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Effect of concentration of hydrofluoric acid and etching time on microtensile bond strength of Zirconia-reinforced lithium silicate ceramics

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ABSTRACT

Introduction: Etching the internal surface of ceramic restorations with hydrofluoric (HF) acid and silane is a well-accepted technique to enhance the bond strength. The aim of this study was to assess the effect of concentration of hydrofluoric acid and etching time on microtensile bond strength (µTBS) of zirconia-reinforced lithium silicate (ZLS) ceramics in 2021.

Materials & Methods: This in vitro study was conducted on 8 Celtra-Duo ceramic blocks size 14 measuring 12×14×18 mm. Each ceramic block was divided into three equal pieces by a cutting machine to obtain a total of 24 specimens. The specimens were randomly divided into 6 groups for etching with 5% and 10% HF acid for 30, 60, and 120 seconds. Silane (Clearfil porcelain activator) and bonding agent (Clearfil SE Bond) were applied to the etched specimens. Panavia F2 resin cement was applied on the surfaces and light-cured. The µTBS of resin cement to porcelain was measured by a universal testing machine. The mode of failure was determined under a stereomicroscope at x40 magnification. Data were analyzed by one- and two-way ANOVA ($P<0.05$).

Results: The mean µTBS of Celtra-Duo ceramics subjected to etching for 30, 60, and 120 seconds was not significantly different in the use of 5% and 10% HF acid concentrations ($P>0.05$). Two-way ANOVA showed that the effects of HF acid concentration and etching time, and their interaction effect were not significant on µTBS of CAD/CAM Celtra-Duo ceramics ($P>0.05$). The mode of failure was dominantly adhesive in both concentrations of 5% and 10% HF acid. No mixed failure occurred in both concentrations.

Conclusion: Considering the non-significant difference in µTBS of ceramics subjected to different concentrations of HF acid for different times, the application of HF acid with lower concentration for a shorter period is recommended to prevent possible adverse effects on ceramic strength.

Keywords: Ceramics, Dentistry, Hydrofluoric Acid, Resin Cements

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Introduction

Ceramic restorations are widely used due to their excellent durability, esthetics, and biocompatibility. Dental ceramics can better mimic the appearance of natural teeth compared with other dental materials.[1] Silicate minerals such as quartz and silica are the main constituents of dental ceramics. Modern dental ceramics have a higher content of the crystalline phase which significantly improves their biomechanical properties.[2] Ceramic restorations can be fabricated by the conventional laboratory technique or the computer-aided design computer-aided manufacturing (CAD/CAM) systems.[3-7] The CAD/CAM technology decreases the fabrication time of high-strength ceramics.[4, 5] Moreover, the blocks fabricated by the CAD/CAM technology are more homogenous, and have fewer defects.[8, 9] Long-term success of ceramic restorations depends on the strength and durability of the resin cement bond to porcelain and dental substrates.

In 1983, Horn suggested etching the surface of porcelain veneers with hydrofluoric acid (HA).[10] A two-dimensional assessment of the etched surface indicates that the porcelain surface is selectively dissolved, depending on the porcelain composition. Accordingly, a surface more prepared for bonding is created as such.[9, 10] Etching of the internal ceramic surface with HA followed by silane application is a documented technique for enhancement of bond strength.[11]

Researchers have long been in search of novel restorative materials with favorable mechanical and esthetic properties. This search led to the introduction of zirconia-reinforced lithium silicate (ZLS) ceramics, which can be used for the fabrication of restorations with CAD/CAM technology.[12] Two types of CAD/CAM ZLS ceramics are currently available in the market namely Vita Suprinity (Vita Zahnfabrik, Bad Säckingen, Germany) and Celtra-Due (Dentsply Sirona, DeguDent, GmbH, Hanau-Wolfgang, Germany). Celta-Duo ceramics have 10% zirconia in their structure, resulting in four times smaller lithium silicate crystals. These ceramics can provide a flexural strength as high as 210 MPa if polished manually, and 370 MPa if glazed in a furnace.[13] Evidence shows that HF acid etching has the greatest efficacy for enhancement of the bond strength of porcelain with a glass matrix to resin cement. [11] The kinetics of the reaction between HF acid and ceramic is influenced by the etching time and concentration of HF acid. [10]

Since the introduction of HF acid for ceramic surface treatment prior to resin bonding, different etching times have been proposed. Also, ZLS is acid sensitive[14], and it is important to clarify the ideal acid concentration and etching times for this ceramic type.[12] However, no consensus has been reached on an ideal etching time with HF acid for the treatment of glass-ceramic restorations. Nonetheless, the manufacturer recommends 30 seconds of etching. Also, due to the novelty of these ceramics, it is important to find the shortest etching time that yields maximum bond strength and has no adverse effect on ceramics.[15]Thus, this study aimed to assess the effect of three different etching times with two different concentrations of HF acid on microtensile bond strength (μTBS) of CAD/CAM Celtra-Duo ceramics to resin cement. The null hypothesis was that increasing the etching time and concentration of HF acid would not increase the μTBS of CAD/CAM Celtra-Due ceramics to resin cement.

