

# Advantage of NMR and FTIR Spectroscopy to Determine Structure Role of CeO<sub>2</sub> in Complicated Borosilicate Glasses: New Approach

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## Abstract

Microstructure of complicated glasses in the system 30Na<sub>2</sub>O-2Al<sub>2</sub>O<sub>3</sub>-25 SiO<sub>2</sub>-xCeO<sub>2</sub> (43-x) B<sub>2</sub>O<sub>3</sub>, x changes from 0.5 to 20 mol% have been extensively studied. Structural determination of glasses containing high cerium oxide content (≥8 mol% CeO<sub>2</sub>) was carried out by <sup>11</sup>B NMR and FTIR spectroscopy. On the other hand, <sup>29</sup>Si MAS NMR experiment is hardly to be applied to glasses of CeO<sub>2</sub> > 8 mol%. This is due to the paramagnetic action which is raised by cerium cations causing dilution or delaying in the resonance phenomenon. It is evidenced from NMR data that sodium oxide is high enough to modify the glass forming units which constitute the skeleton of the glass. Ceria is as well as silica and B<sub>2</sub>O<sub>3</sub> all are acting as glass forming species. Decreasing of both fraction of boron tetrahedral units (N<sub>4</sub>) and chemical shift of silicon nuclei (δ) confirm the role of CeO<sub>2</sub> as a glass former. On the other hand, fast decrease in N<sub>4</sub> and chemical shift of Si nuclei with further increasing CeO<sub>2</sub> contents (≥8 mol%) gives a clear evidence that the ability of cerium oxide to participate as a network former increases with increasing its content. New approach is applied to determine the fraction of CeO<sub>4</sub> as a glass forming units. In this approach, we use the common advantage of <sup>11</sup>B NMR and FTIR spectroscopy to obtain Ce<sub>4</sub> fraction. The latter species cannot be determined from NMR spectroscopy, since very high relaxation time and magnetization of ceria cause intensive spectral broadening which prevent resonance spectra to be appeared.

## Keywords

NMR Spectroscopy, Cerium Oxide, New Approach, Glasses

## 1. Introduction

It was reported previously [1] [2] [3] that some types of borosilicate glasses

(BSG) have an interested academic and scientific priority as well as technical applications. Growing achievement in field microelectronics technology requires new types of glasses which may be used as sealants, particularly, for molten carbonate fuel cells (MCFC) [3] [4]. Specific types of borosilicate glasses have long been the subject of structural studies [2] [5] [6] [7]. This is because of their interest in both technical and academics considerations.

The structure of borosilicate glasses is based on the base former units which are the main constituents of the glass network. These units are designed as,  $Q^n$  in silicate  $[\text{SiO}_4]$  and  $N_4$  or  $B_4$  in borate containing  $[\text{BO}_3]$  and  $[\text{BO}_4]$  structural species. The  $[\text{BO}_3]$  species would be formed in both symmetric and asymmetric configurations. While  $[\text{BO}_4]$  units are formed in the symmetric tetrahedral coordination. As a result of mixing between borate and silicate matrices, the oxygen atoms can be bonded to boron and silicon or in some cases to silicon atoms or boron only, and have a  $\text{Na}^+$  and/or  $\text{Ce}^{2+}$  as charge compensators. The distribution of the borate and silicate structural units depends on the ratios of  $\text{Na}_2\text{O}/\text{B}_2\text{O}_3$  and  $\text{SiO}_2/\text{B}_2\text{O}_3$  [2] [5] [6] which are designed as R and K structural factor. For all K values,  $N_4$  increases up to a maximum in between 0.5 and 0.75 for  $R = 1$ , and the value of this maximum increases with increasing K values. At specific value of K, the proportion of  $N_4$  slowly decreases with increasing R values.

In order to test the possible quantitative use of NMR and FTIR spectroscopy, glasses of two individual composition regimes have been prepared and measured. One contains an extremely low concentration of  $\text{CeO}_2$  and the other enriched with it. The first region contains a limited concentration from  $\text{CeO}_2$  as a paramagnetic material. In such a case,  $^{29}\text{Si}$  NMR study can easily be applied to obtain  $Q^n$  values of different borosilicate glasses. On the other hand, the second type of borosilicate glasses contains further high level from  $\text{CeO}_2$  ( $\geq 8$  mol) which limit the advantage of  $^{29}\text{Si}$  NMR measurements to be used. This is because the high spins magnetic moment of magnetic cations such as cerium and iron produces sufficient broadening of Si NMR lines [2] [8]. As a result, different contributions cannot be resolved and broader non featured and unobservable spectra can be considered.

