**Physical, Optical and Structural studies of Molybdenum doped B2O3-CdO-Al2O3-PbF2 glasses**

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**Abstract:**

The Preparation and thorough physical and structural analysis of B2O3-CdO-Al2O3-PbF2 glasses supplemented with MoO3 ions are the topics of this publication. The creation of NBOs in these glasses was confirmed by the incorporation of MoO3, which resulted in an overall reduction in density and band gap parameters and a boost in refractive index. This transformation of Mo6+ ions to Mo5+ ions was confirmed. It was discovered that there were many borate and aluminate components in the FTIR spectrum. The FTIR spectra also showed the transformation of Mo6+ ions into Mo5+ ions. The transformation of BO4 to BO3 units, which increased NBOs and assisted in explaining the density change, was evident in the influence of MoO3 ions on Raman spectra.

**Keywords** Borate glasses, Refractive index, Optical absorption, FTIR, Raman spectra

**1. Introduction**

Because of the growing significance of glasses for both daily necessities and numerous other uses, researchers have been forced to enlarge their field perspective in a variety of chemical compositions containing borate, silicate, phosphate, and other materials [1, 2]. Borate is generally used as a glass-forming material due to its low melting point, clarity, thermal stability, and solubility [3, 4]. The incorporation of Al2O3 into the borate matrix results in significant modifications to its optical, chemical, and physical properties. AlO4 or AlO6 units can be found in the glass network, based on the amount of Al2O3. Elevating the proportion of Al2O3 can significantly enhance its mechanical strength, chemical resistance, and moisture resistance ability. Additionally, aluminum borate glasses can combine with transition metal ions (TMI) and other metal oxides [5-7].

One of the most common metal oxides utilized to boost density in glass compositions is CdO. It is also widely recognized that CdO is utilized as control rods in nuclear reactors. Borate glasses are treated with fluorides to increase their transmission window, solubility, and fluorescence longevity, among other benefits [7-9]. PbF2 is included in borate glasses to enhance certain optical properties and infrared transparency, which are used in laser engineering and communication systems [10].

However, it was found that molybdenum ions are excellent catalysts for examining catalytic properties since they show remarkable activity and selectivity in a variety of oxidation activities in different oxide glasses. The presence of molybdenum ions in various valence states (+3, +4, +5, and +6) could potentially enhance the optical, electrical, and electrochromic properties of the glasses. The presence of molybdenum ions in multivalence states enhances the electronic contribution to conduction because of electron hopping around molybdenum ions [11–13].

According to their analysis of MoO3's mol%, Ali et al. [19] determined that MoO3 had a former and moderating effect in PbO–Sb2O3–As2O3– MoO3 glasses. Alrowaili et al. [20] synthesized the P2O5–B2O3–Li2O– MoO3 glasses and found that when the MoO3 proportion increased, the Urbach energy decreased and the density, refractive index, metallization criterion, and shielding ability increased. Other researchers, including Shaaban et al. [21], Rada et al. [22], Manisha et al. [23], and others, created glasses with different ratios that included MoO3, and they verified that the glasses that included MoO3 had superior uses concerning optical and shielding properties. Because of this, glasses may be formed when borate and MoO3 combine. Moreover, recent studies have demonstrated that the glass network is significantly altered by the injection of even a tiny quantity of TMI into glasses. Furthermore, the best features, aside from the best optical endowments, are revealed in borate doped cadmium alumina matrix with varying mole percentages of metal fluoride and transition metal ions, for electronic, optoelectronic, and electrochromic applications.

