

Comparative Studies of Cerium and Iron Borosilicate Glass systems: Structural and Magnetic Properties

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دراسة مقارنة بين أنظمة زجاج البوروسيليكات المحتوية على السيريوم والحديد: الخصائص التركيبية والمغناطيسية

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Abstract:

The comparison of the structural and magnetic properties of boro-silicate glasses with the addition of two different oxidic ingredients was discussed in this work. The density of these glass systems was observed to increase within the range of 2.49-2.95 g/cm³, accompanied by a rise in the molar volume as cerium and iron oxides increased. The results have been primarily interpreted on the basis of the variation in the structural role played in both glass systems. Cerium oxide exhibits a strong tendency to reduce the number of non-bridging oxygen (NBO) atoms in the glass network, acting predominantly as a glass former in most content regions. In contrast, Fe₂O₃ behaves mainly as a glass modifier at low concentrations, while it increasingly contributes as a glass network former at higher contents. The magnetic behavior was examined using a vibrating sample magnetometer (VSM) at room temperature under an applied magnetic field ranging from -20,000 to 20,000 G. The studied glass samples exhibited significant changes in magnetic behavior with the incorporation of both oxides into borosilicate network. The obtained results revealed that, ferrimagnetic signals were detected for most of the investigated samples and their magnetic response is attributed to two main factors: the order of network structure and concentration of different oxidation states of iron and cerium oxides. Besides, there is a noticeable variation in the magnetization appeared at the highest Fe₂O₃ concentration (10mol%) in the glass system compared with other glass samples. This indicates a strong correlation between the structural characteristics and magnetic properties of iron borosilicate glass system. Such behavior, could be attributed to the high iron content, which reduces the distance between Fe³⁺ ions, thereby strengthening stronger magnetic interactions. At higher Fe₂O₃ concentrations, Fe-O-Fe linkages become dominant, promoting superexchange interactions between neighboring Fe³⁺ ions. Consequently, the magnetic response can be characterized by ferromagnetic behavior.

Keywords: Borosilicate; Rare earth; Transition metal; Structural; Magnetic characterization.

المخلص:

تناولت هذه الدراسة مقارنة بين الخصائص البنيوية والمغناطيسية لزجاج البوروسيليكات المعدل بإضافة أكسجين مختلفين. وُجد أن كثافة هذه السلسلة الزجاجية تزداد في نطاق 2.49-2.95 جم/سم³، بالتزامن مع ازدياد الحجم المولي للزجاج، وذلك مع زيادة أكاسيد السيريوم والحديد. وقد فسرت النتائج بشكل أساسي بناءً على اختلاف الدور البنيوي الذي يلعبه كل من أكسيد السيريوم وأكسيد الحديد في النظامين الزجاجيين. يتميز أكسيد السيريوم بقدرة عالية على إزالة ذرات الأكسجين غير الرابطة من الشبكة الزجاجية، ويعمل بشكل رئيسي كمكوّن للزجاج في معظم مناطق التركيز. في المقابل، يلعب أكسيد الحديد الثلاثي (Fe_2O_3) دور المعدّل الرئيسي للزجاج في منطقة التركيز المنخفض، ويعمل كمكوّن رئيسي للشبكة الزجاجية عند التركيز العالي. تمّت دراسة الخصائص المغناطيسية باستخدام مقياس المغناطيسية (VSM) عند درجة حرارة الغرفة تحت تأثير مجال مغناطيسي يتراوح بين 20000- و20000 غاوس. أظهرت عينات الزجاج المدروسة تغيرات ملحوظة في سلوكها المغناطيسي مع دمج كلا الأكسجين في نظام زجاج البوروسيليكات. وكشفت النتائج عن رصد إشارات مغناطيسية حديدية في معظم العينات المدروسة، ويعزى استجابتها المغناطيسية إلى عاملين رئيسيين: انتظام بنية الشبكة وتركيز حالات الأكسدة المختلفة لأكاسيد الحديد والسيريوم. علاوة على ذلك، لوحظت زيادة ملحوظة في المغنطة عند أعلى تركيز لأكسيد الحديد الثلاثي (10 مول%) في نظام الزجاج، يشير هذا إلى وجود ارتباط قوي بين الخصائص البنيوية والمغناطيسية لنظام زجاج البوروسيليكات الحديدي. يُعزى هذا التحسن إلى المحتوى العالي من الحديد، الذي يُقلل المسافة بين أيونات Fe^{+3} مما يُعزز التفاعلات المغناطيسية. عند تركيزات عالية من Fe_2O_3 ، تُصبح روابط $Fe-O-Fe$ هي السائدة، مما يُعزز تفاعلات التبادل الفائق بين أيونات Fe^{+3} المتجاورة. ونتيجة لذلك، يُمكن وصف الاستجابة المغناطيسية بالسلوك الفيرومغناطيسي.

