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# Luminescence and Lifetime of CaO-$Al_2O_3$-$B_2O_3$ : $Yb^{3+}$ and CaO-$Al_2O_3$-$B_2O_3$: $Ce^{3+}$ Rare-Earth-Doped Borate Glasses

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**Abstract:** This recherche aimed to investigate the characteristics related to the photo luminescence properties of borate glasses CaO-$Al_2O_3$-$B_2O_3$ doped with 1.0% of $Yb^{3+}$ and $Ce^{3+}$. These glasses were prepared by using a melt-quenching technique and characterized through X-Ray Diffraction (XRD) and Fourier-transform infrared spectroscopy and photo luminescence and decay spectral studies to determine the feasibility of using these glasses as luminescent devices in the visible region. The XRD standard confirmed the amorphous nature of the prepared glasses. By means of the infrared spectra the vibrational characteristics of the borate glass groups were verified. The characterization showed that the levels of rare-earth ions did not compromise the structural properties of the glassy matrixes. Excitation and luminescence spectra and fluorescence lifetime of the rare-earths ions were also detected. The excitation and emission spectra of both glasses show a broad PL profile due to the $^2F_{5/2}\rightarrow$ and 5d→4 ftransitions for Yb and Ce dopant, respectively.

**Key words:** Excitation, luminescence, spectra and fluorescence, life time of the rare-earths, ions, characterization

## INTRODUCTION

Glasses doped with Rare-Earth Ions (REIs) have attracted great attention in the scientific community because of their capability to control properties such as refractive index, density, structure, luminescence and optical absorption by variation of oxide admixtures and composition (Barbi *et al*., 2018; Mahamuda, 2013). The simplicity of synthesis of these glassy materials, the durability of their thermal, mechanical and chemical properties, the ability to manufacture optical elements of any shape and size, their relatively low cost, the possibility to incorporate impurities and their high optical homogeneity makes them ideal for application in devices such as photo chromic sun screen lenses, phosphor lamps, active laser hosts, scintillators and dosimeters (Jafari *et al*., 2014; Sontakke *et al*., 2016; Tripathi *et al*., 2016).

Borate glasses are one of the most promising oxide glasses for applications in optical devices because of their spectroscopic and structural characteristics (Parandamaiah *et al*., 2015; Swapna *et al*., 2014; Luo *et al*., 2016; Barrera *et al*., 2019). These glasses have high linear refractive indices and marked optical non line arities when compared to other vitreous materials (Kassab *et al*., 2002; Mallur *et al*., 2015).

The optical properties of REIs in glasses depend on the chemical composition of the glass matrix, its structure and the nature of the chemical bonds (Kadono, 2007; Mahamuda *et al*., 2013). $Ce^{3+}$-doped inorganic materials such as glasses and crystals are widely used as phosphors for displays and lighting as well as high-energy radiation scintillations (Wisniewski *et al*., 2008; Bachmann *et al*., 2009; Ueda *et al*., 2011). This is due to the superior light yield of $Ce^{3+}$ ions arising from their parity-allowed 5d-4f transitions (Sontakke *et al*., 2016). The absence of cross-relaxation mechanisms and an insignificant multiphonon relaxation of the excitation population, due to the wide gap between 5d and 4f energy level have established $Ce^{3+}$ as one of the most efficient luminescent ions (Barbi *et al*., 2018). $Yb^{3+}$ glass is a promising material with numerous attractive properties (Biswal *et al*., 1999) including good spectroscopic and laser performance parameters with a broad fluorescence spectrum (compared to and amplify ultrashort laser pulses (Kassab *et al*., 2002).

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Addition of $Al_2O_3$ to the oxide glass structure affects different physical properties such as glass transition temperature (Tg), density and refractive index (Yue *et al*., 2018). The structural role of $Al_2O_3$ in oxide glasses is important because this oxide has insuficiente oxygen to form a tetrahedral network. $Al^{3+}$ ions become 4-coordinated with oxygen if there are enough oxygen ions from October 18, 2019 the modifier oxides in the glass (El-Moneim *et al*., 2003). Also, the simultaneous filling up of the vacancies (empty spaces) amidst the borate network present by the interstitial $Ca^{2+}$ metal ions increases the compactness of the glass and consequently the measured density. Therefore an appropriate addition of CaO to the glass contributes to a dense glass body (Luo *et al*., 2016; El-Moneim *et al*., 2003).

