

Effect of post etching cleansing on surface microstructure, surface topography, and microshear bond strength of lithium disilicate

Nikita Agarwal, Sanchit Bansal, Umesh Yeshwanth Pai, Shobha J. Rodrigues, Thilak B. Shetty, Sharon J. Saldanha
Department of Prosthodontics, Manipal College of Dental Sciences, Manipal Academy of Higher Education, Mangalore, Karnataka, India

Abstract

Aim: This study assessed the effect of postetch cleansing on the surface microstructure, surface topography, and microshear bond strength (μ SBS) of lithium disilicate and the resin cement.

Setting and Design: *In Vitro* analytical study.

Materials and Methods: Fifteen discs (10 mm diameter and 2 mm thickness) were fabricated from highly translucent lithium disilicate IPS Emax 2 ceramic (Ivoclar Vivadent, Schaan, Liechtenstein). Four resin cement (RelyX Ultimate, 3M ESPE) cylinders (0.9 mm diameter and 4 mm high) were placed on each ceramic disc (total $n = 60$). The samples were divided into three groups based on the surface treatment of the ceramic discs (20 resin cement cylinders on 5 discs in each group). Group I (HF) (control) etched with 9.6% HF with no postetch cleansing, Group II (HFP) etched with 9.6% HF for 20 s followed by rinsing with water and postetching cleansing with 37% phosphoric acid, and Group III (HFPU) etched with 9.6% HF followed by active application of 37% phosphoric acid followed by postetch cleansing in ultrasonic bath for 5 min. μ SBS of resin cement to ceramic surfaces was tested following a standard protocol. Surface roughness was evaluated using an atomic force microscope. Surface topography and elemental analysis were analyzed using SEM/EDX. Mode of failure was also assessed.

Statistical Analysis Used: The data were analysed using one way analysis of variance and post hoc tukeys test.

Results: The μ SBS were found to be highest for Group III (HFPU), followed by Group II (HFP) followed by Group I (HF) and were statistically significant. There was a difference in the surface topography and surface microstructure between the three groups. Mode of failure was predominantly adhesive.

Conclusion: The μ SBS, surface topography, and surface microstructure were found to be superior in the groups, in which postetch cleansing was done as compared to the control in which no postetch cleansing was done.

Keywords: Ceramic, hydrofluoric acid, lithium disilicate, phosphoric acid, ultrasonic cleansing

Address for correspondence: Dr. Umesh Yeshwanth Pai, Department of Prosthodontics, Manipal College of Dental Sciences, Manipal Academy of Higher Education, Mangalore - 575 001, Karnataka, India.

E-mail: pai.umesh@manipal.edu

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INTRODUCTION

Recent advances in all ceramic restorations have helped in achieving excellent esthetic results.^[1,2] All ceramic restorations have emerged as a viable alternative to metal ceramic restorations for anterior esthetic restorations. The long-term survival rate of these restorations is dependent on the adhesion between the ceramic material and tooth structure.^[3]

The invent of simultaneous phosphoric acid etching of enamel and hydrofluoric acid (HF) etching of ceramics by Horn in 1983 provided a major breakthrough in adhesive dentistry.^[4] The most effective method of treating the intaglio surface of ceramic restoration is etching with hydrofluoric acid followed by application of silane coupling agent.^[5] Hydrofluoric acid causes preferential dissolution of glassy matrix^[6] and helps in the formation of “honeycomb-like structures” which enhances the micromechanical retention.^[7] Silanation increases the wettability and also forms a covalent bond with resin cement at one end and ceramic at the other.^[8]

However, there are various disadvantages of HF. It is a highly toxic substance and diffuses in the cell and poses serious health hazards like tissue necrosis.^[9] Newer glass ceramics have a very fine crystalline structure that gets no benefits from HF etching.^[10] Furthermore, HF etching of glass ceramics can result in the formation of insoluble silica fluoride salts^[9,11] which precipitate on the surface of ceramics acting as a barrier for the resin penetration, thereby hampering adequate bonding. This, in turn, can affect the resin ceramic bond strength.^[12,13]