الكلمات المفتاحية: بوروسيليكات، العناصر الأرضية النادرة، المعادن الانتقالية، التركيب، الخصائص المغناطيسية.

Introduction:

Borosilicate glasses are considered as the most promising among many types of materials owing to their important physical and chemical characteristics. In particular, sodium borosilicate glasses belong to a broad glass family that has many uses, from optical, sealing, and nuclear waste to technical glass that is resistant to radiation and heat.

Numerous researchers have thoroughly examined the structural and physical properties of the sodium borosilicate system [1- 4]. According to spectroscopic observations, borosilicate is composed primarily of silicon and boron oxides as the main glass-forming constituents and its local structure is mainly built from silica Q_n (SiO_4) units (Q_n notation indicates n bridging oxygen per tetrahedron) and the triangular (BO_3) and tetrahedral (BO_4) units. The (BO_3) units are triangular boron in both ring and non-ring configurations whereas the four coordinated (BO_4) units are boron atoms in tetrahedral coordination.

Additionally, borosilicate glass containing transition metal ions (TMI) including iron, copper, zinc, silver, and vanadium are widely researched for their unique behavior in terms of their physical, electrical, and magnetic properties. Numerous studies have investigated the environment of iron oxide in various glass systems [5 – 7]. It was reported that, iron ions commonly exist in two valence states (Fe^{2+} and Fe^{3+}) in borosilicate glasses. The trivalent Fe^{3+} ferric ions act as network formers strengthening the network structure meanwhile divalent Fe^{2+} ferrous ions behave primarily as network modifiers, promoting structural depolymerization and the formation of non-bridging oxygen (NBO) species. El-Damrawi G. et al [2] proposed that, iron oxide with initial content is consumed to produce NBOs in silicate and triggers the conversion of trigonal (BO_3) to tetrahedral (BO_4) groups. Meanwhile, Fe_2O_3 with higher concentrations (≥ 6 mol%) consumes modifier ions to form (FeO_4) groups and Si-O-Fe bonds.

On the other hand, rare earth (RE) elements are among the most attractive components [8, 9] since they have the potential to be incorporated into the glass composition and improve their properties. Glass systems containing RE ions with their different valence states offer a variety of intriguing properties, including high hardness and reactivity, magnetic qualities, and a special capacity to absorb UV light. In particular, Cerium is one of the rare earth elements belonging to the lanthanide group that possesses a vacant 4f electronic structure (fluorite structure) with its multivalent states between cerous (Ce^{3+}) and ceric (Ce^{4+}). These characteristics make cerium a valuable material for its optical, shielding and magnetic properties along with other physical characteristics.

Previous studies concerning the impact of adding cerium oxide on the structural and electric characteristics of alkali borosilicate. Fu et al, Deshpande et al and El-Damrawi. et al [10 – 12] have stated that, the presence of both Ce^{3+} and Ce^{4+} ions lead to a significant enhancement in the electrical conductivity, density, and molar volume. Cerium in the corresponding glass system present in Ce^{+3} state giving rise to a greater number of non-bridging oxygens (NBO). The mobility of the sodium ions will increase with increasing the number of NBOs since there will be more empty sites available.

However, the glass with ≥ 1 mol% cerium oxide has more bridging oxygens at the expense of NBOs due to the presence of cerium in Ce^{+4} form. This behavior may be interpreted in term of the predominant structural role of cerium which functions primarily as a glass former. Cerium ions are presumed to possess the capability to form its own structural units within the glass matrix and preferentially link to BO3 rather than enhancing the conversion to the tetrahedral BO4 species [13].

