EFFECTS OF ETCHING DURATION ON THE SURFACE ROUGHNESS, SURFACE LOSS, FLEXURAL STRENGTH, AND SHEAR BOND STRENGTH TO A RESIN CEMENT OF E.MAX CAD GLASS CERAMIC

by

Hanan Al-Johani

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_____
Jeffrey A. Platt

_____
Norman B. Cook

_____
Tien-Min Gabriel Chu
Chair of the Research
Committee

_____
Marco C. Bottino
Program Director

_____
Date

DEDICATION

I dedicate this thesis to my beloved parents, Dr. Aouda Al-Johani and Dr.Hessa Almusaad; my supportive husband, Mohanad, my sweet son, Saleh; and my siblings, Wafa, Abdulaziz, Mohammad, Hala and Abeer. You were the wind beneath my wings that made all of this possible.

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INTRODUCTION

Following the introduction of the first all-ceramic crown,[1] there has been a growing interest in the use of all-ceramic restorations as an alternative to traditional porcelain-fused-to-metal restorations due to their improved esthetic appearance, biocompatibility, and acceptable strength. Developments in ceramic material science have led to improvements in physical and optical properties and subsequently a considerable increase in the clinical use of all-ceramic restorations.[2-3]

Long-term retention of ceramic restorations can be dependent on the bond strength of the luting resin to both the tooth and ceramic substrates. In order to achieve successful bonding, the surface of the ceramic substrate must be modified to increase the surface roughness by hydrofluoric (HF) acid etching, mechanically by means of diamond burs, air abrasion with aluminum oxide or silica, or by a combination of the previous methods.[43-46] For ceramic surface treatment with HF acid, the acid reacts with the glass matrix and selectively removes the matrix exposing the crystalline structure. As a result, the surface of the ceramic becomes rough, which allows for micromechanical retention on the ceramic surface and increased surface energy prior to combining with the silane solution.[47-49] However, it has been reported that HF acid may also have a weakening effect on the ceramic surfaces[50] due to the presence of these surface flaws. Microcracks may initiate and propagate, weakening the dental ceramic under tensile strain.[51,52] Therefore, it is essential to quantify the required etching duration of HF acid to minimize the possible deleterious effects on ceramic strength while maximizing the bond strength to tooth structure.

Regarding the influence of etching duration, many studies have evaluated the effect of HF acid etching on lithium disilicate glass ceramics other than IPS e.max CAD.[31,33,34] Though HF acid etching can increase the surface roughness, which provides the necessary mechanical interlocking of ceramic to the luting cement, prolonged HF acid etching has shown to have a weakening effect on the evaluated lithium disilicate glass-ceramics.[31,33]

Although many studies have been conducted to compare the effect of different surface treatments on the properties of IPS e.max CAD,[30,46,55,56,28] little has been done in regards to evaluating the effects of different durations of HF acid etching protocol on the surface loss, roughness, and subsequent bonding to IPS e.max CAD per se.[29,30]

In this study, the effects of different etching durations on both the morphological and mechanical properties of IPS e.max CAD lithium disilicate glass-ceramic were evaluated. Qualitative analysis of the etched surfaces was done to evaluate surface morphology. Quantitative analysis was done to assess the relation between etching duration and surface roughness, surface loss, flexural strength, and shear bond strength to a specific resin cement of IPS e.max CAD.

The objective of this study was to evaluate the effect of different hydrofluoric acid etching durations on the surface roughness, surface loss, flexural strength, and shear bond strength to the resin cement used in the study of IPS e.max CAD lithium disilicate glass ceramic.

HYPOTHESES

The null hypotheses of this study were: 1) The difference in HF acid etching durations does not have a significant effect on the surface roughness and surface loss of

IPS e.max CAD; 2) The difference in HF acid etching durations does not have a significant effect on the shear bond strength of IPS e.max CAD to the resin cement used in the study; 3) The difference in HF acid etching durations does not have a significant effect on the flexural strength of IPS e.max CAD.

The alternative hypotheses were: 1) The difference in HF acid etching duration will significantly increase the surface roughness and surface loss of IPS e.max CAD, and 2) The difference in HF acid etching durations does have a significant effect on the shear bond strength of IPS e.max CAD to the resin cement used in the study, and 3) The difference in HF acid etching durations does have a significant effect on the flexural strength of IPS e.max CAD.

REVIEW OF LITERATURE

HISTORY

The term ceramic is derived from the Greek word “keramikos” meaning “pottery” or “potter.” In the early 1700s many European countries were importing porcelain from China and Japan[13] and since then it has been implemented in various aspects of our daily lives, in the form of glass, porcelain, pottery, and bricks. In dentistry, the first porcelain tooth was originated by de Chemant in 1789 and Fonzi invented a "terrometallic" porcelain tooth that was fixed in place by a platinum pin in 1808.[9] The first all-ceramic crown was later introduced by Land in 1903.[1] These feldspathic crowns had excellent esthetics but very low strength limiting their use to crowns with a cast metal core or metal-foil coping. In 1965 McLean and Hughes initiated the concept of adding aluminum oxide particles to the original feldspathic composition to enhance its mechanical and physical properties. However, the main drawback of those crowns was their opaque appearance and large sintering shrinkage. In 1984 Adair and Grossman established the principle of controlled crystallization improving the all-ceramic systems immensely.[8-10]

DEFINITION

“Dental ceramics” are referred to as nonmetallic inorganic structures, composed mainly of oxygen with one or more metallic or semi-metallic elements such as sodium, potassium, calcium, aluminum, zirconium, magnesium, lithium, and phosphorus.[9] The term “porcelain” refers to ceramics with a specific composition of kaolin (hydrated aluminosilicate), quartz (silica) and feldspars (potassium and sodium aluminosilicates)

fired at high temperatures.[11] Ceramics have also been described as composites considering their composition of two or more separate phases.[12]

CLASSIFICATION

Some ceramics contain a combination of both a glassy phase and a crystalline phase and can be classified according to their microstructure, depending on their glass-to-crystalline ratio into four groups: (1) predominantly glass-based, (2) glass-based with crystalline fillers, (3) crystalline-based systems with glass fillers, and (4) polycrystalline. Ceramics can also be classified according to their processing technique into four groups: (1) powder condensation, (2) slip casting (3) heat pressing, and (4) CAD-CAM machining.[4-6] (Table I)

More recently dental ceramics have been classified depending on whether a glass-matrix phase is present or absent, or on whether the material contains an organic matrix that is highly filled with ceramic particles from three groups (1) glass-matrix ceramics (nonmetallic inorganic ceramic materials that contain a glass phase); (2) polycrystalline ceramics (nonmetallic inorganic ceramic materials that do not contain any glass phase), and (3) resin-matrix ceramics (polymer matrices containing mainly inorganic refractory compounds).[7]

CLASSIFICATION BASED ON MICROSTRUCTURE [4,5,14]

Predominantly Glass-based

Glass is referred to as non-crystalline-containing material with atoms arranged in an irregular amorphous pattern. Glass-based dental ceramic systems are composed of feldspar minerals containing mainly silica (SiO_2) and alumina (Al_2O_3) in addition to

sodium and potassium. These glass-based systems are best known for their high translucency mimicking the optical properties of dental enamel. However, feldspathic porcelains have low flexural strength values (ranging from 60 MPa to 70 MPa) limiting their use as veneering materials for metal or ceramic frameworks.

Glass-based with Crystalline Fillers

This class of glass-ceramics has a wide range of glass-to-crystalline ratios as well as different types of crystals; therefore, there are three distinct subcategories containing the same glass composition as the previously described predominantly glass-based ceramics.

- Low-to-moderate leucite glass ceramic. Leucite fillers were first added (17 vol% to 25 vol%) to raise the coefficient of thermal expansion to resemble that of the underlying metal frameworks. Leucite has also been shown to inhibit crack propagation and increase the glass-ceramic's flexural strength. Commercial ceramic systems containing low concentrations of leucite fillers are available as powder ceramics and can be used as veneering material for metal and ceramic frameworks, in addition to porcelain veneers, inlays, and onlays.
- High-leucite glass ceramic. These ceramics contain up to 55-percent leucite crystals, with a surrounding matrix of amorphous glass. The ceramics undergo a special heat treatment that nucleates the leucite crystals and increases their size, generating compressive stresses around the crystals due to the difference in coefficients of thermal expansion between the leucite crystals and the glassy matrix. As a result, crack propagation is hindered and the flexural strength increased significantly (160 MPa in IPS Empress). High-leucite containing glass ceramics are available in both machinable and

pressable forms and can be used to fabricate inlays, onlays, anterior veneers, and anterior crowns.

- Lithium-disilicate glass ceramic. These glass ceramics have lithium disilicate crystals (70 vol%) incorporated in the glassy matrix. Due to the relatively low refractive index of the lithium disilicate crystals, these ceramics exhibit high translucency despite their large crystalline content. In comparison with the leucite-containing glass ceramics, the lithium disilicate glass ceramics have higher flexural strength values ranging between 360 MPa to 400 MPa, allowing these ceramics to be used as inlays, onlays, veneers, anterior or posterior crowns, implant crowns, and as three-unit anterior bridges extending to the second premolar.

Crystalline-based Systems with Glass Fillers

Also called "interpenetrating phase ceramics," first a porous matrix is created, and then it is penetrated with lanthanum aluminosilicate glass to generate a dense interpenetrating ceramic material. An example for these systems is In-Ceram, initially developed as an alternative to porcelain-fused-to-metal restorations due to their high flexural strengths ranging from 350 MPa (In-Ceram Spinel) to 450 MPa (In-Ceram Alumina) to 650 MPa (In-Ceram Zirconia). They are available in slipcast or machinable forms and have a wide variety of uses including veneers, inlays, onlays, anterior and posterior crowns and bridges.

Polycrystalline Systems

This class of ceramics is formed by sintering the crystals (95 vol% to 99 vol%) together without a surrounding glassy matrix, resulting in dense glass-free polycrystalline

ceramic systems. The frameworks of these ceramics are usually either solid-sintered aluminous-oxide or zirconia-oxide frameworks. They exhibit significantly higher flexural strength in comparison with the previously mentioned ceramic systems ranging from 600 MPa (Procera AllCeram Alumina) up to 1200 MPa (LAVA Zirconia) allowing them to be used as multiple unit anterior and posterior bridges. However, the high crystalline content contributed to less than optimal esthetics (high opacity and low translucency), resulting in restorations that may require veneering when used in esthetic regions.

CLASSIFICATION BASED ON FABRICATION METHOD [5,6,14,24]

Powder Condensation

This is the traditional method commonly used to fabricate feldspathic ceramic restorations. It involves manually mixing porcelain powder with de-ionized water to produce slurry, which is then applied with a brush layer-by-layer, vibrated, and condensed to remove any air or water. The porcelain is then fired in a vacuum to further remove any remaining air and enhance the density of the restoration. The end result is feldspathic porcelain with a high glassy phase and a low crystalline content leading to high translucency excellent for veneers. However, the main drawback of this method is the inherent residual porosity in the fired porcelain.