Removal of HF is, therefore, highly advantageous and many techniques have been proposed to remove them such as brushing the fitting surface of the restoration with a clean tooth-brush,^[14] thoroughly rinsing with water,^[8] immersing in an ultrasonic bath with distilled water or 95% alcohol for either 5 or 10 min,^[15,16] application of 37.5% phosphoric acid with gentle agitation using a microbrush for 1 min^[16] (active application of phosphoric acid is better than passive application),^[17] and combination of these techniques.^[16]

These techniques might seem time-consuming because of added steps and equipment required to complete the bonding procedure. However, there exists no concurrence in the literature regarding the necessity of these techniques in the routine all ceramic bonding protocol. Therefore, the purpose of this *in vitro* study was to assess the effect of postetching cleansing on the surface microstructure,

surface topography, and microshear bond strength (μ SBS) of lithium disilicate. The null hypothesis is that there will be no effect of postetching cleansing on the surface microstructure, surface topography, and μ SBS of lithium disilicate.

MATERIALS AND METHODS

The study was approved by institutional ethical committee, Manipal college of dental sciences, Mangalore (Protocol ref no. 18023). A total of 60 resin cement cylinders ($n = 60$) on 15 lithium disilicate discs were tested in this study. The samples were divided into three groups based on the surface treatment of the ceramic discs (20 resin cement cylinders on 5 discs in each group).

Fifteen disc specimens with 10 mm diameter and 2 mm thickness were fabricated from highly translucent lithium disilicate IPS Emax 2 ceramic (Ivoclar Vivadent, Schaan, Liechtenstein).

The discs were divided into three groups randomly as follows:

Group I (HF) (Control): 9.6% HF (Porcelain Etch, Ultradent Products, Jordan) was applied with a microbrush for 20 s and rinsing was done with water for 30 s. No postetching cleansing was done.

Group II (HFP): 9.6% HF (Porcelain Etch, Ultradent Products, Jordan) was applied with a microbrush for 20 s and then rinsed with water for 30 s. Using a microbrush, the specimens were then cleaned with 37% phosphoric acid (3M ESPE Scotch Bond Multi-purpose Etchant, St. Paul, MN, USA) using a gentle brushing motion for 30 s followed by rinsing with water for 30 s.

Group III (HFPU): 9.6% HF (Porcelain Etch, Ultradent Products, Jordan) applied with a microbrush for 20 s and rinsed with water for 30 s. Using a microbrush, the specimens were then cleaned with 37% phosphoric acid (3M ESPE Scotch Bond Multi-purpose Etchant, St. Paul, MN, USA) using a gentle brushing motion for 30 s. Cleaning was completed by immersion of the samples in an ultrasonic bath for 5 min.

Scanning electron microscopy for surface microstructure analysis

Two discs from each group were chosen randomly for microstructure analysis. Samples were desiccated for 48 h (Dry Keeper Simulate Corp., Tokyo, Japan) and sputtering was carried out with a platinum layer of 10 nm (Polaron Equipment Ltd., Hertfordshire, England, UK). Following

sputtering, a scanning electronic microscope (SEM-Zeiss EVO MA 25; Carl Zeiss, Jena, Germany) was used for surface analysis in all the three groups.

Scanning electron microscopy (SEM) was supplemented with energy-dispersive X-Ray spectroscopy (EDS) to analyze the elemental composition of the discs subjected to different surface treatments.

Atomic force microscopy for surface topography analysis

Two ceramic discs from each group were selected randomly for surface roughness measurement using an atomic force microscope (AFM) (Bruker USA). Specimens were tested under a noncontact mode utilizing an AFM cantilever with magneto-resistive sensors incorporated in its tip. The measurements were made on each surface-treated ceramic disc at three random locations using a standardized rectangular spot ($50\ \mu\text{m} \times 50\ \mu\text{m}$). The average or arithmetic surface roughness (Ra), root mean square value roughness (Rq), and peak height/maximum roughness (highest value – Rmax or Z) of the ceramics were noted as numeric values in nanometers.

