

Evaluation of the crack formation of feldspathic ceramic reinforced with bor chemicals

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The purpose of the current study was to improve the mechanical strength and reduce the micro-cracks on the microstructure of the dentin ceramic through addition of various boron compounds (borax, boric acid). Following addition of borax and boric acid (1, 2, 3% of the weight percent) to the traditional feldspathic porcelain; crack lengths, flexural strength and fracture toughness were analyzed. The data analyses were performed by using one-way ANOVA ($p < 0.05$). Differences between groups were determined by Tukey HSD. The results of the present study suggested that the crack lengths were decreased and the fracture toughness were increased in all boric acid added groups ($p < 0.05$). The group containing 1% boric acid had significantly decreased the biaxial flexural strength value compared to the control group ($p < 0.05$). The Vicker's hardness value of the group containing 1% borax was found to be significantly lower ($p < 0.05$). The present study demonstrated that various proportions of borax and boric acid addition in dentin ceramic had reduced the formation of cracks. The current study could be a good starting point on reinforcement of dental ceramics with a possible outcome that will reduce the failures of dental restorations.

Keywords: boric acid, borax, feldspathic ceramic, mechanical features

Introduction

The function, phonation and aesthetic deficiencies resulting from damage or loss of teeth and surrounding tissues are restored with appropriate prosthetic restorations. For nearly a hundred years, dentists have been searching for the ideal restoration material. Although in recent years, amalgam, composites and restorative cements have been used successfully, they are not suitable for multi-faceted restorations. Currently, dental ceramics are widely used in restorative dentistry. They are used as monolithic restorations, porcelain inlays, onlays and metal veneering materials due to the material quality and their aesthetic appearance. Moreover, ceramic materials are chemically stable, have long-lasting color stability, excellent biocompatibility and have an acceptable wear resistance.

Dental ceramics are different from other restorative materials, including metal and acrylic resin, by their chemical, physical, mechanical and thermal properties. Even though conventional dental ceramics are resistant to heat, they are fragile. Therefore, they have limited strength and fracture toughness. Sudden changes in temperature and/or force can result in fractures [1].

The ceramic used in dentistry is a not fully fused

glass derivative formed by sintering process. The sintering process can be defined as a complex sequence of high temperature reactions that occur above the softening point of the porcelain and lead to the partial melting of the glassy matrix by combining the powder particles [2]. However, during the cooling of the sintered dental ceramic to room temperature, micro-cracks occur on the ceramic surface due to the volumetric contraction of the material. Due to the shortcomings that occur during the preparation and modeling of the ceramic as well as the faults that occur during the sintering and cooling in the oven, these micro-cracks may spread to even the deeper layers. The number, depth, width and even direction of these micro cracks play an active role on the mechanical strength of the material [3]. Generally, these cracks or defect regions act as zones where stress is concentrated. Previous cracks or subsequent cracks may result in larger cracks and their propagation in the presence of stress. Such an expansion of cracks causes catastrophic failure in material [4]. Fracture toughness is the mechanical resistance of the brittle material to crack formation and the destructive propagation of cracks under stress [5]. Therefore, improving the fracture toughness of porcelain is an utmost important criterion for extending the clinical performance of porcelain restorations by improving low fracture strength in the mouth [1]. Recently, there has been a focus on reinforcing ceramic materials by introducing particles, whiskers, or fibers

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into matrix structure to improve the mechanical properties and prevent crack propagation.

Boric oxide is a glassy solid, does not exist in nature and is never used in the free state. The value of the boron mines is generally measured by the amount of B_2O_3 (boric oxide) and the ones with a high content of B_2O_3 are considered to be more valuable. Borates (Boric oxide) plays an important structural function as a network former, which is similar to that of silica. Borates with its flux and binding properties can be used as an initiator for glass formation in that they reduce the glass viscosity, decrease surface tension and crack propagation. Moreover, they help to reduce thermal expansion, and, hence, cause an increase in thermal shock resistance. They increase the mechanical strength and scratch resistance of ceramic structure and provide resistance to chemicals. Substituting boric oxide for a portion of the silica content is a sure means of reducing the melting temperature of the glaze without causing a devitrification. Although small amounts of boric oxide improve the mechanical properties of glaze, larger quantities have adverse effects on crazing resistance [6-8].

Boric acid and sodium tetraborates are used for the inserting boric oxide into ceramic structure. Boric acid is an inorganic binder that is widely used in ceramic industry. Boric acid contains 56.3% boric oxide [6, 7]. Borax is the most produced refined product after boric acid, which is used as a strong melting agent in glaze. It reduces the viscosity and surface tension and creates resistance to scratches, cracks and surface staining. Since borax and boric acid are resistant to heat, they are used for making heat resistant materials. Borax contains 36.52% of boric oxide [6-8]. Boric acid and borax are widely used in glass, porcelain, and ware industry, especially in the production of special glasses, where certain properties, including heat resistance, surface hardness and durability, are desired [8].

The aim of the current study is to evaluate the differences in the mechanical resistance of feldspathic ceramics with the addition of borax and boric acid in varying amounts because they are so widely used in glass and porcelain industry.

Experimental

In this study, different proportions of borax and boric acid were added into the feldspathic porcelain to examine the crack length, hardness, flexural strength and fracture toughness. A feldspathic porcelain [Ceramco 3 (Dentsply Degudent GmbH, USA)] dentin powder, which is a Type 1 dental ceramic (DIN EN ISO 6872 standard) containing opacifier, color pigment, 80-95% sodium potassium aluminasilicate and 0-20% tin oxide, was used as a test material.

