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Original article

Effect of Bi_2O_3 on electrical properties of lithium borate and lithium borosilicate glass systems

AL-Sh.Ramadan[a], Sh.Neseem[a], M. Farouk[b], E.Nabhan[a].

[a] *Physics Department, Faculty of Science, (Girls') branch ,Al-Azhar University, Nasr City 11754 Cairo, Egypt .*
[b] *Physics Department, Faculty of Science, (Boys') branch, Al-Azhar University, Nasr City 11884 Cairo, Egypt.*

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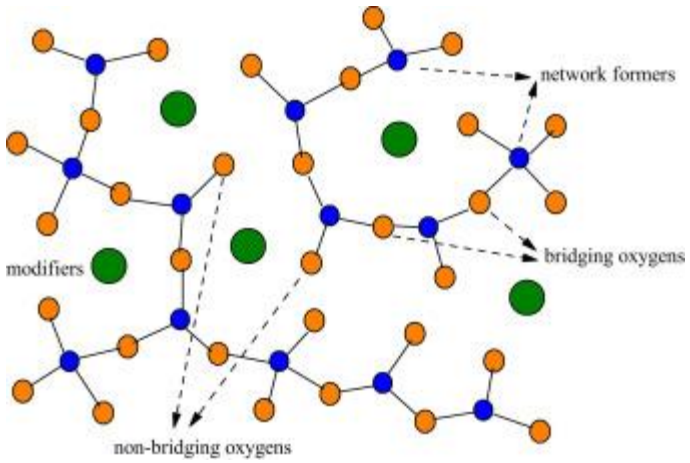
ABSTRACT

This study explores the dielectric characteristics and alternating current conductivity of bismuth glasses that contain borate and borosilicate, which are doped with a fixed amount of CoO and Li_2O, while the concentration of Bi_2O_3 varies $((59-x)B_2O_3-xBi_2O_3-40Li_2O-1CoO)$&$((49-x)B_2O_3-xBi_2O_3-10SiO_2-40Li_2O-1CoO$) where x=(0,5,10,20). The glass samples were synthesized by the normal melt quench technique. The electrical conductivity and dielectric response have been analyzed in the range of frequencies (0.1Hz- 10^8Hz) and temperature range (303K– 463K) by employing impedance spectroscopy technique with the aim of knowing the effect of Bi^{+3} ions on ac conductivity and dielectric properties. It was found that the presence Bi^{+3} facilities Li^+ ions movement because of dual role of Bi^{+3} as glass modifier in $[BiO_6]$ octahedral units and as glass former with $[BiO_3]$ pyramidal units which modify the glass matrix structure and as a result affect on electrical conductivity (creating non-bridging oxygen which leads to open pathways in glass matrix for ion migration). Ac conductivity of all prepared glass systems was well understood on the basis of Jonsher's universal power low with The frequency exponent (s) reduces as the temperature increases suggesting that the Cor-related Barrier Hopping model is the model for clarifying conduction mechanism of the studied glass systems. In the low frequency range, electrical conductivity is independent of frequency having nearly flat plateau region as frequency increases, dispersion starts. It was found that the increase in frequency is matched by a decrease in dielectric constant (ε`) and dielectric loss (ε``) for all examined glass specimens. The peak frequency f_{max} can be illustrated from dielectric modulus spectra.

Graphical abstract



∗ *Corresponding author*
E-mail address: alshaymaaramadan@yahoo.com

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1. Introduction

Ionic transport in oxide glass materials is widely used in solid state electrolytes for example in all rechargeable-solid-state batteries, fuel cells, sensors and laptops. Because glassy materials have distinctive properties which make them utilized in technological applications. They are superior to their crystalline counterparts due to high melting point, isometric structure high chemical resistance, hardness, transparency, corrosion resistance and lack of grain boundaries [1-8].

Because of their high energy density, safety, chemical and thermal stability, lack of electron leakage, extended cycle life; low self-discharge, broader operating potential and temperature window, and reduced flammability, solid electrolytes are widely used compared to liquid electrolytes. Because there are no gaps in the glass materials, which results in greater ion transport, Electrolytes based on glass have shown significant efficiency among other solid electrolytes. As a result, glass-based electrolytes now have greater benefits than ceramic-based one. These characteristics of glassy materials provide a steadier and more consistent route for ion movement and improving their ability to conduct electricity and their effectiveness. Ionic conductivity relays on the movement and concentration of ions. The most important feature of glassy- based electrolytes is the ability of alteration the physical properties by changing composition that cover the demand for various applications [9].

Among glass formers, Borate glasses have higher forming ability in addition to unique characteristics for instance high thermal stability and chemical durability which make them the first choice in many technological applications like in solar energy converters, supersonic conductors, mechanical and thermal detectors etc[10].

