

Effect of hydrofluoric acid surface treatments on micro-shear bond strength of CAD/CAM ceramicsFaraneh Mokhtarpour¹, Homayoon Alaghehmand², Soraya Khafri³

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Abstract

Introduction: Dental ceramics are appreciated as highly esthetic restorative materials that can simulate the appearance of natural dentition better than other materials. The aim of this study was to evaluate the effect of hydrofluoric acid concentration and etching time on micro-shear bond strength (μ SBS) to IPS e.max CAD and Vita Mark II of a dual cured resin cement (Panavia F2.0).

Methods: This study was an experimental in vitro study, performed in the dental material research center of Babol University of Medical Sciences in 2016. Two hydrofluoric acid concentrations (5% and 10%) and three different etching times (20, 60 and 120 seconds) were used to etch the specimens respectively. A silane coupling agent (Clearfil porcelain activator) and priming and bonding agent (Clearfil SE bond) were used on the etched surfaces in accordance to the manufacturer's instructions of use. Then resin cement was applied on the prepared ceramic surfaces and light cured. μ SBS between resin cement and the porcelains were measured with a universal testing machine. Mode of failure was observed with 40x magnification by means of a Stereo microscope. Data were analyzed with ANOVA and independent-samples t-test and Chi-square tests.

Results: In both e.max and Vita Mark II groups, μ SBS were not significantly different when different etching times (one-way ANOVA) and HF acid concentrations (Independent-samples t-test) were used ($p>0.05$), but the highest μ SBS was shown in e.max specimens etched 60 s with 5% HF and Vita Mark II specimens etched 20 s with 10% HF. μ SBS of e.max was significantly higher than Vita Mark II ($p=0.00$).

Conclusion: Best surface treatment for e.max and Vita Mark II ceramics is 20 s etch using 5 % hydrofluoric acid.

Keywords: Glass ceramic; Hydrofluoric acid; Resin cement; CAD/CAM; Adhesion

1. Introduction

Dental ceramics are appreciated as highly esthetic restorative materials that can simulate the appearance of natural dentition better than other materials (1). The use of all ceramic prosthesis in restorative treatments has become popular, and many of these restorations can be fabricated by both traditional laboratory methods and computer aided design/computer aided manufacturing (CAD/CAM) machining. The traditional methods of ceramic fabrication have been described to be time-consuming, technique sensitive and unpredictable due to the many variables and CAD/CAM may be a good alternative for both the dentists and laboratories (2). CAD/CAM may also reduce the fabrication time of high strength ceramics by up to 90%. Furthermore, industrially fabricated blocks are more homogenous with minimal flaws (3). Lithium disilicate and feldspathic ceramic are silica-based ceramics. Feldspathic porcelain is a composite of leucite (potassium aluminosilicate) and glass and lithium disilicate ceramics comprised of approximately 65% volume of highly interlocking lithium disilicate crystals discharged in a glassy