Boric acid and borax compounds and dentin porcelain powder were dispersed in ethyl alcohol in a concentration of 1, 2 and 3%. "Ceramco 3" commercial

dentin porcelain powder by weight was homogenized by sonication for half an hour in an ultrasonic bath and stirred for 4 hours with a magnetic stirrer (Heidolph MR Hei-Standart, Nuremberg, Germany). The alcohol was evaporated off and the samples were dried under vacuum for 24 hours.

Powder mixtures were prepared in 6 groups. Boron acid and boron free dentin porcelain powder were used as a control group. In each group, 20 disc samples were prepared by using metal molds (thickness 1.2 ± 0.2 mm; diameter 15 mm), where the sizes were determined by the standards for the flexure test of Type 1 ceramics. Ten of these samples were used to determine the biaxial flexural strength, and the rest of the samples were used to detect Vickers' stiffness and crack size.

The samples were prepared by a baking process, where the samples were first kept at 650 °C for 4 min with a temperature rise of 50-70 °C per minute. When the temperature reached 910 °C, baking process was finished within 2 minutes. The final surface conditioning of the samples was performed using the diamond burs with a grain size of 15-20 μ m. A total of 10 test specimens were prepared for 7 groups with a thickness of 1.2 ± 0.2 mm and a diameter of 15 mm. After final surface treatment, the samples were cleaned for 3 minutes in ultrasonic bath (Euronda; Erosonic Energy, Italy) that contained distilled water.

Biaxial Flexural Strength Test

For the biaxial flexural strength test, a testing machine (Instron 5565A, Instron UK, England) with the lower part containing 3-stainless steel balls (diameter of 2.5 mm) with an angle of 120° between them, was placed on a table (diameter of 10 mm) and the piston head with a pin having a diameter of 1.4 mm was used. The load was applied vertically to the center of the sample at a speed of 1 mm /min. A thin and soft film layer was placed between the sample and the piston head to obtain an even distribution of the applied forces.

The force at the time the sample got fractured was recorded in Newton by the computer and then converted to MPa.

Vickers Hardness Test and Fracture Toughness

After the samples were embedded in acrylic molds, their surfaces were polished for 30 seconds on a sanding machine (Phoenix Beta, Buehler, USA) at a speed of 300 rpm by pouring 0.3 μ m grain size of Al_2O_3 solution on a velvet cloth under cooling water.

Vickers micro hardness tester (Shimadzu HMV-2000, Germany) was used to measure the Vickers hardness value (VHN) after a 19.6 N load. Following the hardness tests, acceptable crack models were examined with SEM (LEO, EVO 40, Cambridge-England). The samples were plated with gold-palladium (Au-Pd) to determine crack lengths in the coating apparatus (BAL-TEC; SCD50, Liechtenstein). Chemical analysis of the sintered

samples was done by EDS (Bruker, 125 eV, Minden-Germany) connected to SEM unit.

Acceptable crack models were created with a 19.6 N load with Vickers micro hardness tester (Shimadzu HMV-2000, Germany). It was noted that the cracks were removed from the corners of the vickers indentation, but not from the branch. Moreover, the average of the cracks was at least twice the diagonal length. Reindentations were made for unacceptable crack models. Crack lengths were determined by SEM device (LEO, EVO 40, Cambridge-England).

For each samples, traces of cracks that did not branch out of the four corners of the Vickers indentation were measured. The crack length was measured from the center of indentation in accordance with the literature. The length of the fissures extending from all four corners were determined by means of arithmetic mean and the crack length was determined by means of arithmetic mean of the length of the cracks stretching from the four corners [5, 9]. Then, for the 10 samples in each group, the fracture toughness value was calculated with the formula found by Anstis et al. [10] and it was expressed in $\text{MPa}\cdot\text{m}^{0.5}$.

In this study, due to the fact that boric acid and borax additions were as low as 1, 2 and 3% by weight and that the value of the elastic modulus could not be found exactly, the calculation of fracture toughness was performed by assuming the constant was 0.88 instead of using E/H . [11].

$$K_{IC} = 0.016 (E/H)^{0.5} (P/c^{1.5})$$

(K_{IC} : Fracture toughness value ($\text{MPa}\cdot\text{m}^{0.5}$); E: Elastic Modulus; H: Hardness; P: Applied Force (N); c: Crack Length (m).

Crystallographic analyzes of the prepared samples were analyzed using computer controlled X-ray diffractometer (RigakuRadB-DMAX-II, Woodlands-USA).

IBM SPSS for Windows version 22 software was used for statistical evaluation. The quantitative data definition for the variables were presented as arithmetic mean (mean) \pm standard deviation (SD). Shapiro Wilk normality test was used to determine the normal distribution of quantitative data ($p > 0.05$). One-way analysis of variance was used for intergroup comparison and Tukey HDS test was used to determine the group that caused the difference. Statistical significance was accepted as $p < 0.05$.

Results and Discussion

Biaxial Flexural Strength Test

Biaxial flexural strength test results were presented in Table 1. The results demonstrated a significant difference between the groups ($p = 0.003$; $p < 0.01$). The biaxial flexural strength values of the group boric acid 1% was found to be significantly lower than the control group ($p = 0.034$; $p < 0.05$), the borax 1% ($p = 0.048$; $p < 0.05$)

Table 1. Comparison of groups according to Biaxial Flexural Strength

Biaxial Flexural Strength (MPa)*	
	Mean \pm SD
Control	72.06 \pm 11.24
Boric acid %1	57.52 \pm 7.41
Boric acid %2	66.47 \pm 6.93
Boric acid %3	67.06 \pm 11.42
Borax %1	71.46 \pm 9.56
Borax %2	67.08 \pm 11.53
Borax %3	77.77 \pm 11.94
p	0.003

Table 2. Comparison of groups according to Vickers hardness

Vickers Hardness Value (VHN)*	
	Mean \pm SD
Control	541.9 \pm 34.83
Boric acid %1	548.5 \pm 32.64
Boric acid %2	564.0 \pm 16.50
Boric acid %3	512.3 \pm 19.63
Borax %1	507.5 \pm 37.94
Borax %2	510.6 \pm 24.95
Borax %3	530.5 \pm 32.63
p	0.001

and the borax 3% ($p = 0.001$; $p < 0.01$). The mean values of biaxial flexural strength of the other groups were similar to each other ($p > 0.05$).

