



# Fabrication, structure, physical properties and FTIR spectroscopy of zirconate doped-borophosphate bioglasses

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Received: 21 June 2023 / Accepted: 30 August 2023 / Published online: 20 September 2023  
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## Abstract

Samples of base calcium sodium borophosphate bioglasses doped with different amounts of zirconium oxide ( $ZrO_2$ ) were prepared via melt quenching technique. Synthesized samples of nominal composition  $xZrO_2-45B_2O_3-(24.5-x)CaO-24.5Na_2O-6P_2O_5$   $x=0$  (S0)—5 (S6) mol% was investigated using fourier transforms infrared (FTIR) spectroscopy and deconvolution analysis technique (DAT) was employed to investigate the variation of the four coordinated borons. X-ray diffraction (XRD) technique to approve the isotropic character of the prepared glasses. Density ( $\rho$ ) of samples varied from 2.606 g/cm<sup>3</sup> to 2.642 g/cm<sup>3</sup> and molar volume ( $V_m$ ) enhanced from 25.565 cm<sup>3</sup>.mol<sup>-1</sup> to 26.649 cm<sup>3</sup>.mol<sup>-1</sup>. FTIR spectroscopy revealed that only reliable bands were considered and the ( $r^2$ -value) of the regression was nearly 0.99. The maximum value of the difference between experimental and theoretical data not exceeds  $\pm 0.015$  from the normalized spectral data. The fraction of the four coordinated boron was slightly changed due to the addition of  $ZrO_2$  in the doping level. The decrease in the N4 values can be attributed to the structural changes combined with the conversion of BO<sub>4</sub> to BO<sub>3</sub> units.

**Keywords** Boron bioglasses · Zirconium oxide · XRD · FTIR

## 1 Introduction

The biomedical applications of glasses and glass–ceramic materials have received increased attention since Hench et. invention’s of bioglass in 1971 (Hench et al. 1971). Due to their biocompatibility and strong interaction with bone, these materials are widely used for biomedical purposes as orthopaedic implant and bone filler materials (Salinas et al. 2000; Hench 1993). Moreover, bioactive glass–ceramics play a significant role in the biomedical application because of their peculiar microstructure, greater mechanical strength compared to derived glass, and superior thermal conductivity (Hench 1993, 1991; Bairo et al. 2016). Furthermore, by allowing healthy tissue to grow on their surface through a layer of biologically active hydroxycarbonate apatite, they are capable of forming a direct link with the living bone without the development of surrounding fibrous tissue (HCA). The HCA phase that forms on the bioactive glass–ceramic surfaces is chemically and

physically similar to the mineral phase of bone. This correlation plays a significant part in establishing an interfacial connection between bone and bioactive substance (Hoppe et al. 2011).

Abo-Mosallam and Mahdy (Montazerian and Dutra Zanotto 2016) reported on the crystallisation characteristics of glasses with various amounts of fluorocanaseite and lithium disilicate. They came to the conclusion that fluorocanaseite and lithium disilicate glass–ceramics have potential use in dental application. The degradation and biocompatibility characteristics of the Na–Ca–phosphate and fluorapatite mixture in wollastonite–diopside glass–ceramic were studied by Mahdy et al. (Abo-Mosallam and Mahdy 2019).

Boron oxide ( $B_2O_3$ ) is the most common oxide that can be applied as a glass basis. Borate-based glasses have a number of physical and chemical advantages, including a low melting point and high transparency (Mahdy et al. 2021; Kirdsiri et al. 2009). Khattari et al. (Yasaka et al. 2014) reported that the insertion of  $ZrO_2$  in borate glasses structure leads to enhance their mechanical and optical properties. The insertion of rare earth ions such as  $Dy_2O_3$ ,  $Sm_2O_3$ , and  $Nb_2O_3$  into glass structures enhance their luminescence, physical, elastic, optical, and gamma ray shielding properties (Khattari et al. 2023; Divina et al. 2021; Bassam et al. 2023).