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matrix (4). Vita Mark II is CAD/CAM feldspathic ceramic that was introduced for the CEREC system in 1991 (5). IPSTM E.max CAD (Ivoclar Vivadent) is lithium disilicate ceramic that was introduced in 2006 for chairside use (3, 6). For survival of the porcelain laminate veneers in the oral environment, bonding achieved between the porcelain and resin cement is essential (7, 8). Modification of the internal porcelain surfaces by acid etching or air abrasion, resulted in improved bonding (9, 10). A well-known method for increasing bond strength is etching the ceramic surfaces with hydrofluoric acid and then applying a silane coupling agent on the etched surfaces (11). A routine surface pre-treatment for the restoration with dental porcelains is the combination of appropriate etching process following by the application of silane coupling agent (12). Hydrofluoric acid etching on porcelain structures leads to increased surface roughness and higher surface energy (13). Surface modification of the porcelain increases the surface area available for bonding and creates undercuts that increase the bond strength to the resin cement. Subsequent examination of the etched porcelain surfaces, showed that different processes of etching of porcelain surfaces dissolved different porcelain phases preferentially depending on the porcelain composition, and created a more conductive surface for bonding (14, 15). There is a reaction between the Hydrofluoric acid and the silica containing glass matrix and that forms hexafluorosilicates. Following removal of the glass matrix, the crystalline structure is exposed resulting in the surface of the ceramic becoming rough, which is required for micromechanical retention (16-18). Its rough surface also provides more surface energy prior to incorporating the silane solution (17, 19). Several different etching periods have been suggested and used since the introduction of HF acid etching as a ceramic surface pretreatment for resin bonding, (1). It was found that increasing etching duration time (HF) resulted in higher shear bond strength between resin adhesive and dental CAD/CAM porcelain within a range of 0 to 120 s (18). The manufacturer's Advocated etching time for cementation of the IPS e.max Press glass ceramic restorations with a resin cement is about 20 s. J.H Chen in 1998, recommended 120 s etching time with 5% HF acid for Vita Mark II to obtain highest bond strength (18). However, clinically, the optimal HF acid etching time and concentration to treat the glass ceramic restoration surface is not clear and there are not enough articles about appropriate etching time for CAD/CAM ceramics. Therefore, it is important to know the adequate and optimal HF etching time for resin cement bonding without weakening the ceramic (1). Null hypotheses of this study were: 1) Micro-shear bond strength of resin cement with resin cement increase with increasing etching time, 2) Micro-shear bond strength of resin cement with resin cement increase with increasing HF concentration.

2. Material and Methods

2.1. Research design

This study was an experimental in vitro study, which was conducted at the dental material research center of Babol University of Medical Sciences in 2016.

2.2. Specimen preparation

Two types of CAD/CAM chairside ceramics were used in this survey, Feldspathic (Vita Mark II (M)) and Lithium disilicate (IPs e.maxTM CAD(E)) (Table 1). Six blocks No.14 of each ceramic were used. Each block sectioned in width to five pieces using a water-cooled diamond disk with a low-speed saw machine (Delta precision sectioning machine, Mashhad, Iran). For creating a standard surface, blue diamond bur was used for grounding the ceramic surface. Then all specimens were finished to 600 grit silicon carbide paper to simulate CAD/CAM milled surfaces. e.max specimens were heated under vacuum in an oven in order to complete their crystallization. Two different concentrations of handmade HF acid, 5% (a) and 10% (b) (Table 1), were used to etch ceramics. Three etching times were evaluated in this study: 20 s, 60 s and 120 s. Therefore, we had six subgroups for each ceramic (30 pieces): Ma20, Ma60, Ma120, Mb 20, Mb60, Mb120 (Vita Mark II subgroups) and Ea20, Ea60, Ea120, Eb20, Eb60, Eb120 (e.max subgroups). After etching specimens according to their subgroup etching time, they were rinsed with air-water spray for 30 seconds. Then they were ultrasonically cleaned in distilled water for five minutes. For removing any contamination from the ceramic's surface, we used phosphoric etchant gel (Table 1) for five seconds on the pieces and then they were washed and air dried and placed in 99% alcohol, then dried with compressed hot air.

2.3. Bonding method

Thirty pieces for each ceramic were used. The ceramic pieces were equally and randomly assigned to six study groups and etched according to their subgroup. One layer of equal mix of silane coupling agent and clearfil SE bond primer was applied onto all ceramic pieces and allowed to be air dried for 60 seconds. Then one layer of clearfil SE bond was applied onto the ceramics. After that, an equal amount of paste A and B of Panavia (Table 1) were mixed for more than 20 seconds and applied onto the prepared ceramics. Cement interfaces were cured for 20 second from each side with VALO LED (Ultradent USA) with 1000 mw/cm² intensity.