Materials & Methods

This study was approved by the Ethics Committee of Babol University of Medical Sciences (ethical number: IR.MUBABOL.REC.1399.440). This in vitro experimental study was conducted at the Dental Materials Research

Center of School of Dentistry, Babol University of Medical Sciences in 2020-2021 on CAD/CAM Celtra Duo ceramic blocks.

•Specimen preparation: A total of 8 CAD/CAM Celtra-Duo ceramic blocks (#14) were selected. The sample size was calculated based on a previous study and the below formula.[16]

$$n = \frac{\left(Z_{1-\frac{\alpha}{2}} + Z_{1-\beta}\right)^2 (S_1^2 + S_2^2)}{(d)^2} = 20\ ,\ \alpha = 0.05\ ,\beta = 0.20\ ,S_1 = 4\ ,S_2 = 2\ ,d = 2.8$$

A total of 8 Celtra-Duo (Dentsply Sirona, DeguDent, GmbH, Hanau-Wolfgang, Germany) (#14) measuring 12×14×18 mm were used in this study. Each ceramic block was sectioned into three equal specimens by a cutting machine (Delta Precision Sectioning Machine, Mashhad, Iran). A total of 24 specimens were obtained as such (n=4 in each group). The surface of ceramic blocks was finished with a blue long fissure bur (D & Z) for standardization.[17] Next, 5% and 10% concentrations of HF acid were manually prepared. To prepare 5% HF acid, 1 unit of 40% HF acid (Merck, Darmstadt, Germany) was mixed with 7 units of deionized distilled water. To prepare a 10% concentration of HF acid, 1 unit of 40% HF acid was mixed with 3 units of deionized distilled water. The ceramic specimens were randomly divided into two groups for use of 5% and 10% HF acid. Each group was then randomly divided into three subgroups for etching for 30, 60, and 120 seconds (a total of 6 subgroups). Each ceramic group then underwent etching with either 5% or 10% HF acid for 30, 60, or 120 seconds.

After etching, the ceramic specimens were rinsed with air and water spray for 30 seconds and placed in an ultrasonic bath (BioSonic UC50D, Coltene, Whaledent, USA) for 5 minutes to eliminate the residual salts. To eliminate the excess moisture, the specimens were immersed in 99% alcohol and dried with air spray. Table 1 presents the characteristics of the materials used in this study.

•Bonding procedure: One layer of silane (Clearfil Porcelain Bond Activator; Kuraray Medical Inc., Osaka, Japan) was applied to the etched ceramic specimens, dried, and thinned with air spray such that no additional liquid remained on the surface. This was done to create a single layer of porcelain primer for a stronger bond to the bonding agent. Next, one layer of bonding agent (Clearfil SE Bond; Kuraray Medical Inc., Osaka, Japan) was applied to the specimen surface.[17] Panavia F2 cement (Kuraray Medical Inc., Osaka, Japan) was then applied on the prepared ceramic surfaces according to the manufacturer's instructions such that equal amounts of pastes A and B were mixed. The minimum mixing time was 20 seconds. The mixture with paste-like consistency was directly applied into a transparent mold with 6 mm diameter and 2 mm height, and the mold was placed on the silanized ceramic. Afterward, the cement surface was cured by a LED curing unit (Valo Corded, Ultradent, South Jordan, UT, USA) for 20 seconds with a light intensity of 1000 mW/cm^2.

Preparation process and microtensile bond strength test:

To prepare the micro-bars, ceramic-cement blocks were mounted in transparent epoxy resin in stainless steel molds (1×1 mm^2). The mounted specimens were sectioned using a sectioning machine (Delta Precision Sectioning Machine, Mashhad, Iran) with a disc under running water. The sections had a slice interval of 1 mm and were made in two planes perpendicular to each other. Accordingly, micro-bars were obtained with a 1 mm^2 cross-sectional area and 4 mm height (2 mm of ceramic and 2 mm of resin cement). Five microbars were selected from each sample (each subgroup included 20 microbars). The microbars were subjected to a tensile force at a speed of 0.5 mm/minute in a universal testing machine (Koopa, Sari, Iran) until failure. The tensile load in Newton (N) was divided by the cross-sectional area in square millimeters (mm^2) measured by a digital caliper (Shinwa Rules Co., Niigata, Japan) to calculate the bond strength in megapascals (MPa). The μTBS of each specimen was calculated using the formula below: $\alpha=L/A$

Where L indicates load at failure, and A indicates the bonded surface area.

