

Effect of Potassium Nitrate and Boric Acid on the Bond Strength of Veneering Ceramics to Zirconia

Keywords

Boric Acid
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ABSTRACT

In this study, the effects of boric acid addition to the veneer ceramics and treatment of the ceramic cores immersed in potassium nitrate solution were evaluated to reduce the micro cracks that may occur in the internal structure of the ceramic, increase the mechanical properties and improve the chemical bonding strength of core ceramics. In the data obtained after the experiment, the average MPa values showed statistically significant differences according to the groups ($p < 0.001$). The average value in the control group (C) was lower than the others. There was no statistically significant between the mean values of the boric acid application in the veneer ceramic (IB) group and zirconia core immersed in the potassium nitrate solution (IN) group. The highest mean values were observed between the veneer ceramic and the zirconia core by application of boric acid and potassium nitrate (IBN) group. As a result potassium nitrate and boric acid application affects the bond strength between zirconia core and veneer ceramic and increases mechanical properties of ceramics.

INTRODUCTION

Increasing esthetic requirements have led to an increase in the use of all ceramic materials for crowns and fixed dental prostheses and have become a main goal of dental science interest.¹ The properties of all ceramic restoration are the ability to mimic the natural tooth in color and translucency along with strength. All ceramics have excellent intraoral stability and wear resistance adding to their durability, low thermal conductivity, no risk of causing metal allergy² optimal biocompatibility,³ decrease in plaque accumulation and low bacterial adhesion.⁴ Therefore, different core materials and fabrication technique have been developed to increase mechanical strength of dental ceramics.⁵⁻⁷

The sintering of all ceramics is obtained by melting of particles.⁶ As a result of sintering, the different shrinkage of the layers during the cooling of the restoration after firing leads to the formation of residual stresses within the restoration. The significant residual stress may occur in ceramic restorations consisting of multiple layers. It is important to determine the extent and distribution of residual stress.⁸⁻¹⁰ The residual stress in ceramic restorations can occur as a result of rapid firing or incompatibility of thermal expansion coefficients of the layers.¹⁰⁻¹² This thermal stresses initiate the low temperature degradation of zirconia at the frame work veneer interface, reducing the zirconia-porcelain interfacial bond strength.¹³⁻¹⁵

Furthermore, all ceramic dental crowns and especially dental bridges are exposed to high bending stresses.

On the other hand, the transformation of crystals from tetragonal phase to monoclinic phase during the firing process in zirconia ceramic restorations can lead to failures such as macro and micro cracks in zirconia cores.¹⁶ The most common complication in all-ceramic restorations based on zirconia is the chipping and delamination of the veneer porcelain.^{1,5,16-18}

Boron compounds and potassium nitrate can be used to increase the clinical success rates of all ceramic restorations and to reduce fracture complications. These compounds are used to strengthen the mechanical properties of ceramics.¹⁹

Potassium nitrate ion exchanging in low temperature forms a press layer on the ceramic surface to decrease micro-fractures. The ion-exchange process results in the compresses on the silicate system introduction of compressive stresses into the surface layer of ceramic components.¹⁹ There are two different process concerning ion-exchange efficient. The first process influences to the phase structure of the surface. At defined phase balance temperatures, one material phase will be stabilized down to room temperature by a diffusive process. The second process is surface layers again to achieve a lower expansion coefficient in relation to the rest of the material. In consequence compressive stresses will be alerted into the surface layer,²⁰⁻²³ due to potassium ions larger than sodium ions.^{19,24} This stabilized material phase has a lower thermal expansion coefficient in relation to the rest of the material. After cooling down to room temperature, a thin surface layer will be loaded by compressive residual stresses.²⁴ For this reason, this chemical strengthening treatment should be suitable for improving the load limits of dental crowns and bridges.^{25,26}

The boron is a bioactive trace element²⁷ which has metal and nonmetal properties and generally used in the weak inorganic acid form as boric acid, as a bactericide, fungicide and antiseptic.²⁸ There are researches which use the antibacterial activity of different concentrations of boric acid.²⁹ It is reported that besides the temperature reducing effect of boron

addition to the structure of ceramic, it improves dimension change behaviour, provides resistance to thermal shocks and improves electrical and mechanical properties³⁰.

