at room temperature by using the measured values of density (ρ), longitudinal velocity (U_1), shear velocity (U_s) and attenuation (α).

(i) Longitudinal modulus (L)

$$L = \rho U_1^2$$
(1)

(ii) Shear modulus (G) $G = \rho U_s^2$

(iii) Bulk modulus (K)

$$K = L - \left(\frac{4}{3}\right)G \tag{3}$$

(iv) Poisson's ratio (σ)

F

$$\sigma = \left(\frac{L - 2G}{2(L - G)}\right) \tag{4}$$

(v) Fractal bond connectivity (F)

$$= 4G/K$$

- (vi) Young's modulus (E) $E = (1+\sigma)2G$
- (vii) Acoustic impedance (Z) $Z = U_1 \rho$
- (viii) Internal friction (Q⁻¹)

$$Q^{-1} = \frac{\alpha}{8.66\pi f U_l} \tag{8}$$

where, α is the attenuation coefficient and f is the frequency of the quartz crystal.

(ix) Microhardness (H_y)

$$H_{v} = (1-2\sigma) \frac{E}{6(1+\sigma)}$$
(9)

(x) Debye temperature (θ_D)

$$\boldsymbol{\theta}_{\boldsymbol{D}} = \frac{h}{k_{\boldsymbol{B}}} [9N/4\pi V_{\rm m}]^{1/3} U_{\rm m} \tag{10}$$

where, h, k_B , N, V_m and U_m are the Planck's constant (6.626x10⁻³⁴JS), the Boltzmann's constant(1.38x10⁻²³ JK⁻¹), the Avogadro's number (6.023x10²³ mol⁻¹), the molar volume and mean sound velocity of the sample respectively where, $U_m = [\frac{1}{3}(\frac{2}{u_s^s} + \frac{1}{u_l^s})]^{-1/3}$ (xi) Thermal expansion coefficient (α_p)

$$\alpha_{p=23.2(U_1 - 0.57457) \tag{11}$$

RESULT AND DISCUSSION

X-ray diffraction spectrum of the studied glass systems reveal the absence of any discrete or continuous sharp crystalline peaks, but show homogeneous glassy characters. The powder X-ray diffraction spectrum of glass samples of BL, BLK3, and BLC3 are as shown in Fig 1. The experimental values of density (ρ) , longitudinal ultrasonic velocity (U_i) , shear ultrasonic velocity (U_{α}) , and attenuation (α) , of the borate lithium glasses with respect to the change in the mol percentage of K₂O and CaO used as network modifier are listed in the Table 1. The calculated values of longitudinal modulus (L), shear modulus (G), bulk modulus(K), Young's modulus (E), Poisson's ratio(σ), acoustic impedance(Z), internal friction(Q⁻¹), microhardness (H₂), Debye's temperature (${}^{\Box}D$), and thermal expansion coefficient (α_{r}) are presented in the Tables 2-3. Figures 2-18 show the variation of density, molar volume, longitudinal ultrasonic velocity, shear ultrasonic velocity, attenuation, longitudinal modulus, shear modulus, bulk modulus, Young's modulus, Poisson's ratio, fractal bond connectivity, acoustic impedance, internal friction, microhardness, Debye's temperature and thermal expansion coefficient of K₂O and CaO alkali and alkaline earth oxides doped with borate lithium glass and the curves are drawn using least square fitting.

The density and molar volume measurements are very important tool to detect the structural changes in the glass network and also it can be used to describe the network structure and arrangement of the building unit, because it deals with the spatial structure of the oxygen network. In the studied glasses, it is noted that the density decreases 2524.7 to 2079.2 and 2581.3 to 2232.7 Kgm⁻³, congruent with increase in the molar volume from 28.423 to 36.988 and 27.504 to 32.737 m³.mol⁻¹ as the K₂O and CaO content increases on the expense of Li₂O content as shown in Figs 2-3, and listed in Table 1. In general, the density and molar volume should show opposite behaviour to each other, and so in studied system the molar volume increase monotonically with the

decrease in density.⁽¹³⁾ The decrease in density with increase in K_2O and CaO is due to the increase in molar volume in comparison to the concurrent increase in molecular mass due to the Li₂O partial replacement by heavier K_2O and CaO. The molar volume will increase as a result of the formation of non-bridging oxygens (NBOs) which will break the bonds and increase space in the network.⁽¹⁴⁾ This result indicates that the glass structure becomes loosely packed. ⁽¹⁵⁾ Addition of small amounts of K_2O and CaO as a third component into the binary borate lithium glasses results in splitting of Li-O-Li and O-B-O bonds and hence, the bridging oxygens (BOs) are converted into NBOs. A further addition K_2O and CaO into glass interstices, more and more ions being open up in the network. Thus, weakening of the glass structure or reduction in the rigidity of the network takes place.⁽¹⁶⁾