The use of borate glasses in biological applications is currently the subject of in-depth research (Arunkumar et al. 2023) as a result of the investigations conducted by Day et al. (M. Ottomeyer1 et al. 2016; Day et al. 2003; Huang et al. 2006; Brown et al. 2009). Because they are less chemically durable, borate glasses have the potential to be bioactive. These glasses can convert to hydroxyapatite (HA) partially or entirely more quickly thanks to the presence of borate in them. The osteogenic effects of borate on bone, namely on trabecular and alveolar type bone formation and maintenance, have already been demonstrated by many investigators and researchers working on bioactive glasses (Jia et al. 2010; Nielsen and Meacham 2011).

Krishnamacharyulu et al. (Gallardo-Williams et al. 2003) studied the physical properties of  $B_2O_3$ - $SiO_2$ - $P_2O_5$ - $Na_2O$ - $CaO$  doped with  $Ag_2O$  as bioglass systems for biomedical applications. They claimed that the increase of silver concentration leads to enhance the capacity of formation of hydroxyapatite (HA) layer on the surface of the samples. Kamal (Krishnamacharyulu et al. 2018) reported on physical and structure features of  $Ag_2O$ - $B_2O_3$  bioactive glasses. Abo-Naf et al. (Kamal 2014) examined the structure and mechanical properties of  $Na_2O$ - $CaO$ - $B_2O_3$ - $P_2O_5$  glasses, they concluded the mentioned glasses can be applied as bioactive glasses. Abdelghany et al. (Abo-Naf et al. 2015) proved that the zinc-borate glasses and glass–ceramics were promising biomedical materials. Herein, bioactive glass samples of zirconium doped Hench bioglass ( $ZrO_2$ - $B_2O_3$ - $CaO$ - $Na_2O$ - $P_2O_5$ ) have been fabricated. The structure, physical characteristics, and FTIR spectroscopy of the fabricated bioactive glasses have been investigated.

## 2 Experimental work

### 2.1 Sample preparation

Bioglass samples of nominal composition  $xZrO_2$ -45 $B_2O_3$ -(24.5-x) $CaO$ -24.5 $Na_2O$ -6 $P_2O_5$ ;  $x = 0$ –5 mol% were prepared using melt quenching technique. Analytical grade zirconium oxide used as received while calcium and sodium oxide was obtained from their carbonate partner. Ammonium dihydrogen phosphate and orthoboric acid was used

as a source of phosphorus pentoxide and boron oxide respectively. Weighted batches were put in porcelain crucible and sintered at 450 °C to remove carbonate, ammonia, and water. Temperature of the oven was raised gradually to 1200–1300 °C depending on glass composition. Obtained melts were swirled many times to obtain bubble free samples. The melts were then poured into stainless steel molds of required dimensions. Table 1 reveals sample nomination and composition.

## 2.2 Measurements

X-ray diffraction scans on a Cu K target with a secondary monochromator wave were performed using a PANalytical XPert PRO XRD system (where  $\lambda = 1.540$  and the tube operated at 45 kV-40 mA) (Holland).

Density of the samples was measured using Archimedes principle adopting Xylene of density ( $0.868 \text{ g/cm}^3$ ) as bayonet fluid.

Fourier Transforms Infrared (FTIR) spectral data was recorded using Nicolet is10 Thermo Fischer Co. within the range extended between ( $4000\text{--}400 \text{ cm}^{-1}$ ) adopting 64 scans with resolution  $2 \text{ cm}^{-1}$ .

## 3 Results and discussion

### 3.1 Sample nature

Figure 1 illustrates a photo of the prepared bio-glass samples in this work. The mentioned photo confirms that all prepared samples are clear, transparent, and free of bubbles.