These aspects will be investigated in the present study: Firstly, the effect of potassium nitrate for ion exchange mechanism will be evaluated in core material. Secondly, the coating ceramic properties reinforced chemically with boric acid will be examined.

The aim of this study was to measure the shear bond strength between veneering ceramic (treated with boric acid) and zirconia specimens (treated with potassium nitrate).

MATERIALS AND METHODS

In this study, zirconia blocks for core ceramic (IPS e.max CAD, MO block Ivoclar Vivadent, Schaan, Liechtenstein) and dentin porcelain powder for veneer ceramic (IPS e.max Ceram Dentin, A2/TI 1 Ivoclar Vivadent, Schaan, Liechtenstein) were used. Materials used in this study are shown in Table 1. One hundred and sixty zirconia samples were prepared from zirconia block (IPS e-max CAD) in 15x10x0.7 mm dimensions cutting by special diamond disk (Diamond Wafering Blade Series 15 HC Diamond No. 11-4244, IL USA) under water cooling in a Isomet (Low Speed Saw, Buehler Lake Bluff, IL USA) precision cutting device.

The eighty core ceramics from one hundred and sixty samples were immersed in 150 g potassium nitrate solution for 24 hours. The one hundred and sixty samples were then sintered in a high-temperature sintering furnace (InFire HTC Speed; Sirona) at 1581°C for 86 minutes according to the manufacturer's instructions. The grouping of one hundred and sixty samples is shown in Table 2

For the ceramic cores, IPS e.max Ceram dentin porcelain powder was weighed to 3% of the weight of boric acid on a sensitive balance (AND GR 200, Tokyo, Japan), then put into a glass container with alcohol and then magnetic stirrer (Heidolph MR Hei Standard Nuremberg Germany) was put into the

Table 1. The materials used in this study

Material	Manufacture	Content	Thermal expansion	Elasticity module (GPa)
Zirconia Block	IPS e.max CAD	Yttrium 5% to 7%, Hafnium 3% to 5%, trace elements alumina, silica and sodium 0.4% to 1,63%	10.75 ± 0.2	210
Veneer ceramic	IPS e.max Ceram Dentin	SiO ₂ , Al ₂ O ₃ , ZnO ₂ , Na ₂ O, K ₂ O, ZrO, CaO, P ₂ O ₅ , fluoride and pigments	9.5	65
Potassium nitrate	Sigma-Aldrich	KNO ₃		
Boric acid	Merck, Germany	H ₃ BO ₃ or B(OH) ₃		

Table 2. Ceramic groups used in this study

Group	n	Materials
C	40	IPS e.max CAD+ IPS e.max Ceram
IB	40	IPS e.max CAD + (IPS e.max Ceram + %3 Boric asit)
IN	40	(IPS e.max CAD+ Pttassium nitrate solution) + IPS e.max Ceram
IBN	40	(IPS e.max Ceram + 3% Boric acid)+ (IPS e.max CAD+ Potassium nitrate solution)

container. The mixture was sonicated in ultrasonic bath for half an hour to homogenize and stirred with magnetic stirrer for four hours. The alcohol was removed by evaporation and the samples were dried in a vacuum oven for 24 hours. The homogeneity of the components in the mixture was checked by SEM-EDX analysis. After the mixture was homogeneous, the one hundred and sixty ceramic cores were divided into four groups. Boric acid-free porcelain dentin powder was used for C and IN groups, and porcelain dentin powder with boric acid addition for IB and IBN groups (Table 2).