**2. Experimental**

The glass specimens, where x = 0, 0.5, 1, 1.5, and 2mol%, were made with an unusual mixture of 60B2O3-20CdO-5Al2O3-(15-x) PbF2-x MoO3. Melt quenching was the technique used in the creation of glass samples. Every necessary chemical was of annular grade and weighed according to the necessary mole proportions for five different sets of glass combinations. The different mole percentages of the compounds were physically combined in an agate mortar until they were evenly blended, after which they were calibrated using an electronic monopan balance. This chemical mixture was placed in a porcelain crucible and allowed to melt uniformly for one hour at a temperature of 1000°C in an electronic furnace that operates under standard atmospheric conditions. Further melts are swirled to gain homogenization. After the resultant melt was quenched for a few hours at 200°C on a steel plate to relieve internal tensions, the glass samples were prepared for additional examination.

To determine the nature of the samples, X-ray diffraction (XRD) spectra are obtained using a Philips Xpert Pro X-ray diffractometer between 10o and 80o for 2θ. Next, density is calculated using the Archimedes principle with xylene serving as the dipping liquid. Three experimental fragments were taken out of each glass to be measured for density; the final density was calculated using the aggregate density of the three sample pieces. The computation's accuracy is 0.005 g/cc. Using a Bruker FTIR spectrometer, Fourier transform infrared (FTIR) spectra were captured with a precision of 0.01 cm-1 and a resolution of 0.5 cm-1. The Raman spectra of BCAPM glasses were obtained in the 200 –1800 cm-1 wavenumber range using a J. Y. H. LABRAM-HR Raman spectrometer. A wavelength range of 200nm to 1000nm was covered by scanning the UV-Visible spectra of BCAPM glasses using a Shimadzu UV-1800 Spectrometer in absorption mode. Using a Bruker EPR spectrometer, EPR spectra were obtained at room temperature between 2500 and 4000G.

**3. Results and discussions:**

**3.1. X-Ray Diffraction (XRD)**

The BCAPM glasses patterns from an XRD scan are displayed in Figure 1. The illustration unequivocally shows broad humps around 2θ = 20°C and 30°C. Moreover, the lack of distinct peaks suggests that the BCAPM specimens are non-crystalline.



**Figure 1: XRD Spectrum of BCAPM glasses**

**3.2. Physical and optical properties**

Density is indeed an important physical property that provides information about solids; nevertheless, the more clearly the structure is known, the more useful this evidence will be [24, 25]. The density information of the samples can be used to calculate the volume of structural units embedded in the glass.

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**Figure 2: Variation of density and molar volume with MoO3 mole percentage in BCAPM glasses**

Density values in the BCAPM glass system were nonlinearly decreased from 4.072g/cc (BCAPM0) to 4.009g/cc (BCAPM4) in the current system. Using the obtained density and molecular weight (M) information, the molar volume (Vm) was computed (Table 1). The variation of Vm and ⍴ with MoO3 is seen in Figure 2.

**Table 1: Density, molar volume, Urbach energy, optical bandgap, refractive index, molar refraction and molar electronic polarizability of BCAPM glasses**

|  |  |
| --- | --- |
| **Physical & Optical parameters** | **Sample Code** |
| **BCAPM0** | **BCAPM1** | **BCAPM2** | **BCAPM3** | **BCAPM4** |
| **Average molecular weight** | 141.754 | 141.248 | 140.742 | 140.235 | 139.729 |
| **Density (ᵨ) gm/cc** | 4.072 | 4.057 | 4.041 | 4.025 | 4.009 |
| **Molar Volume (Vm) cc/mol** | 34.80 | 34.81 | 34.82 | 34.83 | 34.84 |
| **Optical band gap (Eopt) eV** | 3.063 | 3.049 | 3.013 | 3.002 | 2.951 |
| **1-sqrt(Eg/20)(Rm)** | 0.6084 | 0.6095 | 0.6119 | 0.6126 | 0.6158 |
| **Refractive index (n)** | 2.379 | 2.383 | 2.393 | 2.396 | 2.410 |
| **Dielectric constantnt (ε)** | 5.660 | 5.682 | 5.728 | 5.743 | 5.809 |
| **Molar refractivity (Rm) cm-3** | 21.17 | 21.21 | 21.30 | 21.34 | 21.45 |
| **Electronic polarizability (αm) 10-21 cm-3** | 8.38 | 8.40 | 8.44 | 8.45 | 8.50 |
| **Urbach energy (ΔE) eV** | 0.257 | 0.191 | 0.213 | 0.216 | 0.223 |