Figure 2 reveals the XRD pattern of the S0, S1, S2, S4, S5, and S6 borate bio-glasses containing variable amounts of zirconium oxide measured within Bragg's angle extended between 5–70 degrees. The obtained pattern characterized by a broad hallow centered at about 25 degree characterize the non-crystalline of all prepared samples and pointing to the isotropic nature of the glasses.

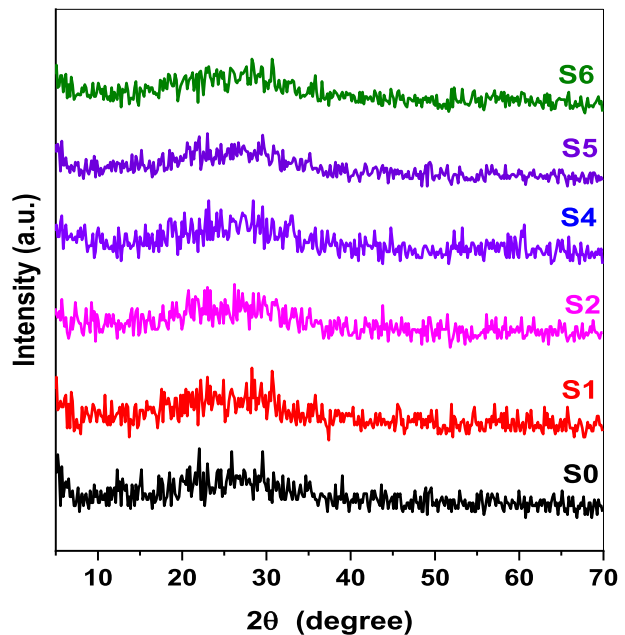
**Table 1** Samples code, chemical composition, density, and molar volume of the prepared samples in (mol%)

Sample code	Chemical composition in mol%					Density, $\rho$ (g/ $\text{cm}^3$ ) $\pm 0.001$	Molar volume, $V_m$ ( $\text{cm}^3/\text{mol}$ ) $\pm 0.001$
	ZrO <sub>2</sub>	CaO	NaO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	B <sub>2</sub> O <sub>3</sub>		
S0	0.0	24.5	24.5	6.0	45	2.606	25.565
S1	0.5	24	24.5	6.0	45	2.615	25.772
S2	1.0	23.5	24.5	6.0	45	2.618	25.910
S3	2.0	22.5	24.5	6.0	45	2.622	26.085
S4	3.0	21.5	24.5	6.0	45	2.632	26.248
S5	4.0	20.5	24.5	6.0	45	2.637	26.512
S6	5.0	19.5	24.5	6.0	45	2.642	26.649



**Fig. 1** A photo of the prepared borate bioglasses

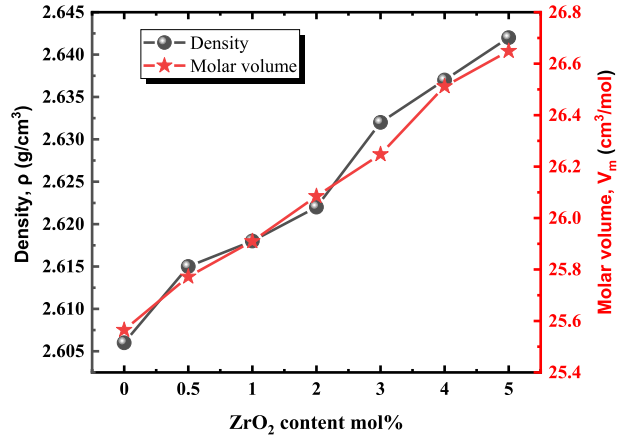
**Fig. 2** XRD pattern of the S0, S1, S2, S4, S5, and S6 prepared borate bioglasses