Borate glasses including heavy metal and transition metal are of significant concern because of their different available implementations. Boron trioxide (B_2O_3) is defined as excellent glass former and is described by many structural units [11, 12]. Units like diborate, triborate, and tetraborate introduce the borate glasses as the favorite host medium for both heavy metal and transition metal oxides [13-16]. Unlike others heavy metal oxides, Bi_2O_3 has been defined as an unorthodox glass former due to the weak field strength of Bi^{3+} ions and possessing applicable physical properties such as high density, high polarizability, special electric characteristics, high thermal stability a wide range of transparency [17-18]. The two possible structure of Bi_2O_3 in glass networks are coordination [BiO_3] pyramidal and [BiO_6] octahedral units due to its dual function, BiO_6 as modifier and BiO_3 glass former with structural units [12]. For these merits, borate glasses with Bi_2O_3 oxides gain spread usage especially in technological applications such as superconducting materials reflecting windows and thermo-mechanical sensors [19].This dual role makes Bi^{3+} ions are expected to alter the electrical characteristics of oxide glasses. Replacement of B with Bi leads to reduction of glass activation energy [20]. Bi_2O_3 quantity has important effect on the glass system conductivity [21, 22].

By the addition of alkali ions (as Li^+,K^+,Na^+),bismuth borate glasses conductivity will be enhanced and they will be suitable for use in sensors and batteries as solid electrolytes with high energy densities characterized by thermal stability, wide available composition ranges, isotropic conductivity, and ease of fabrication.

Lithium borate glasses systems used as solid electrolytes in storage batteries due to significant features like compact size, lightweight nature, and the highly electropositive nature of lithium ions, which contribute to generating high voltage and high energy density in microbatteries. Li^+ ions are believed to take up interstitial sites and balance the extra- negative charge in B_2O_3, and their higher concentrations enhance the glass electrical conductivity because of their small ionic radius; it has been observed that Li^+ forms ionic bonds with non-bridging oxygen. The conduction mechanism for Li^+ ions is influenced by the structural changes resulting from the addition of Li_2O into the B_2O_3 glass network. When Li_2O is added, the trigonal BO_3 units within the boron trioxide B_2O_3 glass begin to transform into tetrahedral BO_4^- up to approximately 30 mol% of Li_2O, and exceeding this concentration results in the non-bridging oxygen formation (NBO) within the glass network. This phenomenon was referred as "boron anomaly" [23]. This behavior is noticed from structure investigation by IR-Spectroscopy and from calculating N_4 (the proportion of four coordination boron atoms) from IR-data it has been found that N_4 decreased which means more open structure [24].

In 1991, Sony Corporation launched lithium-ion batteries designed to energize portable electronic devices like laptops, mobile phones, computers, and more [25].

Lithium borosilicate glasses possess a unique structure due to the inclusion of two glass-forming components. When SiO_2 is added as the second glass former in a lithium borate binary glass, it can lead to greater depolymerization of the borate glass, as SiO_2 acts as a network modifier. This modification results in increased the proportion of orthoborate or pyroborate units, that could increase the conductivity of the ternary glass containing a small fraction of SiO_2 [9]. According to Tatsumisago et al. [26], lithium borosilicate glasses electrical conductivity would be enhanced through the combination of two glass formers, like B_2O_3 and SiO_2, due to the phenomenon known as the mixed former effect. Lee et al. [9].

Due to structure modification is a main influence point affecting ionic conduction; we are going to synthesis two glass systems (bismuth borate & bismuth borosilicate) with fixed proportion of lithium and cobalt varying Bi_2O_3 concentration to investigate the effect of adding alkali oxide, heavy metal oxide and mixing two glass formers on conductivity.

This study will be performed using Impedance Spectroscopy (IS) as it is an effective approach for evaluating

various electrical characteristics and the relaxation processes of materials and their interfaces [27]. This technique proves to be highly beneficial for exploring the bound dynamics and free charge within the bulk and surface areas of the glass matrix, as well as for examining the ionic, semiconducting, mixed electronic–ionic conductivity, and dielectric properties of these materials.

Understanding the conduction mechanisms within these disordered materials offers deep understanding of the processes of system dynamics and transport. This study will examine several dielectric properties, including ac conductivity, loss factor, and dielectric constant throughout wide range of temperatures and frequencies, providing insights into the conduction mechanisms of the glasses and shedding light on their structural characteristics.

2. Experimental:

2.1. Preparation of glasses:

Lithium bismuth borate and lithium bismuth borosilicate glass systems doped with 1% of CoO were synthesized by the traditional melt quenching method. The starting materials have been used were high purity materials of [Li_2CO_3, H_3BO_3, CoO, SiO_2, and Bi_2O_3]. The chemical composition (mol %) of glass systems details are in table (1) and named as BBiLC and BBiSLC, respectively.

Table (1) the chemical composition (in mol %) details of glass systems.