Sample preparation

Following surface treatments, a silane coat was applied (Silane; Ultradent Products, Jordan) to all the discs with a microbrush for 60 s and gently air-dried. After silane application, 2 coats of adhesive (Single Bond Universal Adhesive; 3M ESPE, St. Paul, MN, USA) were applied, gently agitated and dried with a stream for evaporation of the solvent. Then, according to manufacturer's instructions, the adhesive was light-cured for 10 s. Four tygon tubes (Angiocath BD, Cundinamarca, Colombia) with a diameter of 0.9 mm and a height of 4 mm were placed on each surface-treated disc (4 tygons per ceramic disc; total $n = 60$) at a distance of 5 mm from each other and loaded with resin cement (RelyX Ultimate, 3M ESPE St. Paul, MN, USA). The samples were light-cured for 40 s at an intensity of $1,200\ \text{mw}/\text{cm}^2$ (Blue Dent Smart, BG Light, Bulgaria), according to the manufacturer's instructions. A sharp blade was used for the removal of the tygon tubes from the disc. This resulted with each ceramic disc having four resin cement cylinders [Figure 1].

After the sample preparation, thermocycling (MSCT-3, Marcelo Nucci– ME, São Carlos, SP, Brazil) was done to simulate aging in the oral environment. 3500 cycles were done at 5 degrees and 55 degrees with a dwell time of 5 min simulating an intraoral aging of approximately 1 year

Microshear bond strength test

For universal testing, a heat cure PMMA block (Coltene Heat cure Denture Material; Coltene Whaledent, Switzerland) of



Figure 1: Specimen fabrication with resin cement cylinders on lithium disilicate discs

dimension $40\ \text{mm} \times 15\ \text{mm} \times 15\ \text{mm}$ was constructed to stabilize the ceramic discs. This was then positioned in the universal testing machine (Instron, Instron Engineering Corporation, Massachusetts). A blade was positioned at an angle of 90° at the junction of the resin/ceramic interface [Figure 2]. A shear load was applied to each resin cement cylinder, at a crosshead speed of 0.5 mm/minutes, until specimen fractured.^[18] The values were expressed in MPa.^[19]

Fractographic analysis

A compound zoom microscope (CH 20I, Olympus, Olympus scientific Solutions America Corp) was used at $\times 40$ magnification to classify the failure mode as adhesive (at the resin cement/ceramic interface, including pretesting failure), cohesive (within the resin cement or within the ceramic), or mixed (with both adhesive and cohesive failures).

Statistical analysis of the results for the μSBS values was performed by one-way analysis of variance and *post hoc* Tukey's test. A 95% confidence interval was used for all the statistical tests ($\alpha = 0.001$). The statistical analysis was done with SPSS software (Version 15.0, SPSS Inc., Chicago, IL, USA).

RESULTS

Microshear bond strength test and fractographic analysis

The μSBS values (mean and standard deviation) and a statistical comparison of the different groups are shown in Tables 1 and 2, respectively. μSBS values were significantly higher in Group III (HFPU) ($58.88 \pm 2.5\ \text{MPa}$) when compared with Group II (HFP) ($49.52 \pm 2.23\ \text{MPa}$) and Group I (HF) ($42.11 \pm 1.41\ \text{MPa}$) ($P < 0.001$) [Figure 3].

During μ SBS measurements, there were 5, 1, and 2 pretesting failures in the groups HF, HFP, and HFPU, respectively. Fractographic analysis showed that adhesive failures were predominant in all the three groups (Group HF – 70%, Group HFP – 70%, and Group HFPU – 85%) as seen in Figure 4.

