

Spectroscopic and Optical Studies of Fluorescein Dye-Doped Boric Acid Glass System (BAG)

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Abstract

Fluorescein, an important dye, has been incorporated into a glass matrix of $\text{H}_3\text{BO}_3\text{-Pb}_3\text{O}_4\text{-ZnO}$. The glass (denoted as BAG) was synthesized in the temperature range of 1050°C to 1150°C . The X-ray diffraction (XRD) pattern confirmed the glassy nature of the synthesized samples. Laser Raman spectra have been recorded using 532 nm wavelength excitation to study the vibrational nature of the glass structure. Fluorescence dye-doped boric acid glasses show BO_3 and BO_4 structural units in FTIR studies. The Tauc's Plot calculations show the optical band gap of the synthesized sample to be nearly 3 eV which is approximately equal to other Fluorescence dye-doped glasses. Fluorescence spectra have been recorded using 532 nm wavelength excitation. Also, an energy level diagram has been developed.

Keywords

Boric Acid Glass, Fluorescein, FTIR, Tauc Plot, Optical Limiting, Optical Band Gap, Refractive Index

1. Introduction

Fluorescein dye-doped boric acid glass is a type of luminescent glass that combines the optical properties of Fluorescein dye with the structural benefits of boric acid glass. This hybrid material has garnered significant attention in recent years due to its potential applications in various fields, including optics, photonics, and biomedicine [1] [2].

Fluorescein is a synthetic organic dye known for its high fluorescence efficiency,

broad absorption spectrum, emission spectrum, and photostability. The combination of fluorescein dye and boric acid results in a material with enhanced luminescence efficiency, improved photostability, tailorable optical properties, and potential for biocompatibility. Fluorescein dye-doped boric acid glass has potential applications in optical amplifiers, laser materials, biomedical imaging, sensors, and display devices. Fluorescein dye has numerous applications across fields such as medical applications, ophthalmology, fluorescein angiography, cancer research, neurosurgery, fiber optics, optical fiber sensors, amplifiers, laser technology, and optical imaging [3].

In the present work, we have synthesized Fluorescein dye-doped boric acid glass with composition $70\text{H}_3\text{BO}_3-(10-X)\text{Pb}_3\text{O}_4-20\text{ZnO}-(X)\text{C}_{20}\text{H}_{12}\text{O}_5$ where $X = 0.05, 0.1, 0.5$ and 0.3 respectively.

The optical band gap was determined from UV spectra using tauc plot method. XRD pattern confirms the glassy nature of the synthesized sample. In XRD plots, the Broad peaks confirm this and see that as the concentration of Fluorescein is increased, the peaks are sharp and shifted to a higher degree angle. The FTIR spectra show the BO_3 and BO_4 ions in the glass system.

Fluorescein dye-doped glasses have a higher refractive index than other glass systems. Fluorescein dye-doped glasses also have low values of indirect band gap which lies within the range of 2.80 eV to 3.00 eV and have sharp absorption spectrum in UV and IR regions.

2. Experimental

Sample Preparation

Fluorescein dye-doped boric acid glasses were prepared by the rapid cooling method using an appropriate mixture of H_3BO_3 , Pb_3O_4 , ZnO , and $\text{C}_{20}\text{H}_{12}\text{O}_5$ in the composition range $70\text{H}_3\text{BO}_3-(10-X)\text{Pb}_3\text{O}_4-20\text{ZnO}-(X)\text{C}_{20}\text{H}_{12}\text{O}_5$ where $X = 0.05, 0.1, 0.5$ and 0.3 respectively. **Table 2** shows the glass compositions for various prepared samples. Suitable amounts of each chemical were taken and weighed in a weighing machine. After weighing, all the chemicals were thoroughly mixed using a mortar and pestle. The composition of each sample was taken in an open silica crucible and kept in an electric muffle furnace for heat treatment. To decompose the boric acid, initially, the samples were maintained at room temperature and then the temperature was raised to $1050^\circ\text{C} - 1150^\circ\text{C}$ [4]. Base glass $70\text{H}_3\text{BO}_3-(10-X)\text{Pb}_3\text{O}_4-20\text{ZnO}$ was first melted at 1050°C for 1 hour and quenched. The resulting frit was ground, mixed with fluorescein (0.05 - 0.30 mol %) and re melted at 350°C for 15 minutes.

After that the molten liquid was poured quickly between two stainless-steel plates kept at room temperature to form a glassy composition of the required shape. The obtained glasses were light greenish colored in nature. After that, the synthesized glass samples were polished to make them transparent. The obtained glasses and the experimental procedure are shown in **Figure 1**.