The samples are classified into four groups according to applied to potassium nitrate and boric acid procedure (Table 2):

C group (Control Group). The forty zirconia cores (without potassium nitrate) were placed in an adjustable plexiglass mold. The veneering ceramic powder (without boric acid) was mixed with an appropriate amount of the respective liquid according to the manufacturer's instructions. The mold was filled and ultrasonically condensed. The layered zirconia samples were fired according to the firing program of the manufacturer in a firing furnace (Programat P300, Ivoclar Vivadent AG, Schaan, Liechtenstein).

IB group. In this group, boric acid was added to the ceramic veneer and the veneer porcelain was build up zirconia cores (without potassium nitrate) by layering technique and then fired.

IN group. Ceramic veneer (without boric acid) was build up forty zirconia cores (being immersed in potassium nitrate solution) and fired.

IBN group. The forty samples of zirconia ceramics cores were prepared same way as in IN group and veneer porcelain was prepared same way as in IB group and then fired.

According to Guess *et al* method of the samples were subjected to thermocycling for 20,000 cycles at temperatures alternating between 5 and 55°C.³¹ After the thermal cycle, samples were immersed in 37°C distilled water for 24 hours. A special mold made of 30x30mm pattern resin was prepared for shear test. After the samples were placed in the PMMA resin (Meliodent, Heraeus Kulzer GmbH, Hanau, Germany) mold, the tip of the blade in the Instron device was inserted into the veneer core interface and applied until the fracture occurred

at a speed of 0.5 mm/min in usage of universal test machine (TLCLO, Dartec Ltd., Stourbridge, England).

Using the SBS (Short-Beam-Shear) test to determine the core veneer bond strength resulted in more standardized data because the applied forces are perpendicular to the bonding area and the small cross-sectional area of the bonded surface eliminates incorporation of structural flaws, which significantly affect test readings.³²

The failure of ultimate load was recorded in Newton (N) and then was calculated with suitable mathematical method to express the bond strength in MPa. All fracture surface were analyzed by a stereomicroscope (MP 320; Carl Zeiss, Jena, Germany) at 20x magnification for failure analyses.

Furthermore, the obtained data were analyzed statistically using IBM SPSS V23 (IBM Corp.). Firstly, Kolmogorov Smirnov test was used to evaluate whether the data were suitable for normal distribution. It was determined that the data showed normal distribution. After calculating the mean values and standard deviations of the groups, the mean values were made using One-Way Analysis of Variance and then multiple comparisons were performed using Tamhane T2 test. The significance level was taken as $p < 0.05$ in all tests.

RESULTS

The shear bond strength (MPa) of zirconia ceramic cores immersed in potassium nitrate solution and veneer ceramic restorations added boric acid were examined. The mean and standard deviations values of the groups and the results of statistical analysis are shown in Table 3.

The average MPa values differed by groups ($p < 0.001$). The highest mean value was 22.32 in IBN group, the lowest value was 13.43 in C group. The mean values were 18.18 in IN group and 18.98 in IB group. There was no statistical difference between the mean values of IB and IN groups (Table 3). The ratio of fracture types according to groups was given in Table 4.

When the SEM image of the control group was examined, trace lines at moderate depth and a moderately rough surface were observed during cutting (Figure 2a). The other SEM micrographs are shown in Figure 2b, Figure 2c and Figure 2d.

The fracture types obtained in shear bond test results are evaluated. Mixed type fracture surface was observed more or less when SEM images of C group were examined (Table 4). Dark areas show veneer ceramics. When this image is examined, it is observed that veneer ceramic is very irregular and there are many pores and defects in its structure. This situation may weaken the structure of porcelain and cause fractures. The fracture line between zirconia and veneer ceramics is clearly visible (Figure 3).

When the SEM images of IB group sample were examined, a mixed type rupture surface was also observed. Dark areas show veneer ceramics and light areas show zirconia cores. Smooth and homogeneous veneer ceramics have been observed to wet the zirconia core very well (Figure 4)

Table 3. Comparison of MPa average values by groups.