SEM analysis

The results of SEM analysis at $\times 1000$ and $\times 3000$ magnification are demonstrated in Figures 5 and 6,

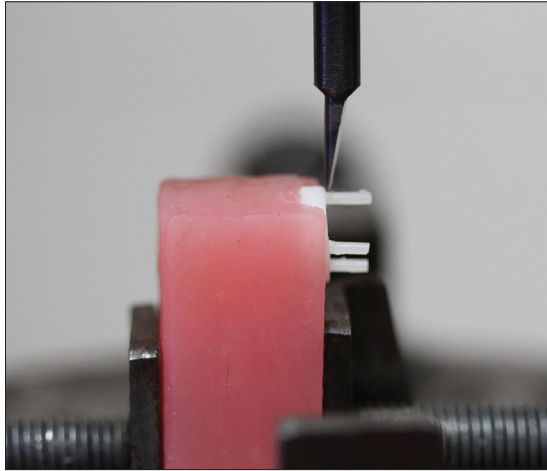


Figure 2: Microshear bond strength testing

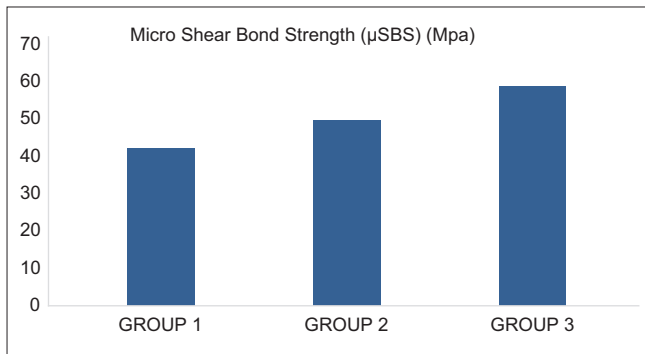


Figure 3: Bar diagram showing fractographic analysis

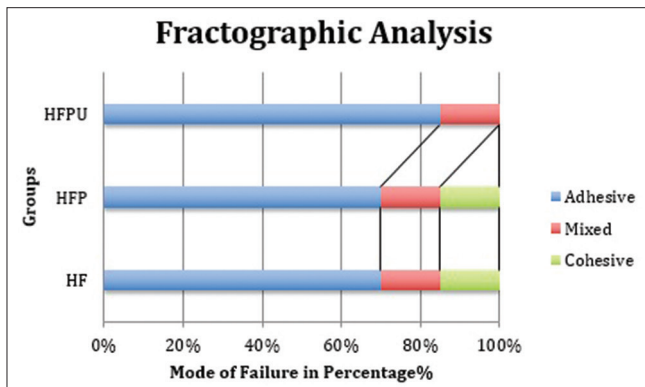


Figure 4: Bar diagram showing mean microshear bond strength values

respectively. Group HF showed needle-like structures with vitreous islands. Microsurface of Group HFP showed lesser vitreous islands and increased density of needle-like structures, giving a dry earth appearance suggestive of increased roughness. Group HFPU showed a uniform interlocked networks of needle-like structures suggestive of most dense etching pattern. From Group HF to Group HFPU, an increase in the density and the uniformity of etching pattern was noted.

Energy-dispersive X-ray spectroscopy analysis

The elemental composition in weight percentage for all the groups is summarized in Table 3. Traces of fluorine were found in group HF (2.1%) and group HFP (1.2%), but no fluorine was found in the group HFPU suggestive of no silica fluoride salts in that group.

Atomic force microscope analysis

The AFM results are presented in Table 4 and Figure 7. Group HFPU showed the highest value of surface roughness (Ra, Rq, and Rmax) as compared to other two groups.

DISCUSSION

The study was conducted to compare the effect of postetching cleansing on surface microstructure, surface topography, and shear bond strength of lithium disilicate. The efficacy of bond strength was evaluated using μ SBS test. The effect of different surface treatment on microstructure of lithium disilicate substrates was evaluated by SEM supplemented with EDS and AFM topographic analysis. The mode of failure was assessed using a compound microscope.