$^{11}\text{B}$  and FTIR spectroscopy are not affected by adding even more concentration from paramagnetic species from  $\text{CeO}_2$ . Therefore, these tools would be simply applied to obtain complementary data. It can be used as a quantitative tool applied to determine structural fractions, such as  $N_4$  in borate network. In this study,  $^{11}\text{B}$  NMR & FTIR spectroscopy can be applied for all glass compositions while  $^{29}\text{MAS}$  NMR technique is limited to low  $\text{CeO}_2$  concentration (8 mol%).

## 2. Experimental

### 2.1. Sample Preparation

The glasses were prepared from reagent grade  $\text{SiO}_2$ ,  $\text{H}_3\text{BO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{Al}_2\text{O}_3$  and  $\text{CeO}_2$ . The melting process was carried out using alumina crucibles at a temperature ranging from  $1250^\circ\text{C}$  to  $1520^\circ\text{C}$  depending on composition. After swirling

the melt several times to ensure good homogeneity and air bubble free, the melt was quenching over a stainless steel plate and pressed to obtain the desired shape.

## 2.2. Nuclear Magnetic Resonance

All NMR measurement have been carried out on glass sample in form of powder. The measurements were carried out via JEOL GSX-500 high-resolution solid-state MAS NMR spectrometer (Mansoura University-EGYPT) in a magnetic field of 11.75 T. A specific frequency of 99.3 MHz is applied to record  $^{29}\text{Si}$  MAS NMR spectra. A spinning frequency of 6 kHz is applied to a cylindrical zirconia sample holder to rotated at a speed depends on the type of the measured nuclei. The Signal of pulse length of 2.62  $\mu\text{s}$  and a recycle delay of 30 s is applied to record  $^{29}\text{Si}$  NMR signal. Around 1000 - 2000 scans were accumulated to get good spectrum.  $^{11}\text{B}$  MAS NMR spectra were recorded at a frequency of 160.4 MHz and spinning rate of 15 KHz. The glass samples were measured with a single pulse length of 0.5 - 1.0 ms and a pulse delay of 2.5 s, and an accumulation of 100 - 200 scans.  $^{27}\text{Al}$  Mas NMR spectra were recorded at a frequency of 130.3 MHz and spinning rate of 6 KHz.

## 2.3. FTIR Measurements

Fourier transform infrared absorption signals of the studied glasses were measured at room temperature in the wavelength range 4000 - 400  $\text{cm}^{-1}$  using a computerized recording FTIR spectrometer (Mattson 5000, USA). Fine powdered samples were mixed with KBr in the ratio 1:100 for quantitative analysis and the weighed mixtures were subjected to a load of 5  $\text{t}/\text{cm}^2$  in an evocable *i.e.* to produce clear homogenous discs. Then, the IR absorption spectra were immediately measured after preparing the discs to avoid moisture attack.

## 3. Results and Discussion

The studied borosilicate glasses are investigated by  $^{23}\text{Na}$ ,  $^{29}\text{Si}$ ,  $^{27}\text{Al}$  and  $^{11}\text{B}$  NMR to offer deeper insight into the structure of a given glass. The fraction of bridging (BO) and nonbridging oxygen atoms (NBO) as a consequence can simply be determined. The NMR results also make a distinction for three-fold coordinated boron B3 between symmetric (B3 with 3BO or 3NBO) and asymmetric (B3 with one or two NBOs) boron, respectively B3s and B3a.