### 3.2 Physical characteristics

Table 1 and Fig. 3 depicts the measured densities ( $\rho$ ) of the prepared born bio-glasses (S0-S6). Results reveal that ( $\rho$ ) was ranged of values between  $2.606 \text{ g/cm}^3$  and  $2.642 \text{ g/cm}^3$ . Also, molar volume ( $V_m$ ) of the proposed samples was varied from  $25.565 \text{ cm}^3 \cdot \text{mol}^{-1}$  to  $26.649 \text{ cm}^3 \cdot \text{mol}^{-1}$ . As shown in Table 1 and Fig. 3, results of ( $\rho$ ) and ( $V_m$ ) indicate an increase in the same order. This may be attribute to the presence of heavy components such as  $\text{P}_2\text{O}_5$  and  $\text{Na}_2\text{O}_2$  in the glass networks may be partially responsible for this trend (Abo-Naf et al. 2015). The substitution of  $\text{CaO}$  with low density ( $3.34 \text{ g}\cdot\text{cm}^{-3}$ ) by  $\text{ZrO}_2$  with higher density ( $5.68 \text{ g}\cdot\text{cm}^{-3}$ ), may also contribute to the observed increase in physical quantities. In addition, the enhancement in the  $\rho$  and  $V_m$  of S0-S6 samples can be ascribed to the greater molecular weight of  $\text{ZrO}_2$  when compared to sodium oxide and the increase

**Fig. 3** Density ( $\rho$ ) and molar volume ( $V_m$ ) as a function of  $ZrO_2$  content of the prepared borate bioglasses

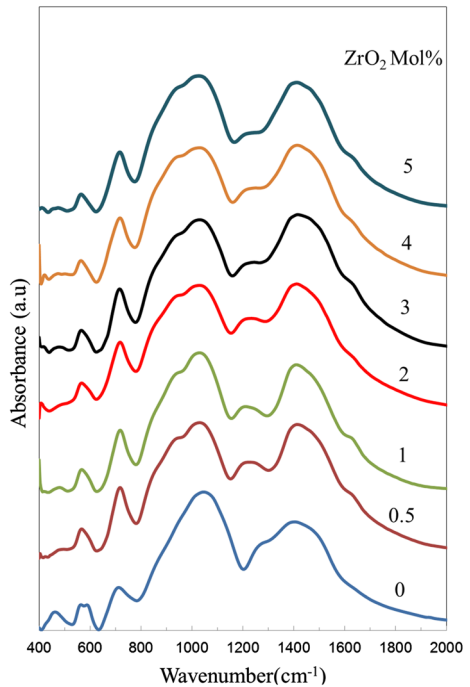


in oxygen atoms in the mixture. These observations indicate that the glass network has become more open with a less dense structure (Abdelghany et al. 2014; El-Batal 2008).

### 3.3 FTIR spectroscopy

Figure 4 shows the FTIR spectrum data of the prepared glasses in the range 4000–400  $cm^{-1}$ . Around 3434  $cm^{-1}$ , the broad band resembling a water molecule was seen and was attributed to the O–H stretching vibrational modes. While the band at 1200–1600  $cm^{-1}$  BO

**Fig. 4** FTIR spectral data within the region 4000–400  $cm^{-1}$  of the prepared bioglasses



stretching vibrations and the peak seen at 930–1197  $\text{cm}^{-1}$  BO tensile and stretching vibrations, respectively, of the trigonal BO-3 units of the tetrahedral (BO-4) units. Correlated with 700–720  $\text{cm}^{-1}$  was the borate network's B-O-B bond bending. The P-O-B function group is represented by the band at 1228  $\text{cm}^{-1}$ . The cation modifier is identified by the band at 400–600  $\text{cm}^{-1}$ .