In addition to the incorporation of oxide additives into the parent glass system, the characteristics of the nucleating agents-which enhance or inhibit the formation of particular phases and impose a particular growth pattern- play a crucial role in modulating the magnetic behavior of the glass materials.

It is widely acknowledged that the base glass matrix with B_2O_3 , SiO_2 and Na_2O exhibits diamagnetic behavior [14, 15]. It is demonstrated that when iron is added to borosilicate glasses producing a magnetic response due to the presence of specific concentrations from both Fe^{3+} and Fe^{2+} ions in glass network [16, 17].

In contract, the ability of cerium to switch between its valence states Ce^{3+} and Ce^{4+} gives also rise to enhance the magnetic properties of the host glass matrix along with other physical characteristics. A number of recent studies [18 – 20] have noted that magnetism may originate from the formation and evolution of oxygen vacancies and associated structural defects. As a result, these vacancies play a crucial role in facilitating exchange interactions between unpaired electron spins, thereby giving rise to the observable magnetic behavior of cerium nanomaterial. It is evident that the Ce^{3+} and Ce^{4+} conversion leads to the emergence of ferrimagnetic behavior of the studied materials [20]. This phenomenon can be attributed to an indirect exchange interaction between cerium ions mediated by oxygen vacancies and strongly influenced by the valence states of the surrounding (Ce^{3+}/Ce^{4+}) ions.

The changes in behavior from diamagnetic to ferrimagnetic of lead borate glass system containing CeO_2 have recently investigated by El-damarawi et al [20]. It observed that, the presence of magnetic crystalline phases in the samples as the crystallinity rises is the reason for the values of magnetic parameters show a growing trend up to around 20% CeO_2 . However, further CeO_2 increase led to a drop in crystallinity, which in turn caused a decrease in magnetism. On the other hand, additional cerium oxide addition up to 50 mol% increases the mixed valence of Ce^{3+}/ Ce^{4+} capacity, which leads to a higher corresponding magnetism.

On this background it was thought interesting in this paper to compare the structural and magnetic properties of ternary Boro-silicate glasses induced with the addition of cerium and iron oxides. The main aims of this research are: (i) to compare the structural changes of B_2O_3 - SiO_2 glasses by adding Fe_2O_3 and CeO_2 , (ii) to compare the density and molar volume of these two different glass systems and (iii) to examine the magnetic properties of ternary borosilicate glasses using vibrating sample magnetometer (VSM) with both of cerium and iron additions.

Material and methods:

The glass system of $[(xFe_2O_3. (43-x) B_2O_3.25SiO_2.30Na_2O.2Al_2O_3$ and $[(xCeO. (43-x) B_2O_3.25SiO_2.30Na_2O.2Al_2O_3$ were prepared over a wide range concentration varied from $x= 0$ to 20 mol% using a melt quenching method. The raw materials of high purity chemical compounds: CeO_2 , Fe_2O_3 , Al_2O_3 , and $SiO_2.Na_2O$ were added as sodium carbonate and B_2O_3 as orthoboric acid (H_3BO_3) were well mixed together to obtain a fine powder. Each batch mixture was then transferred to an alumina crucible and fused in an electric furnace. The molten glass was subjected to repeated stirring to ensure complete homogenization then was cast into preheated stainless-steel molds with the specified dimensions.

The glass density has been estimated at room temperature from standard Archimedes formula **as follow:**

$$D = (W_{SA} - W) * D_L / (W_{SA} - W_{SL}) \quad (1)$$

where D is the sample' density, W_{SA} and W_{SL} are the weights of the sample in the air and the xylene liquid respectively, W is the weight of the wire and D_L is the xylene's density.

The density was used to obtain the molar volume (V_m) **using the relation:**

$$V_m = M/D \quad (2)$$

Where M is the relative molecular mass of glass sample.