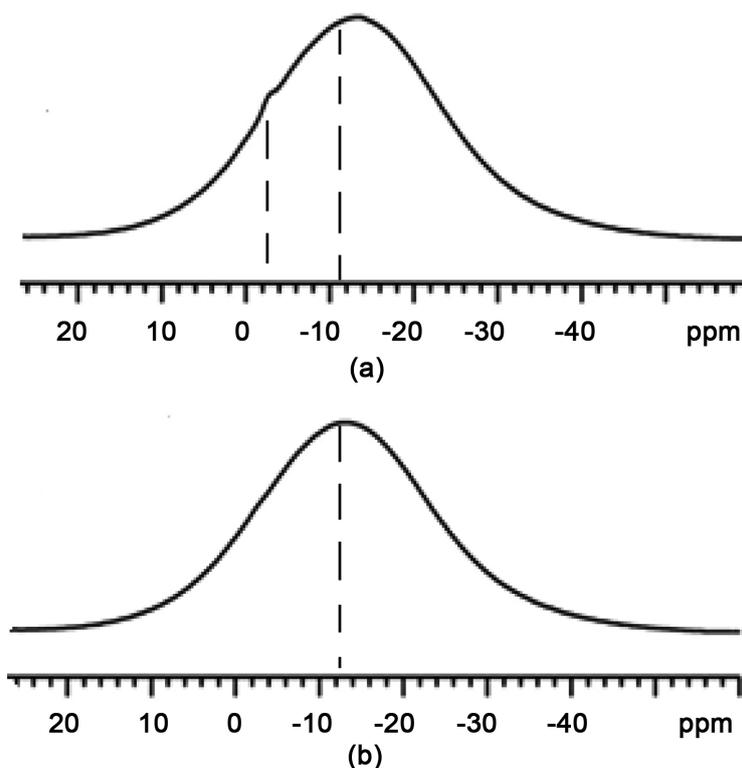
The structural features of modified borosilicate' glasses have been shown to depend on the field strength (*i.e.* charge/radius) of the cation introduced. It is evidenced from NMR investigations that increasing  $\text{CeO}_2/\text{Na}_2\text{O}$  molar ratio will result in promotion the capacity of bonds between different forming species in the glass matrix. This is appeared from a continues decrease in both NBO and  $\text{N}_4$  fraction species. These features are correlated to the higher field strength of the  $\text{Ce}^{2+}$  cation as compared to  $\text{Na}^+$  one. NBOs are preferentially associated with the higher field strength cation  $\text{Ce}^{2+}$ . which results in reducing its content in the investigated glasses.

### 3.1. Cerium Free Borosilicate Glass

#### $^{23}\text{Na}$ , $^{27}\text{Al}$ , $^{11}\text{B}$ and $^{29}\text{Si}$ MAS NMR

NMR spectroscopy of sodium in glasses can be considered as a powerful measuring tool to follow the level of precipitation, crystallization and verification of structural species in glass matrix. For example, **Figure 1(a)** presents  $^{23}\text{Na}$  NMR spectra corresponding to  $\text{Na}_2\text{O}$  distribution as a modifier in glass matrix, since  $\text{Na}_2\text{O}$  is participated between the different species which forming the glass network. The feature of appeared spectrum is clearly differed from that of spectrum (b), leading to formation of additional structural groups in glass network. Presence of weak peak at 2 ppm is considered to be due to accumulation and precipitation of even little concentration from modifier cations in glass phase to form some types of clusters. This consideration is previously reported, since  $\text{Na}_2\text{SiO}_3$  crystalline phase is structurally identified in ternary alkali silicate glasses by different techniques [2] [9] [10] [11]. The broadening of spectra is considered as good evidence for incorporation of sodium in the network as a glass modifier [2] [6].

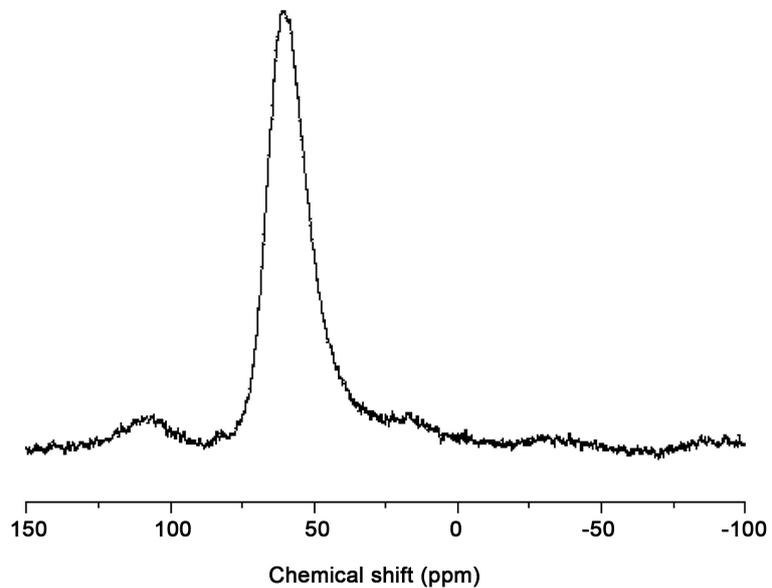
$^{27}\text{Al}$  NMR spectrum presented in **Figure 2** showed that  $\text{Al}_2\text{O}_3$  enters the glass in tetrahedral configuration with oxygen atoms as a first neighbor. The chemical shift of the  $^{27}\text{Al}$  spectrum is 59.6 ppm which is referred to  $\text{AlO}_4$  species as glass forming units. The well formed  $\text{AlO}_4$  units aren't looking as isolated but it was linked with  $\text{SiO}_4$  groups and therefore, Al-O-Si mixed bridges are the product [2] [5].