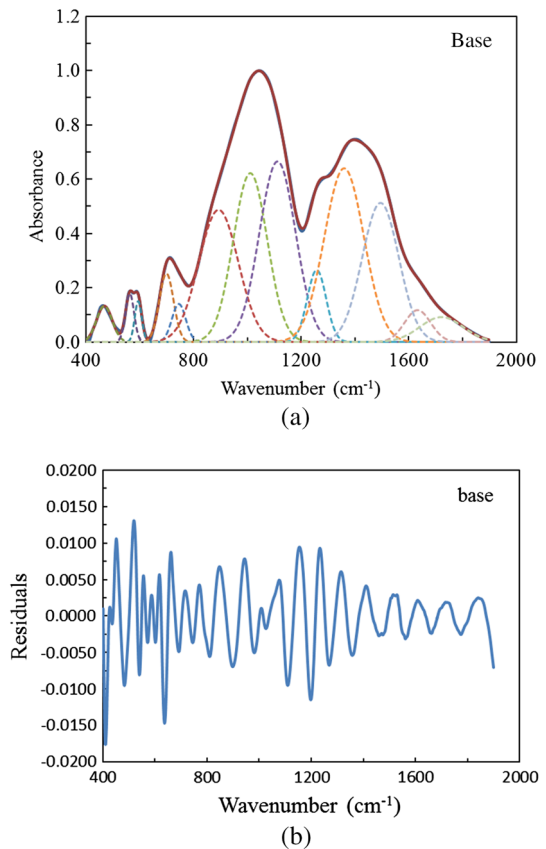
The  $\text{BO}_3$  and  $\text{BO}_4$  peaks relative areas were integrated to calculate the N4 using the next formula:

$$N_4 = \frac{BO_4}{BO_4 + BO_3} \quad (1)$$

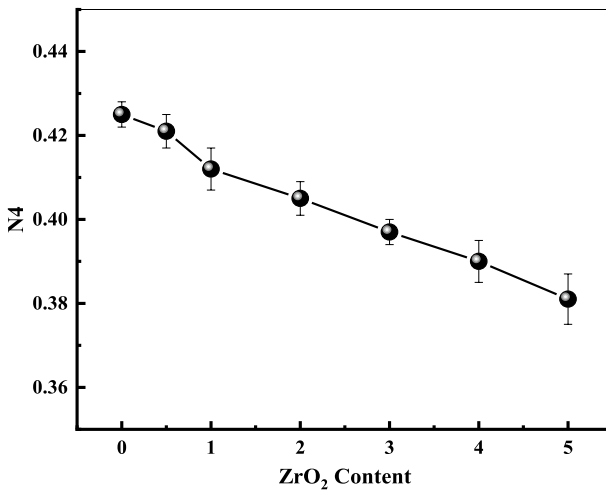
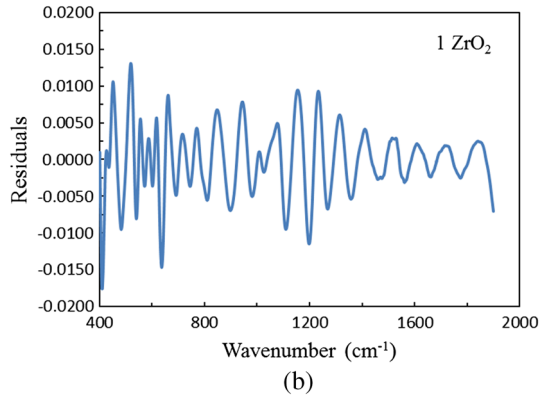
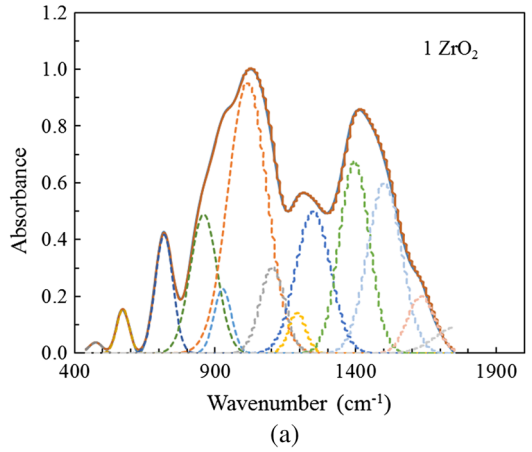
Figure 5a and b shows the deconvoluted spectral data of the base bioglass sample (0  $\text{ZrO}_2$ ) and the variation of residuals within the studied spectral range. Figure 6a and b shows the deconvoluted spectral data of the S2 bioglass sample (1 mol%  $\text{ZrO}_2$ ) and the variation of residuals within the studied spectral range. It was noticed that only reliable bands were considered and the ( $r^2$ -value) of the regression was nearly 0.99. In addition, the maximum value of the difference between experimental and theoretical data not exceeds  $\pm 0.015$  from the normalized spectral data.