Measurement of ultrasonic velocities is a beneficial tool to study the elastic properties of glasses. Further, it provides more information of structural modification and the rigidity of glass network. In the studied system, it is observed from Figs 4-5, the variation of longitudinal velocity (U₁) and shear velocity (U) decreases with increasing of K₂O and CaO contents, but the rate of increase of U₁ is greater than that of U₂. The large difference between U₁ and U₂ arises from volume effects. The change in volume due to compressions and expansions involved in longitudinal strain is pronounced while no change in volume is involved in shear strains. The decreasing behaviour of ultrasonic velocity is related to the increase in the number non-bridging oxygens which makes the glass soft and consequently the decrease in connectivity of the glass network. In general, the decrease of ultrasonic wave velocity is related to the increase in inter-atomic spacing of the material.⁽¹⁷⁾ Therefore, the decrease in ultrasonic wave velocities is due to the fact that alkali and alkaline earth metal ions are involved in the glass network as modifiers by breaking up the tetrahedral bonds of BO, units (i.e. the oxygen of alkali and alkaline earth metal ions breaks the local symmetry while the cations occupy interstitial position). It is known that the Li₂O breaks up the B₂O₂ tetrahedral network with the creation of negatively charged NBO atoms.⁽¹⁸⁾ The variation of ultrasonic velocity and attenuation with composition is related to the structural change of the glass network. The attenuation coefficient describes the total reduction in the intensity due to absorption of energy by the medium and the deflection of energy from the path of the beam by reflection, refraction and scattering. Figs 6-7 illustrates the composition dependence of the attenuation coefficient of the longitudinal and shear ultrasonic waves in the present glass system. It is clear from the figures that the behaviour of the attenuation with composition is opposite to that observed in the case of the measured density, and velocities. The continuous increase in the attenuation with increase in mol% of K₂O and CaO in the glass is the result of decreasing the rigidity (connectivity) of the network. (19)

Elastic moduli such as longitudinal, shear, Young's and bulk modulus is sensitive for any changes in the nature of the chemical bond and its strength and also it is more sensitive is exploring the change in the cross-link density which characterize the glass structure. The compositional dependence of the elastic moduli may be discussed in terms of the glass structure as the network rigidity.⁽²⁰⁾ As seen from the table 2 and Figs 8-11the elastic moduli (longitudinal, shear, Young's and bulk modulus) values decreases due to the composition effect. In general, the elastic moduli reduce when the density or ultrasonic velocities decrease. From the above observation, the decrease of these moduli is due to the increase in formation of non-bridging oxygen atoms (composition effect) that cause the formation of a loose packed structure.⁽²¹⁾ The observed decrease in the elastic properties of the studied glasses can be interpreted through two approaches. The first one is related to the breaking of B-O-B bridges and the formation of K₂O and CaO units with NBO atoms which leads to the loosening of the network structure. The second approach can be explained that the increase in molar volume would lead to the decrease in elastic moduli with the increase in modifier contents of K₂O and CaO.⁽²²⁾

Poisson's ratio can be explained on the basis of the effect of tensile stress on an oriented chain of atom or ions. If strain is lateral to the chain, its effect is maximum for lowest crosslinks. Rajendran et al (23) reported that the Poisson's ratio is affected by the changes in the cross-link density of the glass network, and the structure with high cross-link density has Poisson's ratio in the order of 0.1-0.2, while structure with low cross-link density has Poisson's ratio in the order of 0.3-0.5. In the studied glass systems, the value of Poisson's ratio (Table 2) increase and it is vary from 0.34 to 0.39. The increase in Poisson's ratio is due to breaking of network linkages and formation of smaller structural units in the glass samples.⁽²⁴⁾ Further, a low cross-link density leads to an increase in Poisson's ratio. Furthermore, from the above Table, it is seen that the values of Poisson's ratio increases with increasing of K₂O and CaO content,⁽²⁵⁾ while the fractal bond connectivity F decreases. Therefore, increasing of Poisson's ratio shows that the reducing of cross-link density due to more number of non-bridging oxygens. The Poisson's ratio values are increasing behaviour due to the increase in molar volume which means that the structure becomes more open. Therefore, the change in behaviour of Poisson's ratio as a sensitive tool for the glass compositions is attributed to the change in the type of bonding.