Sample No.	**((59-x)B_2O_3-xBi_2O_3-40Li_2O-1CoO)**				
BBiLC0	59B_2O_3	0 Bi_2O_3	0SiO_2	40Li_2O	1CoO
BBiLC 5	54B_2O_3	5 Bi_2O_3	0SiO_2	40Li_2O	1CoO
BBiLC 10	49B_2O_3	10 Bi_2O_3	0SiO_2	40Li_2O	1CoO
BBiLC 20	39B_2O_3	20 Bi_2O_3	0SiO_2	40Li_2O	1CoO
	((49-x)B_2O_3-xBi_2O_3-10SiO_2-40Li_2O-1CoO)				
BBiLSC0	49B_2O_3	0Bi_2O_3	10SiO_2	40Li_2O	1CoO
BBiLSC5	44B_2O_3	5Bi_2O_3	10SiO_2	40Li_2O	1CoO
BBiLSC10	39B_2O_3	10Bi_2O_3	10SiO_2	40Li_2O	1CoO
BBiLSC20	29B_2O_3	20Bi_2O_3	10SiO_2	40Li_2O	1CoO

Batches corresponding to approximately 15 g were mixed homogeneously through ground in an agate mortar and melted in a porcelain crucible in an electrical muffle furnace until reaching 1100°C and keeping steady at this temperature for about 90 min to allow a complete homogenization of the melt and acquired the desirable viscosity. The molten was stirring several times to ensure greater homogeneity. The free-bubble melts were subsequently rapidly cooled (quenched) at room temperature by being poured and pressed between two pre-heated copper plates (poured on a copper plate and another copper plate was put on the first one, i.e., sandwiching) to obtain thin disk-shaped glass samples with smooth and flat surfaces by polishing perfectly by emery paper for dielectric measurements. The samples are covered with silver paste to guarantee effective contact between the surfaces of the samples and the stainless steel electrodes of the capacitor cell. Using porcelain crucibles and low melting temperatures are important features.

2.2. Dielectric measurements

The electrical conductivity and dielectric properties are studied by a computerized LRC meter (Hioki model 3553 Japan (0.1Hz-10^8Hz) as a broadband spectroscopy) and over temperature range of (303–463) K at intervals of 40 K.

The A.C conductivity and dielectric constant were calculated as reported in literature [28-30] from:

$$\sigma_{ac}(\omega) = 2\Pi f(L/a)C\ D \quad (1)$$

Where f is the frequency in Hertz, L represents the sample thickness measured in meters, while (a)denotes the sample cross-sectional area, C is the capacitance in farad and D is loss factor.

To assess these characteristics at various temperatures, the sample is positioned between two electrode cells and then placed inside a non-inductive furnace, which heats the samples at a consistent rate. The temperature of the samples is monitored using a thermometer that is in contact with the sample.

The dielectric constant is given by [31]:

$$\varepsilon` = \frac{CL}{a\varepsilon_o} \quad (2)$$

Where ε_o ($8.854 * 10^{-12}$ Fm^{-1}) refers to the permittivity of free space.

An alternative method for examining the glasses electrical response involves analyzing the findings through the real (M`) and imaginary components (M``) of the electric modulus M* (the electric modulus formalism has the benefit of extraction the material bulk response occurring during relaxation process in the glass) as detailed in references [32-35]:

$$M^* = 1/\varepsilon^* = M` + j\ M`` \quad (3)$$

$$M` = \varepsilon`/ \varepsilon`^2 + \varepsilon``^2 \quad (4)$$

$$M`` = \varepsilon``/ \varepsilon`^2 + \varepsilon``^2 \quad (5)$$

In the given context, M′ represents the real component of the complex electric modulus M, while M″ denotes its imaginary component.

3. Result

Dielectric studies

The dielectric characteristics of electrolytes that are based on glass have drawn significant interest from researchers as they offer valuable insights into the material's internal mechanisms for energy storage and various electronic applications.