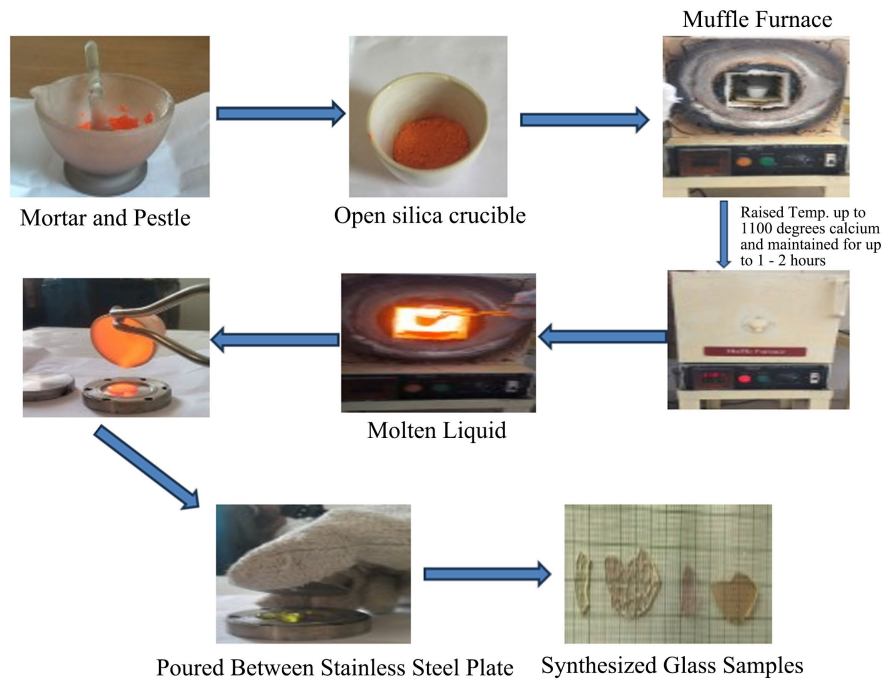


Figure 1. Schematic diagram of synthesis glass by using the rapid cooling process in muffle furnace.

The physical parameters, such as the density, number of rare earth ions, refractive index, electronic polarizability [5] [6], and molar refractivity, are calculated using the following equations and tabulated in **Table 1**.

Table 1. Physical parameter.

Sr. No.	Properties	A	B	C
1	Density, ρ (g/cm ³)	2.79	2.81	2.80
2	Molar volume, V_m (cm ³ /mol)	30.74	30.50	30.60
3	Average Molecular weight, M (g/mol)	85.77	85.73	85.69
4	Refractive index, n	2.51	2.48	2.43
5	Molar Refraction, R_m (cm ³)	21.36	21.30	22.25
6	Electronic Polarizability, α_e (10 ⁻²⁴ cm ³)	-	1.70	0.88

The refractive index (n) of the prepared glass is calculated using,

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}}$$

The electronic polarizability (α_e) is determined following the expression,

$$\alpha_e = \frac{3(n^2 - 1)}{4\pi N_{RE}(n^2 + 2)}$$

The molar refractivity R_m of the prepared glasses is obtained by,

$$R_m = \frac{n^2 - 1}{n^2 + 2} \frac{M}{\rho}$$

where M is the average molecular weight and ρ is the density in $\text{g}\cdot\text{cm}^{-3}$.

Briefly, the glass composition is shown in **Table 2**. We have synthesized samples (A, B, C, and D) as below:

Table 2. $\text{C}_{20}\text{H}_{12}\text{O}_5$ dye doped BAG composition.

GLASS SAMPLE	H_3BO_3 (mol%)	Pb_3O_4 (mol%)	ZnO (mol%)	$\text{C}_{20}\text{H}_{12}\text{O}_5$ (mol%)
A	70	10	20	-
B	70	9.95	20	0.05
C	70	9.9	20	0.10
D	70	9.7	20	0.30

3. Result and Discussion

3.1. X-Ray Diffraction

The XRD spectra of the synthesized sample have been recorded by using the SmartLab 3 kW/Rigaku model diffractometer. The broad peaks show the glassy nature of the synthesized sample [7]. The XRD plot show one broad characteristic of peak amorphous materials and there are no signs of crystalline phases. No crystalline fluorescein peaks observed indicating dissolution. As the concentration increases the peaks are broader as compared to the undoped sample and peaks shifted to some but higher angle [8]. Corresponding XRD patterns of Fluorescein dye-doped and undoped boric acid glasses are shown in **Figure 2**.