Hardness Test

The mean and standard deviation values of the hardness test results are given in Table 2. The Vickers hardness values of the groups were significantly different ($p = 0.001$; $p < 0.01$). The mean Vickers hardness value of the group boric acid 2% was significantly higher than the groups of boric acid 3% ($p = 0.004$; $p < 0.01$), borax 1% ($p = 0.001$; $p < 0.01$) and borax 2% ($p = 0.003$; $p < 0.01$). Moreover, the mean Vickers hardness value of the 1% of boric acid group was significantly different than borax 1% group ($p = 0.042$; $p < 0.05$) The Vickers hardness value was not significantly different between the other groups ($p > 0.05$).

Fracture Toughness

Fracture toughness values were determined by using crack length values that were monitored by SEM. Mean and standard deviation values of fracture toughness are given in Table 3. SEM images obtained from the groups are also presented in Figure 1.

Although, 1%, 2% and 3% boric acid addition and 1% and 2% borax addition had increased fracture toughness values, the mean fracture toughness between the groups were similar to each other ($p = 0.112$; $p > 0.05$).

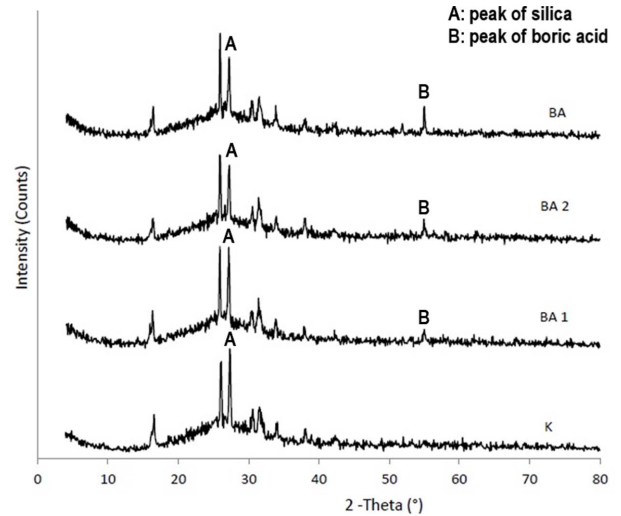
Table 3. Comparison of groups according to Fracture Toughness

Fracture Toughness (MPa/m ^{0.5})*	
	Mean ± SD
Control	1.06 ± 0.14
Boric acid %1	1.11 ± 0.11
Boric acid %2	1.14 ± 0.14
Boric acid %3	1.15 ± 0.10
Borax %1	1.11 ± 0.09
Borax %2	1.10 ± 0.12
Borax %3	1.00 ± 0.16
p	0.112

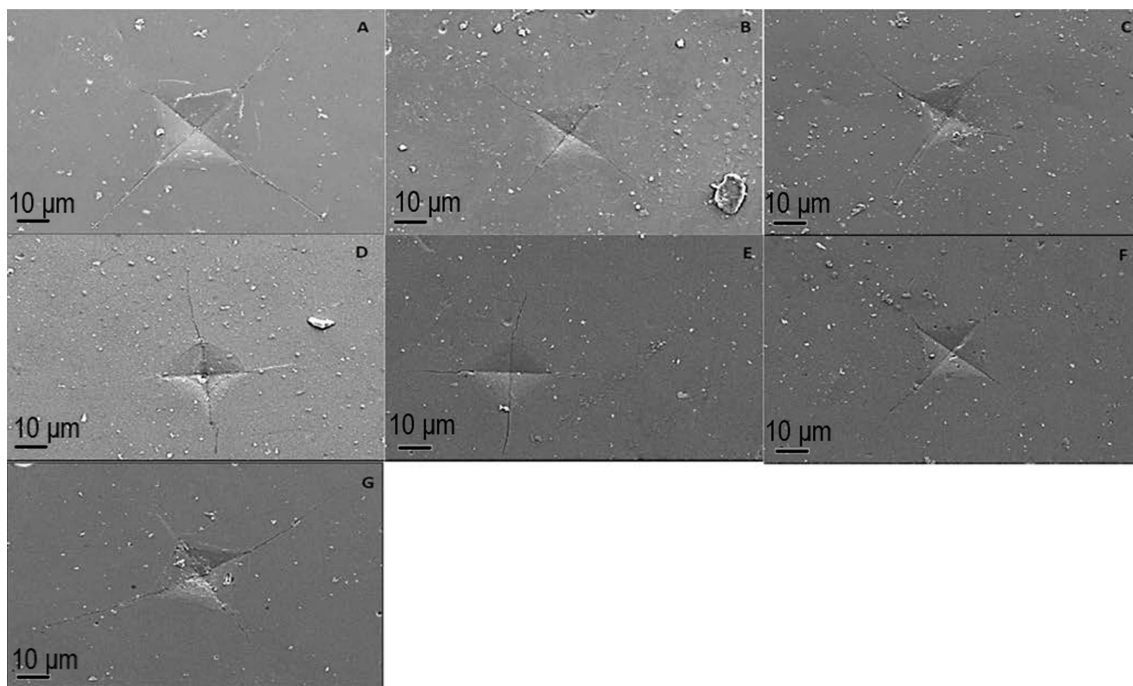
XRD Analysis

When XRD patterns of boric acid added groups and control groups in Figure 2 as well as borax added groups and control groups in Figure 3 were examined, it was observed that the main phase was silica. The results demonstrated that boric acid and borax added to the feldspastic matrix enters the structure from the decreased intensity of 2θ peaks of silica at 27° and the increased intensity of 2θ peaks of boron compounds at 55°. Since the position and shape of the XRD peaks of the feldspastic phase were similar, addition of boric acid and borax did not change the crystal structure of the main phase.