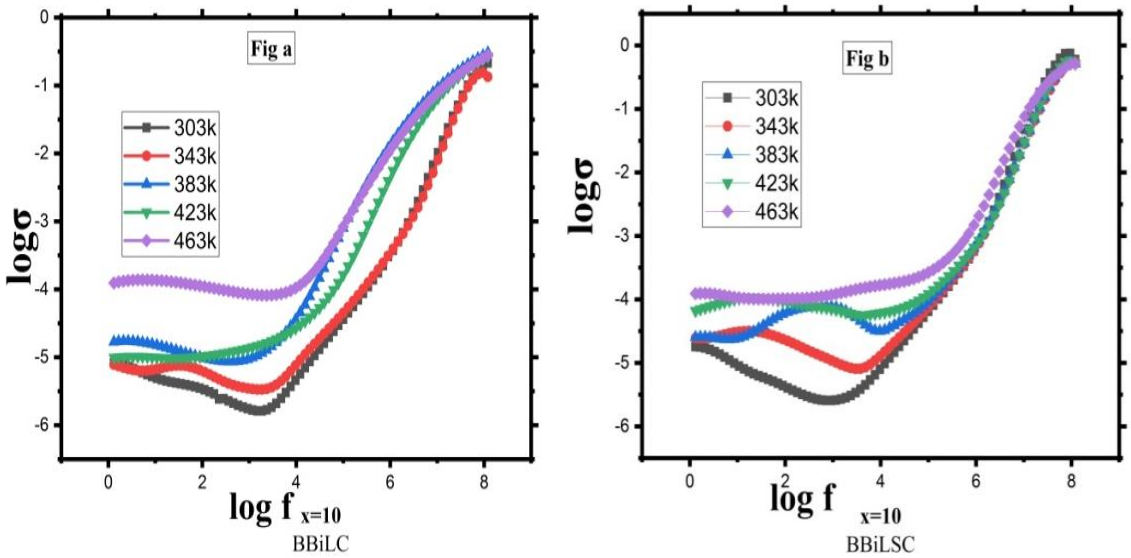
3.1. Ac conductivity

The frequency-dependent electrical conductivity $\sigma_{ac}(\omega)$ in solids is attributable to the current inflow through the hopping of localized charge carriers, which can be either electrons or ions. For the examined samples, the conductivity $\sigma_{ac}(\omega)$ of each glass composition is examined in relation to both frequency and temperature. The sample that contains 10 mole% of Bi_2O_3 serves as a representative example, as illustrated in Fig (1 –a,b).

The obtained ac conductivity is distinguished by two separate parts: the first part recognized at lower frequencies and referred to as the plateau region, remains constant with frequency. The second part, at higher frequencies, shows an increase in conductivity as the frequency rises, known as the dispersion region.

At low frequencies, σ exhibits a frequency-independent characteristic (plateau nature), which results in dc conductivity (σ_{dc}), stemming from the random diffusion of ionic charge carriers through an activated hopping mechanism (long-range order transport and effective diffusion of mobile ions in response to the applied electric field) [36-37]. This phenomenon arises from electrode polarization due to the buildup of mobile ions at the interface. The accumulation of this space charge is more pronounced at lower frequencies. As a result, the overall conductivity (σ) of the sample diminishes as the frequency decreases. A transition from the frequency-independent flat region to higher frequencies signifies the beginning of the conductivity relaxation phenomenon.

At elevated frequencies, σ exhibits a dependence on frequency that leads to ac-conductivity $\sigma_{ac}(\omega)$. In this situation, $\sigma_{ac}(\omega)$ increases approximately following a power law (Jonscher's universal power law); $\sigma_{ac}(\omega) = A\omega^s$ and ultimately approaches linearity at temperatures that are even greater. Here, A is a temperature-dependent constant and s is a frequency exponent that varies with temperature.



Fig(1-a,b) variation of ac conductivity with frequency for different temperatures for x =10 for both BBiLC& BBiLSC glass systems.

As the temperature rises, the motion of mobile ions intensifies, leading to an overall increase in the conductivity of BBiLC and BBiLSC glass systems, as shown in figures (1-1a, b).These findings align with earlier studies on various semiconducting glasses and amorphous materials [38-39].

From the above figures (2-a, b), at lower temperatures, the conductivity remains almost constant with respect to temperature. In contrast, at elevated temperatures, the conductivity becomes dependent on temperature, leading to an increase in conductivity.

The activation energy (E_{dc}) is determined from the slope of the linear regression of log σ against 1000/T in the low temperature range for all glass samples prepared, as illustrated in figures (3 – a,b) using the Arrhenius equation:

$$\sigma = \sigma_o \exp(E_{dc}/K_B T) \quad (6)$$

In the given equation(6), σ_o represents the pre-exponential factor that is influenced by the characteristics of the glass, E_{dc} refers to the energy required for thermal activation in electrical conduction, and k stands for Boltzmann's constant[40-41].

The activation energy values (E_{dc}) demonstrate a declining trend as the concentration of Bi_2O_3 rises, suggesting an enhancement in conductivity [42].