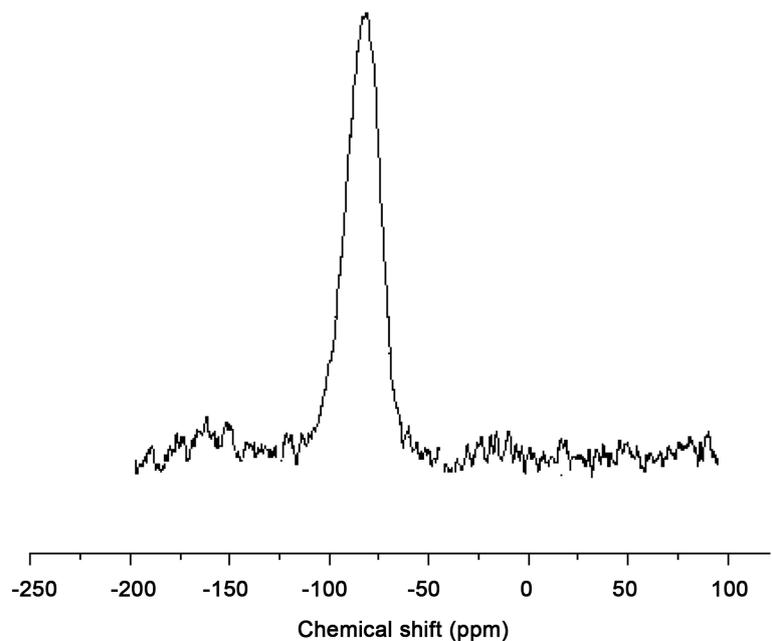
**Figure 1.**  $^{23}\text{Na}$  NMR spectra of borosilicate glass. (a) for glass containing clustered Na atoms, (b) represent Na as a modifier only.

**Figure 3** is  $^{29}\text{Si}$  NMR spectrum of borosilicate glass free from  $\text{CeO}_2$ . The chemical shift ( $\sigma$ ) of base glass is listed at  $-86.6$  ppm and is related to  $\text{Q}^2$  species [2] [5] [6]. This leads that  $\text{Na}_2\text{O}$  as modifier oxide is consumed to break two bridging bond as an average per each tetrahedral  $\text{SiO}_4$  unit. Some of tetrahedral aluminum may shield silicon nuclei through forming  $\text{Si-O-Al}$  bonds. Then the chemical shift of ( $-86.6$ , ppm) would be related to  $\text{Q}^2$  species or  $\text{Q}_3^4$  which means the 3 from oxygen atoms around the silicon are NB type, one connected to Al atom via BO.

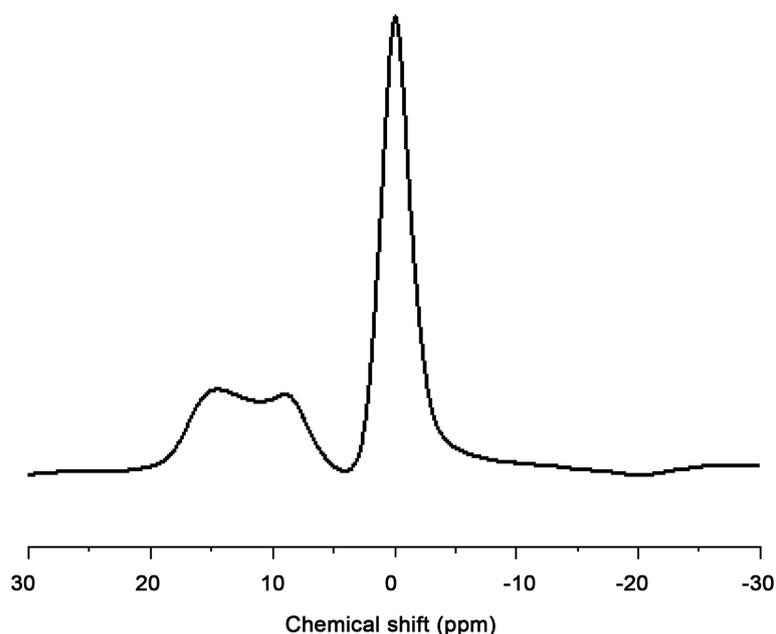
**Figure 4** presents  $^{11}\text{B}$  NMR spectrum of cerium free borosilicate glass. The