Slip Casting

The slip-casting technique (also known as glass infiltration) uses ceramic slips and glasses in a two-stage heat treatment to form the final ceramic restoration. The slip is a liquid suspension of ceramic particles; it is applied over a gypsum die that absorbs the water from the slip through capillary action, resulting in a framework of ceramic

particles. After the first heat treatment, the ceramic particles are sintered and a porous microstructure is created. During the second heating treatment, molten glass penetrates into the porous framework, surrounding the ceramic microstructure, forming the core of the dental prosthesis. Subsequently, the core is veneered with feldspathic porcelain. Ceramics fabricated by slip casting exhibited higher fracture resistance than those fabricated by powder condensation due to the strengthening crystalline particles that form a continuous network throughout the framework. The main drawback of this fabrication method is an excessive number of complicated steps, which may result in internal defects due to incomplete glass infiltration.

Heat Pressing

The heat-pressing method is a lost-wax method used to fabricate molds for pressable dental ceramics, which are available as prefabricated ingots. The first step in this method is designing a wax model, followed by creating a mold out of gypsum materials. The ingots are then heated to a temperature at which they become a highly viscous liquid, and then are slowly pressed into the lost wax mold cavity. Restorations can be fabricated up to their full contour or as frameworks later veneered with feldspathic porcelain. The advantage of this method is the relative similarity to the lost wax method used with metal castings, thus fewer technical problems are faced in the dental laboratory.

CAD-CAM Machining

This method is the most recent ceramic fabrication method. It uses a scanning device, design software, and a milling machine to fabricate ceramic restorations from

prefabricated partially sintered ceramic blocks. The ceramic blocks exhibit moderate strength in their partially sintered state allowing them to be easily milled by the CAD/CAM system. Subsequently, the blocks are exposed to heat treatment upon which they become fully sintered and acquire their maximum strength and esthetic properties.[25] CAD/CAM systems can also be used by dentists to take digital impressions using the scanning device and to fabricate the ceramic restorations in the dental clinic in a single appointment. Also, conventional impressions can be taken by the dentist and sent to the laboratory, where they are fabricated by the CAD/CAM system and sent back to the dentist for a subsequent visit.[26] The main advantages of this fabrication method are the reduced time and cost required to fabricate the ceramic restorations in comparison with the conventional laboratory-based techniques.

HISTORICAL PERSPECTIVE OF LITHIUM DISILICATE GLASS CERAMICS

Lithium disilicate glass ceramics were first discovered in 1959 by Stookey from precipitated of $Li_2Si_2O_5$ in glass with clusters of Ag as nucleating agents for crystallization, creating a binary lithium disicilate glass-ceramic system.[15,16] Later in 1998, lithium disilicate glass ceramics were introduced by Ivoclar Vivadent as IPS Empress II, a pressable glass-ceramic with a multi-component system, containing 30% by volume of glass matrix and 70% by volume of lithium disilicate crystals. In 2001 the same manufacturer introduced a lithium disilicate glass ceramic with superior processing mechanisms, IPS e.max Press, a castable lithium disilicate glass ceramic with improved mechanical and optical properties. The latest generation introduced in 2005 by the manufacturer is IPS e.max CAD, a machinable lithium disilicate glass ceramic developed

to accommodate the recent advances in CAD/CAM technology.[17,18]

PROCESSING OF LITHIUM DISILICATE GLASS CERAMICS

Pressable lithium disilicate (IPS e.max Press) is produced by a bulk casting production method; glass powders are heated simultaneously until the proper viscosity of the glass melt is achieved. The melt is then poured into a mold and left to cool slowly. This cooling process minimizes the voids and internal defects improving the mechanical and optical properties of the glass ingots. IPS e.max Press blocks are then processed using the lost-wax hot pressing technique; the pressable ingots are heated to a temperature at which they become a highly viscous liquid, and then are slowly pressed into the lost wax mold.[19-21]

Machinable lithium disilicate (IPS e.max CAD) is initially produced by a pressure casting procedure to fabricate the transparent lithium disilicate glass ingots, which then undergo a partial crystallization process. The partial crystallization ensures easy processing and machining of the blocks with CAD/CAM systems. The partially crystallized blocks have an acquired blue tint. Subsequent to tempering of the blocks, the crystallization is complete, and IPS e.max CAD blocks obtain their desired mechanical and optical properties.[22-23]

MICROSTRUCTURE OF LITHIUM DISILICATE GLASS CERAMICS

The microstructure of IPS e.max CAD consists of approximately 65% volume fraction of lithium disilicate crystals ($Li_2Si_2O_5$), 34% volume fraction of residual glass matrix, and 1% volume fraction of porosity after heat treatments. The chemically composition of IPS e.max CAD is mainly quartz (SiO_2) and lithium dioxide (Li_2O) and in

lesser amounts phosphor oxide (P_2O_5), potassium oxide (K_2O), and other oxides as coloring components. High-temperature x-ray diffraction studies revealed that IPS e.max CAD presintered ingots are fabricated of lithium metasilicate crystals (Li_2SiO_3) and crystobalite. Upon a second stage of crystallization as the restorations are tempered, lithium disilicate crystals ($Li_2Si_2O_5$) are formed.[16-19]

EFFECT OF HYDROFLUORIC ACID ETCHING ON THE MICROSTRUCTURE OF LITHIUM DISILICATE GLASS CERAMIC

Lithium disilicate glass ceramics are acid susceptible materials; subsequent to surface treatment with HF acid, morphological changes occur in the glass ceramic surface allowing for micromechanical retention necessary with the luting agents used for cementation.[34] Prochnow et al.[27] reported qualitative changes in the surface topography of lithium disilicate glass ceramics when exposed to different HF acid etching regimens. SEM and AFM images revealed that untreated ceramic surfaces (IPS e.max CAD, Ivoclar Vivadent) were smooth and homogeneous in comparison to etched surfaces, which became increasingly porous and irregular as the HF acid concentration increased. Authors also stated that there were no significant differences in the mean roughness values among the different etching groups. Kalavacharla et al.[28] evaluated the effect of different HF acid etching protocols with and without silane application on the bond strength between lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) and a resin composite. The SEM images showed that lithium disilicate glass ceramic specimens etched with 5-percent HF acid for 20 seconds exhibited elongated crystals after disintegration of the silica matrix while the specimens etched with 9.5-percent HF acid for 60 seconds exhibited a more distinct etching pattern. Zogheib et al.[29] examined the

effect of different HF acid etching times on the surface roughness and flexural strength of a lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent). Authors reported that the untreated surfaces displayed homogenous configurations in comparison with the etched groups, which displayed irregular and porous etching patterns, with voids and channels between the lithium disilicate crystals increasing gradually as the etching time increased, especially in specimens etched for 90 s and 180 s. Similar findings were reported by Ramakrishnaiah et al.,[35] who attributed this finding to the dissolution of the glassy phase at a faster rate than the crystal phase. Menees et al.[30] studied the influence of alumina particle abrasion and HF acid etching on the flexural strength lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) when using different abrasion pressures and different etching protocols. The authors stated from the SEM analysis that specimens etched for 20 s for both HF acid concentrations (5% and 9.5%) was sufficient to remove an adequate portion of the glass matrix; however, more extensive glass removal was noticed at the 120-second etching groups of both concentrations. Hooshmand et al.[31] assessed the effect of HF acid etching (9% for 2 minutes) on the biaxial flexural strength of two hot-pressed glass ceramics (IPS Empress and IPS Empress 2, Ivoclar Vivadent). Authors noted when comparing SEM images of untreated and treated ceramic surfaces that the treated surfaces became porous and irregular as a result of the dissolution of the glass phase and elongated lithium disilicate crystals were seen protruding from the glassy matrix in IPS Empress 2 (Ivoclar Vivadent).

EFFECT OF SURFACE CONDITIONING ON MECHANICAL PROPERTIES OF LITHIUM DISILICATE GLASS CERAMIC

Xiaoping et al.[33] examined the effects of different 9.5-percent HF acid times on the flexural strength of a pressable lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent). Ceramic specimens were etched for 0 s, 20 s, 40 s, 60 s, and 120 s and the mean flexural strength values were 384 ± 33; 347 ± 43; 330 ± 53; 327 ± 67, and 317 ± 41 MPa, respectively. Authors concluded that increasing the HF acid etching times significantly reduced the mean flexural strength; however, the mean flexural strength values of all etched specimens increased significantly after the application of dual-curing resin cement. Similarly, Zogheib et al.[29] reported that increasing the HF acid etching durations significantly decreased the flexural strength of lithium disilicate glass ceramics; when etching with 4.9-percent HF acid for 0 s, 20 s, 60 s, 90 s, and 180 s, the mean flexural strength values were: 417 ± 55; 367± 68; 363 ± 84; 329 ± 70; and 314 ± 62, respectively. Menees et al.[30] compared the flexural strength of a machinable lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) after alumina abrasion at different pressures and HF acid etching at different concentrations and times. Authors found that the flexural strength in specimens etched with HF acid was not significantly different from that found in the control group regardless of the etching time or concentration. However, the lithium disilicate glass ceramic specimens abraded with alumina displayed significantly lower flexural strength values due to highly concentrated areas of mechanical stress and microfractures created in the surface microstructure. Stawarczyk et al.,[36] examined the effect of different HF acid etching times on the fracture load of three different machinable glass-ceramics (IPS Empress CAD, KLEMA CAD/CAM and IPS e.max CAD). Crowns were fabricated, divided into 6 groups

according to their HF acid etching times (0 s, 30 s, 60 s, 90 s, 120 s, and 150 s) and adhesively cemented on the metal abutment. After 24 hours, crowns were loaded until fracture and the fracture load was documented. The study showed that unetched IPS Empress crowns exhibited lower fracture loads than etched crowns for 150 s. Regarding KLEMA CAD/CAM crowns, both unetched and etched for 150-s crowns exhibited lower fracture loads than those etched for 90 s, while IPS e.max CAD (Ivoclar Vivadent) crowns displayed no effect of etching time on the fracture loads.

MATERIALS AND METHODS

PREPARATION OF LITHIUM DISILICATE SPECIMENS

As shown in Table II, specimens were divided into 4 groups (n = 42/group) according to the etching duration, and then further divided into 3 subgroups according to the properties tested. Following the ISO Specification 6872,[40] IPS e.max CAD (Ivoclar Vivadent) blocks were sectioned using a low speed diamond wheel saw (Isomet 1000, Buehler, Lake Forest, IL) (Figure 1). For subgroup 1 and 2 specimens (n = 16/subgroup), ceramic blocks were sectioned into 5 x 5 x 3 mm square shaped bars. For subgroup 3 specimens (n = 10/subgroup), ceramic blocks were sectioned into 1.3 x 4 x 18 mm rectangular bars. All surfaces of the bars were smoothed and polished under running water using 400-, 600-, 800- and 1000- grit silicon carbide papers (EXAKT Technologies, Oklahoma City, OK) at 300 rpm on a polishing machine (EXAKT 400 CS, EXAKT Technologies, Oklahoma City, OK). According to the manufacturer's recommended two-stage heating schedule (Table III), the specimens were fired in a vacuum pump furnace (Ivoclar Vivadent, Programat CS/CS2) (Figure 2). Subsequently, the specimens were rinsed with distilled water and stored dry until the etching procedures took place.