To calculate the influence of BO4 on the change in the relative population of tetrahedral units BO4 and triangular units BO3, Fig. 7 plots the N4 as a function of BO4 concentration.

**Fig. 5** **a** Deconvoluted spectral data of the base sample; **b** Variation of residuals of the base sample within the studied spectral range



**Fig. 6** **a** Deconvoluted spectral data of the S2 sample; **b** Variation of residuals of the S2 sample within the studied spectral range



**Fig. 7** The N4 as a function of ZrO<sub>2</sub> content in the fabricated bioglasses

The levels of N4 were found to vary depending on the BO4 concentration. It would seem that the presence of BO3 tends to reduce the number of BO4 units.

The presence of BO<sub>3</sub> and BO<sub>4</sub> structural groups was combined with the appearance of specific peaks within the FTIR spectra of the studied glasses related to such vibrational groups that are usually overlapped leading to the smearing of the less intense peaks.

Gaussian convolutions can be observed in the studied FTIR spectral data in the form of broadening or shoulders in descending lobes. The inverse problem, deconvolution of such data can help in understanding the structural variation resulting from the change in the composition of the studied glassy sample for specific applications. Spectral bands are usually considered as multiple overlapping Gaussian peaks where their sum can correlate with the experimental measured one. The suggestion of the number and position of the convoluted peaks based both on previously analyzed spectral data of similar glasses containing nearly similar forming oxides presented in literature and/or the second derivative of the experimental normalized FTIR data after corrections for background and dark current noises (Goel et al. 2008; Abdelghany 2010). Some authors (Abdelghany and Behairy 2020; Moustafa et al. 1994; Hammad et al. 2016) suggest too many peaks to calculate the relative area of each broad band based on statistical measures representing the proportion of the variance of dependent variables correlated with independent on in the regression model (R-squared or  $r^2$ ) which are considered inaccurate. The examined parameters of all detected peaks using Gaussian deconvoluted for S1-S6 samples are tabulated in Table 2.

In addition, it was observed that the fraction of the four coordinated boron was slightly changed due to the addition of the zirconium oxide in the doping level. The decrease in the N4 values can be attributed to the structural changes combined with the conversion of BO4 to BO3 units. Such changes are usually combined with destroying BO4 units that may be converted to either BO<sub>3</sub> or/and BO<sub>2</sub>O<sup>-</sup> units with the generation of non-bridging oxygens. Therefore, the addition of zirconium (ZrO<sub>2</sub>) ions in such glass appears to influence the glass network neighboring BO3 and zirconium cation which leads to the formation of both