Besides the excellent esthetic features, low tensile and shear strength of ceramic restorations makes them fragile during chewing. The main reason for the failures

**Fig. 2.** Comparative XRD results of boric acid added groups compared with control group.

is the formation of cracks and their propagation [12]. Feldspathic porcelain is the most commonly used ceramic in dentistry. However, containing large amounts of glassy phase reduces its durability [13, 14]. Therefore, ceramic restorations are subjected to firing in the laboratory. During the cooling of the restorations to the room temperature, volumetric contractions in the material caused the formation of micro cracks on the surface of the porcelain. These micro cracks can propagate

**Fig. 1.** SEM images of crack formation on sintered samples. (A) SEM Image (×1000) of crack formation in control group at (B) SEM Image (×1000) of crack formation in %1 boric acid added group (C) SEM Image (×1000) of crack formation in %2 boric acid added group, (D) SEM Image (×1000) of crack formation in %3 boric acid added group, (E) SEM Image (×1000) of crack formation in %1 borax added group, (F) SEM Image (×1000) of crack formation in %2 borax added group, (G) SEM Image (×1000) of crack formation in %3 borax added group.

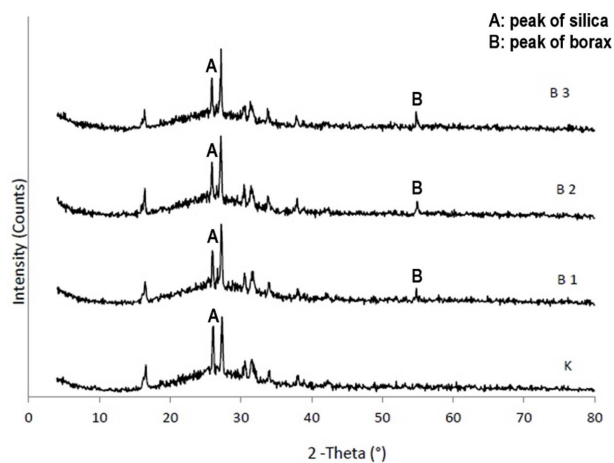


Fig. 3. Comparative XRD results of borax added groups compared with control group.

even deeper. In addition, the final conditioning of the restoration surface also adversely affects the micro cracks. As a result, the negative impact of the layer containing the micro-cracks further changes the mechanical properties of the ceramic material [3, 15].

Boron compounds are used to strengthen the mechanical properties of ceramics. Boron oxide may undergo normal glass form, but it is generally used as a binder and network former. Borate plays a significant role in glazing technology and is the second most important constituent after the silica. It has been reported that strength to the mechanical resistance and scratch formation increases with the amount of borate in the content of ceramic glazures [6].

Boron oxide (B_2O_3) acts as a glass modifying agent by reducing the viscosity and softening temperature of the silica network, such as alkali metal ions. Boron oxide has its own network structure, which enters between the three-dimensional silica network structures. Such silica tetrahedrons is partially degraded by the boron-oxygen (-Si-O-B-) interaction occurred between oxides in silica tetrahedral and boron compounds. This allows coefficient of softening temperature and thermal expansion of silica network structure to decrease compared with pure silica [16].

Boric acid and borax are inorganic binders which are widely used in ceramic industry [7, 17]. It is stated in the literature that they provide resistance to scratches, cracks and surface staining by decreasing viscosity and surface tension [7, 17, 18]. In previous studies, the amount of boric acid used ranged between 1% and 3% by weight [7, 18].

Structures with a low coefficient of thermal expansion have higher thermal shock resistance due to less stress [19]. Therefore, dental ceramics have low thermal shock resistance, since they have high expansion coefficient. In the current study, the addition of boric acid and borax, which has its own network structure, was expected to reduce thermal expansion resistance

and decrease the formation of cracks by lowering the thermal expansion coefficient in the silica structure of feldspathic porcelain.

It was determined that the crack lengths were generally reduced by 1, 2 and 3% boric acid addition, respectively. The crack lengths demonstrated a tendency to reduce with the addition of 1% and 2% borax, but the difference was not statistically significant. Moreover, the addition of 3% borax was found to increase the crack length. Since each group was treated with similar final surface conditioning process, it could mimic the difference between the groups. However, glaze application in future studies can provide better understanding.

One of the factors affecting the mechanical behavior of ceramics against forces is the presence of crystal structures such as alumina, leucite and zirconium oxide [20-22]. These crystals arrest the progression of cracks within the structure, since it is difficult to penetrate into the crystal [1]. Because dental ceramics contain a high proportion of glassy phases, cracks can easily progress further. However, alumina particles, which are added to the ceramic structure, are more difficult to break than glass, a higher amount of energy is required for the formation of fracture. Hence, it makes the crack progression more difficult. Therefore, alumina-glass composition is twice as endure as the glass phase [22-24].

Hammouda and Beyari [25] added yttrium, which is a partially stable zirconium, to the conventional dental porcelain by 3, 5 and 7% by mass and reported that 3 and 5% addition elevated the flexural resistance but decreased the hardness and increased its radiopacity. Some researchers have added wollastonite, which is an industrial mineral and defined as natural calcium metasilicate, and reported that the addition of 2% wollastonite to the porcelain powder increases the flexural resistance of the dental alumina or porcelain [26]. Moreover, Medeiros et al. [27] performed reinforcement studies with porcelain and reported that addition of Gadolinium Aluminum Perovskit ($GdAlO_3 / Al_2O_3$) fibers increased the resistance of porcelain. Bayca [17] reported that addition of ulexite, is a boron mineral, to ceramic body decreased the sintering temperature of the tiles, however there is an increase in the bending strength of the ceramic tiles.