As the mole proportion of MoO3 increases, the density of the BCAPM glasses decreases non-linearly, as seen by the crystal densities of MoO3 (4.70gm/cc) and PbF2 (8.44gm/cc). The decrease in density may be explained by the fractional substitution of MoO3 (143.94 g/mol) with PbF2 (245.2 g/mol). The literature suggests that the decrease in density values could be caused by the transformation of [BO4] units to [BO3] units, where [BO4] is denser than [BO3] [26, 27].



**Figure 3: Absorption spectra of BCAPM glasses**

Figure 3 shows the absorption spectra of BCAPM glasses. An intriguing technique is the investigation of the valence and conduction band transitions in the UV-visible zone utilizing the primitive absorption edge. The absorption coefficient α(v), which has decreasing values as a function of photon energy hν, is the primary feature of the absorption edge in non-crystalline substances [28].

$α\left(ϑ\right)= Cexp(\frac{hϑ}{∆E})$ (1)

where ΔE is Urbach energy and C is a constant. The α (ν) can be obtained from the absorbance A by using the equation below;

$α\left(ϑ\right)=2.303\frac{A}{d}$ (2)

where ‘d’ is the thickness. Davis et al [29] and Tauc et al [30] suggested relation:

 ($αhυ$) =$B^{2}(hυ-E\_{g})^{2}$ (3)

The BCAPM glasses Tauc plot is displayed in Figure 4. It is possible to obtain the optical band gap (Eg) measurements (refer to Table 2) by extending the linear portion of the curve. In the current BCAPM glasses, MoO3 enrichment from x=0 to 2 mole% causes Eg to decrease from 3.063 eV (BCAPM0) to 2.951 eV (BCAPM4). The bandgap decreases upon the addition of MoO3, signifying the establishment of localized states for electrons in close proximity to the conduction band. It is possible to lower the bandgap by connecting these localized states to the conduction band. The decrease in band gap could have been brought on by the process of transformation of Mo6+ ions to Mo5+ ions. A decrease in bandgap and a rise in non-bridging oxygens can result from a boost in Mo5+ ions, which act as modifiers in the glass network and occupy octahedral positions [11, 31].



**Figure 4: Tauc plot of BCAPM glasses**

Urbach energy is found by calculating the inverse slope of the linear section of the Urbach plot, as shown in Figure 5. The Urbach energy provides the ability to determine the degree of disorder in the glass network. These values were discovered to be significantly lower for the BCAPM glass system, suggesting that there was less disorder in the glass structure [32].



**Figure 5: Urbach energy plot of BCAPM glasses**

The relationships that follow [33–35] are used to estimate the remaining variables, such as molar refraction (Rm), molar electronic polarizability (αm), and refractive index (n).

$\frac{n^{2}-1}{n^{2}+2}=1-\sqrt{\frac{E\_{g}}{20}}$ (4)

$R\_{m}=\frac{n^{2}-1}{n^{2}+2}\*V\_{m}$ (5)

$α\_{m}=\left(\frac{3}{4ПN\_{A}}\right)\*R\_{m}$ (6)

The n parameters were increased from 2.379 (BCAPM0) to 2.410 (BCAPM4) due to an overabundance of MoO3 in BCAPM glasses. Rm and ρ exhibit an increasing trend with mixture since they are both correlated with molar volume.