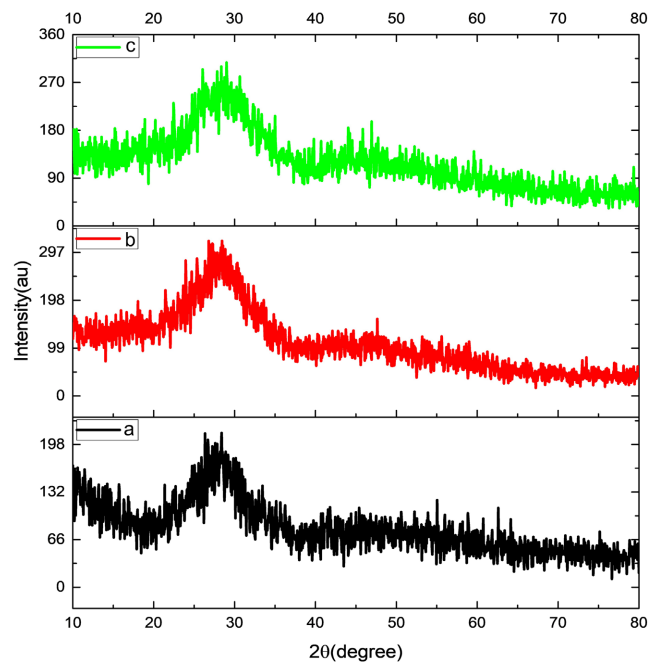


Figure 2. XRD spectra of undoped and fluorescein dye doped Boric Acid Glass (BAG).

3.2. FTIR Spectra

Figure 3 shows the FTIR spectra of Fluorescein dye-doped BAG glass which is recorded using the Spectrum Two/Perkin Elmer model. The various [9] absorption bands and their assignments obtained from FTIR spectra are shown in **Table 3**. The bands are assigned in the region $425.84 - 449.95 \text{ cm}^{-1}$ due to the bending vibrations of B-O-B linkages [10], in the region $649.76 - 918.20 \text{ cm}^{-1}$ the bands are obtained due to ZnO vibrations due to ZnO units, the bands are identified in the region $918.20 - 1153.95 \text{ cm}^{-1}$ due to the B-O symmetric stretching vibrations of BO_4 units. At around 1504 cm^{-1} the vibration band [11] was observed due to the vibrational modes of the hydroxyl or water group in the glass system matrix. COO^- of fluorescein proof of retention are present in the region around 1691 cm^{-1} due to low concentration in the glasses [12]. In the region, $3400 - 3500 \text{ cm}^{-1}$ bands are assigned due to CH banding vibrations, and in the region, 3600 cm^{-1} bands are due to OH banding vibrations. **Figure 3** shows the FTIR spectrum for $\text{C}_{20}\text{H}_{12}\text{O}_5$ doped and undoped BAG glass for various concentrations [13]. The corresponding assignment of FTIR is shown in **Table 3**.

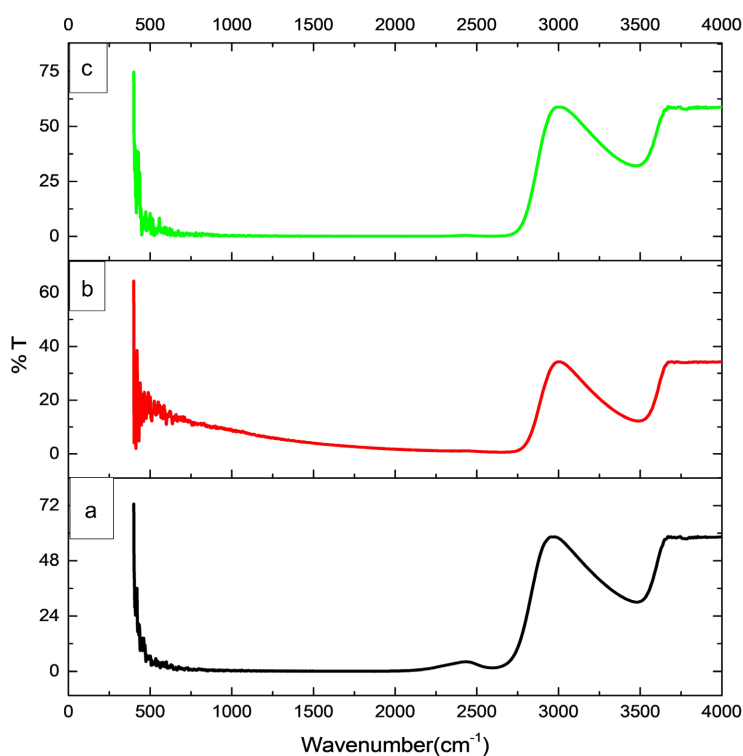


Figure 3. FT-IR spectra of undoped and $\text{C}_{20}\text{H}_{12}\text{O}_5$ ion doped boric acid glass.

Table 3. The FTIR assignments and their band positions (cm^{-1}) of Fluorescein dye-doped glass samples.

Sr. no.	Undoped	$(\text{C}_{20}\text{H}_{12}\text{O}_5)$ (0.05%)	$(\text{C}_{20}\text{H}_{12}\text{O}_5)$ (0.1%)	Band Assignments
1	3660.2	3685	3868	O-H bending vibrations.