Boric acid and borax have a similar structure with silica and they are used as a dissolver in ceramic industry. Therefore, the aim of the present study was to investigate the effects of the addition of crystalline boric acid and borax compounds on the mechanical properties of feldspathic porcelain.

The International Organization for Standardization (ISO 6872) proposed and assigned the biaxial flexural strength examination with a three-point bending test to measure the flexural resistance (flexing endurance) of dental ceramics [28]. The three point bending test is susceptible to cracks and superficial pits formed on the sample surface and edges as the samples are prepared.

The formation of these cracks, defects and pits on the surface significantly affect the results that will be analyzed [9, 29, 30]. In addition, the fact that the dimensions and volumes of the samples are very different from those of dental restorations and the risk of porosity formation during the preparation of samples are also disadvantages of this method [31]. For these reasons, in the present study, biaxial flexural strength test was preferred to determine the flexural resistances of the disc-shaped samples in accordance with the 6872 standard prepared by ISO for durability testing.

Feldspathic porcelain is the most widely used dental ceramic, but its flexural resistance is only between 60 and 70 MPa; therefore, it requires a metal infrastructure to be strengthened [32]. Many studies have been conducted to investigate the mechanical properties of feldspathic porcelain [33-35]. In our study, piston test was used on three balls and in parallel with the literature, the flexural resistance of conventional feldspathic Ceramco 3 porcelain used for the control group was established to be 72.06 ± 11.24 MPa. It is determined that all values found in the test result are above 50 MPa, which is the lowest value according to ISO 6872 "Dental Ceramics" standard, and it is compatible with dental ceramic standard.

The atomic bonds in the ceramic crystal are both covalent and ionic and have no free electrons. These strong atomic bonds provide stability, hardness and resistance to chemicals. However, they adversely affect other conditions, including low thermal conductivity and increased fragility [36].

In our study, it was observed that 2% and 3% boric acid additions did not cause a significant change in flexural resistance ($p = 0.003$; $p < 0.01$). The flexural resistance of the feldspathic porcelain group with 1% boric acid was found to be 57.52 ± 7.41 MPa and was significantly lower than the control group ($p = 0.034$; $p < 0.05$). The results of the current study showed that the addition of 1% boric acid did not reduce the hardness of the structure. It is thought that the silica tetrahedrals in the three-dimensional network structure may have reduced the amount of Si-O binding. As a result, the atomic bonds in the ceramic crystal may stay stronger.

Although flexural strength was similar between the control and borax added groups ($p = 0.003$; $p < 0.01$), a numerical increase was observed with the addition of 3% borax compared to the control group. The addition of 3% borax may have entered the complex with its own cage structure by greatly reducing the Si-O connections and covalent bonds in the three-dimensional network composition of the silica tetrahedrals. While it decreases the stability and hardness of the material, borax slightly increases flexural resistance due to its unique lattice structure. The hardness of the materials are directly related to the crystal structures and bond strength between atoms. As the bond strength increases,

the hardness of the minerals increases[37].

The hardness value of the restorative materials used in the mouth is important. If their hardness is higher than that of dental enamel, they can cause damage to the natural teeth. Therefore, the hardness value of the material should be close to the enamel. On the other hand, if the hardness value of the ceramic material is higher than that of the enamel, then the abrasion effect is reduced with the glaze layer[37,38]. In the light of these findings, in the present study, Vickers hardness test method was used considering the fragile and sensitive structure of dental ceramics. Moreover, cracks were determined by Vickers hardness measuring device.

In this study, the hardness value of Ceramco 3 feldspathic porcelain and the hardness value of boric acid and borax added feldspathic porcelain samples were compared. The hardness value of 1% and 2% of borax added groups were significantly lower than the control group ($p = 0.042$; $p < 0.05$). The decrease in the 3% borax added group was not statistically significant ($p > 0.05$). The addition of 1, 2, and 3% borax through its cage structure reduced Si-O connections in the three-dimensional network structure of silica tetrahedrals. However, it is believed that this application reduced the strength of the atomic connections in the ceramic crystal and decreased the hardness. In addition, the addition of 1, 2, and 3% of borax did not have any effect to increase the abrasiveness of feldspathic porcelain.

On the other hand, the addition of 1 and 2% of boric acid showed no significant difference in the hardness value of the control group. It is thought that it did not decrease the stability and hardness of the structure by reducing the Si-O connections that affect the stability of the ceramic structure. However, its addition improves the amount. It can be claimed that the addition of 3% boric acid greatly reduced the Si-O connections in the three-dimensional network of the silica tetrahedrals. Moreover, its addition also reduced the softening temperature, the expansion coefficient, and atomic bonds while decreasing the intermittent space, thus reducing the hardness.

The indentation fracture toughness technique is relatively simple to apply; it only requires a smoothly polished surface for the sample to be measured. When the literature is examined, it is seen that this technique is generally used to determine the fracture toughness of ceramic materials [39-42]. Therefore, in the current study, indentation fracture toughness test method was used to evaluate fracture toughness.

Hammouda and Beyari [25] evaluated the fracture toughness of conventional dental porcelain, which was treated with 3, 5 and 7% of yttrium stabilized tetragonal zirconium polycrystalline (Y-TZP). Their results showed that the fracture toughness value of the conventional feldspathic porcelain was 0.538 ± 0.049 MPa.m^{0.5}. However, the fracture toughness values of 3, 5 and 7% yttrium partially stable zirconium addition groups were $1.424 \pm$

0,064 MPa.m^{0.5}, 0.782 ± 0.106 MPa.m^{0.5}, 0.489 ± 0.069 MPa.m^{0.5} respectively. These results suggest that increased particle size reduces the fracture toughness when other microstructural variables are assumed constant.