ETCHING PROTOCOL

According to the etching duration, IPS e.max CAD (Ivoclar Vivadent) specimens were randomly divided into 4 groups; Group A was not etched (control). Group B, C and D were etched with 5-percent HF acid gel (IPS Ceramic Etching gel, Ivoclar Vivadent)

for 20 s, 60 s and 90 s respectively. In subgroup 1 specimens, for adequate interpretation of the etched surfaces during subsequent analysis, etching was carried out using a taping technique, in which both sides of the square shaped specimen (1.5 mm from each side) were taped with Scotch tape and the HF acid was applied on the exposed center of the bar (2mm) (Figure 3). Subsequent to the allotted etching time in each group, the IPS Ceramic Etching Gel was rinsed from the ceramic surface under running water. Then, the tape was removed and the ceramic bonding surface was thoroughly dried.[42] For subgroup 2 and subgroup 3 specimens, the entire surface of a single side of the bars was etched following the same etching protocol previously described, according to the allotted etching time in each group (Figure 4 and Figure 5).

NON-CONTACT PROFILOMETRY

Surface roughness values (R_a and R_q) were determined using non-contact 3D optical profilometry (Proscan 2000, Scantron, Taunton England) (Figure 6) of eight specimens from subgroup 1 of each group (A1, B1, C1 and D1). R_a is the average roughness value of a surface and R_q is the square root of the mean of all the height deviations, which magnifies odd spikes in an otherwise smooth surface. Both R_a and R_q exhibit similar interpretation, the lower the roughness values, the smoother the surface. Each specimen was scanned in four different areas to calculate the average R_a and R_q values in each group.[29,37,57]

SURFACE LOSS

Subsequent to roughness characterization, the surface loss was calculated by measuring height loss using non-contact optical profilometry (Proscan 2000) (Figure 6).

[36,38] The S5/03 sensor was used for scanning at a 10-µm step size and 300 steps in the x direction, and at a 100-µm step size and 10 steps in the y direction. For height loss measurement, eight areas along the etched plane were measured by comparing it to the adjacent un-etched surface of the ceramic bar.[57]

SCANNING ELECTRON MICROSCOPY (SEM)

Subsequent to roughness and surface loss characterization, microstructural analysis of the topography of both the etched and non-etched surfaces of three random specimens in each group (A1, B1, C1 and D1) were examined under a Field Emission-SEM (JSM 7800 F JEOL, Tokyo, Japan). The surfaces were sputter-coated with gold (Denton Vacuum Desk V, Denton Vacuum) prior to scanning. The microstructure of the lithium disilicate crystals was examined.

SHEAR BOND STRENGTH (SBS)

Sixty four specimens of subgroup 2 of each group (A2, B2, C2 and D2) were individually embedded in Teflon molds using acrylic resin.[39,40] Etched surfaces of the specimens were silanated with a thin coat of Monobond Plus (Ivoclar Vivadent), allowed to react for 60 s and blow dried. Subsequently, a resin cement (Multilink Automix, Ivoclar Vivadent) button (diameter = 2.38 mm, height: 2 mm) was bonded to the glass ceramic samples according to the manufacturer's instructions (Figure 7). The resin cement was then polymerized for 10 s using an LED light curing system (Bluephase Style, Ivoclar Vivadent, irradiance = 1008 mW/cm^2, radiant exposure = 10.3 J/cm^2). Half the specimens of each group were aged by thermocycling (TH) (5000 cycles, 5°C to 55°C, dwell time: 30 s, transfer time of 10 seconds). The other half of the specimens were

stored in distilled water for 24 h (24H). After the assigned storage time for each group, the specimens were individually placed in a stainless steel jig for SBS testing (Ultradent) and loaded by a custom notched fixture (Ultradent) in compression using a Universal mechanical testing machine (Sintech ReNew 1123, MTS, Shakopee, MN) (Figure 8) at a crosshead speed of 1 mm/min (Figure 9). The SBS was calculated using the following formula:

SBS (MPa) = Load (N) / Area (mm^2)

FLEXURAL STRENGTH

Specimens of subgroup 3 (A3, B3, C3 and D3) were placed on the 3-point flexure test fixture as recommended by ISO Specification 6872 with the etched surface facing downwards.[41] The 3-point flexure test fixture consists of two cylinders with a radius of 0.8 mm (span distance = 15 mm) and a loading cylindrical head with a radius of 0.8 mm. Specimens were loaded to failure (1.0 mm/min cross-head speed) using a universal testing machine (Sintech ReNew 1123, MTS, Shakopee, MN) (Figure 8 and Figure 10). Upon load fracture, flexural strengths (F) were calculated from the following equation:

$$F = 3PL/2bh^2$$

P is the load at fracture; L is the test span; b is the thickness of the sample, and h is the height of the sample.

STATISTICAL ANALYSIS

Summary statistics (mean, standard deviation, standard error, range) were calculated for the different tested properties for each experimental group. Data for surface

roughness (R_a and R_q), surface loss and flexural strength test were analyzed using one-way analysis of variance (ANOVA) to identify the significant effects of different HF acid etching durations. Data for shear bond strength test were analyzed using two-way ANOVA to test the effects of etching duration, storage for 24 hours/thermocycling, and their interaction. All pair-wise comparisons from ANOVA were made using Fisher's Protected Least Significant Differences to control the overall significance level at 5 percent.

RESULTS

SURFACE ROUGHNESS

Means, respective standard deviations (± SD), standard errors (± SE) of surface roughness values (R_a and R_q) are shown in Table IV and Figure 11. When comparing surface roughness in etched groups, highest roughness values were found in the 90-s group (R_a= 1.78 μm, R_q=2.41 μm), followed by the 20-s group (R_a= 1.34 μm, R_q= 1.85 μm) and least roughness was found in the 60-s group (R_a= 1.15 μm, R_q= 1.47 μm). One-way ANOVA followed by a pair-wise Fisher's Protected Least Significant Differences test revealed that among the control and etched groups, HF acid etching durations did not have a significant effect on the surface roughness R_a or R_q values ($p = 0.3408$; $p = 0.3245$), respectively.

SURFACE LOSS

Means, respective standard deviations (± SD), and standard errors (± SE) of surface loss values are shown in Table V and Figure 12. When comparing surface loss in etched groups, highest surface loss was found in the 90-s group (-3.49 μm), followed by the 60-s group (-1.31 μm). The 20-s group showed a height gain (0.84 μm). One-way ANOVA followed by a pair-wise Fisher's Protected Least Significant Differences test revealed that among the control and etched groups, HF acid etching durations had a significant effect on the surface loss ($p = 0.0006$). Surface loss for 20-s etching duration was significantly lower than 90-s ($p = 0.0035$). Surface loss for 60-s and 90-s etching duration was significantly greater than the control group ($p = 0.0060$ and $p < 0.0001$).

Non-contact profilometry scans of subgroup 1 specimens are presented in Figure 13 to Figure 16.

SCANNING ELECTRON MICROSCOPY

SEM micrographs, at different magnifications, of the non-etched and etched ceramic surfaces are presented in Figure 17 to Figure 20. With increasing etching times, the etched ceramic surfaces became increasingly porous and irregular as the glass matrix was selectively removed, leaving the lithium disilicate crystals protruding. Specimens etched for 60 s and 90 s displayed greater voids and porosities in comparison with specimens etched for 20 s.

SHEAR BOND STRENGTH

Means, respective standard deviations (± SD), standard errors (± SE) of shear bond strength (SBS) values are shown in Table VI and Figure 21. In samples stored in distilled water for 24 hours, highest shear bond strength values were found in the 20-s group (7.94 MPa), followed by 60-s group (7.61 MPa), the 90-s group (6.65 MPa) and least SBS was found in the control group (2.88 MPa). In samples thermocycled, an opposite pattern was observed in the different etching groups, as the highest SBS values were found in 90-s group (4.01 MPa), followed by the 60-s group (3.31 MPa), the 20-s group (2.42 MPa) and least SBS values were found in the control group (0.1 MPa). Among the control specimens that were thermocycled, only one specimen survived the thermocycling process and did not lose the resin button. The two-way ANOVA followed by a pair-wise Fisher's Protected Least Significant Differences test revealed that among the control and etching groups, the SBS values were not significantly different between

different HF acid etching durations ($p = 0.4650$). Additionally, SBS values after 24-h storage were significantly higher than thermocycling ($p = 0.0166$) among different etching durations. The two-way interaction between group and storage method was not significant ($p = 0.8412$).

FLEXURAL STRENGTH

Means, respective standard deviations (± SD), standard errors (± SE) of flexural strength values are shown in Table VII and Figure 22. Highest flexural strength values were found in the control group (291.48 MPa) followed by the 60-s etching group (267.11 MPa), the 90-s group (246.69 MPa) and least flexural strength was found in the 20-s group (239.06 MPa). One-way ANOVA followed by a pair-wise Fisher's Protected Least Significant Differences test revealed that among the control and etched groups, HF acid etching durations did not have a significant effect on the flexural strength values ($p = 0.1260$).

TABLES AND FIGURES

TABLE I

Classification of dental ceramics

	Composition	Manufacturing method	Commercial examples
Glass-based	Feldspathic (SiO_2-Al_2O_3-Na_2O-K_2O)	Powder condensation	Vita VMK Vitadur alpha Vita VM7 Ceramco3
		CAD/CAM	VITABLOCS Mark II VITABLOCS TriLuxe
Glass- based with crystalline fillers	Leucite (SiO_2-Al_2O_3-K_2O)	Powder condensation	Vita VM9, Vita VM13
		Heat pressing	IPS Empress Fortress Pressable Finesse All-Ceramic
		CAD/CAM	IPS ProCAD IPS EmpressCAD
	Lithium disilicate (SiO_2-Li_2O)	Heat pressing	IPS Empress 2 IPS e.max Press
		CAD/CAM	IPS e.max CAD
	Fluorapatite	Powder condensation	IPS e.max Ceram
		Heat pressing	IPS e.max ZirPress
Crystalline-based with glass fillers	Aluminum oxide	Slip casting	In Ceram Alumina In Ceram Spinell
		CAD/CAM	In Ceram Alumina In Ceram Spinell

TABLE II

Distribution of experimental groups

Group (n = 42)	Etching time	Subgroup	Tested property
Group A Control	None	A1	SEM Surface loss Non-contact surface profilometry
		A2	Shear bond strength
		A3	Flexural strength
Group B	20 s	B1	SEM Surface loss Non-contact surface profilometry
		B2	Shear bond strength
		B3	Flexural strength
Group C	60 s	C1	SEM Surface loss Non-contact surface profilometry
		C2	Shear bond strength
		C3	Flexural strength
Group D	90 s	D1	SEM Surface loss Non-contact surface profilometry
		D2	Shear bond strength
		D3	Flexural strength

TABLE III

Recommended two-stage heating schedule

	Stage 1	Stage 2
B (◦C)	403	
S (min)	0.3	
t (◦C/min)	90	30
T (◦C)	820	840
H (min)	0:10	7
V1 (◦C)	550	820
V2 (◦C)	820	840
Heating time (min)	12.77	

B (◦C) = Furnace stand-by-temperature, S (min) = furnace door closing time, t (◦C/min) = heating or ramp rate, T (◦C) = holding temperature, H (min) = holding time, V1 (◦C) = vacuum-on temperature, V2 (◦C) = vacuum-off temperature.