It is a well-known fact that adhesion is a key factor in strengthening of esthetic restorations.^[17] The unique mechanical and physical properties of materials involved along with the optimal surface enhancement of the bonding substrates interact to form a strong bond between the restoration and the tooth.^[20] The materials include the tooth structure, bonding agent, resin cement, and porcelain restoration. Hydrofluoric acid causes preferential dissolution of the glassy phase of ceramic and provides an ideal microstructure for bonding. On the other hand, silane coupling agent provides a chemical covalent hydrogen bond that is an essential factor in creating a sufficient resin bond to silica-based ceramics.^[21] There is a development of good bond strength between the ceramic surface and the luting agent if the surface of the ceramic is clean and rough.^[22,23] Stretched crystals and superficial

Table 1: Different letter (A/B/C) shows that there is statistical significant difference between the groups (P<0.001)

	Groups	n	Mean	Std. deviation	Welch statistics (*)/F (ANOVA)	P
Micro	HF	17	42.11 ^A	1.41	306.20*	<0.001
Shear bond	HFP	19	49.52 ^B	2.23		
Strength	HFPU	18	58.88 ^C	2.52		
(µSBS) (MPa)	Total	54	50.30	7.14		

*Suggests that the welch test is used which is a variant of ANOVA. It is used when the variances are not equal

Table 3: Elemental composition (in weight %) of all the three groups

Groups	Si	C	O	F	Na	Mg	Al	K	P
HF	36.1	6.7	47.5	2.1	0	0	1.9	3.9	1.8
HFP	38.5	6.2	48.3	1.2	0.3	0.2	1.5	2.5	1.3
HFPU	39.8	5.5	49.9	0	0	0	1.3	2.3	1.2

irregularities were observed when HF was used to treat the surface of lithium disilicate. According to Höland *et al.*,^[24] elongated crystals of lithium disilicate form the main crystalline phase and lithium orthophosphate constitutes the second phase of glass ceramic. A glassy matrix surrounds both these phases. HF, thus, creating irregularities in the lithium disilicate crystals removes this glassy matrix and the second phase. The present study showed similar results.

According to Della Bona *et al.*, there is an increase in the potential for bonding of the ceramic surface if the surface area available is more, which, in turn, depends on the surface cracks and irregularities formed on the intaglio surface of the ceramic.^[25] In the present study, surface roughness was highest for HFPU group, signifying more surface area available for bonding and hence better bond strength as compared to other groups. Phosphoric acid acts as a neutralizer. The bond strength increases when active application of phosphoric acid is done as it aids in the removal of the precipitates formed after etching with HF.^[23] This allows for a deeper penetration of the resin cement in the ceramic.

In the present study, it is seen that the ultrasonic group has the highest value of bond strength and surface roughness, signifying that the postetching residues were cleaned properly. Use of ultrasonic helps to eliminate the surface fluoride residues, thus allowing a proper etched surface and hence increased surface roughness. Group HFPU showed an increase in roughness of 11.5% and 3.5% over HF and HFP, respectively. This was confirmed with the AFM images that show the highest value of peak in the Group HFPU. SEM images also show more dense and uniform pattern in the Group HFPU when compared with the other two groups. Clinicians who do

Table 2: Post Hoc Tukey Test showing significant difference between different groups

Dependent Variable	(I) group	(J) group	Mean difference (I-J)	Std. error	P
Microshear Bond Strength (µSBS) (Mpa)	Group HF	Group HFP	-7.41*	0.70	<0.001
	Group HF	Group HFPU	-16.78*	0.71	<0.001
	Group HFP	Group HFPU	-9.36*	0.69	<0.001

*Suggests that the welch test is used which is a variant of ANOVA. It is used when the variances are not equal

Table 4: Roughness values of all groups at 50 µm where Ra - is mean arithmetic mean, Rq - square root mean and Rz - maximum peak value or roughness value

Type of roughness	Group HF (In nm)	Group HFP (In nm)	Group HFPU (In nm)
Arithmetic roughness (Ra)	527	575	596
Root mean square roughness (Rq)	653	729	761
Max. peak value (Rz)	4667	5556	6371

not have an ultrasonic bath may use 37% phosphoric acid for the same purpose in order to increase the bond strength.