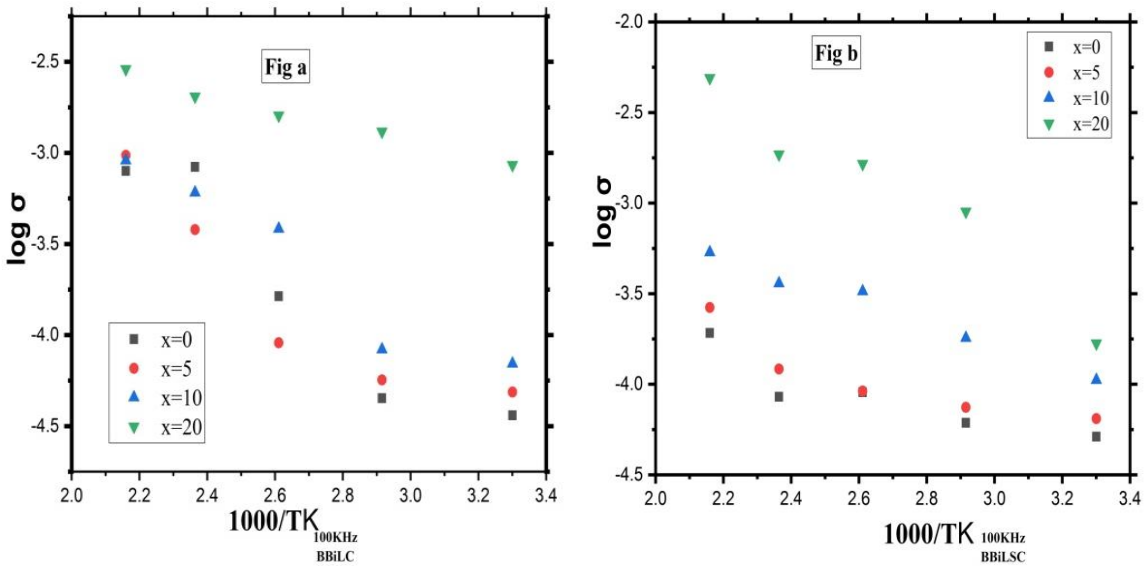


Fig (2- a,b) Variation of ac conductivity (log σ_{ac}) with the inverse of temperature at different composition & f=100KHZ for both BBiLC& BBiLSC glass systems.

The differences in E_{dc} values observed with varying amounts of Bi_2O_3 can be explained by the structural modifications taking place in the analyzed glasses, which favor the rise in non-bridging oxygen (NBO) ions[24- 43].

Numerous models, including the quantum mechanical tunneling model (QMT), correlated barrier hopping model (CBH), small polaron hopping (SPH), and the overlapping large polaron tunneling (OLPT) model, have been suggested by various investigators to explore the conduction mechanism by examining how the frequency exponent 's' changes with temperature [44-47].

Based on hopping theory, when hopping occurs between localized states that are randomly distributed, $\sigma_{ac}(\omega)$ is directly proportional to ω^s, with the condition that $0.5 < s < 1$. It is understood that lower s values suggest a multi-hopping process, whereas higher s values indicate a single hopping process [48].

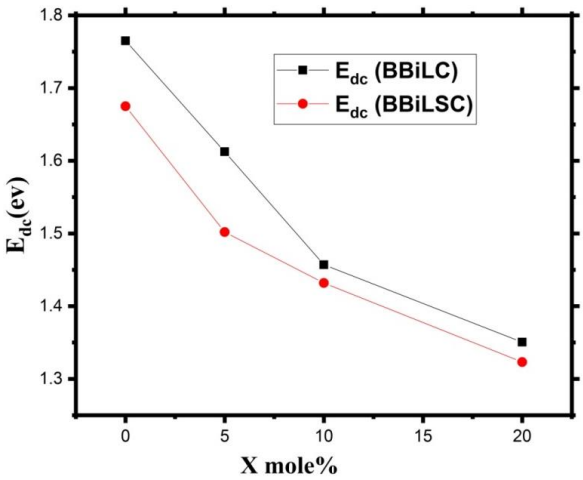
For our studied glass systems (BBiLC&BBiLSC) ac conduction follows correlated barrier hopping model (CBH) as exponent 's' is influenced by temperature (s decreases as the temperature increases) as shown in fig (4-a,b).

3.2. Dielectric properties:

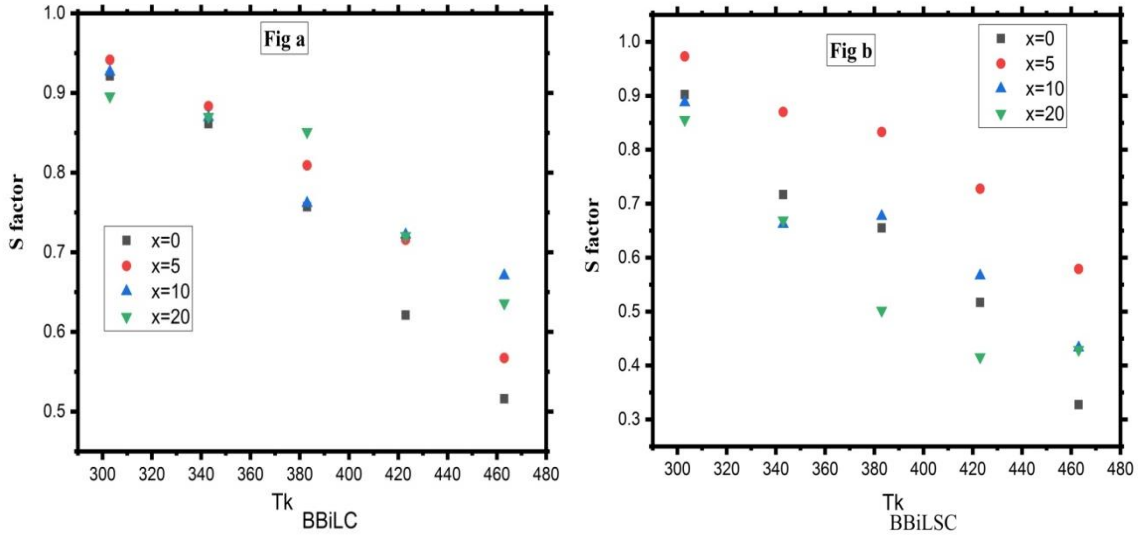
To advance our inquiry, we analyzed the dielectric properties of the studied glass samples.