TABLE IV

Means (μm) ± SD, ±SE and range of surface roughness of experimental groups

Etching group	Roughness parameter	Mean	Std Dev	Std Error	Minimum	Maximum
20s	R_a	1.3414	0.7878	0.2785	0.6850	2.8890
	R_q	1.8468	1.2305	0.4350	0.9010	4.4860
60s	R_a	1.1476	0.6013	0.2126	0.4760	2.4840
	R_q	1.4705	0.4983	0.1762	0.8550	2.4690
90s	R_a	1.7826	0.8884	0.3141	0.8580	3.6440
	R_q	2.4861	1.5096	0.5337	1.1760	5.9250
Control	R_a	1.8778	1.2583	0.4449	0.3690	4.0620
	R_q	2.4046	1.4393	0.5089	0.6270	4.8480

TABLE V

Means (μm) ± SD, ±SE and range of surface loss of experimental groups

Etching group	Mean	Std Dev	St Error	Minimum	Maximum
20s	0.8429[a,b]	2.7667	0.9782	-3.0460	5.3700
60s	-1.3093 [b,c]	1.9899	0.7035	-5.6230	0.9230
90s	-3.4914[c]	2.9933	1.0583	-9.8710	-0.0770
Control	2.7300[a]	2.9907	1.0574	-0.1210	8.4650

*Means sharing the same superscript are not significantly different from each other.

TABLE VI

Means (MPa) ± SD, ±SE and range of shear bond strength of experimental groups

Etching group	Storage method	Mean	Std Dev	Std Error	Minimum	Maximum
20s	24h	7.9375[a]	6.8128	2.4087	0.4000	21.1000
	TH	2.4200[b]	1.0663	0.4769	1.0000	3.7000
60s	24h	7.6143[a]	7.0860	2.6783	1.0000	20.6000
	TH	3.3125[b]	1.3809	0.4882	1.6000	5.4000
90s	24h	6.6500[a]	2.9549	1.0447	2.8000	10.8000
	TH	4.0143[b]	1.4206	0.5369	1.3000	5.4000
Control	24h	2.8750[a]	3.9978	1.4134	0.0000	12.2000
	TH	0.1000[b]	.	.	0.1000	0.1000

*Means sharing the same superscript are not significantly different from each other.

TABLE VII

Means (MPa) ± SD, ±SE and range of flexural strength of experimental groups

Etching group	Mean	Std Dev	Std error	Minimum	Maximum
20s	239.0600	53.8241	17.0207	173.2000	325.1000
60s	267.1100	29.1624	9.2220	213.1000	309.6000
90s	246.6900	53.3445	16.8690	165.5000	321.4000
Control	291.4800	64.9909	20.5519	115.9000	334.8000

FIGURE 1. Isomet 1000, a cutting machine.

FIGURE 2. Vacuum pump furnace (Programat CS/CS2).

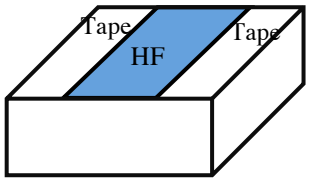


Figure 3

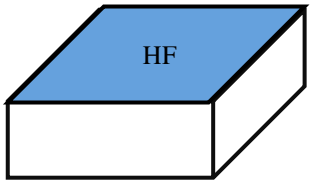


Figure 4

Figure 5

FIGURE 3. Schematic representation of etching method for subgroup 1 specimens.

FIGURE 4. Schematic representation of etching method of subgroup 2 specimens.

FIGURE 5. Schematic representation of etching method for subgroup 3 specimens.

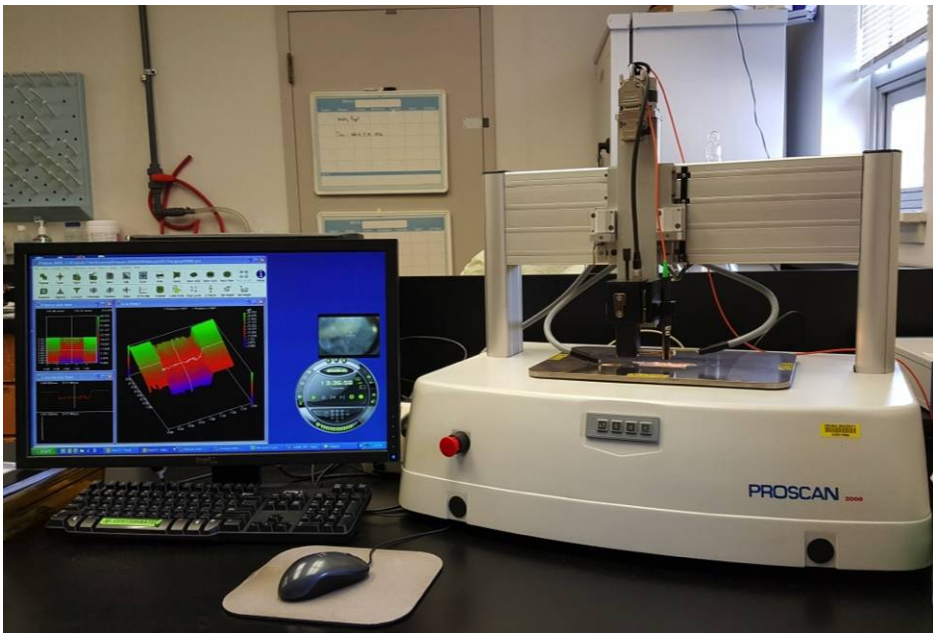


FIGURE 6. Non-contact optical profilometer (Proscan 2000).

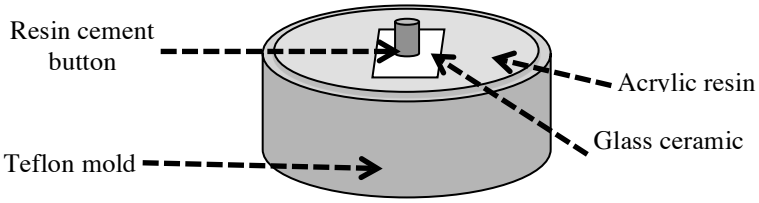


FIGURE 7. Schematic representation of mounting specimens for SBS testing.

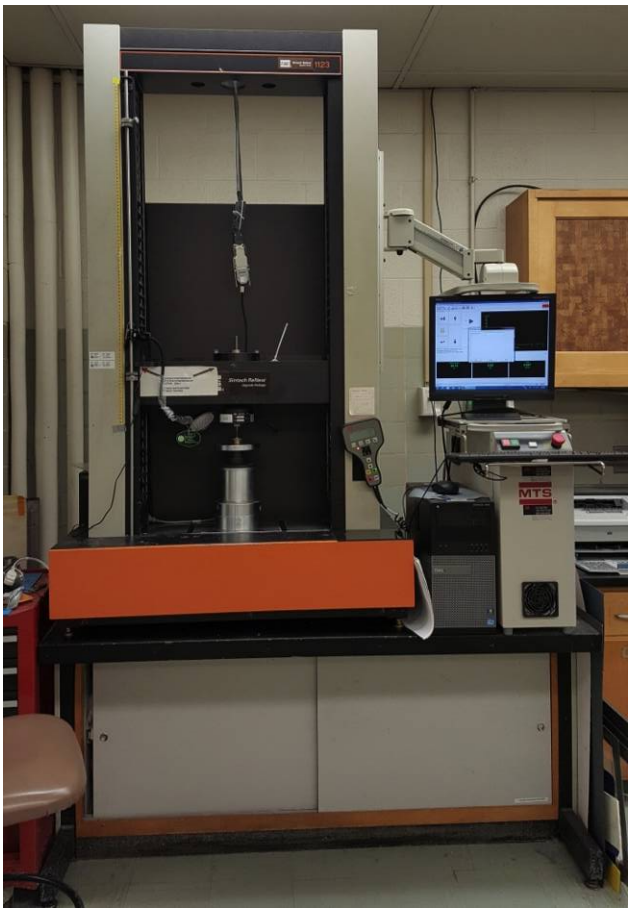


FIGURE 8. Universal testing machine (MTS).

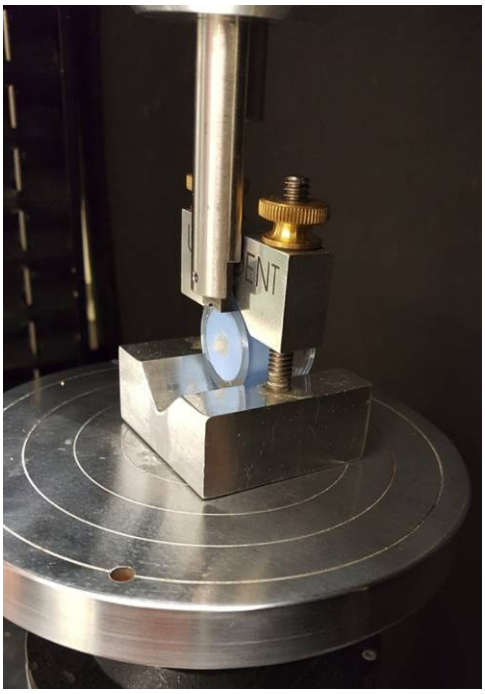


FIGURE 9. Shear bond strength testing.

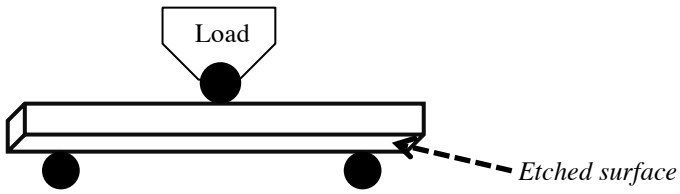


Figure 10

FIGURE 10. Schematic representation of mounting specimens for flexural strength testing.