The acoustic impedance and internal friction (Figs 14-15) values are decreases, due to the decrease in compactness and rigidity of the structure of the glass. The behaviour of internal friction is a measure of heat produced with in a material by conversion of mechanical strain energy, when it is subjected to fluctuating stress. The smaller values of internal friction indicates the slower atomic or molecular move-

ments.^(26,27) However, the increasing behaviour of internal friction is noticed in BLC series of glasses posses higher rigidity, strength and compactness of the glass network over the BLK glasses. The Debye temperature is a very important parameter for solid materials which is related to atomic vibrations as it represents the temperature at which nearly all modes of vibrations are excited.⁽²⁸⁾ The Fig 16 shows the decreasing behaviour of Debye temperature indicates weakening of the structure and decreasing rigidity of the glass systems.⁽²⁹⁾ The observed decreases in Debye temperature in this study is mainly attributed to change in the number of atom per unit volume and also to the existence of non-bridging oxygen.⁽³⁰⁾ Microhardness (H₂) expresses the stress required to eliminate the free volume of the glass. The free volume in the glass is the openness of the glasses over that of the corresponding glasses. Srivastava and Srinivasan⁽³¹⁾ have stated that the thermal expansion coefficient of materials depends on the strength of bonds. From the Figs 17-18, shows the continuous decrease in microhardness and thermal expansion coefficient reveals the presence of non-bridging oxygen ion (NBO) and this causes the formation of soft glassy network.

CONCLUSION

Effects of concurrent addition of K₂O, CaO and partial reduction of Li₂O on elastic and structural properties of 80B₂O₂-(20-x)Li₂O-xK₂O and 80B₂O₂-(20-x)Li₂O-xCaO glasses have been studied. The amorphous nature of glasses was reflected from XRD study. The molar volume increases monotonically with the decrease in density. This results causes the formation of non-bridging oxygens (NBO_s). Ultrasonic velocities(both longitudinal and shear) values decreased with the addition of small amounts of K₂O and CaO, which is interpreted as being due to the decrease in connectivity of the network structure. Furthermore, the variation of elastic moduli, Poisson's ratio, acoustic impedance, internal friction, Debye temperature, micro hardness and thermal expansion coefficient are indicating the decrease in rigidity of the network structure. However, the increasing behaviour of internal friction is noticed in BLC series of glasses posses higher rigidity, strength and compactness of the glass network over the BLK glasses.

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Abbreviation

Non-bridging oxygen (NBO)

X-ray Diffraction spectrum (XRD)

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Table 1: Composition, measured values of density (ρ), molar volume (V_m), longitudinal velocity (U_l), shear velocity (U_c) and attenuation (α) of BL, BLK, and BLC glasses at room temperature.

Sample Label	Composition (mol %)	Density ρ/(×kg. m ⁻³)	Molar volume V _m / (×10 ⁻⁶ – m ³ .mol ⁻¹)	Ultrasonic Velocity U/(m.s ⁻¹)		Attenuation α /(nepers. unit length ⁻¹)	
				Longitudinal (U ₁)	Shear (U _s)	Longitudinal (U _l)	Shear (U _s)
B_2O_3 -Li ₂ O	(BL)						
BL	80 - 20	2334.6	30.197	2906.12	1412.28	459.66	255.32
B ₂ O ₃ -Li ₂ O-	K ₂ O (BLK)						
BLK 1	80-18-02	2524.7	28.42	2684.62	1396.00	588.57	388.65
BLK 2	80-16-04	2437.6	29.97	2646.15	1333.33	541.95	752.24
BLK 3	80-14-06	2403.3	30.93	2638.46	1323.08	525.39	712.08
BLK 4	80-12-08	2323.2	32.55	2615.38	1254.55	515.32	725.55
BLK 5	80-10-10	2079.2	36.99	2603.77	1106.45	493.13	766.33
B ₂ O ₃ -Li ₂ O-	CaO (BLC)						
BLC 1	80-18-02	2581.3	27.50	2883.33	1383.67	347.69	707.31
BLC 2	80-16-04	2506.3	28.54	2841.67	1207.14	540.98	810.20
BLC 3	80-14-06	2455.9	29.34	2816.67	1155.95	604.38	841.83
BLC 4	80-12-08	2356.4	30.79	2712.00	1116.13	673.75	875.59
BLC 5	80-10-10	2232.7	32.74	2664.00	1091.80	703.72	931.84