**Figure 2.**  $^{27}\text{Al}$  NMR spectrum of cerium free borosilicate glasses.



**Figure 3.**  $^{29}\text{Si}$  MAS NMR spectrum of cerium free glass.



**Figure 4.**  $^{11}\text{B}$  NMR spectrum of borosilicate glass.

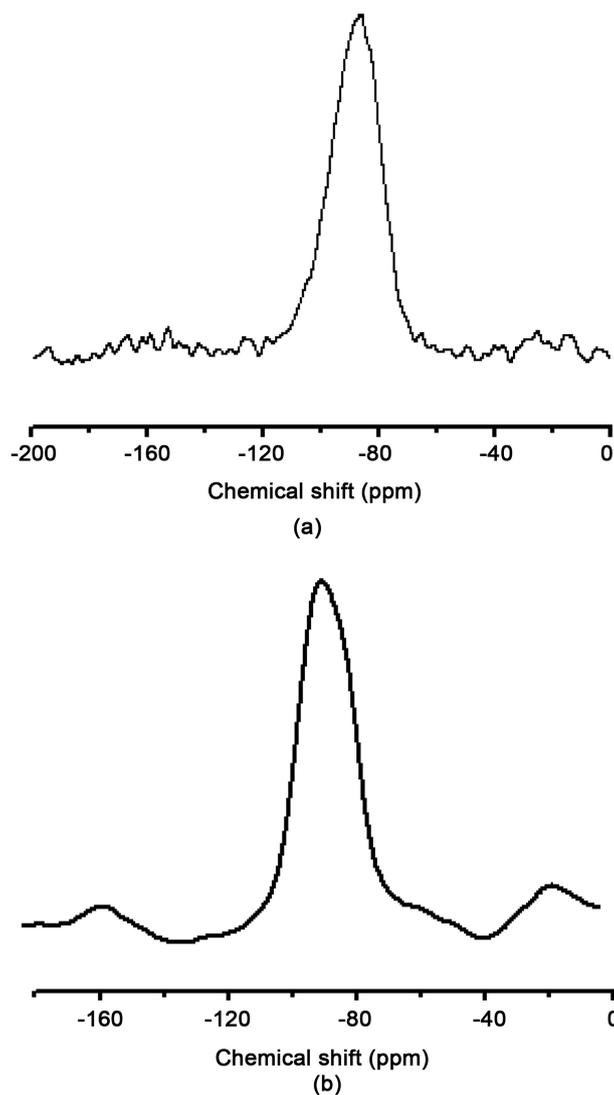
chemical shift ( $\sigma$ ) of the main signal is appeared at 0 ppm and the broad signal is appeared between 5 - 22 ppm which is assigned to both symmetric and asymmetric  $\text{BO}_3$  species [11]. The determined value of fraction of transformed borons ( $N_4$ ) is equal to 0.64 which means that 64% from total boron is found in tetrahedral configuration with oxygen atoms.

### 3.2. Cerium Containing Borosilicate Glasses

#### $^{11}\text{B}$ and $^{29}\text{Si}$ NMR Results

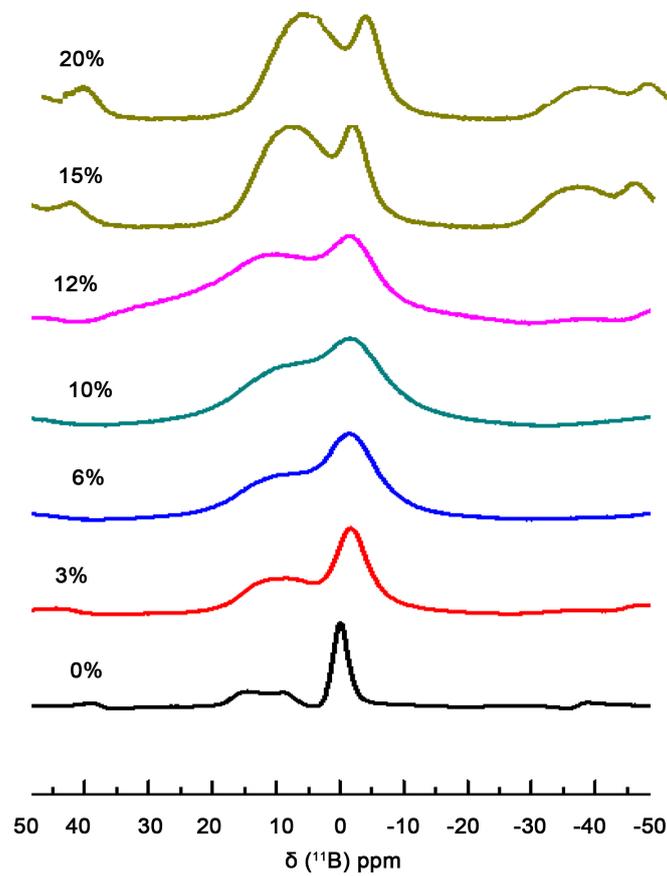
**Figure 5** showed  $^{29}\text{Si}$  NMR spectra for two glasses containing different concentration from  $\text{CeO}_2$  (3 and 6 mol%). The chemical shift of glass containing 6 mol%  $\text{CeO}_2$  is lower ( $-92.4$  ppm) than that of glass containing 3 mol%. ( $-87$  ppm). The difference between the two values of chemical shift is extremely high (6 ppm) which confirm the effective role of cerium in changing the network structure even upon a small addition. In terms of chemical shift consideration, we conclude that  $\text{CeO}_2$  plays a role of strong glass former, since more shielded silicate units are formed upon  $\text{CeO}_2$  addition. In such situation  $\text{Q}^3$  species are the more formed unit. As a consequence, tetrahedral silicate units containing only one non bridging oxygen atom are the main product in sample containing 6 mol%  $\text{CeO}_2$ .