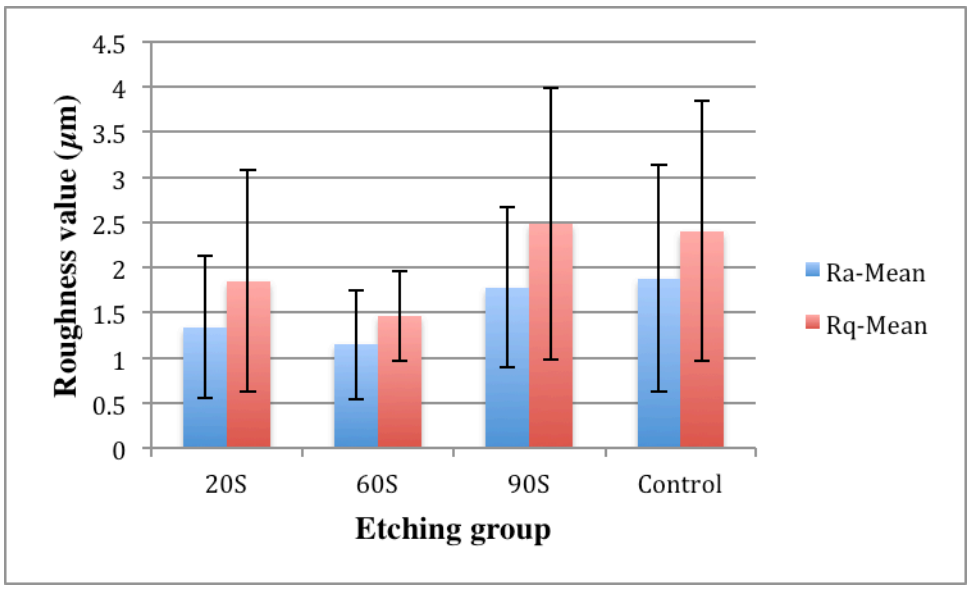


FIGURE 11. Surface roughness means and respective ± SD of experimental groups.

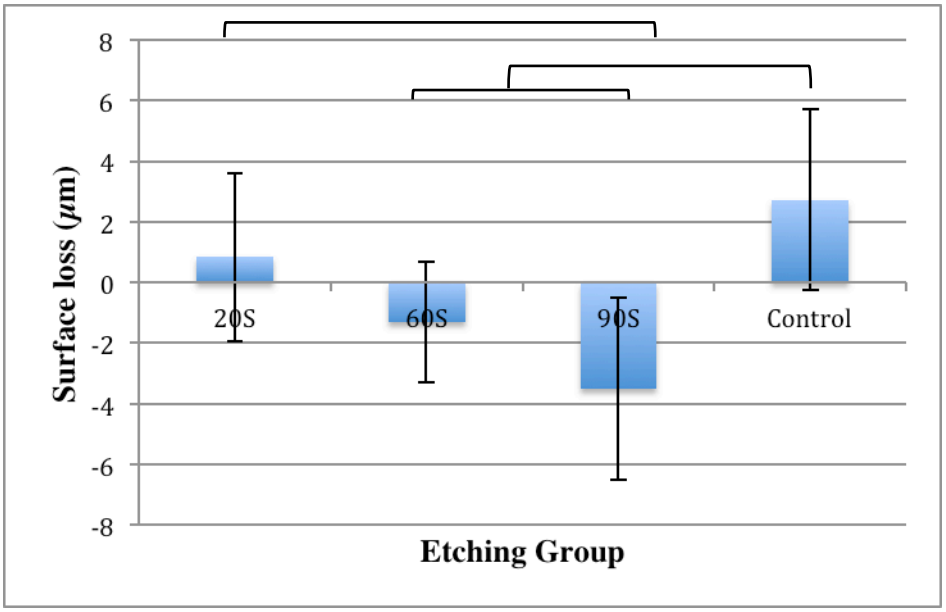


FIGURE 12. Surface loss means and respective ± SD of experimental groups.

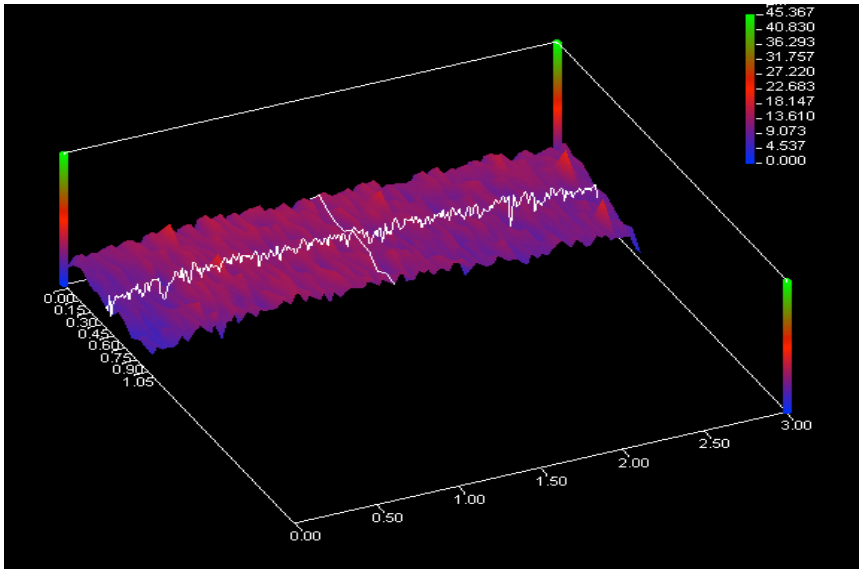


FIGURE 13. Representative non-contact profilometry scan of unetched ceramic group.

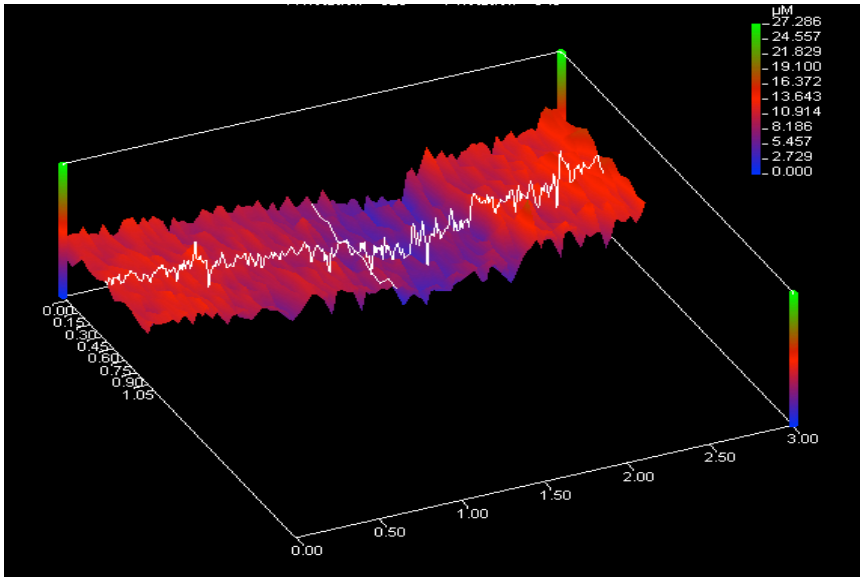


FIGURE 14. Representative non-contact profilometry scan of 20-s etched ceramic group.

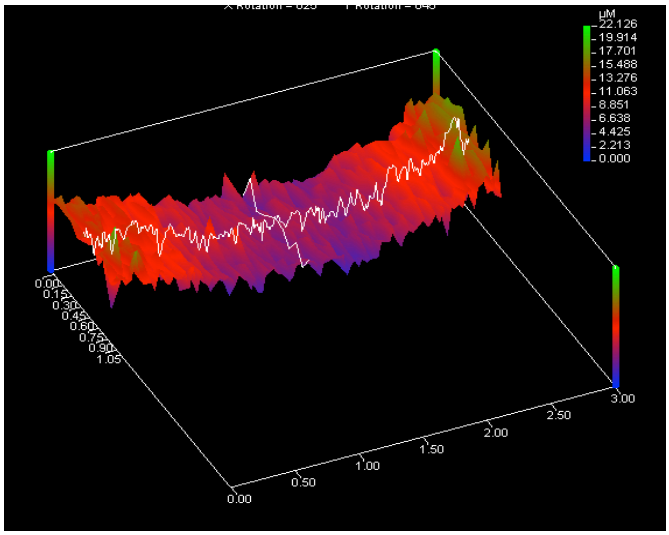


FIGURE 15. Representative non-contact profilometry scan of 60-s etched ceramic group.

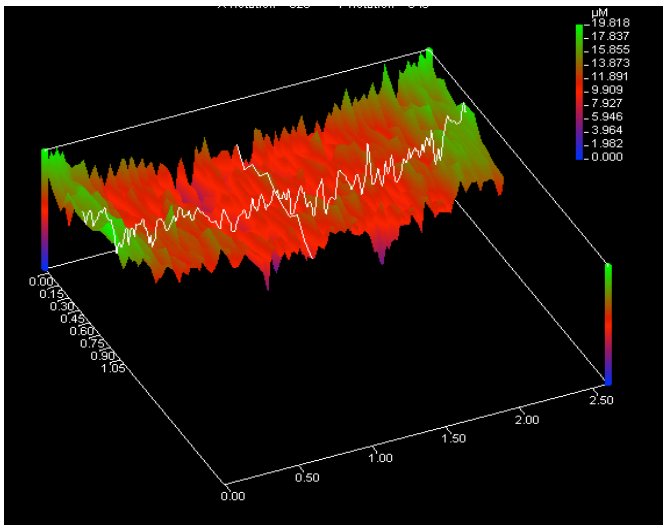
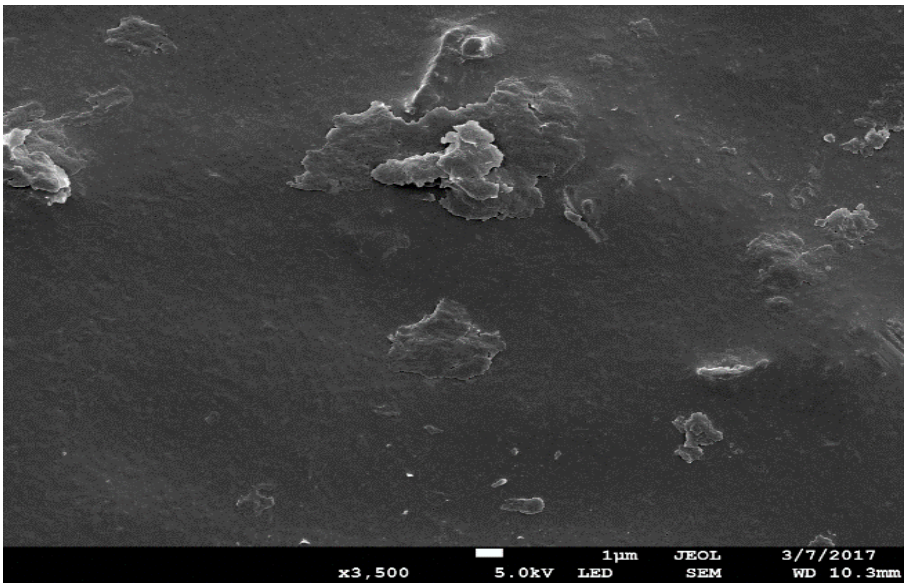
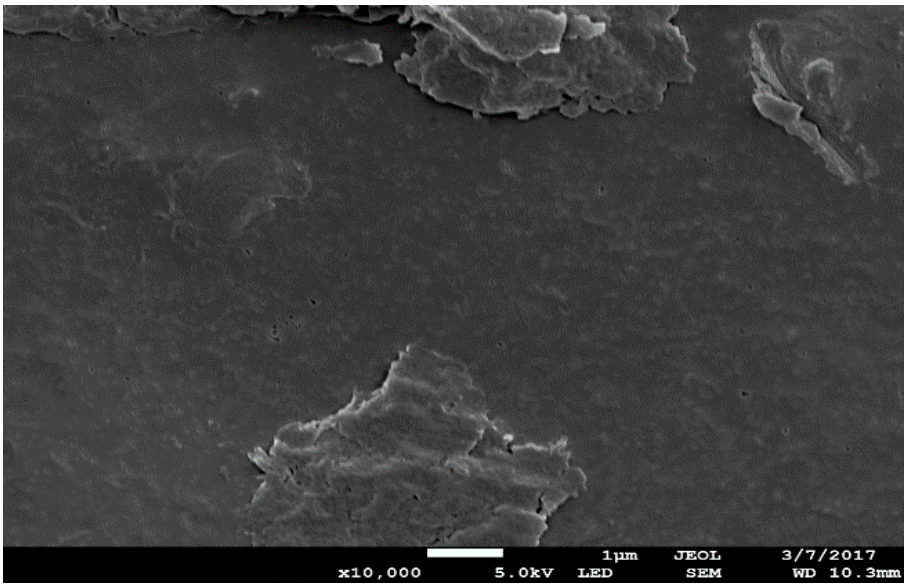