The magnetic hysteresis (M–H) loops were recorded at room temperature under an applied magnetic field ranging from $-20,000$ to $20,000$ G in order to investigate the magnetic properties of the prepared samples. The measurements were carried out using a Lake Shore Vibrating Sample Magnetometer (VSM 7403, Metallurgical Institute, El-Tebbeen, Helwan)

Results and discussion:

Figures 1 and 2 illustrate the dependence of density and molar volume on Fe_2O_3 and CeO_2 contents of the investigated glass samples. As it can be seen in figure 1, the density of both glass systems shows a linear increase with the incorporation of both Fe_2O_3 and CeO_2 in the host network. The increasing in the density may be attributed to the rise in the average molecular weight of the oxide constituents within

the glass network, since both CeO_2 and Fe_2O_3 possess relatively higher molecular masses than that of SiO_2 . Besides, the density enhancement can be interpreted as the result of a competition between the masses and size of the various structural groups formed in glass structure. The structural modifications into the borosilicate network due to the replacement of SiO_2 by $\text{Fe}_2\text{O}_3/\text{CeO}_2$ involved the formation of various tetrahedral units - which possess a larger molecular volume compared to the BO_3 units - and enhanced structural compactness, rather than disrupting the glass network.

In addition, increasing CeO_2 concentration leads to a steeper rise in density compared to Fe_2O_3 -doped glasses. Meanwhile, the molar volume of the Fe_2O_3 -containing glass system increases at a higher rate than that of the CeO_2 -doped glasses. This alteration is correlated with the relatively higher generation of NBOs in the Fe_2O_3 -containing glass system compared to the CeO_2 -containing glass system [2,20]. The increased NBOs concentration causes disruption of the glass network connectivity, producing a more open and less compact structure that contributes to lower density values. In contrast, glass structures enriched with bridging oxygens maintain greater network integrity and compactness, resulting in enhanced density.

The previous results [2] have demonstrated that the substitution of SiO_2 by Fe_2O_3 results in the formation of a more open glass network structure. In this regard, Fe_2O_3 participates in the glass network primarily as a network modifier by occupying interstitial sites within the structure and increasing the number of non-bridging oxygen atoms (NBOs) and accordingly results in a more open structure. This, in turn disrupts the continuity of the glass network, which contributes to, the higher molar volume observed in the obtained results. Cerium, on the other hand, mostly acts as a glass-forming material when added to a glass network even with low content making more linking and reducing NBOs [12].

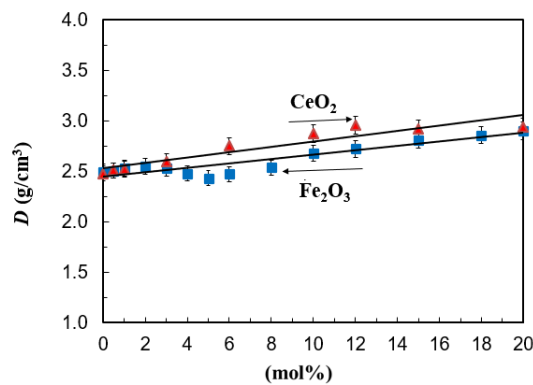


Figure (1): Variation of density with $\text{Fe}_2\text{O}_3/\text{CeO}_2$ Content.

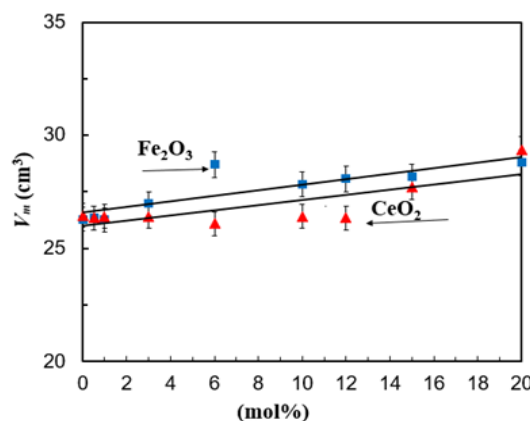


Figure (2): Variation of molar volume with $\text{Fe}_2\text{O}_3/\text{CeO}_2$ Content

The magnetization measurements were carried out through hysteresis cycles, which help evaluating the magnetization in the investigated materials. From these loops, the principal magnetic parameters: the coercive field (H_c), saturation magnetization (M_s), and the total loop area—can be determined. These parameters are highly sensitive to the nature and concentration of structural defects formed within the magnetic material along with the electronic transitions and possible changes in the valence states of iron and cerium ions.