The experimental  $^{11}\text{B}$  MAS NMR spectra for glasses involving different concentration from  $\text{CeO}_2$  are shown in **Figure 6**. The lowest spectrum is related to sodium borosilicate glass (free from  $\text{CeO}_2$ ). NMR spectra for glasses of higher  $\text{CeO}_2$  concentrations are also presented in the same figure. It can be seen from this figure that there is a remarkable changes in the spectral features upon increasing  $\text{CeO}_2$  contents. Increasing  $\text{CeO}_2$  at expense of  $\text{B}_2\text{O}_3$  is noticed to have no remarked effect on the chemical shifts ( $\sigma$ ) of tetrahedral boron ( $\text{BO}_4$ ), since ( $\sigma$ ) is

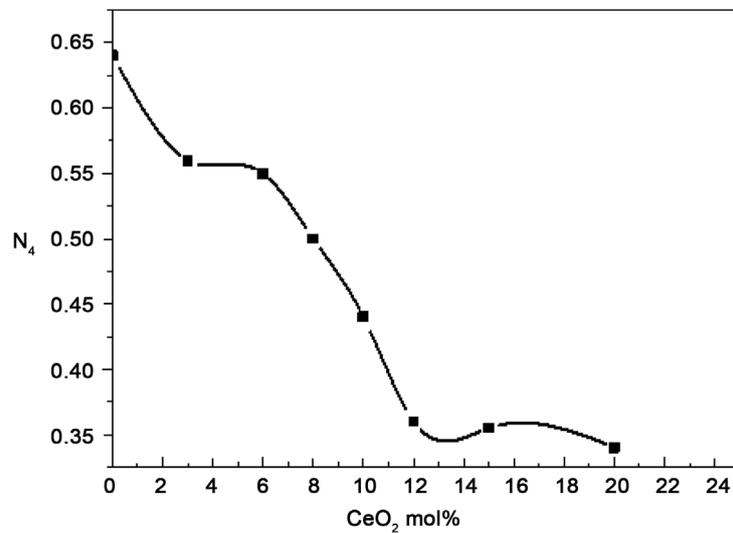


**Figure 5.** (a) and (b) NMR spectrum of glass containing 3 and 6 mol%  $\text{CeO}_2$ , respectively.

still fixed around 0 ppm for all investigated samples. But the fraction of boron tetrahedral units ( $N_4$ ) is only affected, since it changed from 0.63 to 0.32 upon addition of 20 mol%  $\text{CeO}_2$ , See **Figure 7**. This means that substitution of  $\text{B}_2\text{O}_3$  with  $\text{CeO}_2$  should result in decreasing the concentration of  $\text{BO}_4$  units in the borate network through lowering transformation of  $\text{BO}_3$  triangle units to  $\text{BO}_4$  groups. It can be seen from **Figure 7** that the relative area characterizing  $\text{BO}_3$  units increases with increasing  $\text{CeO}_2$  contents and reverse behavior is shown indicating decreasing  $\text{BO}_4$  concentration. This means that portion of  $\text{Na}_2\text{O}$  which is responsible for boron transformation is also decreased. This may be because the major part of the cerium inters as a glass former. As a result, most of modifier would be firstly consumed to form  $\text{CeO}_4$  groups and the rest can be distributed between the borate and silicate network. Therefore, the fraction of tetrahedral boron  $N_4$  is hardly decreased with increasing  $\text{CeO}_2$  content which support that  $\text{CeO}_2$  has great ability to withdraw  $\text{Na}_2\text{O}$  from borate network.  $\text{CeO}_2$  is therefore



**Figure 6.**  $^{11}\text{B}$  NMR spectra of selected composition of modified borosilicate glasses containing  $\text{CeO}_2$ .