Yoshimura et al. [43] evaluated the fracture toughness of high heat alumina core ceramics, feldspathic porcelain, hot press leucite based glass ceramics, glass ceramics and glass infiltrating alumina ceramics reinforced with hot press lithium disilicate according to Astm e399 standards. The fracture toughness values were 0.67 ± 0.08 MPa.m^{0.5}, 0.84 ± 0.07 MPa.m^{0.5}, 0.96 ± 0.03 MPa.m^{0.5}, 1.81 ± 0.18 MPa.m^{0.5} and 2.91 ± 0.20 MPa.m^{0.5}, respectively. Researchers have stated that the materials they used may have different mechanical properties and that the highest fracture toughness value of glass infiltrating alumina ceramic may be related to the high elastic modulus and fracture surface energy.

In this study, the fracture toughness value of the control group was 1.06 ± 0.14 MPa.m^{0.5}, while the fracture toughness values of 1, 2, and 3% boric acid added groups were 1.11 ± 0.11 MPa.m^{0.5}, 1.14 ± 0.14 MPa.m^{0.5}, 1.15 ± 0.10 MPa.m^{0.5}, respectively. Although there was a quantitative difference between the groups, it was not statistically significant. With the addition of boric acid, the Si-O connection amount of the silica tetrahedrals in the three-dimensional network structure could be reduced as well as the softening temperature and thermal expansion coefficient of the porcelain may also be decreased. Thus, the distance between the particles has diminished and the crack propagation may have been more difficult. Therefore, it is thought that the fracture toughness of the structure may be elevated. In addition, boric acid has a less complex structure, which has a better penetration into the silica structure and higher ability to fill the gaps in the structure. As a result, elevated fracture toughness may be resulted in increase of the numerical value.

Similar effects were achieved with 1 and 2% Borax additions. While the fracture toughness of the ceramic structure is increased, the addition of 3% borax may significantly reduce the connections and covalent bonds in the Si-O network which affect the stability of the ceramic structure and damaged it. As a result, the fracture toughness value was reduced. Moreover, with the wider lattice structure of borax, interactions within the structure have been reduced with respect to particle sizes. As a result, the fracture toughness value may be decreased as the percentage by weight in the structure increases.

On the other hand, the fracture toughness values of feldspathic porcelain with 1, 2 and 3% borax added groups were 1.11 ± 0.09 MPa.m^{0.5}, 1.11 ± 0.09 MPa.m^{0.5} respectively. It was determined that the addition of borax in the ratio of 1% and 2% in fracture toughness value was 1.00 ± 0.16 MPa.m^{0.5}, but this increase was not statistically significant. However, the addition of 3% borax was found to cause a relative decrease in fracture toughness.

Several techniques have been proposed in the literature to examine the formation and length of cracks in dental materials. These are SEM, light microscope, a convergent laser scanning microscope and transillumination (posterior light imaging) technique [44-46]. Among them, SEM analysis is an effective and widely accepted method for the examination of properties, such as surface topography, dispenser and connections [47]. Therefore, in the present study, SEM technique was used to determine the crack length. Moreover, chemical analyses of samples were evaluated with EDS detector connected to SEM unit.

It has been observed that using Vickers micro hardness device, traces were formed within cracks that did not progressively advanced through porosities. The image of these traces is consistent with the literature [5]. In addition, images of boric acid and borax added groups were uniform and homogeneous that were consistent with the image of the unadulterated feldspathic porcelain group. These results suggested that boric acid and borax additions did not damage the feldspathic porcelain structure. Moreover, EDS analysis showed that all groups were rich in silicon and oxygen elements (Figure 4-10). The detection of boron in borax and boric acid added groups following the analyses of individual elements suggested the involvement of borax and boric acid in the porcelain structure.

X-ray diffraction method (XRD) is widely used to determine different crystal structures and knitting parameters in the materials. In the present study, XRD analyzes were performed to investigate the effect of boron compounds on ceramic structure. When the XRD graphs were obtained from sintered unadulterated feldspathic porcelain group, the boric acid and borax added groups were examined. It was observed that the silica was the main phase. These findings are compatible with the structure of the feldspathic porcelain and also support the result of EDS analysis. XRD patterns of borax and boric acid added groups were different from the control group. The entrance of boric acid and borax to the Feldpastic matrix was observed from decreased intensity of the 2θ peak of silica at 27° and from the 2θ peaks of boric acid or borax at 55°. As the positions

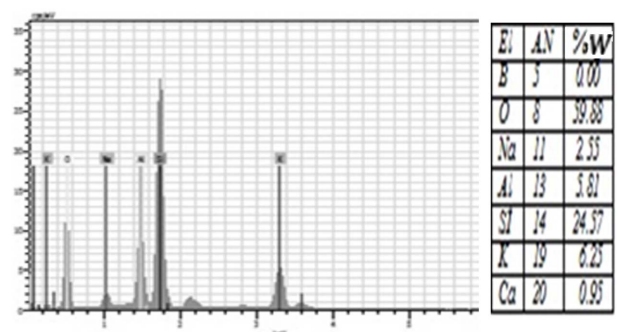


Fig. 4. The amount of elements contained in the control group according to EDS analysis.