3.2.1. Dielectric constant:

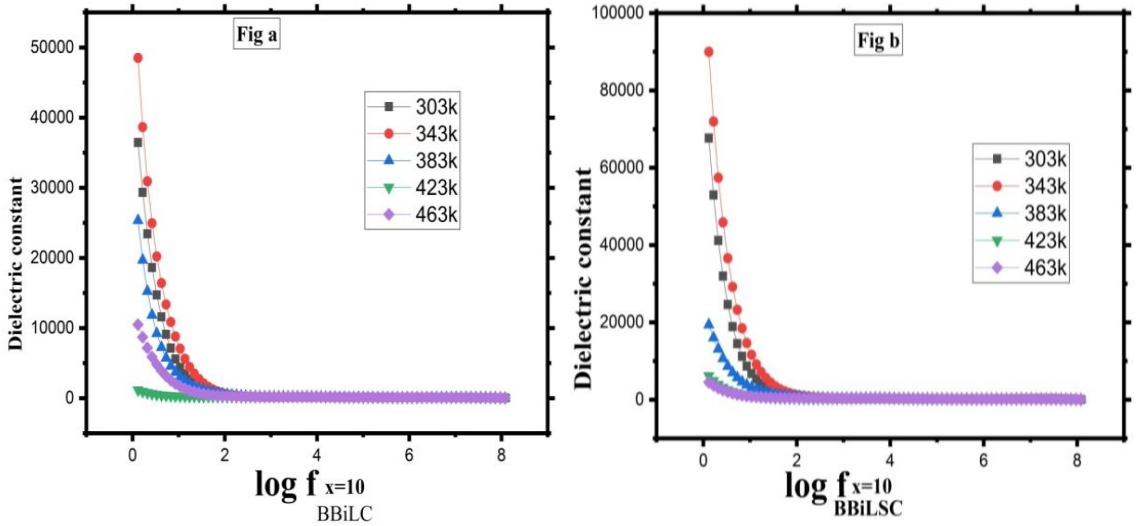
As the frequency increases, ε' reduces and reaches a steady value as illustrated in fig (5-a, b). Such results suggest that at elevated frequencies, AC field periodic reversal prevents charge accumulation at the interface (dipoles can no longer rotate adequately), causing their oscillation to lag behind the applied field, that results in the observed reduction in dielectric, and as the frequency increases, ε' stays steady[49-51]. The movement of charge without accumulation can be understood through the ion diffusion process. The rise of the dielectric constant ε' as frequency decreases could be linked to the effect of charge accumulation at the interface, resulting in a net polarization of the ionic medium and the establishment of a space charge region at the electrode-electrolyte interface [52-54]. The rise in permittivity can largely be attributed to enhanced ionic movement in these glasses when mixed transition metal ions are substituted [55]. The Bi^{3+}-ions (heavy metal cations) have significant polarizability, a result of their large ionic diameter and weaker relative bond/field strengths, which likely explains the high values of ε′ observed [55-56].



Fig(3-a,b)The compositional variations in E_{dc} with Bi_2O_3 content



Fig(4-a,b)Thermal variation of the exponent s for x =10 for both BBiLC& BBiLSC glass systems.



Fig(5-a,b) variation of Dielectric constant with frequency for different temperatures for x =10 for both BBiLC& BBiLSC glass systems.