FIGURE 16. Representative non-contact profilometry scan of 90-s etched ceramic group.

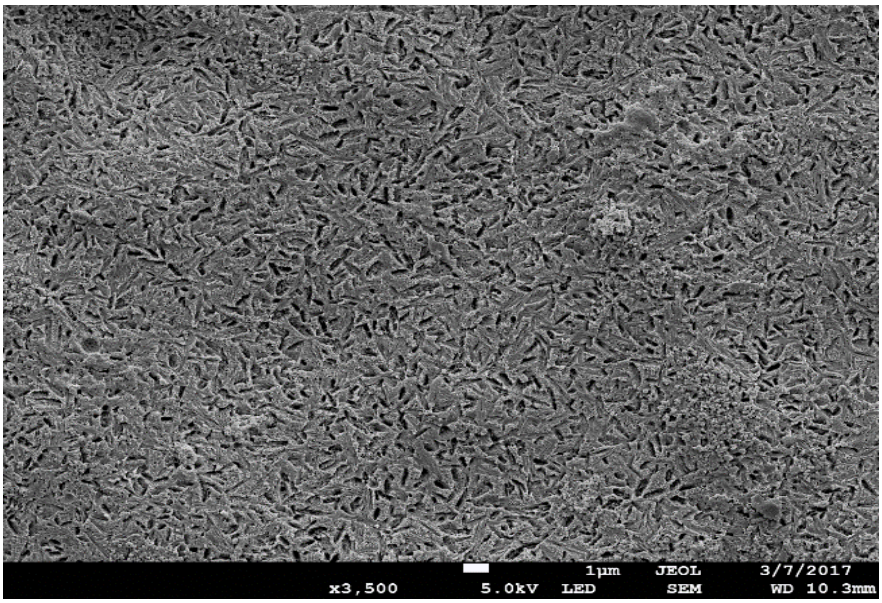


(a)

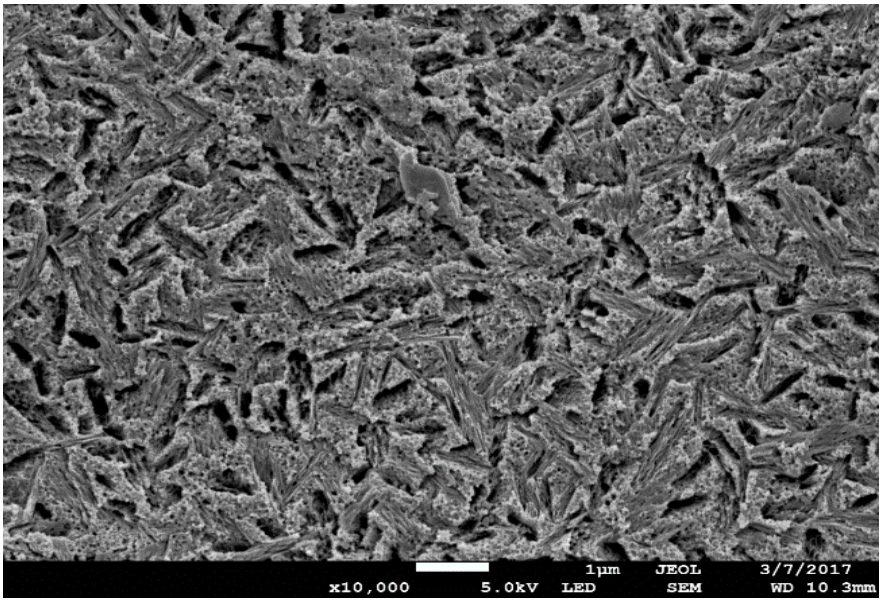


(b)

FIGURE 17. Representative SEM micrograph of the un etched ceramic group at (a) X3500 magnification and (b) X10000 magnification.

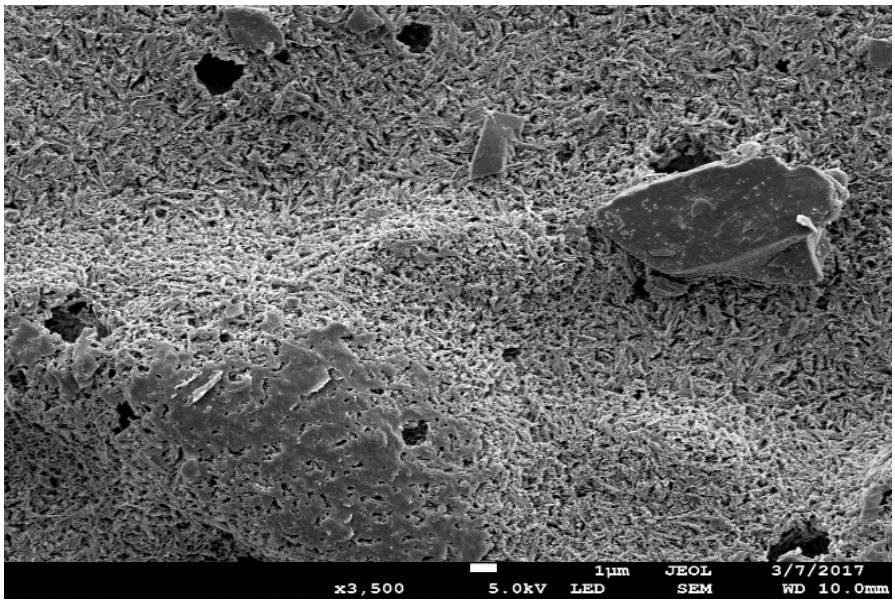


(a)

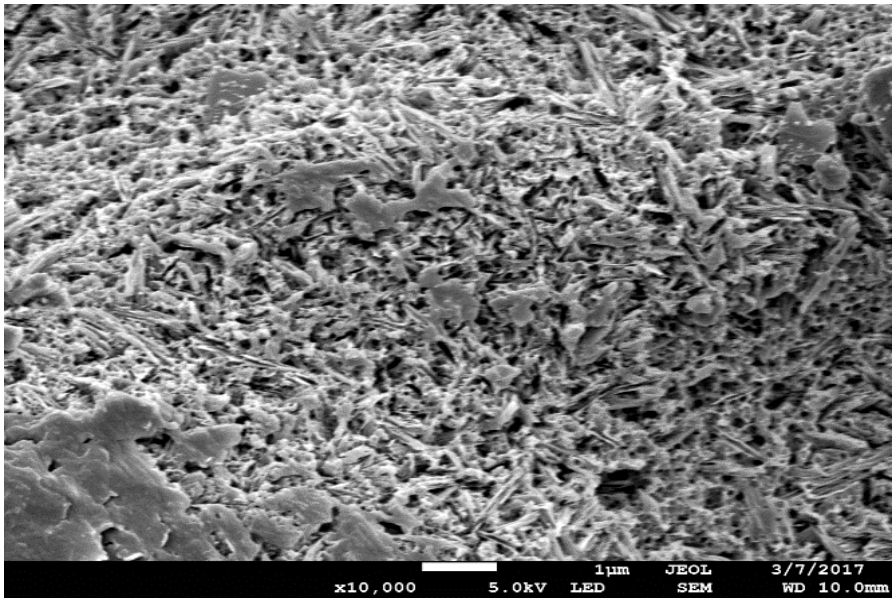


(b)

FIGURE 18. Representative SEM micrograph of the 20 -s etched ceramic group at (a) X3500 magnification and (b) X10000 magnification.

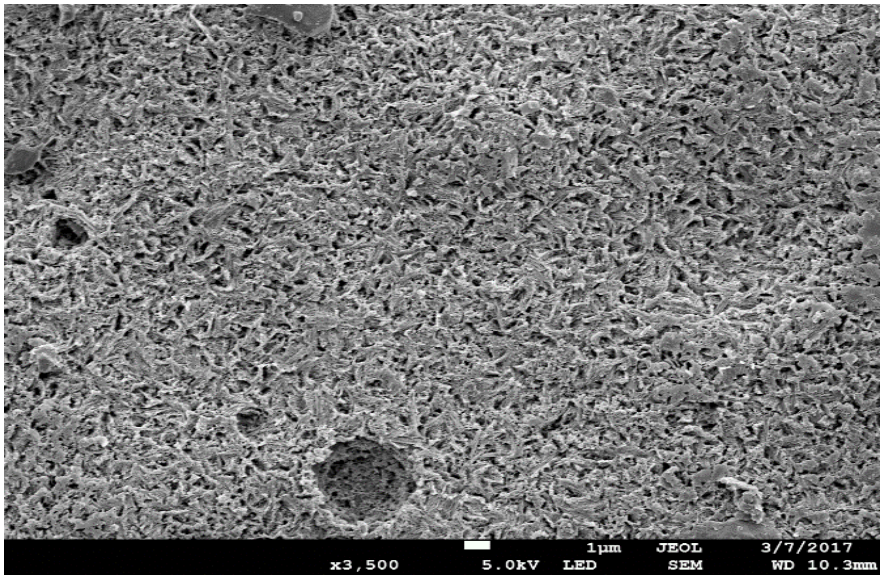


(a)

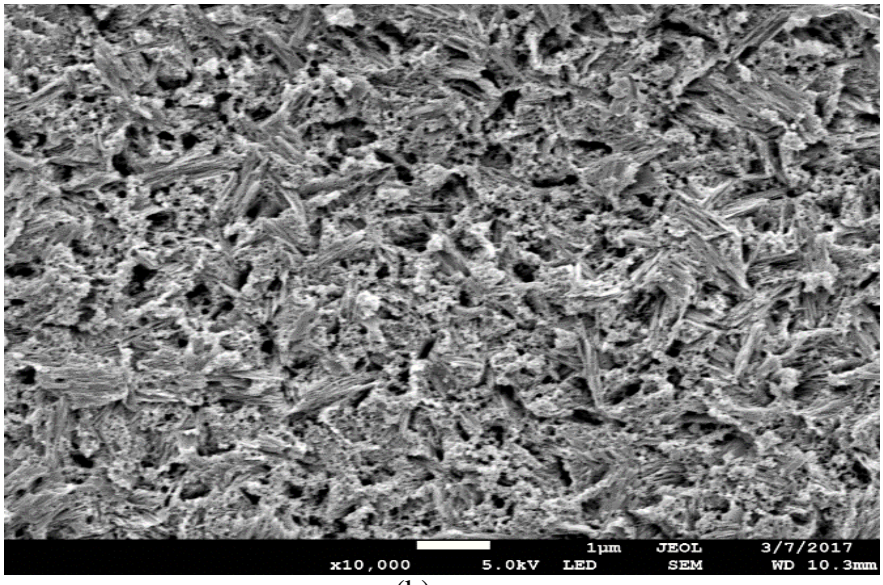


(b)

FIGURE 19. Representative SEM micrograph of the 60-s etched ceramic group at (a) X3500 magnification and (b) X10000 magnification.