**Table 2** Parameters of deconvoluted bands of S1-S6 bioglasses

Sample	S1		S2		S3		S4		S5		S6	
	Center	R.A	Center	R.A	Center	R.A	Center	R.A	Center	R.A	Center	R.A
1	–	–	571.80	0.16	573.14	0.17	570.60	0.16	568.85	0.16	571.89	0.20
2	714.58	0.05	716.00	0.43	716.68	0.48	716.01	0.46	712.83	0.47	716.93	0.50
3	863.05	0.10	847.45	0.37	837.20	0.44	836.88	0.36	852.90	0.62	850.05	0.55
4	933.07	0.04	944.54	0.69	917.40	0.60	919.83	0.54	927.25	0.47	924.00	0.44
5	1027.98	0.29	1054.88	0.83	1026.75	0.92	1049.14	0.95	1009.17	0.89	1016.85	0.95
6	1131.07	0.10	–	–	1111.46	0.32	–	–	1090.25	0.43	1100.72	0.38
7	–	–	–	–	1188.46	0.25	–	–	–	–	1198.56	0.58
8	1262.68	0.09	1227.54	0.59	1249.03	0.59	1225.78	0.47	1223.01	0.65	1272.69	0.38
9	1335.73	0.06	1388.34	0.62	1393.43	0.89	1382.26	0.85	1393.40	0.79	1382.80	0.73
10	1374.71	0.06	–	–	–	–	–	–	–	–	–	–
11	1466.77	0.16	1495.00	0.68	–	–	–	–	–	–	1499.50	0.71
12	1591.63	0.05	–	–	1502.85	0.57	1513.83	0.60	1511.40	0.66	–	–
13	–	–	1629.70	0.22	1609.75	0.29	1634.12	0.11	1632.10	0.20	1630.92	0.14
14	–	–	–	–	–	–	1679.27	0.23	–	–	1691.08	0.18
15	–	–	1728.46	0.11	1736.21	0.14	570.60	0.16	1721.54	0.15	–	–



BO3 and ZrO4 structural groups. Such changes can be correlated with minor changes in both density and molar volume as shown in Table 1 and Fig. 3.

## 4 Conclusion

In this study, the direct influence of zirconate oxide ( $ZrO_2$ ) on structure, density, molar volume, and FTIR spectroscopy of fabricated Hensch bioglasses has been examined. The fabricated bioglass samples had the nominal composition  $xZrO_2-45B_2O_3-(24.5-x)CaO-24.5NaO_2-6P_2O_5$ , where  $x=0-5$  mol% and prepared via the melt quenching route. Fabricated bioglasses were coded as S0, S1, S2, S3, S4, S5, and S6 corresponding to  $x$  values. The amorphous nature of the prepared bioglasses proved via XRD measurements. Density ( $\rho$ ) of samples was changed from  $2.606\text{ g/cm}^3$  and  $2.642\text{ g/cm}^3$  and molar volume ( $V_m$ ) was varied from  $25.565\text{ cm}^3\cdot\text{mol}^{-1}$  to  $26.649\text{ cm}^3\cdot\text{mol}^{-1}$ . Only trustworthy bands were taken into account, according to FTIR spectroscopy, and the regression's ( $r^2$ -value) was close to 0.99. In terms of normalized spectral data, the maximum value of the discrepancy between experimental and theoretical data is limited at 0.015. The addition of  $ZrO_2$  to the doping level slightly altered the proportion of the four coordinated boron. The structural modifications and the conversion of BO4 to BO3 units are to blame for the fall in N4 values.

**Acknowledgements** The authors express their gratitude to Princess Nourah bint Abdulrahman University Researchers Supporting Project number (PNURSP2023R28), Princess Nourah bint Abdulrahman University, Riyadh, Saudi Arabia.

**Author contributions** HA: Assisted in data collection & analysis and rearranging the manuscript first draft; NAMA: Conceptualization, review & editing, writing manuscript first draft; FA: Assisted in data analysis; ZYK: Assisted in data analysis and drawing the figures; AMA: Conceptualization, review & editing, writing manuscript first draft; YSR: Supervision and finalized the last version of the manuscript. All authors read and approved the final manuscript.

**Funding** To Princess Nourah Bint Abdulrahman University Researchers Supporting Project Number (PNURSP2023R28).

**Availability of data and materials** All data generated or analyzed during the study are included in this article.

## Declarations

**Conflict of interest** Authors declare no conflict of interest directly or indirectly related to the work submitted for publication.

**Ethical approval** Not Applicable.

**Consent to participate** Not Applicable.

**Consent for publication** Not Applicable.

**Informed consent** Not Applicable.

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