Rare-earth-doped glasses have shown a good sensitivity for absorbing UV-visible radiation and emitting visible radiation that may make them promising candidates for sensing electromagnetic radiation. Due to this in the current study, Calcium Aluminoborate ($CaO$-$Al_2O_3$-$B_2O_3$) matrix glass was used as host for REIs in both $Yb^{3+}$- and Ce3+ -doped glasses. To this purpose, we scrutinize the optical excitation, luminescence decay kinetics and the structural and amorphous nature of these borate glasses.

## MATERIALS AND METHODS

The proposed glass compositions were prepared using the conventional melt-quenching technique. Raw materials and rare-earth dopants used were: CaO, $Al_2O_3$, $H_3BO_3$, $Yb_2O_3$ and $Ce_2O_3$ (SigmaAldrich, 99.99% purity). The proposed composition ratios used to prepare the glass samples were: 27% CaO-18% $Al_2O_3$-55% $B_2O_3$:1.0 wt. % of $Yb^{3+}$ (CAB:$Yb^{3+}$) and 27% CaO-18% $Al_2O_3$-55% $B_2O_3$:1.0 wt.% of $Ce^{3+}$ (CAB:$Ce^{3+}$). The above mentioned materials were weighed and the powders well mixed for 30 min in an agate mortar before being melted in a porcelain crucible in an electric furnace at 1100°C for 60 min with a heating rate of approximately 20°C/min.

The molten mixtures were stirred frequently to ensure homogeneity and then poured into a pre-heated aluminum mold at 200°C. The amorphous structures were confirmed by X-Ray Diffraction (XRD) in a Rigaku DMAX 2000 CuKα diffractometer with the tube operating at 40 kV/20 mA with steps $2\theta(°) = 0.5$ in the $2\theta$ scan range of $10° < 2\theta < 80°$ with 10 sec per step at room temperature. For these analyses, grain sizes of <75 mm were used.

The local structure of lead borate glass and lead aluminum borate were studied by Fourier Transform Infrared (FTIR) spectroscopy. The spectra were obtained in the range of 4000-400 $cm^{-1}$ at room temperature in a varian 640 spectrometer (typical resolution of 4 $cm^{-1}$ and 64 accumulations). The samples were in the form of KBr pellets (discs). The excitation and emission spectra were obtained using a spectrofluorometer (Nanolog-Horiba) with an R928P photo multiplier tube equipped with a xenon lamp (CW 450 W) as excitation source and a 399 nm filter. Lifetime was also obtained with the nano log using the same lamp and the same detector.

## RESULTS AND DISCUSSION

**XRD spectral analysis:** The glasses are amorphous materials with absence of crystalline periodicity. The XRD patterns of the two glass samples are shown in Fig. 1. The spectra show the absence of any sharp peak and the presence of broad humps which confirms the amorphous nature of the rare-earth-doped borate glasses. This behavior indicates that the samples are not crystalline and corroborate with the formation of vitreous structures. The XRD patterns of both materials are very similar but with a difference in intensity located between 20 and 40° for CAB: $Yb^{3+}$ sample. The presence of dopant is only factor to caused this difference in intensity, since, the same synthesis procedure for both samples, this will also be discussed in section 3.3.

**FTIR spectral analysis:** Information related to the arrangement of structural groups of the studied glasses was explained with the help of FTIR spectroscopy. The FTIR spectra of the glasses CAB:$Ce^{3+}$ and CAB: $Yb^{3+}$ are shown in Fig. 2. The broad composite bands extending to