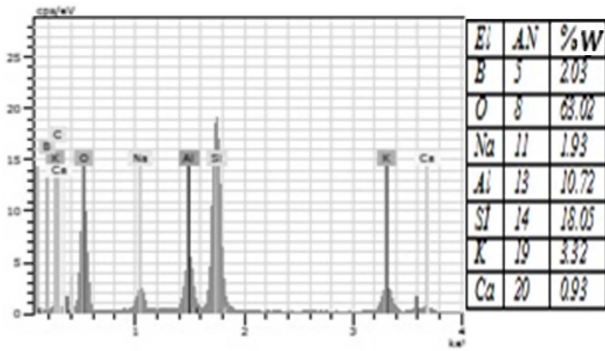


Fig. 5. The amount of elements contained in the %1 Boric Acid group according to EDS analysis.

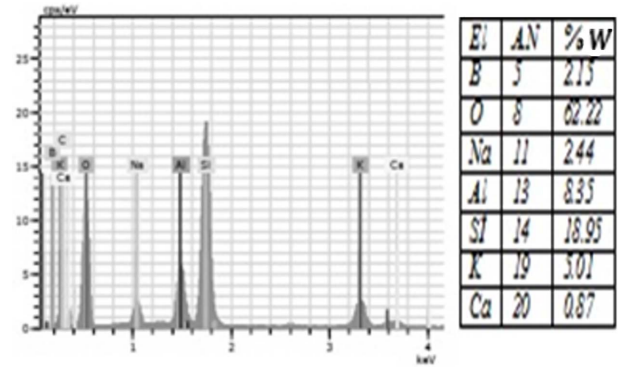


Fig. 8. The amount of elements contained in the %1 Borax added group according to EDS analysis.

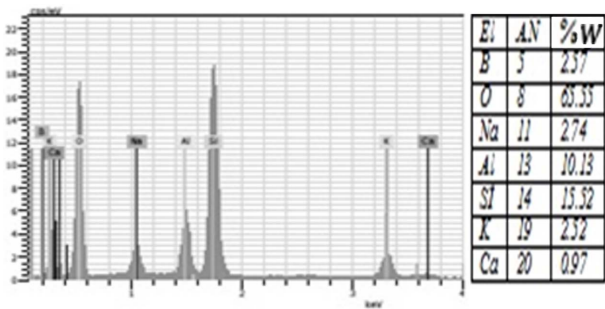


Fig. 6. The amount of elements contained in the %2 Boric Acid group according to EDS analysis.

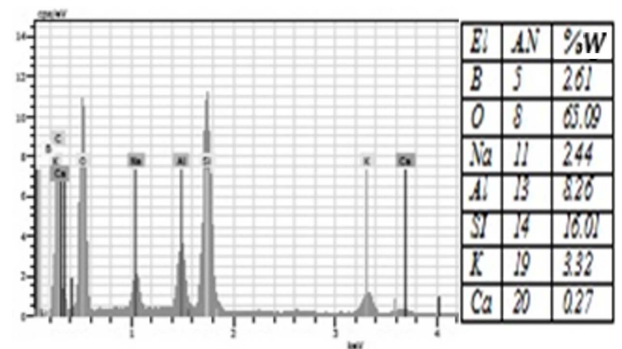


Fig. 9. The amount of elements contained in the %2 Borax added group according to EDS analysis.

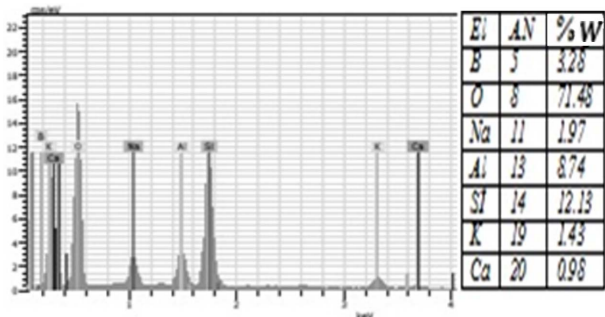


Fig. 7. The amount of elements contained in the %3 Boric Acid group according to EDS analysis.

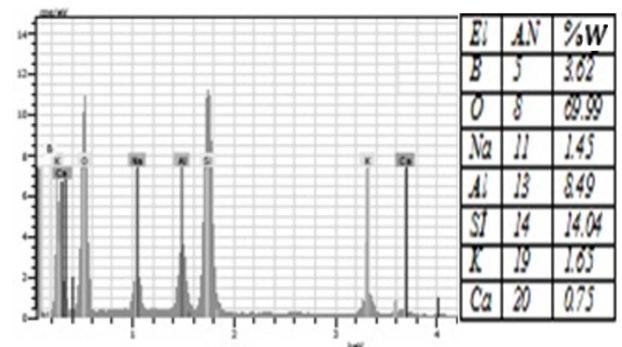


Fig. 10. The amount of elements contained in the %3 Borax added group according to EDS analysis.

and shapes of XRD peaks of the Feldspastic phase did not change, it was concluded that addition of borax and boric acid did not change the crystal structure of the main phase. The SEM images of the sintered porcelain samples obtained from the control, borax and boric acid groups support that the crystal structure did not change. In addition, XRD results are consistent with EDS analysis results.

Conclusions

Within the limits of current study, reinforcement of feldspathic ceramic with small quantities of boric acid or borax resulted in an increased fracture toughness

value compared to the control group. This may indicate that boron products also have fluxing effect on dental ceramics. On the other hand, biaxial flexural strength of feldspathic ceramic decreased with the addition of varied amounts boric acid and borax but was not significant compared to the control group. It is considered that the current study could be a good starting point for future studies on reinforcement with boron compounds of feldspathic dental ceramic and innovation of different high technology dental ceramics with strengthening properties.