Groups	N	Average± SD	Test Statistic	P
Group C	40	13,43 ± 0,90 b	F=153,244	<0.001
Group IB	40	18,98 ± 1,26 a		
Group IN	40	18,18 ± 1,80 d		
Group IBN	40	22,32 ± 0,95		

Table 4. The ratios of fracture types of samples.

Groups	Adhesive	Cohesive	Mixed
Group C	95%	-	5%
Group IB	37%	-	63%
Group IN	23%	77%	-
Group IBN	33%	-	67%

When the SEM images of the IN group were examined, a cohesively failed surface was observed in the veneer ceramic. The rupture started in veneer ceramics and continued in veneer ceramics. It was observed that the veneer ceramics had a homogeneous structure and a small amount of porosity (Figure 5).

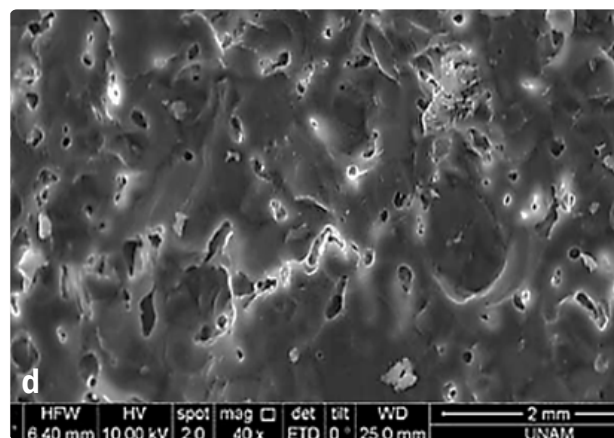
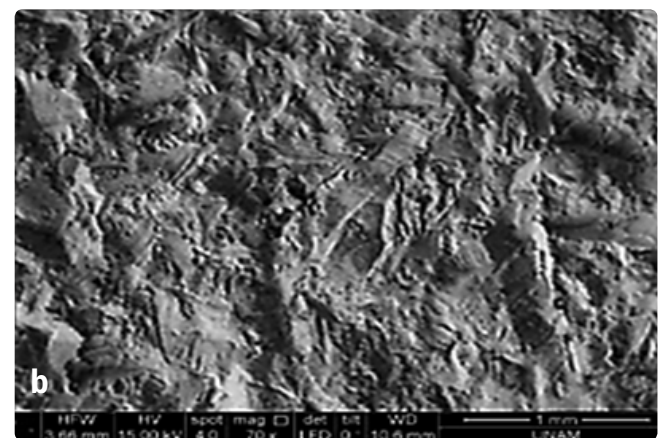
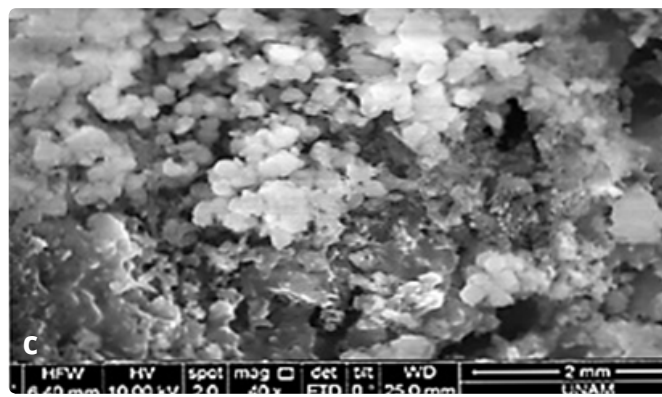
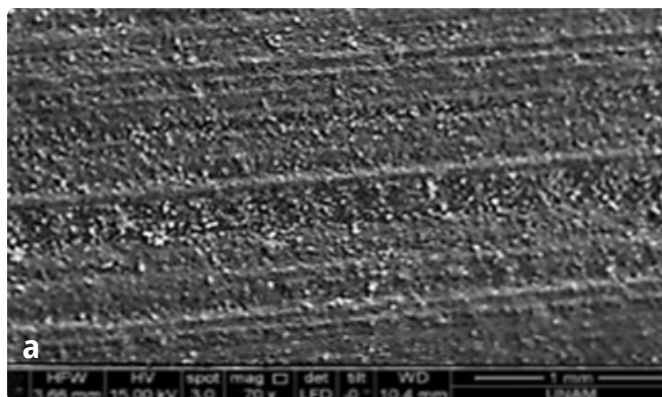