**3.3. FTIR spectroscopy:**

The method of analysis used to get infrared absorption or transmission spectra and identify different functional groups in materials is called Fourier transform infrared spectroscopy or FTIR spectroscopy. FTIR spectra can be used to investigate the impact of MoO3 on the structural properties of BCAPM glasses having lead fluoride. Figure 6 displays the BCAPM glasses infrared absorption spectra in the 400–1600 cm-1 wave number range. Table 3 lists the assignments of the numerous infrared bands (IR bands) detected from FTIR spectra: 423, 461, 517, 579, 673, 724, 901, 995, 1061, 1116, 1168, 1279, 1376, 1466, and 1549 cm-1. The IR analysis generally identifies two separate frequency zones. According to references 36–38, the stretching vibrations BO3 and BO4 borate units are associated with ranges of 1200–1600 cm-1 and 800–1200 cm-1, correspondingly. Three main broad bands emerged throughout the examination, with substantial variations in the infrared spectra found at approximately 670, 1050, and 1380 cm-1. The spectra showed that the core of the bands remained the same, but the bandwidths widened as the MoO3 quantity ascended. Molybdenum ions can join the glass network as either network formers or network modifiers, depending on their valence states. Mo5+ ions can claim the former sites, whereas Mo6+ ions can claim the altering sites. When MoO3 is added to BCAPM glasses to compensate for the loss of PbF2, the hexavalent molybdenum ions are converted to pentavalent ions, increasing the amount of Mo5+ ions (which act as modifiers).



 **Figure 6: FTIR Spectra of BCAPM glasses**

The IR Band assignments are shown in Table 2. The BCAPM glass specimen's bands ranging from 423 to 517 cm-1 are ascribed to the vibrations of Pb2+ and Cd2+ as well as the octahedral AlO6 [5, 6, 39]. The Mo-O bond vibrations in deformed MoO4 may be the cause of the peak near 579 cm-1 [13, 14]. The elongations of B-O-B bonds in BO3 unit groups are shown by the IR band locations 673 cm-1 detected in BCAPM glasses [41]. The bending vibrations of B-O links are responsible for the peak, which is located at around 724 cm-1. The Mo–O–Mo bridging bonds inside the structure are what cause the band at 900 cm-1. [13]. The bands between 995 and 1060 cm-1 are caused by the B-O stretching vibrations of tetrahedral BO4 units [42, 43]. The bands within 1116 and 1279 cm-1 are caused by stretching vibrations of tetragonal BO3 units from various borate groups. The band is ascribed to the stretching extensions of the B–O of trigonal (BO3)3- units at about 1376 cm-1. The IR band at 1476 - 1549 cm-1 is caused by anti-symmetrical elongations B-O-B groups [44 - 46].

**Table 2: IR Band Assignments of BCAPM glasses**

|  |  |
| --- | --- |
| IR band position (cm-1) | Assignments |
| 420 - 520 | Pb2+ , Cd2+ and AlO6 octahedral  |
| ~579 | Mo-O bond vibrations |
| ~673 | Stretching vibration of B–O–B bonds in BO3 units  |
| ~724 | Bending vibrations of B–O linkages |
| ~900 | Mo–O–Mo bridging bonds |
| 990 - 1070 | B-O stretching vibrations |
| 1110 - 1280 | Stretching vibrations of tetragonal BO3 units |
| ~1376 | Stretching elongations of the B–O of trigonal (BO3)3- units |
| 1476 - 1549 | Anti-symmetrical stretching vibrations of B–O–B groups |

**3.4 Raman spectroscopy:**

One of the most efficient tools for comprehending the structure of glasses is vibrational spectroscopy. Glasses are non-crystalline structures made up of structural units that are comparable to crystalline forms and can be freely arranged in a three-dimensional network. Raman spectroscopy is one of the finest and most effective vibrational spectroscopy methods for examining the structure of glass.