2.4. Micro-shear bond strength test

Ultimately, the pieces were sectioned perpendicular to the bonding interface area to obtain beams with a bonding area of about 1mm² using a water-cooled diamond disk in a sectioning machine (Delta precision sectioning machine, Mashhad, Iran). Two beams from each piece were obtained. So, 10 beams in each experimental group were tested for μ SBS (60 total beams for μ SBS test). The cross-sectional area of each beam was measured using a digital caliper (Shinwa Rules Co., Niigata, Japan). Micro-shear bond strengths were measured with Zwick Universal Testing machine (Zwick GmbH & Co., Ulm, Germany) at a crosshead speed of 0.5 mm/min until failure. The resultant forces in N were divided into the cross-sectional area and the pressures in Mpa were calculated.

2.5. Failure mode

Failure mode of beams were evaluated with a stereomicroscope with 40X magnification and reported into three groups: 1) Failure in ceramic or resin cement (cohesive); 2) Failure in the interface of ceramic and resin cement (adhesive); and 3) Failure in ceramic or resin cement and the interface (mixed).

2.6. Statistical analysis

Independent-samples t-test was used for μ SBS and Ra comparison between different HF concentrations and type of ceramic. One-way ANOVA was used for comparison between different etching times. Two-way and three-way ANOVA was used for evaluating interactions among the factors. Chi-square test was used for analysis of mode of failure.

Table 1. Material descriptions, manufacturers, compositions and batch number

Material (manufacturer)	Description	Composition and batch number
Panavia F2.0: Kuraray Medical Inc, Osaka, Japan	Dual-cure single-step self-etch resin cement	ED Primer II; Liquid A: HEMA (30%-50%), MDP, Nmethacryloyl-5-aminosalicylic acid, water, accelerator (61185); ED primer II liquid B: N-methacryloyl-5-aminosalicylic acid, accelerator, water, sodium benzene sulfinate (61185); Paste A: hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, sodium aromatic sulfinate (TPBSS), N,N diethanol-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, photo initiator, dibenzoyl peroxide (61185)
Clearfil SE Bond: Kuraray Medical Inc, Osaka, Japan	Light-cure self-etch adhesive	Primer: MDP, HEMA, Hydrophilic dimethacrylate, N, N-Diethanol, p-toluidine, water(00109A) Bonding: MDP, Bis-GMA, HEMA hydrophobic dimethacrylate, dl-Campherquinone, N, N-Diethanol p-toluidine, silanated silicate(00043A)
Clearfil Porcelain Bond Activator: Kuraray Medical Inc, Osaka, Japan	One bottle pre-activated silane	Bisphenol A polyethoxy dimethacrylate, 3 methacryloyl oxypropyl trimethoxy silane (00241A)
VITA BLOCS Mark II: VITA Zahnfabrik, Bad Säckingen, Germany	CEREC/in Lab (2M1C I12) block	Mixture of feldspathic crystalline particles embedded in a glassy matrix Vol % \approx 30(15670)
IPS e.max CAD blocks: Ivoclar Vivadent, Liechtenstein	Lithium disilicate blocks	SiO ₂ (57–80%), Li ₂ O (11–19%), K ₂ O (0–13%), P ₂ O ₅ (0–11%), ZrO ₂ (0–8%), Al ₂ O ₃ (0–5%), MgO 90–5%) and coloring oxides (0–8%)(R64456)
Merk Hydrofluoric acid 40%: Merk, Darmstadt, Germany	Liquid 40% hydrofluoric acid	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)
Scotchbond Etchant gel: 3M ESPE, St. Paul, MN, USA	35% acid phosphoric etchant gel	35% by weight Phosphoric acid, 60% water and 5% Synthetic amorphous silica as thickening agent(N105148)

3. Results

3.1. Micro-shear bond strength

Mean μ SBS and standard deviation of the e.max and Vita Mark II are shown in Table 2. In both e.max and Vita Mark II groups, μ SBS were not significantly different when different etching times (one-way ANOVA) and HF acid concentrations (Independent-samples t-test) were used ($p > 0.05$), but the highest μ SBS was shown in Ea60 and Mb20 groups. The μ SBS of e.max was significantly higher than Vita Mark II ($p = 0.00$, one-way ANOVA). The μ SBS was not significantly different between etching times and HF acid concentrations in Vita Mark II. Statistical analysis showed that μ SBS did not have significant difference between different concentrations, times and ceramics (three-way ANOVA).