Continued

2	3419.45	3576.6	3509	C-H bending vibrations.
3	2441.00	2610	2648.04	-
4	1638.02	1504.50	1691	bending vibrations of water molecules.
5	1384.33	1386	1368.57	COO ⁻ of fluorescein proof of retention
6	-	1271.50	1280.56	B-O stretching vibrations in BO ₄ units from the borate group
7	-	1153.95	1159.70	-
8	1019.94	1013.66	1026.10	-
9	-	917.64	905.21	-
10	-	785.05	670.43	Zn-O vibrations of ZnO units
11	480.82	425.84	428.03	Angle modification of B-O-B linkage

3.3. Raman Spectroscopy

Laser Raman spectroscopy is also useful in the study of vibrational modes of C₂₀H₁₂O₅ doped Boric Acid Glass (BAG). Raman spectral studies using the 532 nm as the exciting sources have been done for undoped and Fluorescein dye-doped boric acid glass using the WITEK model Alpha 300R. Experimental data for the sample are given in **Figure 4** exposed by 532 nm. **Figure 4** shows the Raman spectra of Fluorescein dye-doped boric acid glass for excitation of wavelength 532 nm.

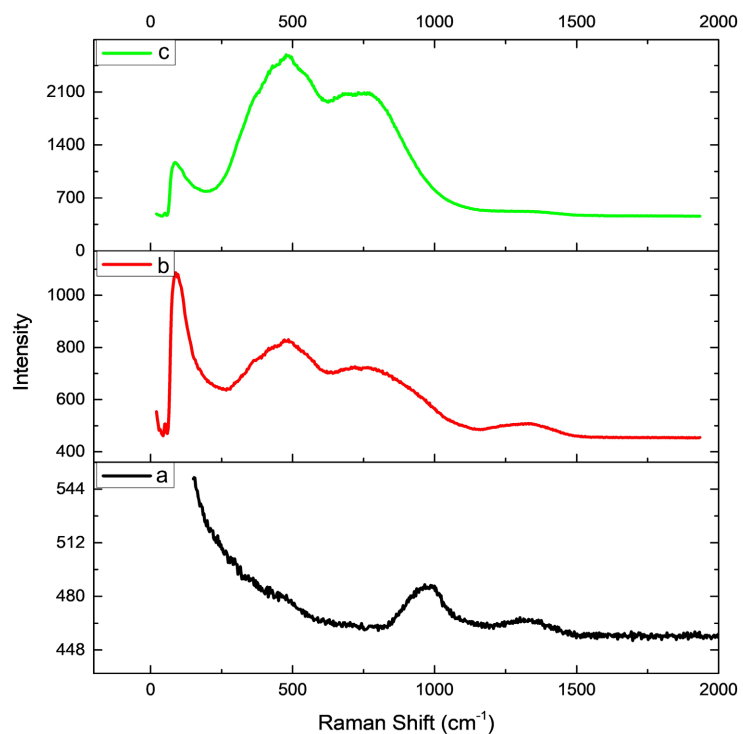


Figure 4. Raman spectra of undoped and fluorescein dye-doped Boric Acid Glass at 532 nm.

Raman spectra show differences in the lines but show five peaks as in **Figure 4**. Raman spectra for undoped glass show 2 to 3 well-shaped peaks as in **Figure 4**. Also, the plot of doped glass shows more peaks than undoped glass and has shifted to a lower wavelength as in **Figure 4**. Raman Peak positions (cm^{-1}) for Fluorescein dye-doped boric acid glass samples at different wavelengths are tabulated in **Table 4**.

Table 4. Raman peak positions (cm^{-1}) for Fluorescein dye-doped boric acid glass samples at different wavelengths [14] are tabulated below.

S. N.	For Wavelength 532 nm			Band Assignment	
	A	B	C	Group	Intensity
1					
2	-	96	88	-	-
3	-	274	220	Lattice Vibration	Strong
4	464	485	482	-	Strong
5	-	795	779	B-O-C interaction	Strong
6	986	-	-	C-C aromatic	Week
7	1184	1147	-	-	-
8	1313	1349	-	-	Very Strong

3.4. UV-VIS Absorption

In the range of 200 nm to 800 nm, UV-VIS absorption spectra have been recorded. A typical Absorption plot for Fluorescein dye-doped boric acid glass is shown in **Figure 5**. As we increased the concentration of $\text{C}_{20}\text{H}_{12}\text{O}_5$, the higher concentrated sample showed more peaks than the low concentration or undoped sample. Furthermore, the undoped [15] sample showed nearly three peaks at 322 nm, 362 nm, and 584 nm but the doped sample with a concentration of 0.05% $\text{C}_{20}\text{H}_{12}\text{O}_5$ shows five to six peaks at 376, 452, 489, 522, 546, and 653 nm, also the concentration with 0.1% $\text{C}_{20}\text{H}_{12}\text{O}_5$ doped sample shows eight peaks at nearly 317, 357, 380, 407, 450, 488, 546 and 653 nm. This can be seen in **Figure 5**, As we increase concentration [16], the peaks increased in strength and shifted to higher wavelengths, whereas some peaks are at the same position in the doped and undoped sample as shown in **Figure 5** [17].