**Figure 7.** Fraction of boron tetrahedral as a function of  $\text{CeO}_2$  concentration.

played the role of glass former, since it consumes some of  $\text{Na}_2\text{O}$  as a modifier to build  $\text{CeO}_4$  groups. Formation of the latter groups is expected to grow at expense of both  $\text{BO}_4$  and  $\text{NBO}$  units which may result in reducing the concentration of fraction of tetrahedral boron  $N_4$  with increasing  $\text{CeO}_2$  concentration. In com-

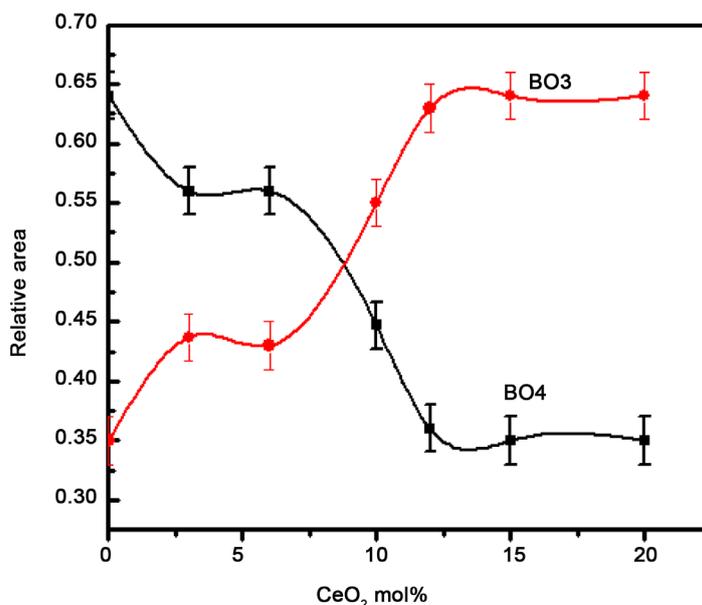
parison, glass of higher CeO<sub>2</sub> content has lower fraction of BO<sub>4</sub> than that of cerium free glass. Generally, N<sub>4</sub> showed abrupt decreasing trend upon increasing CeO<sub>2</sub> concentrations, **Figure 7**.

All NMR spectra are analyzed to obtain a quantitated values representing BO<sub>4</sub> and BO<sub>3</sub> groups. The relative area representing each type (BO<sub>4</sub> and BO<sub>3</sub>) has been determined and presented graphically by **Figure 8**. Reverse behavior is shown between BO<sub>4</sub> and BO<sub>3</sub> concentration with increasing CeO<sub>2</sub> contents. This behavior is closely matches with that presented by **Figure 7**. Both figures showed a reduction in N<sub>4</sub> concentration. In the same time the area representing BO<sub>3</sub> groups is increased with increasing CeO<sub>2</sub> concentration. These changes support that the concentration of Na<sub>2</sub>O which required to modify borate network is deeply reduced upon increasing CeO<sub>2</sub> concentration. As a result, concentration of well transformed BO<sub>3</sub> to BO<sub>4</sub> is also decreased which in turns result in decreasing N<sub>4</sub>.

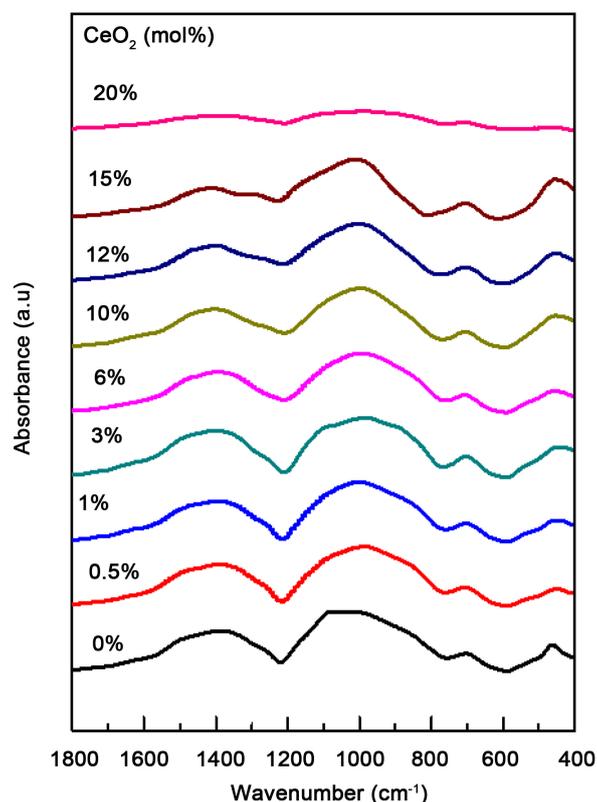
### 3.3. FTIR Spectroscopy

**Figure 9** showed FTIR absorption spectra of borosilicate glasses containing different concentrations from CeO<sub>2</sub>. It is hardly to extract specific information referred to each element shared in performing the spectra. This is because of great overlap between mixed vibration modes of Si-O, B-O, Ce-O which may be hardly to be separated. Therefore, for example, we take the advantage of <sup>11</sup>B NMR spectroscopy in correlation to FTIR [11] [12] [13] [14] to get complete information about structural role of Ce nuclei which cannot measured by NMR. In this respect, we analyzed FTIR spectra and determine the fraction of all four coordinated species, which is termed B<sub>4</sub>, since