SEM is a commonly used method to analyze the microstructure after different surface treatments.^[20,22] This was supplemented with EDX analysis to give the elemental composition. When an electron beam is bombarded, energy present in the X-ray emitted from the specimen is measured and it gives the elemental composition of the specimen. One of the limitations of EDX analysis is that it cannot detect the elements having concentration <1%^[22] The EDX analysis detected the presence of Na, Si, Al, O, C, and F on the specimens. However, the elemental composition of fluorine varied and was minimum in the HFPU group, suggestive of proper removal of the precipitates in that group.

AFM analysis showed that the values of Ra (arithmetic roughness), Rv (maximum valley depth), and Rp (maximum peak height) were found to be highest for HFPU group, signifying that the surface roughness was maximum with the HFPU group; hence, it has the maximum surface area for bonding and hence the highest bond strength. Group HFPU showed an increase in bond strength of 28.48% and 15.89% over HF and HFP group, respectively.

Etching was done with 9.6% HF for 20 s. Several studies have shown that there is a negative effect on the flexural strength of a lithium disilicate material when the etching time is increased^[26] Furthermore, there is no increase in the bond strength on increasing the concentration of HF.^[25] For bonding of ceramic to the resin, universal adhesive was used. It consists of silane and a monomer

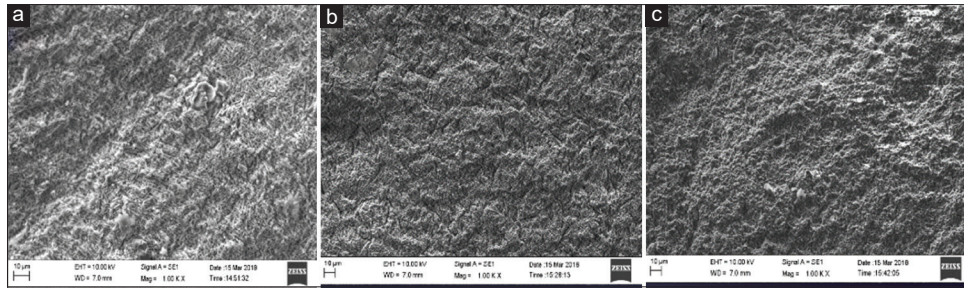


Figure 5: SEM images obtained from each experimental group ($\times 1000$): (a) Group HF, (b) Group HFP, (c) Group HFPU

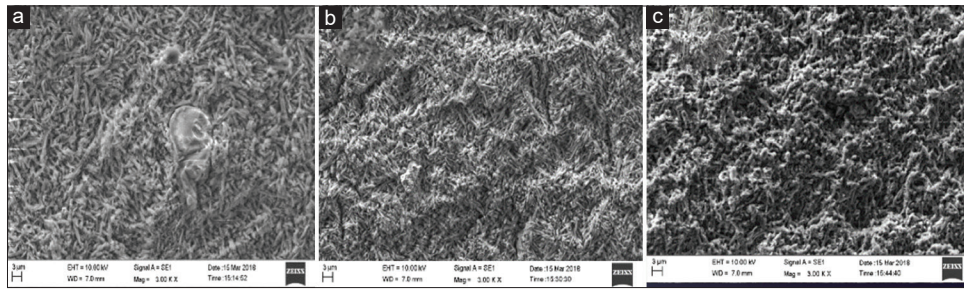


Figure 6: Scanning electronic microscope images obtained from each experimental group ($\times 3000$): (a) Group HF, (b) Group HFP, (c) Group HFPU