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References

1. K.J. Anusavice, C. Shen, and H.R. Rawls, in "Phillips' science of dental materials" (Elsevier Health Sciences, 2013) p.418-474.
2. I.L. Denry, *Crit. Rev. Oral Bio. Med.* 7 (1996) 134-43.
3. K. Anusavice and B. Hojjatie, *J. Dent. Res.* 70 (1991) 1009-1013.
4. M. Albakry, M. Guazzato, and M.V. Swain, *J. Biomed. Mater. Res., Part B* 71 (2004) 99-107.
5. S.S. Scherrer, I.L. Denry, and H.A. Wiskott, *Dent. Mater.* 14 (1998) 246-255.
6. S. Stefanov, *Glass Tech.* 41 (2000) 193-196.
7. Ö. Cengiz and A. Kara, *AKU-J. Sci. Eng.* 9 (2009) 29-35.
8. S. Cook, in "Ceramic engineering and science proceedings" (Wiley Online Library, 2002) p.47-55.
9. H. Yilmaz, C. Aydın, and B.E. Gul, *J. Prosth. Dent.* 98 (2007) 120-128.
10. G. Anstis, P. Chantikul, B.R. Lawn, and D. Marshall, *J. Amer. Cer. Soc.* 64 (1981) 533-538.
11. P. Chantikul, G. Anstis, B.R. Lawn, and D. Marshall, *J. Amer. Cer. Soc.* 64 (1981) 539-543.
12. M.A. Rosenblum and A. Schulman, *J. Amer. Dent. Assoc.* 128 (1997) 297-307.
13. R. Seghi, T. Daher, and A. Caputo, *Dent. Mater.* 6 (1990) 181-184.
14. L.K. Weinstein and A.B. Weinstein, US Patent No. 3,052,982 (1962).
15. R. Cook, B. Lawn, T. Dabbs, and P. Chantikul, *J. Amer. Cer. Soc.* 64 (1981) C-121-C-122.
16. M. Kah, in "Comparison of Surface Treatments on the Strength of Dental Porcelain". (The University of Sydney, 1992) p.11.
17. S.U. Bayca, *J. Ceram. Process. Res.* 10 (2009) 162-166.
18. T. Aydın and A. Kara, *AKU-J. Sci. Eng.* 9 (2009) 53-60.
19. R.E. Loehman, in "Characterization of ceramics" (Momentum Press, 2010) p.169-187.
20. A.S.C. Nathan, R. Tah, and M.K. Balasubramaniam, *J. Oral Biol. Craniofac. Res.* 8 (2018) 221-224.
21. R. Tokunaga, H. Takahashi, N. Iwasaki, M. Kobayashi, K. Tonami, and N. Kurosaki, *Dent. Mater. J.* 27 (2008) 347-355.
22. H. Fischer and R. Marx, *J. Dent. Res.* 80 (2001) 336-339.
23. W.J. O'Brien, in "Dental materials and their selection" (Quintessence Publishing Company, 2002) p.212-229.
24. A. Shenoy and N. Shenoy, *J. Conserv. Dent.* 13 (2010) 195.
25. I.M. Hamouda and M.M. Beyari, *Int. J. Sci. Res. Know.* 1 (2013) 404.
26. N.V. Asar, T. Korkmaz, and E.B. Gül, *Mater. Des.* 31 (2010) 2540-2545.
27. I.S. Medeiros, L.A. Luz, H.N. Yoshimura, P.F. Cesar, and A.C. Hernandez, *J. Mech. Beh. Biomed. Mater.* 2 (2009) 471-477.
28. ISO, No. 6872 (2008) p.11-21.
29. B. Ersu, M. Yenigül, and I. Tulunoğlu, *J. Hacettepe Univ. Fac. Dent.* 31 (2007) 71-78.
30. A. Miller, J. Long, B. Miller, and J. Cole, *J. Prosth. Dent.* 68 (1992) 38-41.
31. J.R. Kelly, *Dent. Mater.* 11 (1995) 103-110.
32. J.W. McLean, *Oper. Dent.* 16 (1991) 149-156.
33. R.A. Giordano, L. Pelletier, S. Campbell, and R. Pober, *J. Prosth. Dent.* 73 (1995) 411-418.
34. M.A. Kılıçarslan and A. Zaimoğlu, *J. Istanbul Univ. Fac. Dent.* 32 (1998) 28-33.
35. R.R. Seghi, J.A. Sorensen, R. Seghi, and J. Sorensen, *Int. J. Prosth.* (1995) 8.
36. A. Zaimoğlu, G. Can, E. Ersoy, and L. Aksu, in "Diş hekimliğinde maddeler bilgisi" (AÜ Publisher, 1993) p.515.
37. J.D. Hudson, G.R. Goldstein, and M. Georgescu, *J. Prosth. Dent.* 74 (1995) 647-654.
38. D. Jagger and A. Harrison, *J. Prosth. Dent.* 72 (1994) 320-323.
39. A.S. Rizkalla and D.W. Jones, *Dent. Mater.* 20 (2004) 198-206.
40. J. Gong, Z. Zhao, and Z. Guan, *J. Euro. Cer. Soc.* 21 (2001) 941-946.
41. G. Elssner, H. Hoven, G. Kiessler, and P. Wellner, in "Ceramics and ceramic composites: materialographic preparation" (Elsevier, 1999) p.151-153.
42. M. Guazzato, M. Albakry, S.P. Ringer, and M.V. Swain, *Dent. Mater.* 20 (2004) 441-448.
43. H.N. Yoshimura, C.C. Gonzaga, P.F. Cesar, and W.G. Miranda Jr, *Ceram. Int.* 38 (2012) 4715-4722.
44. M.K. Etman, *J. Prosthodontics* 18 (2009) 550-559.
45. N. Beck, F. Graef, O. Gerstbrein, and M. Karl, *J. Prosth. Dent.* 104 (2010) 301-305.
46. I. Denry, J. Mackert Jr, J. Holloway, and S. Rosenstiel, *J. Dent. Res.* 75 (1996) 1928-1935.
47. D. Xie, W. Brantley, B. Culbertson, and G. Wang, *Dent. Mater.* 16 (2000) 129-138.