$$B_4 = (BO_4 + CeO_4) / (BO_4 + CeO_4 + BO_3) \quad (1)$$



**Figure 8.** Change of relative areas of both BO<sub>3</sub> and BO<sub>4</sub> units with CeO<sub>2</sub> concentration.



**Figure 9.** FTIR spectra of borosilicate glasses containing different  $\text{CeO}_2$  concentrations.

From the above equation, the fraction of both boron and cerium tetrahedral units can be determined, since  $(\text{BO}_4 + \text{CeO}_4)$  is represented by the spectral area which is resolved between  $800 - 1200 \text{ cm}^{-1}$ . By using the advantage of  $^{11}\text{B}$  NMR spectroscopy, the fraction of boron in tetrahedral coordination

$$N_4 = (\text{BO}_4 / \text{BO}_4 + \text{BO}_3) \quad (2)$$

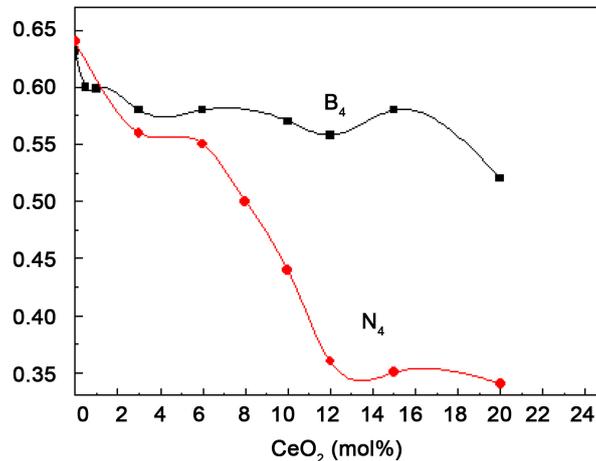
can be simply determined. Subtracting the numerical data determined from Equation (2) from that of Equation (1), the concentration or fraction of  $\text{CeO}_2$  as a former  $\text{CeO}_4$  units can be simply obtained.

$$\text{CeO}_4 = B_4 - N_4 \quad (3)$$

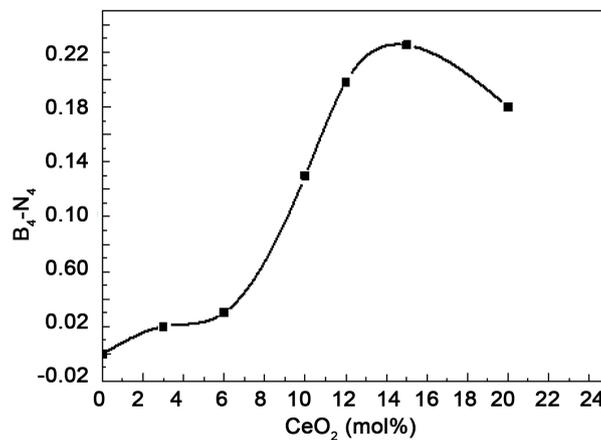
**Figure 10** represented  $B_4$  and  $N_4$  fraction determined from equations 1 and 2 respectively. The difference between the  $B_4$  and  $N_4$  is signed by  $\text{Ce}_4$  ( $\text{CeO}_4$ ) represented by the graph of **Figure 11**. It can be shown from this figure that concentration of  $\text{CeO}_4$  as a glass former is increased with increasing  $\text{CeO}_2$  content.

#### 4. Conclusion

Structural role of cerium is determined in borosilicate glasses by different modern techniques. Cerium enters the network of the investigated glasses as a strong network former. Increasing  $\text{CeO}_2$  concentrations result in decreasing both  $N_4$  and NBO in the whole glass network. New approach has been applied to determine



**Figure 10.** Change of both  $N_4$  and  $B_4$  fraction with  $CeO_2$  concentration.



**Figure 11.** Change of fraction of ceria ( $Ce_4$ ) = ( $B_4 - N_4$ ) with increasing ceria contents.

$CeO_4$  fraction which can not be determined by NMR spectroscopy.

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