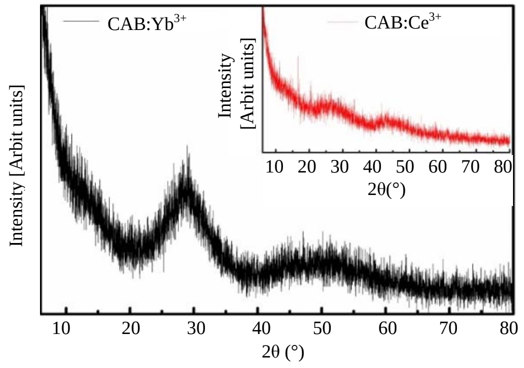

Fig. 1: XRD pattern of glass CAB: $Yb^{3+}$

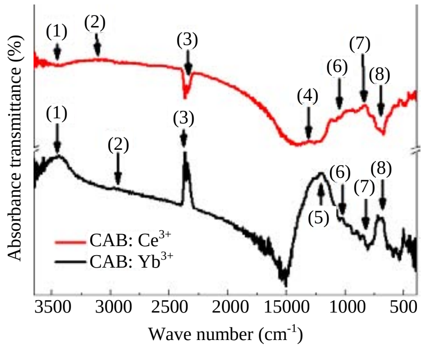

Fig. 2: FTIR spectra of CAB:$Yb^{3+}$ and CAB:$Ce^{3+}$ glasses. The peaks were identified according to Table 1

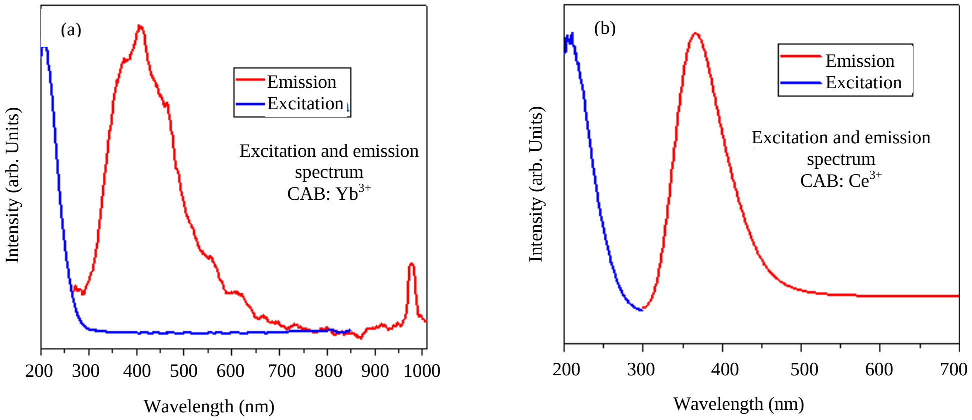

Fig. 3(a, b): (a) Photo luminescence excitation and emission spectra for CAB: $Yb^{3+}$ samples usingexcitation at 250 nm and emission at 410 and 976 nm and (b) Photoluminescence excitation andemission spectra for CAB: $Ce^{3+}$ samples with excitation at 280 nm and emission at 367 nm

the near-infrared region ~3415 $cm^{-1}$ (CAB: $Ce^{3+}$) and ~3419 $cm^{-1}$ (CAB: $Yb^{3+}$) are due to hydroxyl, water or B-OH groups present in the samples. The presence of these hydroxyl or water groups could be attributed to the experimental procedure (KBr technique) and may also arise due to the presence of water in $H_3BO_3$ (Balachander *et al.*, 2013). The spectra show peaks at ~2927 $cm^{-1}$ (CAB:$Ce^{3+}$) and ~2923 $cm^{-1}$ (CAB: $Yb^{3+}$) which are attributed to the presence of hydrogen bonding in the glass sample (Sharma *et al.*, 2006; Balachander *et al.*, 2013). Finally, the borate glasses in the study show three active infrared spectral regions. The region at 600-800 $cm^{-1}$ is attributed to the bending vibrations of borate segments. The other, at 850-1200 $cm^{-1}$ is attributed to the stretching vibration of structural groups containing $BO_4$ tetrahedra such as diborate, triborate, tetraborate and pentaborate groups (Aronne *et al.*, 1999; Galeener *et al.*, 1980). Peaks at ~828 $cm^{-1}$ (CAB: $Ce^{3+}$) and ~826 $cm^{-1}$ (CAB: $Yb^{3+}$) are due to the $BO^{4-}$ stretch (tri, tetra and pentaborates). The bands centered at ~1024 $cm^{-1}$ (CAB: $Ce^{3+}$) and ~1021 $cm^{-1}$ (CAB: $Yb^{+3}$) in the these studied systems confirm the presence of asymmetric stretching of tetrahedral $BO_4$ units and vibrations of diborates bridging to pentaborate (Balachander *et al.*, 2013). The region between 1200 and 1500 $cm^{-1}$ arises from the B-O stretching vibration of $BO_3$ units such as metaborate chains and rings and pyroborate and orthoborate groups (Kamitsos *et al.*, 1990). The broad peak centered at ~1326 $cm^{-1}$ present in the CAB:$Ce^{3+}$ glass spectrum is associated with B(3)-O-B(4) stretching vibrations in the glass net stretching (Saddeek *et al.*, 2010). The shoulder at ~1236 $cm^{-1}$ exhibited by the glass CAB: $Yb^{3+}$ can be attributed to asymmetric stretching vibrations of B-O bond in metaborate's pyroborates and orthoborates (Gautam *et al.*, 2012). It is important to note that $BO_4$ structural units can be formed with the incorporation of CaO and $Al_2O_3$ in the network of the studied glasses (Saddeek *et al.*, 2010).