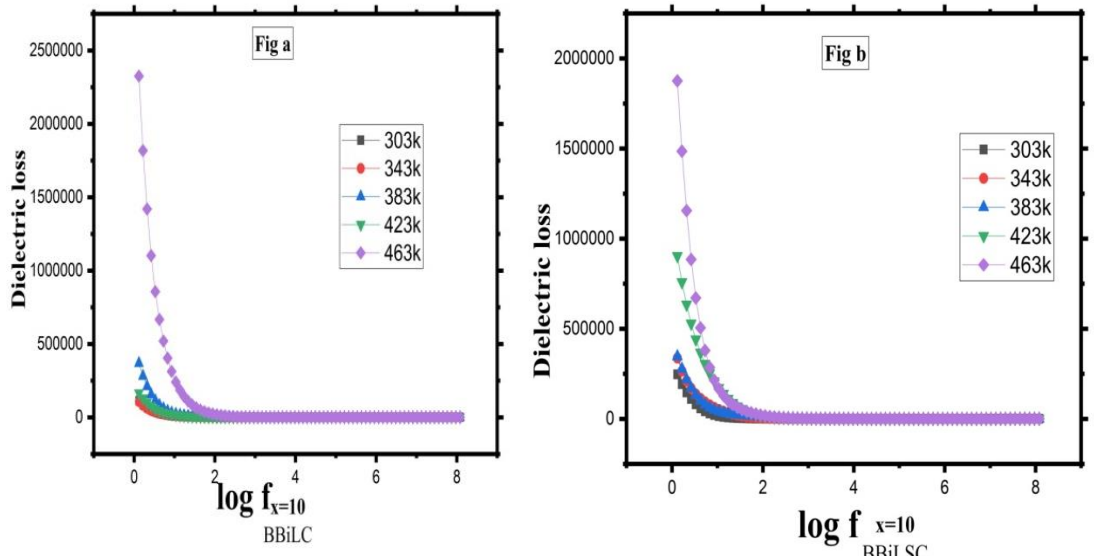
3.2.2Dielectric loss:

In the studied glass sample, at low frequencies, conduction losses are the most significant, and there is a noticeable increment in ε`` as the temperature rises. Nevertheless, the graph of ε`` against frequency does not show any peak, making it challenging to extract information about the frequency of the dielectric loss peak. Consequently, the data were transformed into modulus formalism. This approach has the benefit of diminishing the impact of electrode effects, allowing for the study of conductivity relaxation [49-51, 57-58].

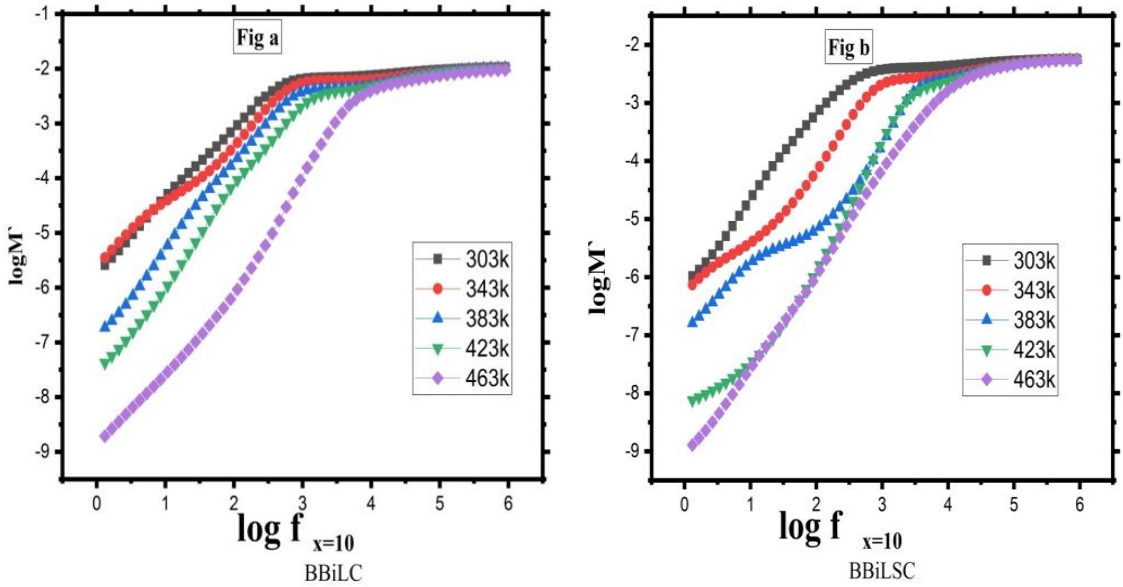
3.2.3Electrical modulus:

A modulus formulation is essential for a deeper knowledge of the conduction mechanism. The framework of the complex electric modulus could assist us in identifying the bulk response of conductive materials.

Figures (7-a, b) illustrate how the real component of the modulus spectra varies with frequency at various temperatures. The real component of the dielectric modulus M` shows minimal values at low frequencies, suggesting that electrode polarization has little effect on M*. The dispersion observed in M` at lower frequencies can attributed to the ease with which conducting ions can migrate; in other words, this dispersion is primarily a result of conductivity relaxation. As the applied field's frequency rises, M` levels off to a steady value [59]. It is evident from figures (8-a, b) that the imaginary component of the dielectric modulus, M``, shows a low value at lower frequencies, likely because of the high capacitance linked to the electrodes.