3.2. Mode of failure

Modes of failure are shown in Table 3. Chi-square test showed for e.max groups that in 20, 60 and 120 s etching times, HF concentration did not have any significant influence on failure mode. Without considering etching times, HF concentration had significant effect on failure mode. In 5% concentration, adhesive failure was more than others and in 10% concentration, mixed failure was dominant. In Vita Mark II groups, only in 60 s etching time, HF concentration had significant influence on failure mode, and cohesive failure was the most in 5% concentration, and in 10% concentration adhesive failure was the most. Without considering etching times, HF concentration had significant effect on failure mode, so in 5% concentration cohesive failure was the most and in 10% concentration adhesive failure was dominant.

Table 2. Micro-shear bond strength (MPa) of e.max & Vita Mark II

e.max		
Acid time	Acid concentration	
	5%	10%
20 s	10.52±5.39 ^{aA}	13.59±6.93 ^{aA}
60 s	16.84±9.85 ^{aA}	14.35±7.02 ^{aA}
120 s	12.76±4.56 ^{aA}	14.94±4.13 ^{aA}
Vita Mark II		
Acid time	Acid concentration	
	5%	10%
20 s	6.32±2.92 ^{aA}	7.18±2.97 ^{aA}
60 s	5.96±3.66 ^{aA}	5.21±1.04 ^{aA}
120 s	6.88±2.64 ^{aA}	5.95±2.4 ^{aA}

The different lowercase letters indicate a significant difference ($p=0.05$) between the etching times maintaining the same acid concentration. Different capital letters indicate a significant difference ($p=0.05$) between Acid concentration maintaining the same time.

Table 3. Mode of failure

Ceramic	HF concentration	Etching time	Adhesive failure	Cohesive failure	Mixed failure
e.max	5%	20 s	7 (70%)	1 (10%)	2 (20%)
		60 s	3 (30%)	4 (40%)	3 (30%)
		120 s	3 (30%)	6 (60%)	1 (10%)
	10%	20 s	3 (30%)	2 (20%)	5 (50%)
		60 s	2 (20%)	1 (10%)	7 (70%)
		120 s	1 (10%)	4 (40%)	5 (50%)
Vita Mark II	5%	20 s	4 (40%)	4 (40%)	2 (20%)
		60 s	2 (20%)	5 (50%)	3 (30%)
		120 s	2 (20%)	5 (50%)	3 (30%)
	10%	20 s	6 (60%)	3 (30%)	1 (10%)
		60 s	8 (80%)	2 (20%)	0 (0%)
		120 s	4 (40%)	4 (40%)	2 (20%)

4. Discussion

In this study, Micro-shear testing results showed that there is no significant difference between different acid concentrations and etching times for each porcelain, so number 1 and 2 null hypotheses were rejected. According to