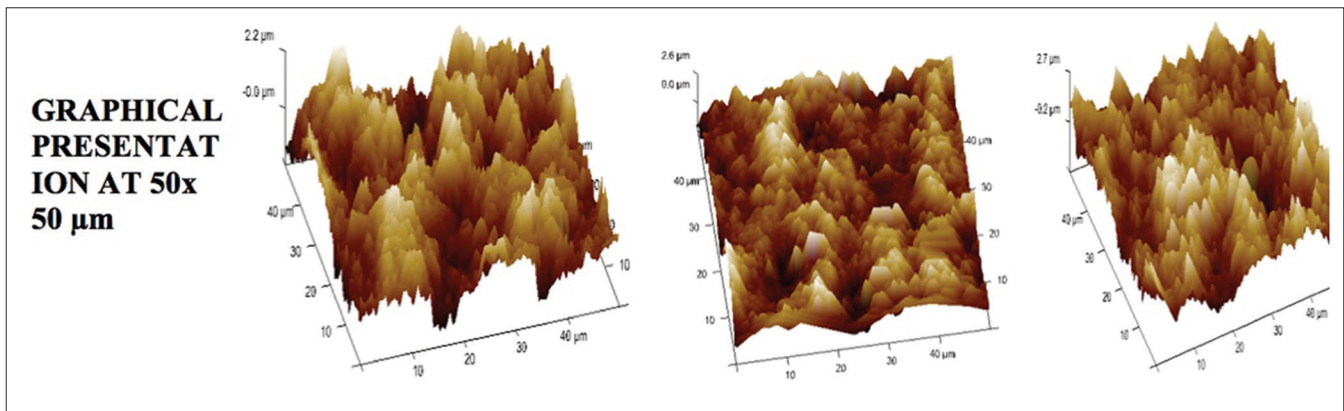


Figure 7: Atomic force microscope images of Group HF, Group HFP, HFPU

10-methacryloyloxydecyl dihydrogen phosphate that helps improve the bond strength between the two.^[26]

The methodology used in the study was to evaluate μSBS values as ceramics are brittle in nature. Moreover, on being compared with the macrotests used, microshear testing needs only small area of bonding, resulting in the uniform distribution of stresses.^[27]

The fracture mode in the present study was adhesive followed by mixed or cohesive, which is an advantage as no sectioning is needed for fabrication of sample for performing the microshear test.^[27] Majority of the specimens showed adhesive type of failure in all the groups as the bond between the resin cement and the restoration was weaker than the cohesive bond between the resin cement particles and/or the ceramic particles.

10% of the samples in group HF and group HFP showed cohesive failure. Cohesive failure in the resin cement occurs due to the forces that are nonhomogeneously distributed and are developed due to the weak bond strength between the luting agent and the restoration. This cohesive failure, in turn, results in the weakened unsupported restoration under the masticatory or the occlusal forces.^[28]

Thermocycling of all the samples was also performed to maintain the oral environment. Samples were thermocycled at 5°C and 55°C at 3500 cycles with a dwell time of 5 min resulting in aging of about a year.^[29]

The results of the present study proved that the postetching cleansing affects the μSBS between the lithium disilicate and the resin cement and also affects

the microstructure and the surface topography of the lithium disilicate. Hence, the null hypothesis was rejected.

Limitations of this study are that only one type of resin cement was used. Comparing between different cements could be interesting and a point of further research. Another limitation could be the preload failures before testing the samples. More studies can be done with addition of groups and materials in the future.

Clinical application

The clinical application of the study is that on following these simple postetching cleansing protocols with readily available materials in most clinical practices, the bond strength increases by almost 29%, which, in turn, will enhance the longevity of the bonded lithium disilicate restorations.

CONCLUSION

Within the limitations of the present study, the following conclusions can be drawn:

- Postetching cleansing significantly increases the bond strength between the resin cement and ceramic restoration
- Cleaning with 37% phosphoric acid followed by ultrasonic cleansing for 5 min results in superior bond strength as compared to cleaning with only phosphoric acid or no cleaning at all.

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Conflicts of interest

There are no conflicts of interest.

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Agarwal, *et al.*: Effect of post etching cleansing on lithium disilicate

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