Table 2: Values of elastic moduli, Poisson's ratio, and fractal bond connectivity of BL, BLK and BLC glasses at room temperature.

Sample Label	Longitudinal Modulus L/(×10 ¹⁰ N.m ⁻²)	Shear Modulus G/ (× 10 ¹⁰ N.m ⁻²)	Bulk Modulus K/(×10 ¹⁰ N.m ⁻²)	Young's Modu- lus E/ (×10 ¹⁰ N.m ⁻²)	Poisson's Ratio (σ)	Fractal bond connectivity (F)	
B ₂ O ₃ -Li ₂ O (BL)							
BL	1.9716	0.4656	1.3509	1.2528	0.3454	1.379	
B_2O_3 -Li_2O-K_2O (BLK)							
BLK 1	1.8195	0.4920	1.1636	1.2935	0.3146	1.691	

BLK 2	1.7068	0.4333	1.1290	1.1524	0.3298	1.535
BLK 3	1.6728	0.4207	1.1119	1.1207	0.3320	1.513
BLK 4	1.5891	0.3656	1.1017	0.9874	0.3505	1.327
BLK 5	1.4096	0.2545	1.0703	0.7070	0.3890	0.951
B ₂ O ₂ -Li ₂ O-CaO (BLC)						
BLC 1	2.1459	0.4942	1.4870	1.3346	0.3503	1.329
BLC 2	2.0238	0.3652	1.5369	1.0151	0.3899	0.950
BLC 3	1.9484	0.3281	1.5110	0.9206	0.4030	0.869
BLC 4	1.7331	0.2935	1.3418	0.8206	0.3980	0.875
BLC 5	1.5845	0.2661	1.2298	0.7445	0.3990	0.866

Table 3: Values of acoustic impedance (Z), internal friction(Q⁻¹), microhardness (H_v), Debye temperature (θ_D) and thermal expansion coefficient (α_p) of BL, BLK and BLC glasses at room temperature.

Sample Label	Acoustic Impedance Z /(×10 ⁷ kg, m ⁻² s ⁻¹)	Internal Friction (Q ⁻¹) /(×10 ⁻¹¹ dB.s ² m ⁻²)	Micro Hardness H _v /(×10º N.m ⁻²)	Debye Temperature θ _D /(K)	Thermal Ex- pansion Coefficient α _n /(×10² m.s ⁻¹)				
B_2O_3 -Li ₂ O (BL)									
BL	0.6784	290.85	0.4795	184.82	674.08				
B_2O_3 -Li_2C	B_2O_3 -Li ₂ O-K ₂ O (BLK)								
BLK 1	0.6777	403.13	0.6077	185.66	622.69				
BLK 2	0.6450	376.58	0.4915	174.57	613.77				
BLK 3	0.6341	366.15	0.4710	171.46	611.98				
BLK 4	0.6076	362.31	0.3641	160.24	606.63				
BLK 5	0.5413	348.25	0.1882	136.15	603.94				
B_2O_3 -Li_2O-CaO (BLC)									
BLC 1	0.7442	221.74	0.4931	186.92	668.79				
BLC 2	0.7122	350.05	0.2679	184.50	659.13				
BLC 3	0.6917	394.55	0.2120	153.85	653.33				
BLC 4	0.6390	456.81	0.1995	146.15	629.05				
BLC 5	0.5947	485.72	0.1789	140.10	617.91				



Figure 1: The powder X-ray diffraction spectrum of glass samples of BL, BLK3, and BLC3 at room temperature.