this study, best surface treatment for e.max and Vita Mark II ceramics is 20 s etch using 5 % hydrofluoric acid. Both chemical and mechanical retention are necessary for a reliable bonding between ceramics and resin cement. Porcelain surface treatments increasing the micromechanical retention of the resin cement by modifying its texture. Silane coupling agents react with the glassy matrix of the ceramic and with the composite organic matrix, so they lead to chemical retention between ceramic and resin cement (20, 21). For these reasons, we used HF acid and silane coupling agent for treating ceramic surfaces. Since the concept of etching porcelain surface was introduced and adhesive cementation of porcelain laminate veneers was reported, many authors have stated that the concentrations and etching periods must be adjusted to each type of ceramic in order to optimize the bond strength (14, 16, 18, 21-23). To avoid reducing the strength of the ceramic, it is important to know the optimal HF etching time for micromechanical retention (17). For this reason, research on the adequate etching protocol for a lithium disilicate-based and feldspathic glass ceramic was carried out in the present study. Regarding etching time, a number of studies have been carried out using various types of ceramic and HF etchants (16-18, 24, 25). Chen et al. (16) evaluated two HF etchants (2.5 and 5%) and seven different etching times (0, 30, 60, 90, 120, 150, and 180 s) for feldspathic porcelain treatment. They determined that etching periods of more than 30 s increased the bond strength to resin and that 2.5% HF yielded higher bond strengths to resin than the 5% HF for all etching time periods, except 180 s. A study by Guler et al. (24) evaluated the effect of a number of 9.6% HF etching times (30 s, 30+30 s, 60 s, 60+60 s, 120 s, and 180 s) on feldspathic porcelain and 2 adhesive systems on shear bond strengths to resin composite. It was concluded that adequate bond strength of porcelain to resin can be produced by HF etching for 120 s. The result of this study showed that there is no significant difference in μ SBS between 5% and 10% HF and 20, 60 and 120 s etching times. In this study, μ SBS of e.max was significantly higher than Vita Mark II, it is because of significant higher fracture resistance of e.max CAD than Vita Mark II ((1378 N/1025 N) versus (405 N/454 N) ($p < .05$) (26). IPS-e.max CAD has up to 70 vol. % crystalline content in glassy matrix. It is composed of 58% silica (SiO₂), lithium-metasilicate, -disilicate and -phosphate crystals and 10% zirconia crystals. Crystallization of e.max is a two-stage process. Primarily, the lithium metasilicate crystals accelerate (40 vol.% of 0.2–1.0 mm crystal size). Eventually, heating under vacuum culminates in the development of a more advanced crystalline content with finer lithium-disilicate crystals (70 vol. % of 1.5 mm grain size) which are incorporated into a glassy matrix. Development of highly interlocked microstructure during final crystallization, results in increased strength of e.max. Vita Mark II is a ceramic material without zirconium, and composed of a weak glass matrix phase and one or more irregularly-shaped crystalline phases, which are more brittle and weaker than zirconia. That is why its fracture strength is lower than e.max (27). Zogheib and Della Bonna (17) stated that the flexural strength of the lithium disilicate-based glass ceramic decreased after HF etching. The reason for this could be explained by the amounts of the glass phase involving lithium disilicate crystals. Several studies examined different ceramics, and the weakening effect of HF etching was confirmed (25, 28, 29). So, by increasing the etching time and HF concentration, disruption of surface increases and it may lead to cohesive or mixed failure more than adhesive. As it has been seen in this study, in e.max groups, in 5% concentration, adhesive failure was more than others, and in 10% concentration, mixed failure was dominant. By increasing HF concentration, the failures turn from adhesive to mixed failure. Adhesive failure means that the strength of the adherend is more than the adhesive, cohesive failure means that the adherend's strength is lower than the adhesive and mixed failure shows that the strength of the adherend and the adhesive is equal. So, by increasing etching time and HF concentration, mixed and cohesive failures might increase comparing to adhesive failure. Our study limitation was the use of a sectioning machine for making of beams, because sectioning stress could decrease real μ SBS.

5. Conclusions

The results showed that in both e.max and Vita Mark II groups, μ SBS were not significantly different when different etching times and HF acid concentrations were used, but the highest μ SBS was shown in Ea60 and Mb20 groups. The μ SBS of e.max was significantly higher than Vita Mark II. For clinical use, we suggest that in both e.max and Vita Mark II ceramic restorations, 20 s etching with 5% HF is sufficient to have acceptable bond strength without weakening of ceramics. Further studies could be performed on the effect of HF surface treatments on the bond strength of other CAD/CAM ceramics such as Vita Suprinity and Vita Enamic to resin cement.

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Conflict of Interest:

There is no conflict of interest to be declared.

Authors' contributions:

All authors contributed to this project and article equally. All authors read and approved the final manuscript.

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