(a)



(b)

FIGURE 20. Representative SEM micrograph of the 90-s etched ceramic group at (a) X3500 magnification and (b) X10000 magnification.

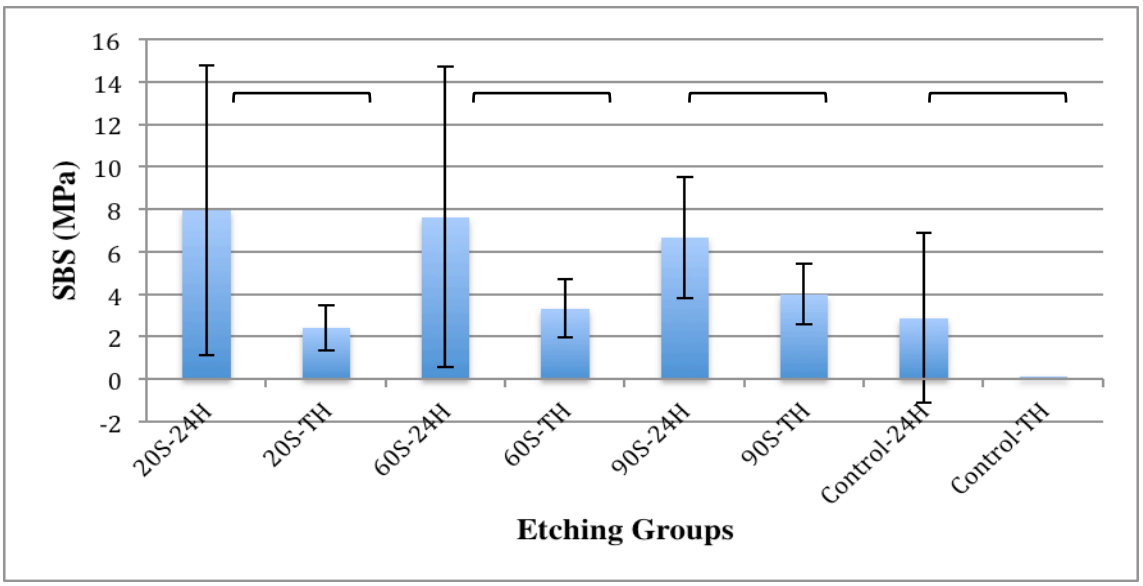
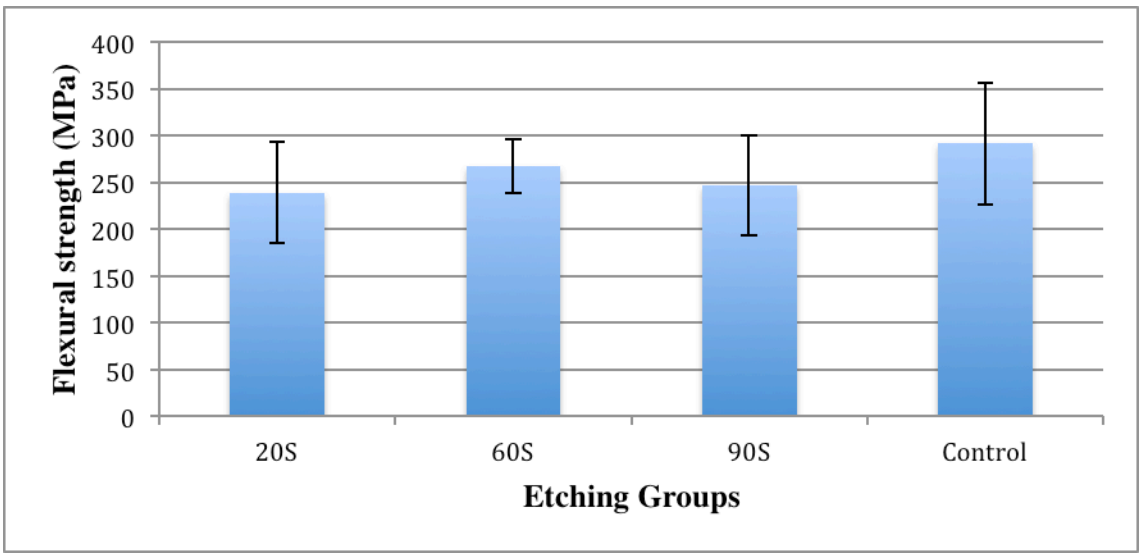


FIGURE 21, (Top) Shear bond strength means and respective ± SD of experimental groups.

FIGURE 22. (Bottom) Flexural strength means and respective ± SD of experimental groups.



DISCUSSION

Adhesion of resin cement to lithium disilicate glass ceramics depends on achieving adequate micromechanical retention within the ceramic substrate. Numerous studies have been conducted comparing the effects of different etchants on the microstructure and bond strengths of glass-ceramics. Della Bonna et al.[54] compared the microstructural pattern changes following etching a glass-ceramic with 9.6-percent HF, 10-percent ammonium bifluoride and 4-percent acidulated phosphate fluoride. The study found that HF produced the most aggressive etching pattern with the most prominent topographic pattern on all dental ceramics examined due to the high roughness values obtained (R_a = 1.4 μm, R_q = 2.1 μm, and R_t = 39.8 μm). Similar findings were reported by Pattanaik et al.[32] and Ayad et al.[66] HF acid etching has been shown to increase the surface roughness of the glass ceramics by selectively removing the glass matrix and exposing the lithium disilicate crystals allowing the resin cement to penetrate and to interlock within these surface irregularities. These interactions result in the greatest bond strength between the ceramic and tooth structures when compared with other ceramic surface treatments.[30,44,55,66] In this study, the effects of different etching durations on both the morphological and mechanical properties of IPS e.max CAD lithium disilicate glass-ceramic were evaluated.

Höland [58] and Belli et al.[59] described IPS. e.max CAD as a multicomponent system with P_2O_5 serving as the nucleating agent for the controlled bulk crystallization of lithium disilicate ($Li_2Si_2O_5$) crystals in a two-step heating process (Table III). Once the glass ceramic ingots undergo an initial firing process, lithium metasilicate crystals

(Li_2SiO_3) are formed that provide the partial strength required for machining with the CAD/CAM systems. Subsequently, a second firing process ensues upon which crystallization is complete as lithium disilicate crystals are formed. XRD patterns of IPS e-max CAD revealed the Li_2SiO_3 phase present in the pre-crystallized blocks, which disappeared completely in the crystallized blocks predominantly composed of a $Li_2Si_2O_5$ phase and a remaining Li_3PO_4 segment. In the present study, SEM images of the etched and unetched ceramic surfaces noticeably represented the effect of the different etching durations on the microstructure of the glass ceramic. SEM images at both magnifications (X3500 and X10000) revealed numerous irregularities and voids in the etched ceramic surfaces as well as elongated lithium disilicate crystals in comparison with the unetched ceramic surfaces, which displayed homogenous patterns. This is explained by the selective removal of the glassy matrix in the treated specimens exposing the underlying crystalline structure. In addition, as the etching periods increased, the size and number of the voids also increased as was seen in specimens etched for 90 seconds versus those etched for 20 seconds, which demonstrated fewer microstructure alterations. These observations are in agreement with some previous studies.[27-32]

Surface roughness of dental ceramics can be measured by contact or non-contact methods. The main disadvantage of the contact method is the possible abrasion of the specimens due to the force applied by the profilometer stylus. However, non-contact profilometers overcome this disadvantage, given they do not touch the surface of the ceramic specimens and that the small diameter of the laser scanner (<100 μm) provides accurate measurement of the surface topography.[69,70] Hence, non-contact profilometry was the method of choice for surface roughness and surface loss measurement. In the

present study, the HF acid etching did not significantly increase the surface roughness for any of the experimental groups, regardless of the etching duration used. This is in contrast to what was reported by Zogheib et al.[29] and Ramakrishnaiah et al.[35] who observed a positive correlation between ceramic surface roughness and increasing HF acid etching duration. Zogheib et al.[29] reported increased Ra surface roughness values for etching durations as short as 20 seconds. Ramakrishnaiah et al.[35] also reported greater S_a roughness values for 20 second etching groups in comparison with the control specimens. On the other hand, Prochnow et al.[27] reported that using HF acid of different concentrations did not significantly affect the mean surface roughness values (Ra) of the lithium disilicate glass ceramic used. Smaller roughness values were previously reported in the literature, which could be explained by the use of different polishing protocols, the use of contact profilometry, or the use of different roughness parameters to report the surface roughness.[27,29,35] However, the non-contact profilometry scans in the present study display a pattern of higher peaks and deeper valleys of the etched specimens in comparison with the untreated group, which could explain the significant difference in the surface loss values (μm) between the different etching groups in the present study. IPS e.max CAD specimens etched for 90 seconds showed greater loss in the height of the specimens in comparison with the remaining experimental groups, and this could be explained by the loss of the lithium disilicate crystals in addition to the loss of the glassy matrix in the superficial layer exposed to the prolonged etching time by HF acid.

Taking into consideration the obtained results of the present study, we fail to reject the first null hypothesis of this study that HF acid etching time would not significantly affect the surface roughness of the lithium disilicate glass ceramic.

Conversely, the second null hypothesis of this study was rejected that the HF acid etching time would significantly affect the surface loss of the glass ceramic.