Table 1. Characteristics of the materials used in this study

Material	Description	Manufacturer and Country	Composition and Batch Number
Panavia F2	Dual-cure self-etch resin cement	Kuraray Medical Inc., Osaka, Japan	Paste A: hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, sodium aromatic sulfinate (TPBSS), N, Ndiethanol-p-toluidine, surface-treated (functionalized) sodium fluoride,10%, silanated barium glass (61185); Paste B: MDP, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica, photoinitiator, dibenzoyl peroxide (61185)
Clearfil SE Bond	Light-cure self-etch adhesive	Kuraray Medical Inc., Osaka, Japan	Primer: MDP, HEMA, Hydrophilic dimethacrylate, N, N-Diethanol, p-toluidine, water(00109A) Bonding: MDP, Bis-GMA, HEMA hydrophobic dimethacrylate, dl-Camphorquinone, N, N-Diethanolp-toluidine, silanated silicate(00043A)
Clearfil Porcelain Bond Activator	one bottle of pre-activated silane	Kuraray Medical Inc., Osaka, Japan	Bisphenol A polyethoxydimethacrylate, 3-methacryloyloxypropyltrimethoxy Silane (00241A)
Celtra-Duo ceramic	Zirconia-reinforced glass-ceramic	Dentsply Sirona, Germany	10% zirconia, 58% silica, lithium metasilicate, and phosphate crystals, SiO2, P2O5, Al2O3, LiO, ZnO, 10% ZrO2 (16006396)
Hydrofluoric acid 40%	Liquid 40% hydrofluoric acid	Merck, Darmstadt. Germany	Chloride:1ppm, Hexafluorosilicate :50 ppm,phosphate:0.5 ppm,Sulphate:2 ppm, Arsenic & Antimony:0.03 ppm, Silver:0.020 ppm,Aluminium:0.050 ppm, Barium:0.050 ppm, Beryllium:0.020 ppm, Bismuth:0.020 ppm, Calcium:0.200 ppm (B0710538231)

Assessment of the mode of failure: The mode of failure of specimens was determined under a stereomicroscope at x40 magnification. The failures were categorized into three categories of cohesive failure (fracture within the ceramic or cement), failure at the ceramic-cement interface (adhesive), and mixed failure (fracture of the ceramic, resin cement, and interface).

•Statistical analysis:Data were analyzed using SPSS version 26. One-way ANOVA and independent sample t-test were applied to compare different etching times. Two-way ANOVA was used to analyze the interaction effect of the variables. $P<0.05$ was considered statistically significant.

Results

The mean µTBS of CAD/CAM Celtra-Duo ceramics subjected to 5% HF acid etching (P=0.211) and 10% HF acid etching (P=0.724) for 30, 60, and 120 seconds was the same with no significant difference (Table 2). The mean µTBS of CAD/CAM Celtra-Duo ceramics etched for 30 (P=0.107), 60 (P=0.707), and 120 seconds (P=0.773) was not significantly different in the use of 5% and 10% HF acid.

According to two-way ANOVA, the effects of HF acid concentration (P=0.166) and etching time (P=0.433), and the interaction effect of etching time and HF acid concentration (P=0.153) on µTBS of CAD/CAM Celtra-Duo ceramics were not significant. The majority of failures in 5% and 10% HF acid groups were adhesive. Mixed failure was not seen in any of the 5% or 10% HF acid groups (Table 3).

Table 2. Mean and standard deviation of microtensile bond strength of CAD/CAM Celtra-Duo ceramics subjected to etching with different concentrations of HF acid for different times

Etching time	5% concentration	10% concentration	P value**
	Mean and standard deviation of bond strength (MPa)	Mean and standard deviation of bond strength (MPa)	
30 s (n=20)	13.08±5.99	10.75±1.53	0.107
60 s (n=20)	11.30±2.69	11.05±1.20	0.707
120 s (n=20)	11.00±2.09	11.22±2.51	0.773
P value*	0.211	0.724	-

*ANOVA; **Independent sample t-test

Table 3. Frequency percentage of different failure modes in the study groups

HF acid concentration	Etching time	Adhesive failure	Cohesive failure (fracture within the ceramic or cement)	Mixed failure
	30 s	14	6	0
	60 s	13	7	0
	120 s	15	5	0
	30 s	11	9	0
	60 s	12	8	0
	120 s	10	10	0

Discussion

This study revealed that no significant difference in µTBS of CAD/CAM Celtra-Duo ceramics etched with 5% and 10% HF acid with different etching times, and the null hypothesis of the study was accepted. Thus, the best etching time for CAD/CAM Celtra Duo ceramics is 30 seconds by using 5% HF acid.