Technically, the undoped glass prepared was colorless and there was no band structure in their spectra in UV and visible regions, but one band was seen nearly at 362 nm in the UV region [18], The samples prepared were transparent, and the doped glass containing $\text{C}_{20}\text{H}_{12}\text{O}_5$ attained a light pink color and obtained some bands in the UV-VIS region. The bands around 320 - 376 nm is due to π - π transition of benzoid rings. The bands around 452 - 490 nm is due to π - π transition of xanthene core (monomer). The bands around 522 - 546 nm is due to J-aggregate/an-ionic form of fluorescein. The bands around 653 nm are due to n- π shoulder due to carbonyl interaction with Pb^{2+} matrix (**Figure 6**).

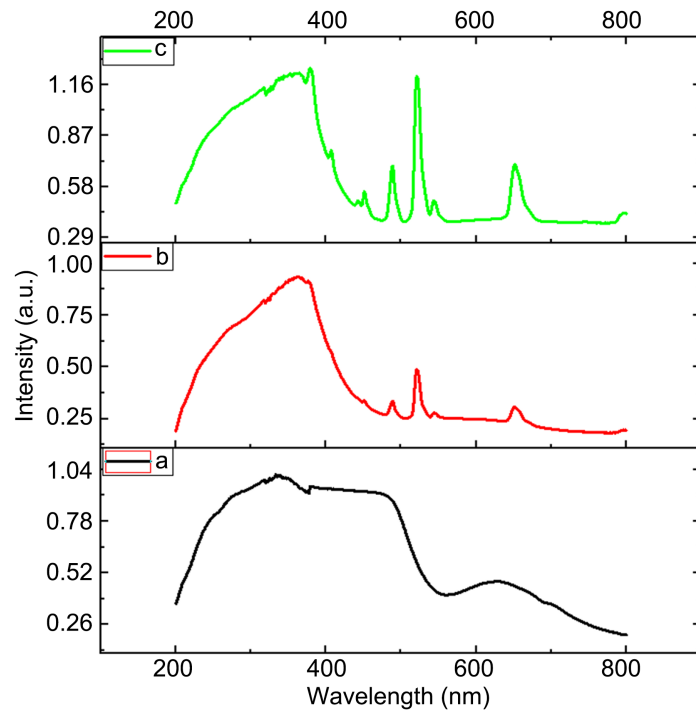


Figure 5. UV-VIS absorption spectra of undoped and $C_{20}H_{12}O_5$ doped Boric Acid Glass.