**Figure 7: Raman spectra of BCAPM glasses**



**Figure 8: Deconvolution spectra for the BCAPM1 sample**

 In order to determine the precise Raman band locations, Figure 7 displays the Raman spectra of the BCAPM glasses, and Figure 8 shows the deconvolution spectra for the BCAPM1 sample. Table 4 lists the numerous Raman bands that have been detected from Raman spectra. These include 344 cm-1, 498 cm-1, 674 cm-1, 789 cm-1, (983 cm-1 shift 925 cm-1), 1079 cm-1, 1229 cm-1, (1347 cm-1 shift 1337 cm-1), 1437 cm-1, and 1738 cm-1. MoO6 octahedra shared by two corners is the source of the rapidly rising Raman band at 344 cm-1 [47, 48]. Isolated diborate groups are the cause of another band that is present at 498 cm-1 [49]. The detection of the Raman band at 674 cm-1 in BCAPM glasses led to the observation of anti-symmetric elongations of O-Mo-O linkages [50]. The stretching vibrations of Mo-O-Mo links and the creation of AlO4 tetrahedra are the sources of another Raman band detected at 789 cm-1 [5, 47]. The vibrations of Mo-O and Mo=O bonds in single and paired MoO6 units are responsible for a significant band that is seen at ~950 cm-1 in BCAPM glasses and increases sharply [51, 47]. In the current BCAPM glass system, it is evident that the two Raman peaks one at 344 cm-1 and the other at 950 cm-1 are growing dramatically as we increase MoO3. In addition, the Raman band at 950 cm-1 is moving from 983 cm-1 to 925 cm-1, and its intensity increases as MoO3 quantities rise. This could be because MoO3, a compound with a low molecular weight, has replaced PbF2. The alteration in BCAPM glasses closer to a lower frequency range and greater intensity could be attributed to the conversion of [BO4] units to [BO3] with a spike in MoO3 contents. This result confirms the observation that density fluctuation among BCAPM glasses is decreased as one raises MoO3 doping content. The presence of diborate groups is responsible for the Raman band at 1079 cm-1. The strength of the Raman bands at 789 cm-1 and 1229 cm-1 was found to decrease from BCAPM0 to BCAPM4 in glass specimens when the PbF2 was substituted with MoO3, and eventually, the Raman bands in the neighbourhood converged. Pyroborate groups and elongated B-O bands related to the borate groups are identified by a strong Raman band in BCAPM glasses at 1340 cm-1 [53, 54]. The B-O- vibrations of the BO3 units are responsible for the bands in this glass system around 1437 cm-1, whereas the metaborate units of the chain and ring type are responsible for the band at 1738 cm-1[54–56].

**Table 3: Raman Band Assignments of BCAPM glasses**

|  |  |
| --- | --- |
| Raman band position | Band assignments |
| ~344 | MoO6 octahedra |
| ~498 | Isolated diborates |
| ~674 | Anti-symmetric elongations of O-Mo-O linkages |
| ~789 | Stretching vibrations of Mo-O-Mo linkages and the formation of AlO4 tetrahedra |
| ~950 | Vibrations of Mo-O |
| ~1079 | Existence of diborate groups |
| ~1340 | Pyroborate groups |
| ~1437 | B–O- vibrations of the BO3 units |
| ~1738 | Chain and ring type metaborate units |

**4. Conclusions:**

Employing the traditional melt quenching procedure, glasses with the following composition: 60B2O3-20CdO-5Al2O3-(15-x) PbF2-x MoO3 (where x = 0, 0.5, 1, 1.5, and 2 mol%) were created. Physical, optical, FTIR and Raman spectroscopy methods were used to examine these materials to determine how MoO3 affected the structural alterations that took place in them. The change from BO4 to BO3 units and the molecular weights of PbF2/ MoO3 are responsible for the specimens' decreased density. A decrease was noted in the optical band gap values, which were calculated using UV-visible spectra. The decrease in MoO3 content is caused by the conversion of Mo6+ ions to Mo5+ ions, which occurs at the cost of PbF2. Distinct borate and aluminate groups have been established by FTIR and Raman spectra. These spectra further validate that MoO3 functions as a modifier in these glasses and assists the transition of Mo6+ ions into Mo5+ ions.

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