Each oxide of CaO or $Al_2O_3$ can convert two $BO_3$ units into two $BO_4$ units (Saddeek *et al.*, 2010). These spectra exhibit broad absorption bands because of the general disorder in the network, due mainly to the wide distribution of structural units occurring in these glasses. The FTIR spectralpeaks are detailed in Table 1.

**Photoluminescence spectral analysis:** The photo luminescence excitation spectrum for an 1 wt.% $Yb^{3+}$ doped CAB glass, corresponding to the $^2F_{5/2}\rightarrow{}^2F_{7/2}$ transition is presented in Fig. 3a. The peaks related to $Yb^{3+}$was observed at 976 nm and is assigned to transitions from the first level of the excited to the ground state. The most intense emission band has a peaked at about 410 nm extending across the visible range. This emission band can be attributed to the formation of $Yb^{3+}$clusters in the glass structure-such a characteristic of clusters formation resulting in visible emission due to energy transfer from ytterbium as a dopant has been confirmed in the literature (Schaudel *et al*, 2000, Oppo *et al.*, 2012).The band of emission cooperative present in the sample of the work published by Schaudel *et al.*, 2000, resemble the spectrum obtained for the CAB: $Yb^{3+}$glasses.

The interesting fact here is that the excitation spectrum does not exhibit a band invisible region (Fig. 2a). As an ultraviolet excitation (250 nm) was used to obtain the emission peak at infrared region, the excitation of the cluster and the isolated ions of ytterbium is attributed to the occurrence of energy transfers from the vitreous matrix to the clusters and ions, since, the electronic transitions of Yb corresponding to the $^2F_{5/2}\rightarrow{}^2F_{7/2}$ transition are excited in the infrared (Biswal *et al*, 1999).

Table 1: Peak table of FTIR spectra of CAB: $Ce^{3+}$ and CAB: $Yb^{3+}$ glasses

| Order | CAB: $Ce^{3+}$($cm^{-1}$) | CAB: $Yb^{3+}$($cm^{-1}$) | Assignment | Researcher |
|---|---|---|---|---|
| 1 | ~3415 | ~3419 | Hydroxyl or water or B–OH groups | Balachander *et al*. (2013) |
| 2 | ~2927 | ~2923 | Hydrogen bonding | Sharma *et al*. (2006) |
| 3 | ~2342 | ~2342 | Presence of atmospheric $CO_2$ | Schadle *et al*. (2016) |
| 4 | ~1326 | ~1236 | B(3)–O–B(4), B–O stretching | Saddeek *et al*. (2010)<br>Gautam *et al*. (2012) |
| 5 | ~1024 | ~1021 | $BO_4$ group stretching | Pal *et al*. (2011) |
| 6 | ~828 | ~826 | $BO^{4-}$ stretching (tri, tetra pentaborates) | Aronne *et al*. (1999)<br>Galeener *et al*. (1980) |
| 7 | ~696 | ~695 | $BO_4$ vibrations | Aronne *et al.* (1999)<br>Galeener *et al.* (1980) |