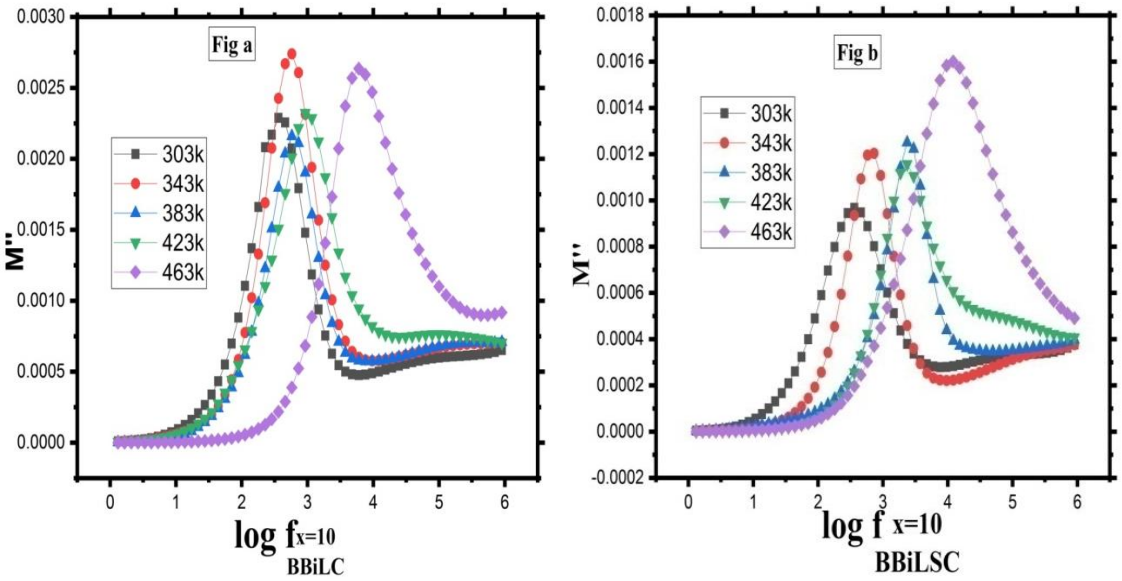


Fig(6-a,b) variation of Dielectric loss with frequency for different temperatures for x =10 for both BBiLC& BBiLSC glass systems.



Fig(7-a,b) variation of real part of electric modulus (M`) with frequency for different temperatures for x =10 for both BBiLC& BBiLSC glass systems

The M`` graph displays a somewhat asymmetric peak at every temperature. With an increase in temperature, the peak position moves towards higher frequencies, and the frequency at which M`` reaches its maximum is referred to as the relaxation frequency, f_M. The peaks exhibit asymmetry on both sides of the maxima. The frequency range below the peak frequency f_M indicates where charge carriers can move over extended distances, while the reduction of M″ values beyond f_{max} in the high-frequency area suggests that mobile ions are becoming restricted to their potential wells. Therefore, the peak frequency signifies a transition from long-range mobility to short-range mobility. The shift in f_{max} with temperature implies that the hopping of charge carriers is influenced by the weakening of bonds, which leads to reduction in relaxation time ($\tau_m \alpha\ 1/f_{max}$)[60-61].



Fig(8-a,b) variation imaginary part of electric modulus (M`) with frequency for different temperatures for x =10 for both BBiLC& BBiLSC glass systems.

4. Conclusion:

All glass samples were perfectly prepared using the traditional melt-quench technique. The ac conductivity adheres to Jonscher's power law (JPL). Based on the dependence of the's' values on temperature, it appears that charge conduction occurs due to the correlated barrier hopping model (CBH). Both the conductivity and dielectric constant increase with a higher concentration of Bi_2O_3 due to enhanced polarizability and non-bridging oxygens (NBO), and the electrical conductivity is predominantly ionic owing to the presence of Li^+ ions. The conductivity values for the examined glasses reached approximately 10^{-1}S/cm in the range of frequencies (0.1Hz- 10^8Hz) and temperature range (303K– 463K). The conductivity values for the BBiLSC system are higher than those for the BBiLC system, reinforcing the idea that mixing two glass formers enhances conductivity. The electric modulus of the studied glass samples shows an asymmetric peak in the imaginary part (M″), indicating a non-Debye type of relaxation. The transport mechanisms are also elucidated based on modifications in glass structure as the amount of Bi_2O_3 was determined to be crucial for the conductivity of this glass system as Bi_2O_3 is increased by reducing the amount of B_2O_3 this alters the dielectric characteristics of the investigated glass systems. The resulting glass samples could be utilized as solid electrolytes in many technological applications.

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