Figure 2: SEM micrograph of (a) control group, (b) added potassium nitrate (c) before added boric acid and (d) after added boric acid.

The SEM images of the group IBN showed a mixed type rupture surface. The half-moon shaped dark areas show veneer ceramics and the light colored areas show the zirconia core ceramics. It was observed that the veneer ceramic was more dense and homogeneous. Therefore, there was no porosity in the structure and the zirconia-veneer ceramic combination was very good. The dark areas seen in zirconia are veneer ceramic residues (Figure 6).

DISCUSSION

The results based on this vitro study were accepted as a hypothesis. Potassium nitrate and boric acid effect on the shear bond strength of zirconia core and veneering ceramic.

The mean shear strength in (control) groups were about 13.43 MPa and (IN) groups were about 18.18 MPa that was in the same reason at the reported by Fischer *et al* that value of four point flexure test showed that treatment of ion exchange below glass temperature with potassium nitrate is highly effective in bonding strength.^{24,25}

Two different strategies for strengthening of ceramics were used in this study.

The first procedure used potassium nitrate influences the phase structure of the surface. In surfaces with potassium nitrate, ion exchange is the process of forming a thin layer with compressive strength on the surface by displacing ions of different sizes such as sodium and potassium.

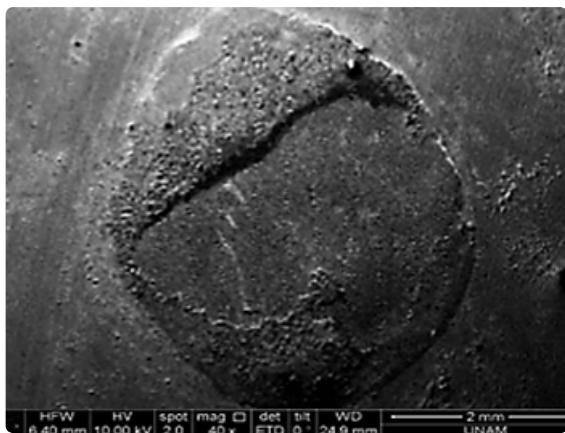


Figure 3: The SEM micrograph of C group.

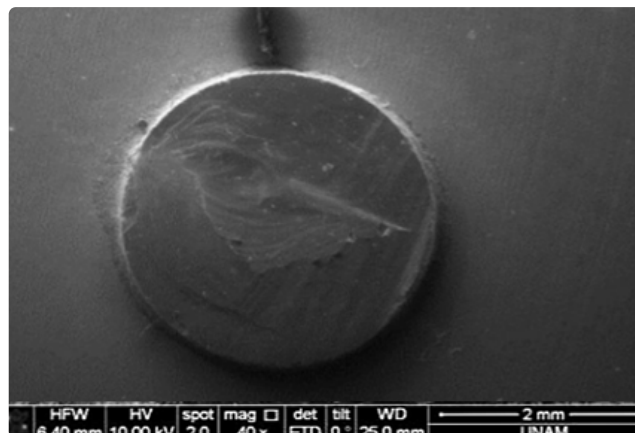


Figure 5: The SEM micrograph of IN group.