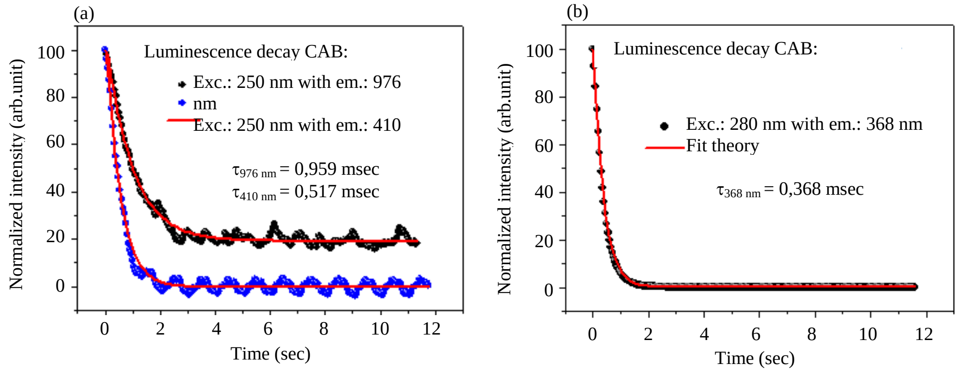

Fig. 4(a, b): The luminescence decay curves for CAB:$Yb^{3+}$ and CAB:$Ce^{3+}$

The ytterbium ions cluster formation and vitreous matrix components can be confirmed by the luminescent decay times shown in Fig. 4a. A decay time of τ = 0.959 ms was observed for the emission at 976 nm and another one of τ = 0.517 ms for the emission at 410 nm. These results corroborate with the decay times observed by Schaudel *et al.* (2000) in the study on the luminescent properties of ytterbium doped inborosilicate glass. Figure 3b shows PL emission and excitation spectra of CAB: $Ce^{3+}$ sample. The Ce-oped samples exhibit an emission band between 320-460 nm, centered at 367 nm under excitation wave length of 280 nm. This emission is assigned to the 5d-4f transitions from the first excited state to the ground state. Such wavelength emission agreed well with reported value for other Ce doped glasses (Nakauchi *et al.* 2017). The luminescent decay time observed for the emission at 367 nm was 0.517 msec as shown in Fig. 4b.

Although, the emission and excitation spectra of CAB: $Ce^{3+}$agree with the one obtained by Nakauchi *et al.* (2017) when studied other aluminoborate glass doped with Ce, the order of magnitude of luminescent decay time of CAB: $Ce^{3+}$ glass is three times greater. Emission $Ce^{3+}$centers have usually fast decay time from 50-60 nsec, approximately (Nakauchi *et al.* 2017). However, the luminescent decay time can be change by the interaction between doping ion and other ions present in the structure by non-irradiative energy transfer (Kucera *et al*, 2013). As spectroscopic properties depend on the lattice and composition of the glass, then this result confirm that cerium ion was added as a dopant to the CAB glass structure.

## CONCLUSION

New Calcium Aluminoborate glasses, CAB: $Yb^{3+}$ and CAB: $Ce^{3+}$ were synthesized and studied using different spectroscopic techniques. The XRD spectra confirmed the amorphous nature of the as-prepared glasses. From the XRD spectra of the glasses investigated, a difference in intensity was observed between 20 and 40°; this difference was related due to the dopant. It was found from the FTIR spectra that different structural borate groups are present in these glasses. By means of the FTIR spectra it was observed that $BO_4$ structural units can be formed with the incorporation of CaO and $Al_2O_3$ in the network of the glasses studied.

Each CaO or $Al_2O_3$ oxide can convert two $BO_3$ units into two $BO_4$ units. The excitation and emission spectra of both glasses show a broad PL profile due to the and 5d→4f transitions for Yb and Ce dopant, respectively. An emission band centered at about 410 nm extending across the visible region was observed to CAB: $Yb^{3+}$ and attributed by the formation of $Yb^{3+}$ clusters. These features enable the use of these new material as candidates for the development of devices that operate in the near visible and infrared spectral regions.

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