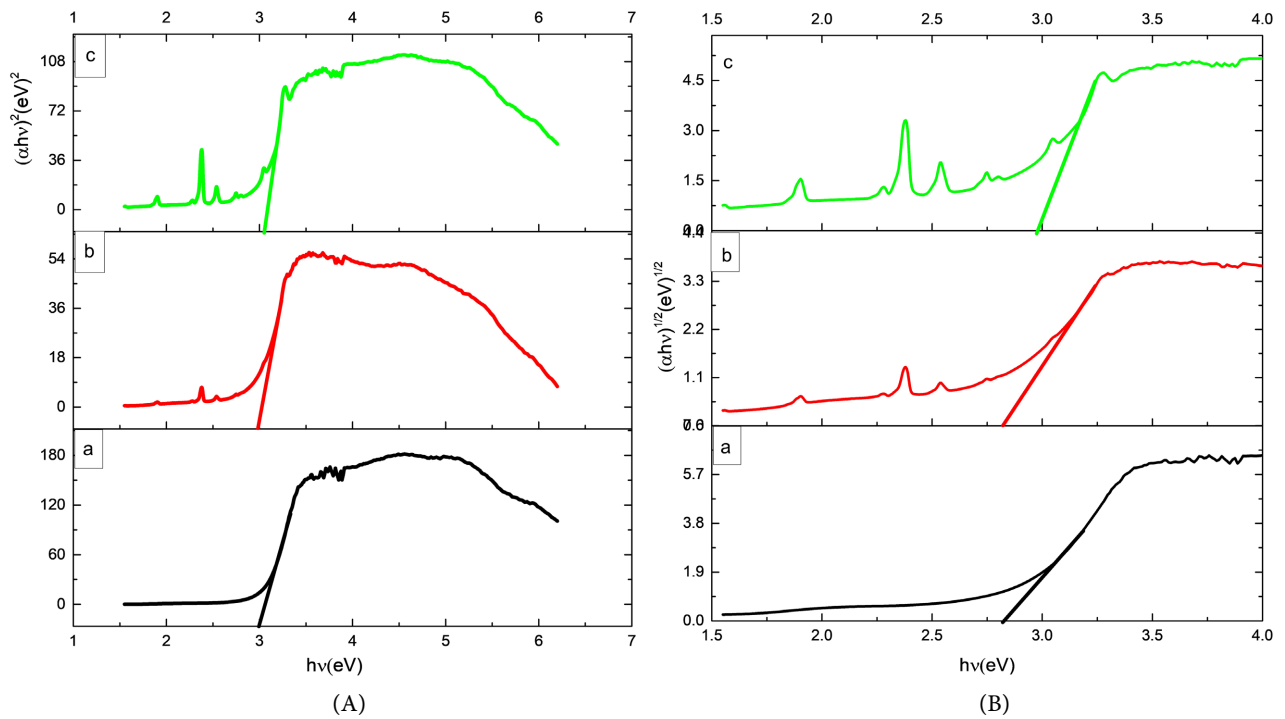


Figure 6. Absorption Tauc's plot of undoped and $C_{20}H_{12}O_5$ doped Boric Acid Glass (A) direct band gap, (B) indirect band gap.

Tauc's Plot can be used to determine the [13] energy band gap (E_g), by using the relation,

$$(\alpha h\nu)^n = A(h\nu - E_g) \quad [13]$$

where α is an absorption coefficient, A is any arbitrary constant and E_g is the energy band gap. For the indirect band gap, we use $n = 1/2$ and direct band gap $n = 2$. The $h\nu$ value can be calculated by using the relation $1240/\text{wavelength}$.

The E_g value for the undoped sample is nearly equal to 3 eV which is compared to other borate glasses as in reference [15]. The Indirect band gap value for the undoped sample is 3.07 eV, 0.05% $\text{C}_{20}\text{H}_{12}\text{O}_5$ doped glass is 3.01 eV, and 0.1% $\text{C}_{20}\text{H}_{12}\text{O}_5$ doped glass is 2.82 eV approximately which is comparable to other $\text{C}_{20}\text{H}_{12}\text{O}_5$ doped Glasses [19] as shown in Table 5. As we increased the concentration of $\text{C}_{20}\text{H}_{12}\text{O}_5$, the optical band gap decreased (Figure 7).

Table 5. Direct band gap, Indirect band gap, and refractive index of the $\text{C}_{20}\text{H}_{12}\text{O}_5$ doped and undoped boric acid glass.

Glass System	E_g (eV) (indirect)	Direct E_g (eV)	Refractive Index (n)
Undoped A	3.07	3.25	2.51
B	3.01	3.18	2.48
C	2.82	3.05	2.43
Fluorescein dye doped glass system [30]	2.80	3.00	2.41

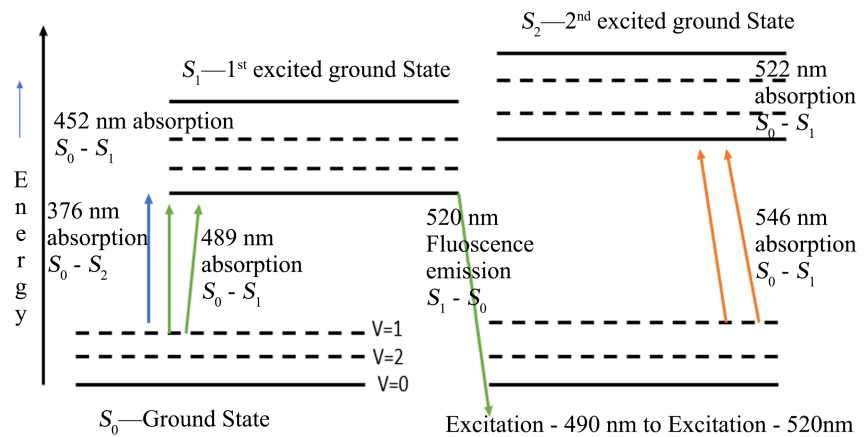


Figure 7. Energy level diagram of fluorescein dye doped glass.

Finally, the refractive index (n) is calculated by using the relation between the refractive index (n) and optical band gap (E_g), using the following relations [6]

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}}$$

where n values are found to be decreased with $\text{C}_{20}\text{H}_{12}\text{O}_5$ content.