Different types of bond strength tests have been utilized to evaluate the bonding efficiency of lithium disilicate glass ceramics to resin cements. Among these methods are tensile, microtensile, shear, and microshear bond strength tests and push-out tests. The shear bond strength test was chosen in the present study because it was the most frequently used bond testing method investigating the adhesion between resin cements and ceramic materials. Furthermore, studies have reported that the shear bond strength test generates stress similar to major stresses responsible for bonding failures in-vivo.[28,39,60-62] Chen et al.[47] evaluated the effect of etching and silane priming on bond strength to a feldspathic porcelain by examining the effect of different HF acid etchant concentrations (2.5% and 5%) and different etching times (0 s, 30 s, 60 s, 90 s, 120 s, 150 s, and 180 s) on the microstructure and bond strengths of feldspathic porcelain (VMK 68, Vita Zahnfabrik) to composite resin (Clearfil APX, J Morita USA, Inc.). Authors concluded that shear bond strength was greater when the porcelain was etched with the 2.5-percent HF than that etched with the 5-percent HF when etched for 150 seconds or less. Authors explained that over-etching porcelain could adversely affect bond strengths due to difficulty in removing the etchant from the etched surfaces, the wettability of intermediate resins, and inherent post-curing stress concentration at the adhesive interface structure. Authors also stated that using silane significantly improved the bond strength of the feldspathic porcelain to resin composite, which is in agreement with several studies.[28,64,65,67]

The present study revealed that the varying etching durations did not significantly

affect the shear bond strength of the lithium disilicate glass ceramic to the resin cement used. This is in agreement with findings reported in the literature. Kalavacharla et al.[28] examined the shear bond strength in lithium disilicate glass ceramics exposed to different etching protocols and concluded that the HF concentration or etching time did not have a significant effect on bond strength for the specimens that were coated with silane while specimens that were not coated with silane exhibited higher bond strength values at higher etchant concentrations and longer etching durations. Authors reported higher SBS values in comparison with the present study, which can be explained by their use of resin composite to avoid cohesive failure of the resin cement.

In addition, the HF etching and polishing protocol in the present study resulted in greater variation in the SBS values evident in the standard deviation statistics.

In the present study, prior to testing the shear bond strength, half the specimens were stored for 24 hours in distilled water and half were thermocycled for 5000 cycles. This was based on evidence in the literature stating the bond strength following thermocycling closely resembles that found in the oral cavity, considering that resin-ceramic bonds might be compromised in the oral environment due to contamination of the luting surfaces by saliva, blood, or other contaminants. In addition, it was found that the resin-porcelain bond tends to be weaker after the thermocycling of bonded specimens.[50] It has been reported in the literature that thermocycling for 10,000 cycles is equivalent to 1 year of *in-vivo* functioning.[71] Accordingly, the 5000 cycles used in the present study would approximate 6 months of clinical performance. The present study revealed a statistically significant difference between water storage and thermocycling on the shear bond strength of the ceramic specimens. This finding coincides with previous

studies in the literature in which thermocycling had shown to significantly decrease bond strength of a resin luting cement to an etched glass ceramic surface.[44,50]

Hence, based on the findings of the shear bond strength test in this study, we fail to reject the third null hypothesis of this study that HF acid etching time would not significantly affect the shear bond strength of the lithium disilicate glass ceramic to a resin cement.

Various types of laboratory tests have been employed to evaluate the flexural strength of dental ceramics that take into consideration their brittle nature, which renders them weaker in tension than in compression. Flexural strength can be measured by uniaxial tests such as 3-point or 4-point bending, or biaxial tests such as ring-on-ring, ball-on-ring, piston-on-ring, and piston-on-three-ball tests. All tests are based on creating tensile stresses at the bottom surfaces of the specimens generating cracks at the surface flaws, which propagate until catastrophic failure occurs. The 3-point bending test was chosen in this study considering it has been the standard test for dental ceramics due to its uncomplicated test design test, and the preparation of specimens regarding shape and dimension is relatively simple. When compared with the 4-point bending test, the flexural strengths obtained from the 3-point bending test were higher because of the smaller flaw-containing area.[69] However, the main limitation of uniaxial flexural strength tests is the inevitable presence of flaws along the surface edges of the rectangular shaped specimens; hence, biaxial flexural tests have recently been used because the central loading eliminates the effects of any surface flaws along the edges.[31]

Within the present study, flexural strength values were higher in the control group compared with the etched groups; however, the difference in HF acid etching durations

did not have a significant effect on the flexural strength values of the IPS e.max CAD specimens. This finding is in agreement with a study done by Menees et al.[30] which compared the effect of different etching protocols on the flexural strength (3-point bending test) of IPS e.max CAD specimens. Authors concluded that the HF acid decreased the flexural strength of the glass ceramic specimens regardless of the protocol used; however, this was not statistically significant. Furthermore Prochnow et al.[27] also stated that etching lithium disilicate glass ceramics with different HF acid concentrations did not affect the flexural strength of the specimens in comparison with the unetched specimens. However, opposite findings were reported by Zogheib et al.[29] and Hooshmand et al.[31] who both stated that etching the lithium disilicate glass ceramics with HF acid significantly reduced the flexural strength of the specimens.

Hence, based on the findings of the flexural strength tests, we fail to reject the fourth null hypothesis that that HF acid etching time would not significantly affect the flexural strength of the lithium disilicate glass ceramic.

Some limitations in the present study include the following: (1) Only one HF acid concentration was examined, and further studies should be done evaluating different etching durations and etchant concentration combinations. (2) The present study was performed in optimum temperature and humidity settings; however, it is necessary to repeat these tests under different environment settings to simulate the clinical conditions. (3) The 3-point bending test used in the present study is a uniaxial loading test that does not accurately reflect the actual fracture strengths found *in-vivo* considering the various loading conditions in the oral cavity.

SUMMARY AND CONCLUSIONS

Within the limitations of this *in-vitro* study, the following conclusions were drawn:

1. The difference in HF acid etching duration does not have a significant effect on the surface roughness of IPS e.max CAD lithium disilicate glass ceramic.

2. The difference in HF acid etching duration does have a significant effect on the surface loss of IPS e.max CAD lithium disilicate glass ceramic.

3. The difference in HF acid etching durations does not have a significant effect on the shear bond strength of IPS e.max CAD to the resin cement used in the study.

4. Thermocycling significantly reduced the shear bond strength of IPS e.max CAD to the resin cement used in the study.

5. The difference in HF acid etching durations does not have a significant effect on the flexural strength of IPS e.max CAD lithium disilicate glass ceramic.

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ABSTRACT

EFFECTS OF ETCHING DURATION ON THE SURFACE ROUGHNESS, SURFACE LOSS, FLEXURAL STRENGTH OF E.MAX CAD GLASS CERAMIC AND SHEAR BOND STRENGTH TO A RESIN CEMENT

by

Hanan Al-Johani

Indiana University School of Dentistry
Indianapolis, Indiana

Background: Long-term retention of ceramic restorations is dependent on the bond strength of the luting resin to both the tooth and porcelain substrates. In order to achieve successful bonding, the surface of the porcelain substrate must be modified to increase the surface roughness, and this can be achieved chemically by hydrofluoric (HF) acid etching. However, prolonged HF acid etching has shown to have a weakening effect on the evaluated lithium disilicate glass-ceramics. Therefore, it is essential to quantify the required etching duration of HF acid to minimize the possible deleterious effects on ceramic strength while maximizing the bond strength to tooth structure.

Objectives: To evaluate the effects of HF acid etching duration on the surface roughness, surface loss, flexural strength, and shear bond strength of IPS e.max CAD (Ivoclar Vivadent) lithium disilicate-based glass ceramic to a resin cement. Hypothesis:

The differences in HF acid etching durations will not have a significant effect on the surface roughness, surface loss, flexural strength, or shear bond strength of IPS e.max CAD to a resin cement. Methods: 168 specimens were prepared from IPS e.max CAD blocks. All specimens were polished and sonically cleaned in distilled water. Specimens were fired in the vacuum pump furnace according to the manufacturer's instructions. Specimens were then divided into 4 groups, according to etching durations, then further divided into 3 subgroups, according to the properties tested. Group A was not etched (control), Groups B, C and D were etched with 5-percent HF acid (IPS Ceramic Etching gel, Ivoclar Vivadent) for 20 s, 60 s and 90 s respectively. The morphologies of both etched and non-etched surfaces in specimens of subgroup 1 of each etching group (n = 16/group) were observed under scanning electron microscopy (SEM). In addition, non-contact surface profilometry (Proscan 2000) was used to calculate the surface loss and to examine the surface roughness of the etched ceramic surfaces and roughness values (R_a, R_q) were documented for each group. Furthermore, etched specimens of subgroup 2 (n = 16/group) were silanated (Monobond Plus, Ivoclar Vivadent) and cemented with a resin cement (Multilink Automix, Ivoclar Vivadent). The shear bond strength (SBS) was measured using a universal mechanical testing machine. For each etching group, subgroup 3 specimens (n = 10/group) were loaded to failure in a three-point bending test to measure their flexural strength values using a universal mechanical testing machine. Data for surface roughness, surface loss, and flexural strength were analyzed using one-way analysis of variance (ANOVA), to identify the significant effects of different HF acid etching durations. Data for shear bond strength test were analyzed using two-way ANOVA to test the effects of etching duration, storage for 24 hours/thermocycling, and

their interaction. All pair-wise comparisons from ANOVA analysis were made using Fisher's Protected Least Significant Differences to control the overall significance level at 5 percent. Results: Difference in HF etching durations did not have a significant effect on surface roughness values R_a or R_q ($p = 0.3408$; $p = 0.3245$) respectively, but had a significant effect on surface loss ($p = 0.0006$). SBS values were not significantly different between experimental groups ($p = 0.4650$); however, SBS values after 24-h storage were significantly higher than that found after thermocycling ($p = 0.0166$) among different etching durations. Finally, different HF etching durations did not have a significant effect on flexural strength values ($p = 0.1260$). Conclusion: Within the limitations of this study, different HF etching durations did not have a significant effect on surface roughness, flexural strength, or shear bond strength of IPS e.max CAD. However, the different etching durations significantly affected the surface loss of the lithium disilicate glass ceramics.

CURRICULUM VITAE

Hanan Aouda M. Al-Johani

November 1988	Born in Boulder, Colorado, USA
June 2006	High School Diploma Alfursan High School, Riyadh, Saudi Arabia
July 2006 to July 2012	Bachelor of Dental Medicine and Surgery (BDS) Faculty of Dentistry, King Abdulaziz University Jeddah, Saudi Arabia
July 2012 to present	Demonstrator (Teaching Assistant) Dental Materials Division, Department of Restorative Dentistry, College of Dentistry, King Abdulaziz University, Jeddah, Saudi Arabia.
June 2014 –July 2017	Master of Science in Dentistry (MSD) Major in Dental Materials and Operative Dentistry Minor in Preventive Dentistry Clinical certificate in Operative Dentistry Indiana University School of Dentistry, Indianapolis, Indiana.

Professional Organizations

The Academy of Operative Dentistry
Saudi Dental Society
The Saudi Commission for Health Specialties