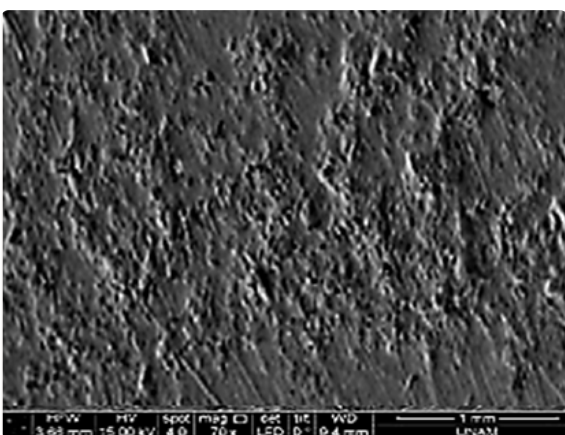
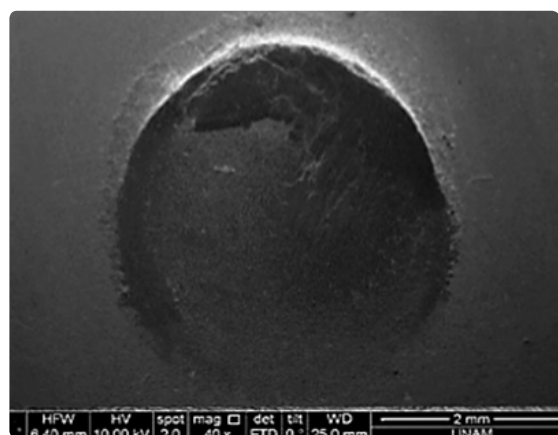
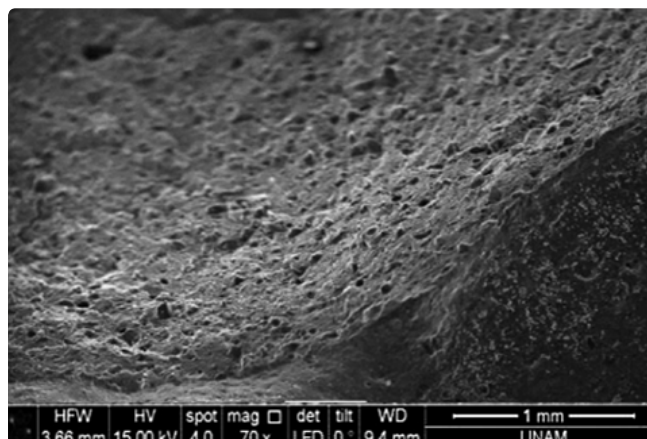
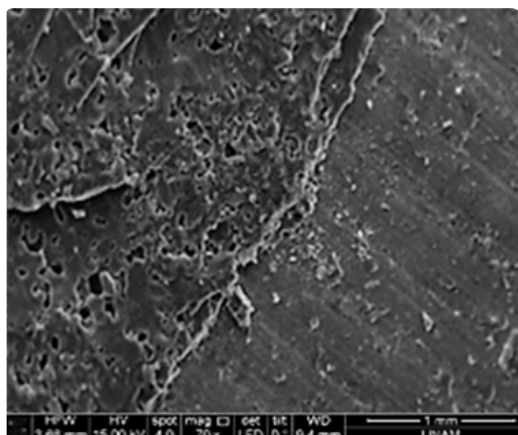


Figure 4: The SEM micrograph of IB group.