The creation of sufficient porosity for a strong bond is influenced by the composition of ceramic.[18] CAD/CAM Celtra-Duo ceramic has a high crystalline content (70 v%) in its glass matrix phase and contains 58% silica and

10% zirconia crystals along with lithium meta-silicate and phosphate crystals. Mokhtarpour et al. (2019) evaluated electron microscopic images and showed that increasing the etching time and concentration of HF acid can cause surface degradation in CAD/CAM ceramics and result in crack formation in them. Thus, a shorter etching time with a lower concentration of HF acid can provide optimal surface porosity for bonding. [17] Similar to the present study, Mokhtarpour et al. (2017) in another study compared 5% and 10% concentrations of HF acid and 20, 60, and 120 second etching times and reported that the mean micro-shear bond strength (SBS) of e.max and Vita Mark II was not significantly different after using different concentrations of HF acid with different etching times. However, the maximum micro-SBS was recorded in e.max specimens etched with 5% HF acid for 60 seconds and Vita Mark II specimens etched with 10% HF acid for 20 seconds.[16] They suggested etching with 5% HF acid for 20 seconds.

Fonzar et al. (2020) [19] assessed the mean micro-SBS of Vita Suprinity ceramic and suggested etching with 4.9% HF acid for 20 seconds. They found a significant difference in bond strength between different concentrations of etchant, which was different from the present results. However, different etching times had no significant effect on the mean micro-SBS, which was in line with the present findings. Straface et al. (2019) [20] found that 15 seconds of etching of Vita Suprinity ceramic with 5% HF acid yielded the maximum SBS. Longer etching times had no significant effect, and the efficacy of 30 seconds of etching was comparable to 60 seconds of etching. Yazarloo et al. (2019). [18] evaluated the μTBS of Suprinity ceramic and found that the best etching time was 120 seconds with 5% HF acid. Duration of etching and concentration of etchant significantly affected the μTBS of Suprinity ceramic in their study, which was different from the present findings. Ramakrishnaiah et al. (2016) used 5% HF acid for 20, 40, 80, and 160 seconds for etching of IPS e.max, Vita Mark II, Suprinity, And Dentsply Celtra ceramics and found that increasing the etching time increased the depth and number of porosities, surface hardness, and surface wetting. [21] Variations in the results of studies on this topic can be attributed to differences in ceramic types.

In the present study, 30 seconds of etching was selected as recommended by the manufacturer; the selection of 60 and 120 seconds of etching times was based on the results of Chen et al.[22] To eliminate the possible confounding effect of other ingredients in the composition of commercially available porcelain etchants (in addition to HF acid), these concentrations were manually prepared in this study.

In this study, the majority of failures were adhesive. In general, cohesive failure had a higher frequency in 10% concentration of acid comparable to the frequency of adhesive failure. This finding indicates that the tensile strength of adhesive was almost similar to the cohesive strength of ceramic and cement. Adhesive failure indicates that the strength of the adhesive is stronger than that of the adherend, while cohesive failure indicates the lower strength of the adherend than the adhesive.[16]

Several factors can affect the bond strength such as the cutting process of specimens, heterogeneity of the substrate, variations in material properties, technical sensitivity of the cement, and expertise of the operator, resulting in differences in the reported bond strength values.[23]

This study had some limitations. The sectioning of ceramic blocks was difficult and time-consuming, and the materials were costly. Considering the different properties of ceramics, it is suggested to perform other tests on them. Also, the flexural strength of ceramics should be measured following the application of different concentrations of etchants with different application times.

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Conclusion

Thus, it appears that the best etching time for CAD/CAM Celtra-Due ceramics with 5% HF acid would be 30 seconds.

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Conflicts of Interest

There is no conflict of interests to declare

Authors' Contribution

Homayoon Alaghehmand developed the original idea and protocol. Morteza Rostami contributed to the development of the protocol, abstracted data and prepared the manuscript, and edited the article. Faraneh Mokhtarpour analyzed the data. Study supervision was conducted by Homayoon Alaghehmand.

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