The magnetic-field dependence of magnetization of $[(x\text{Fe}_2\text{O}_3 \cdot (43-x) \text{B}_2\text{O}_3 \cdot 25\text{SiO}_2 \cdot 30\text{Na}_2\text{O} \cdot 2\text{Al}_2\text{O}_3$ ($x = 1, 2, 3, 6$ and 10 mol%) and $[(x\text{CeO}_2 \cdot (43-x) \text{B}_2\text{O}_3 \cdot 25\text{SiO}_2 \cdot 30\text{Na}_2\text{O} \cdot 2\text{Al}_2\text{O}_3$ ($x = 1, 2, 3, 6$ and 10 mol%) glasses measured at room temperature are presented in figures 3 and 4.

As previously mentioned, the base glass matrix composed of Na_2O , B_2O_3 , and SiO_2 exhibits diamagnetic behavior. Accordingly, the partial substitution of SiO_2 by cerium and iron oxides leads to a substantial alteration in the magnetization behavior of the investigated glass materials... Fig. 5 presents dependence of saturation magnetic moment (M_s) (in emu/g) on $\text{CeO}_2/\text{Fe}_2\text{O}_3$ addition. It can be demonstrated that, M_s changes with different rates upon $\text{CeO}_2/\text{Fe}_2\text{O}_3$ additions. An increasing trend followed by decreases, then start to increase again. This trend could be attributed as follow: The magnetic behavior is believed to originate from the presence of the structural modification induced by the effect of $\text{CeO}_2/\text{Fe}_2\text{O}_3$ addition. Presence of crystallite species has the most predominant effect on the magnetic behavior of the glasses containing low content of both oxides [19-21]. Further increase in CeO_2 and Fe_2O_3 (>1 mol%) leads to reduction in crystallinity where the network structure is mostly found in amorphous state as shown in previous work [21, 22] thereby their magnetization is decreased.

Extra more addition of cerium oxide CeO_2 and Fe_2O_3 (>3 mol%) leads to increasing capacity of mixed valence of $\text{Ce}^{3+}/\text{Ce}^{4+}$ and of $\text{Fe}^{2+}/\text{Fe}^{3+}$. The transition from one oxide state to another is the main factor governing the generation of magnetization in these investigated samples [20]. Therefore, the magnetization exhibited a significant increasing in the samples containing higher concentrations of both oxides.

It also can be a notable, all magnetization curves of CeO_2 glass system are saturated at a higher magnetic field region and the hysteresis loops exhibit an S-shaped behavior, which is characteristic of ferrimagnetic signals. It's well known that; ferrimagnetism is the dominant type of magnetic ordering characterizes the magnetic glasses attributed to the exchange interactions between the two unpaired magnetic sub-lattices containing oxygen atom between them.

In contract, it is obviously seen that the hysteretic loop of sample containing $10 \text{ Fe}_2\text{O}_3$ mol% behaves like ferromagnetic behavior as compared to other iron borosilicate samples, which show ferrimagnetism signals. The ferromagnetic behavior could be because the high iron content reduces the distance between Fe^{3+} ions, enabling strong magnetic interactions between them. In such a case, Fe-O-Fe linkages become dominant, allowing superexchange interactions between neighboring Fe^{3+} ions. The high concentration exceeds the solubility limit of iron in the glass matrix, leading to the formation of iron-rich clusters or ferrite-like phases [23- 25].

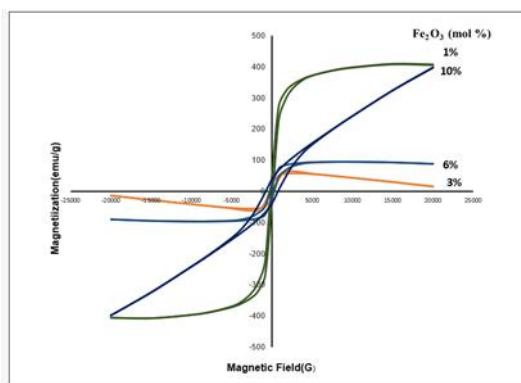


Figure (3): Room-temperature hysteresis curves for samples containing 1, 3, 6, and 10 mol% Fe_2O_3