3.5. Saturation Absorption and Optical Hysteresis of Fluorescein Dye-Doped BAG

The nonlinear transmission behavior of fluorescein dye-doped boric acid glass (BAG) was investigated using a continuous-wave He-Ne laser ($\lambda = 632.8$ nm, 20

mW) as the excitation source. The schematic of the experimental setup is shown in **Figure 8**. The laser beam was focused to a spot diameter of ~ 1.5 mm on the sample, corresponding to a peak intensity of ~ 1.13 W/cm². The sample thickness was measured as 1.2 ± 0.05 mm using a digital micrometer. The incident power was varied using a calibrated neutral density (ND) filter wheel (OD 0.1 - 2.0). The transmitted power was measured using a calibrated photodiode power meter. To avoid thermal damage, a dwell time of 5 sec was maintained at each power level and measurements were performed for three successive cycles, with reproducibility within $\pm 8\%$ [20]-[23].

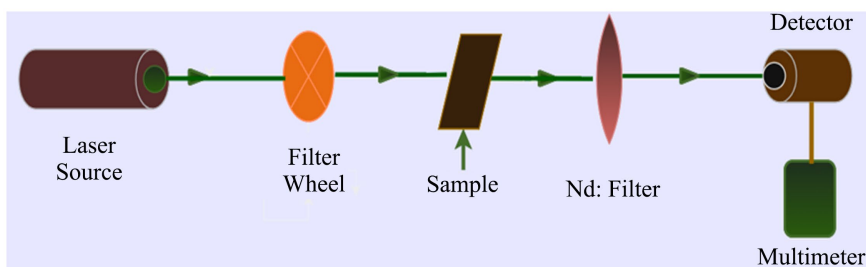


Figure 8. Schematic diagram for measuring saturation absorption using He-Ne laser 632.8 nm 20 mW, ND filter wheel, sample thickness 1.2 mm.

Figure 9 shows the saturation absorption curve (transmitted power vs incident power) for Sample C (0.10 mol% fluorescein). At low incident power (<5 mW), the transmittance increases linearly, following Beer-Lambert law. Above ~ 8 mW, the absorption saturates due to ground state depletion ($S_0 \rightarrow S_1$) of fluorescein molecules, and the transmitted power shows sub-linear increase. This saturable absorption is crucial for optical limiting applications where the material attenuates high-intensity light, protecting sensitive optical components and photodetectors [24] [25].

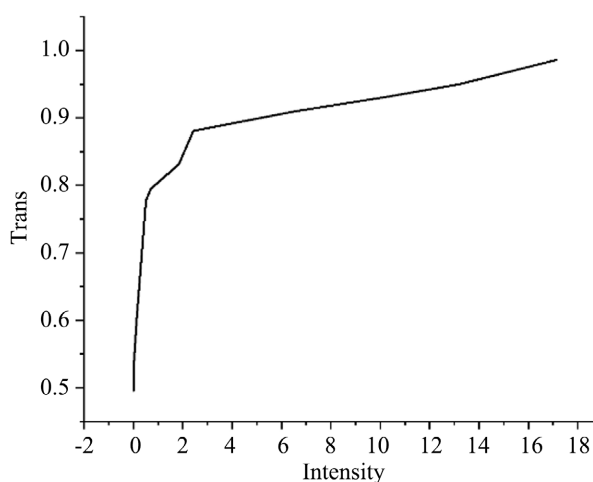


Figure 9. Saturation absorption behavior of fluorescein dye-doped BAG (Sample C) showing transmittance vs incident intensity-sub-linear at high intensity due to ground state bleaching.

Figure 10 shows the incident power vs transmitted power when cycled in increasing and decreasing directions. An optical hysteresis loop is observed with a loop width of $\Delta I \approx 0.8$ mW. At a given incident power (e.g., 10 mW), two distinct transmitted values are observed depending on the history (increasing/decreasing branch).

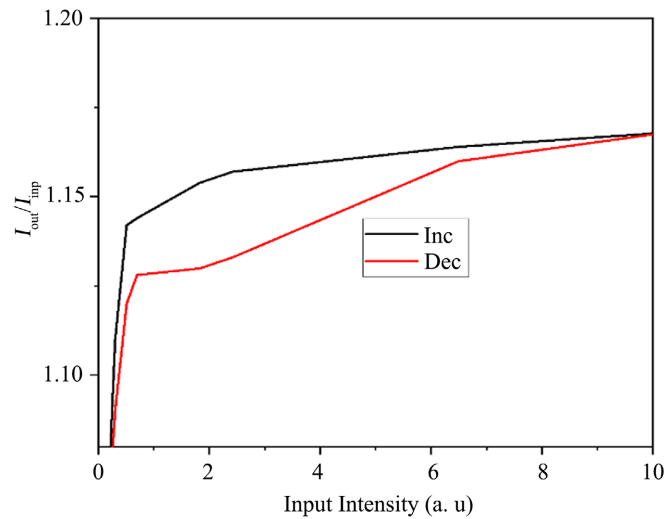


Figure 10. Optical hysteresis loop of fluorescein dye-doped BAG (Sample C) showing thermal hysteresis ($\Delta I = 0.8$ mW)-revised from bistability to photo-thermal hysteresis.