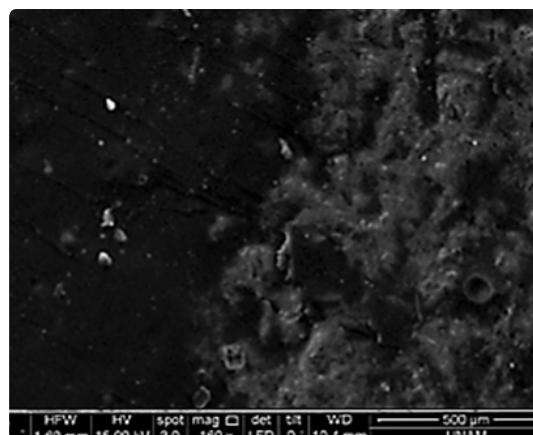
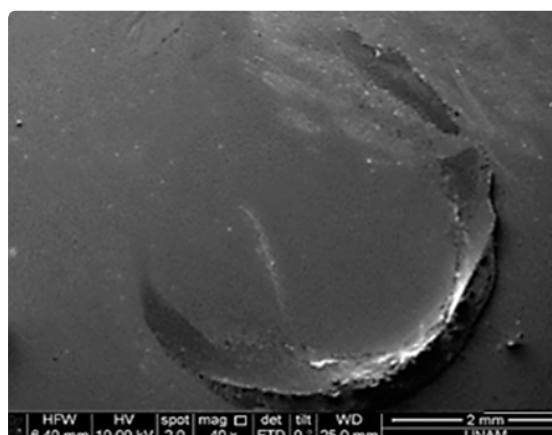


Figure 6: The SEM micrograph of IBN group.

Large ions enter the structure of glass or ceramic by high-temperature diffusion. During cooling, large ions are captured by the ceramic surface and occupy more space due to their high size. Thus, they reduce the shrinkage potential of the surface layer and keep it under a certain pressure. The shrinkage of the deep layers is limited by the fact that the fully filled outer layer causes a certain tension and a compression stress of 700 MPa is achieved.^{26,32}

The second procedure influences the surface of ceramic veneer with added boric acid. The effects of boron treatments on mechanical, biological, and dimensional properties of ceramic based materials have been rarely investigated. Akturk et al²⁹ applied 5% boric acid solution to the cavity before the restoration and they reported that boric acid does not have an impact on the bond strength of composite resin, regardless of the type of bonding system employed (self-etching).

In this study, the mean shear strength in (IB) groups were about 18.88 MPa. Boric acid positively effects on shear bond strength. The first reason is to use 3% boric acid concentration. The second reason is the addition of crystal boric acid due to the silica dissolved in ceramic material would increase the thermal shock resistance and decrease the crack formation by decreasing the thermal expansion coefficient in silica structured feldspatic porcelain which is widely used in porcelain restorations. One of the factors affecting the mechanical behaviour of ceramics against force is the presence of crystal structures added to ceramics.^{32,35,36} This shows that the three-dimensional network structure of silica tetrahedra is partially disrupted by the interaction of boron-oxygen (-Si-O-B-) between the oxygen in the silica tetrahedra and the boron compounds. This allows the softening temperature and thermal expansion coefficient of the silica network to be reduced relative to pure silica.³⁷

The boric acid, which is added to ceramic bodies, has been reported to improve the electrical and mechanical properties as well as to improve the dimensional change behaviour, to improve thermal and shock resistance.³⁸ The boric acid is used as binder in ceramics. As a result of the addition of boric acid, melting and adhesion are at a lower temperature.³⁵ It is stated in the literature that they provide resistance against scratching, cracks and surface staining by reducing viscosity and surface tension.^{30,36,37}

The mean shear strength in (IBN) groups were about 22.32 MPa. It is described that the effect of both potassium nitrate and boric acid properties on bond strength may make potassium nitrate and boric acid as a viable alternative for reinforcements materials.

A limitation of the current study was that the various concentrations of boric acid and potassium nitrate. There is a need for further research to determine which concentration is most effective the bond strength and microleakage of restorations.

Using boric acid and potassium nitrate together, there is no study investigating the connection resistance of ceramics. Therefore, the results will be evaluated in different studies with different concentrations.

CONCLUSION

1. The results showed that boric acid and potassium nitrate increase the mechanical resistance of ceramic and prevent the chipping and delamination in veneer porcelain.
2. Using potassium nitrate and boric acid effect the bond strength between zirconia core and veneer ceramic. The higher shear bond strength values for (IBN) and there was no statistically significant between the mean values of the IB and IN groups.
3. It is thought that the potassium nitrate ions form a compressive layer on the ceramic surface and cause the reduction of micro cracks.

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