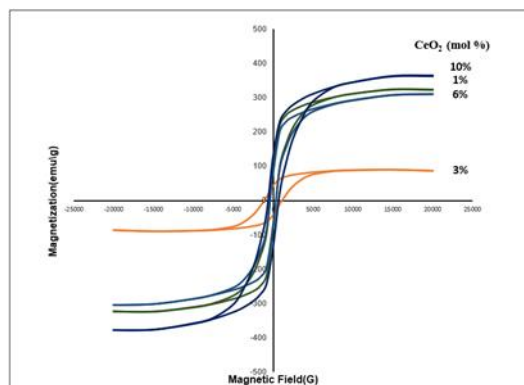


Figure (4): Room-temperature hysteresis curves for samples containing 1, 3, 6, and 10 mol% CeO_2

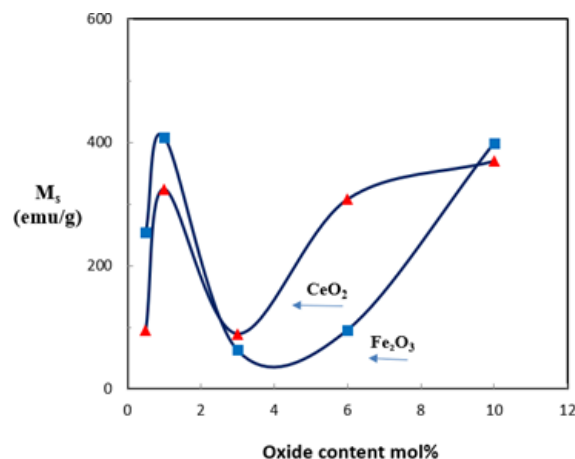


Figure (5): Saturation magnetization (M_s) as function of Fe_2O_3 and CeO_2 concentrations

Conclusion:

In summary, borosilicate glasses with varying concentrations of Fe_2O_3 and CeO_2 were prepared and characterized widely using several analytical techniques. The structural modifications and variations in properties induced by the incorporation of these oxides through the host glass network are primarily attributed to their roles within the glass network. At low Fe_2O_3 concentrations, iron oxide is incorporated into the glass network predominantly as a network modifier meanwhile its dual role becomes evident at higher content. In this case, part of the iron acts as a modifier, generating non-bridging oxygens (NBOs) in the silicate network, while another fraction participates in the formation of bridging bonds within the borate network. In contrast, CeO_2 behaves mainly as a network former even at low concentrations. Consequently, the modifier species present in the glass are initially consumed in the formation of CeO_4 structural units, and the remaining portion is then distributed between the borate and silicate networks. The density and molar volume for both glass series increased as iron oxide and cerium oxide contents increase. The density of glass can be interpreted based on the competition between the mass and the size of the structural units forming the glass network which correlated with the degree of the ionic groups bonding within the structure. On the other hand, the molar volume is more sensitive to structural variation of the tested material, particularly the degree of network connectivity.

The magnetic response of borosilicate glasses varies clearly with both Fe_2O_3 and CeO_2 . Both glass systems showed substantial change by the same trend in the saturation magnetization variation. At low content, the presence of more ordered polycrystalline phases in the samples is mainly responsible for inducing the magnetic behavior. With increasing of both oxides, maximum magnetization is evidenced at about 10 mol%.

For Fe_2O_3 glass system, the magnetic behavior strongly depends on iron concentration. At low Fe_2O_3 content, the loops are narrow and nearly linear. At higher Fe_2O_3 concentration, the hysteresis loops become wider with higher saturation magnetization and coercivity. This is because Fe^{3+} ions interact through Fe–O–Fe superexchange pathways and may form iron-rich clusters or ferrite-like phases, leading to clear ferromagnetic behavior at highest concentration.

In contrast, CeO_2 -doped borosilicate glasses display a weaker magnetic variation. CeO_2 is not intrinsically ferromagnetic, and the magnetic behavior mainly arises from the presence of Ce^{3+}/Ce^{4+} ions and defect centers such as oxygen vacancies within the glass structure. As a result, the hysteresis loops show moderate magnetization compared to Fe_2O_3 -rich samples. Therefore, the observed magnetic response in CeO_2 -containing glasses is primarily related to structural modifications and defect-induced magnetism in the glass network rather than strong magnetic exchange interactions

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