This behavior is attributed to photo-thermal induced refractive index change (thermal lensing) rather than pure electronic optical bistability [26]-[29]. The absorbed 632.8 nm energy by fluorescein dye is partially converted to heat via non-radiative $S_1 \rightarrow S_0$ internal conversion, leading to local temperature rise, change in refractive index (dn/dT) and formation of a thermal lens. This thermal effect produces a hysteresis loop which is often misinterpreted as electronic bistability. Therefore, we have revised the earlier claim of “optical bistability” to “thermal optical hysteresis” in accordance with the observed mechanism. The hysteresis width decreases from 1.2 mW (Sample D, 0.30%) to 0.6 mW (Sample B, 0.05%) with decreasing dye concentration, confirming its dye-concentration dependence. Such thermal hysteresis can be utilized in all-optical switching, optical memory and optical limiting devices operating at low power CW lasers [30] [31].

4. Conclusions

Fluorescein dye-doped boric acid glass system (BAG) with composition $70\text{H}_3\text{BO}_3 \cdot (10-X)\text{Pb}_3\text{O}_4 \cdot 20\text{ZnO} \cdot (X)\text{C}_{20}\text{H}_{12}\text{O}_5$ where $X = 0, 0.05, 0.10$ and 0.30 mol% was successfully synthesized by two-step melt quenching technique to avoid thermal decomposition of organic dye. Base glass $70\text{H}_3\text{BO}_3 \cdot 10\text{PbO} \cdot 20\text{ZnO}$ was first melted at 1050°C for 1 hour, quenched, ground, mixed with fluorescein dye and re-melted at 350°C for 15 min and quenched between stainless steel plates. XRD patterns show a broad hump centered at $2\theta \approx 27^\circ$, confirming amorphous nature of all samples with no crystalline fluorescein peaks, indicating homogeneous dissolution of dye in the borate matrix.

FTIR studies reveal characteristic borate bands: 425 - 450 cm^{-1} (B-O-B bending), 670 - 795 cm^{-1} (Zn-O, B-O-C interaction), 918 - 1153 cm^{-1} (B-O stretching in BO_4 units), 1368 - 1386 cm^{-1} (asymmetric stretching of BO_3 units with COO^- of fluorescein confirming dye retention at 1691 cm^{-1}), 3400 - 3868 cm^{-1} (O-H and C-H stretching). Raman spectra recorded with 532 nm excitation show bands at 96 - 88 cm^{-1} , 220 - 274 cm^{-1} (lattice vibrations), 464 - 485 cm^{-1} , 779 - 795 cm^{-1} (B-O-C interaction) and 1313 - 1349 cm^{-1} (C-C aromatic of fluorescein), confirming incorporation of dye.

UV-VIS absorption (200 - 800 nm) shows concentration dependent evolution: undoped glass shows only a weak band at 362 nm (Pb^{2+} related), while doped glasses show six bands at 376 nm ($\pi\text{-}\pi^*$ benzenoid $S_0 \rightarrow S_2$), 452 and 489 nm (xanthene monomer $S_0 \rightarrow S_1$), 522 and 546 nm (J-aggregate/anionic form $S_0 \rightarrow S_1$) and 653 nm ($n\text{-}\pi^*$ shoulder due to carbonyl interaction with Pb^{2+} matrix). The optical band gap calculated using Tauc relation $(\alpha h\nu)^{1/2} = A(h\nu - E_g)$ with absorption coefficient $\alpha = 2.303 \times A/t$ where $t = 1.2$ mm sample thickness and linear fitting in 3.2 - 3.8 eV range shows indirect band gap decreasing with dye concentration: 3.07 eV (A, undoped), 3.01 eV (B, 0.05%), 2.82 eV (C, 0.10%) and 2.80 eV (D, 0.30%). The refractive index calculated from the Dimitrov-Sakka relation $(n^2 - 1)/(n^2 + 2) = 1 - \sqrt{E_g/20}$ decreases from 2.51 to 2.41.

The fluorescence emission, excited at 490 nm, shows emission at 520 nm ($S_1 \rightarrow S_0$) explained by the Jablonski diagram of fluorescein (monomer and J-aggregate) in a rigid borate host.

Nonlinear transmission studies using a He-Ne laser (632.8 nm, 20 mW, 1.5 mm spot, 1.13 W/cm^2 , sample thickness 1.2 mm) show saturable absorption above 8 mW due to S_0 ground state depletion and thermal optical hysteresis loop width ~ 0.8 mW due to photo-thermal lensing (dn/dT) rather than pure electronic bistability, useful for optical limiting, optical switching and laser safety devices.

Thus, fluorescein-doped BAG glasses are promising candidates for luminescent solar concentrators, optical limiters and photonic switches.

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Data Availability

Data will be made available on request.

Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported

in this paper.

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