

Effects of Surface Treatment Procedures on Bond Strength of Lithium Disilicate Glass Ceramic

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Objective: To evaluate the microshear bond strength (μ SBS) of resin cement to a lithium disilicate glass ceramic conditioned with different surface treatment procedures.

Methods: Crystallised slices of lithium disilicate glass ceramic were randomly divided into five groups (n = 10) according to different surface treatment procedures: the no surface treatment (NT) group was untreated; the hydrofluoric acid (HF) group was conditioned with 4.5% HF; the silane (S) group was conditioned with a silane coupling agent; the hydrofluoric acid and silane (HFS) group was conditioned with HF followed by the silane coupling agent; and the Monobond Etch & Prime (MEP) (Ivoclar Vivadent, Schaan, Liechtenstein) group was conditioned with the one-step self-etching primer MEP. Resin cement was applied to the ceramic surfaces and irradiated. A µSBS test was performed. Failure analysis, surface roughness tests, surface topography examination and elemental analysis were also conducted. The data were analysed with a one-way analysis of variance (ANOVA) and Tukey honestly significant difference test (P < 0.05).

Results: The MEP group resulted in comparable μ SBS to the HFS group (16.9 ± 4.3 MPa and 16.0 ± 2.2 MPa, respectively), but a significantly higher μ SBS than the NT (1.0 ± 0.9 MPa), HF (8.9 ± 3.9 MPa) and S (12.6 ± 2.5 MPa) groups. Adhesive failure was mainly observed in the NT and HF groups, while the S, HFS and MEP groups demonstrated the most mixed failure. Though micrographs revealed a roughened surface in the HF group, no significant difference was found with any other groups.

Conclusion: Within the limitations of this study, it can be concluded that the μ SBS of resin cement to lithium disilicate glass ceramic etched with MEP is as efficient as that treated with HF and silane.

Key words: bond strength, lithium disilicate glass ceramic, self-etching primer, surface treatment

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This study was supported by Clinical Medicine Plus X – Young Scholars Project, Peking University, the Fundamental Research Funds for the Central Universities and the Chinese Stomatological Association Youth Founding (No. CSAR2018-03); the materials used in the study were supplied by Ivoclar Vivadent. Lithium disilicate glass ceramic is a highly aesthetic material for repairing dental hard tissue defects. Over the past decade, CAD/CAM systems have developed into an efficient and error-free tool to produce accurate dental restorations, which contributes to the popularity of lithium disilicate glass ceramic among dental practitioners^{1,2}. With enhanced physicomechanical properties, the longevity of ceramic restorations depends greatly on the quality of cementation^{3,4}. To improve the bonding performance, pretreatment of the ceramic surface is mandatory. The current strategy includes etching with hydrofluoric acid (HF) and subsequent application of a silane (S) coupling agent. HF can induce surface micro-

Material	Туре	Composition	Manufacturer	Lot no.
IPS e.max CAD	Lithium disilicate glass ceramic	SiO ₂ (57–80 wt%), Li ₂ O (11–19 wt%), K ₂ O (0–13 wt%), P ₂ O ₅ (0–11 wt%), ZrO ₂ (0–8 wt%), ZnO (0–8 wt%), Al ₂ O ₃ (0–5 wt%) MgO (0–5 wt%), colouring oxides (0–8 wt%)	Ivoclar Vivadent	S06031
IPS Ceramic Etching Gel	Hydrofluoric acid etch- ing gel	4.5% hydrofluoric acid	Ivoclar Vivadent	X19943
Monobond N	Silane coupling agent	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	Ivoclar Vivadent	Y08580
Monobond Etch & Prime	Self-etching glass- ceramic primer	Ammonium polyfluoride, silane system based on trimethoxypropyl methacrylate, alcohols, water and colorant	Ivoclar Vivadent	Y12749
Multilink N	Dual cure resin cement	Dimethacrylates, HEMA, barium glass filler, Ba-Al- Fluoro-Silicate glass, ytterbium trifluoride, highly dis- persed silica, catalysts and stabiliser, pigments	Ivoclar Vivadent	Y11346

Table 1 Details of materials used.

roughness by reacting with the glassy matrix and expose the crystalline structure⁵. The microroughness provides micromechanical retention. S coupling agent improves surface wettability and achieves chemical bonding with resin cements⁶.

The increase in bond strength of lithium disilicate glass ceramic etched with HF is evidence-based and well accepted by dental practitioners⁷; however, HF is constantly questioned due to its hazardous effects on human health⁸. It is a volatile chemical and, when accidentally released, can diffuse as a dense vapour and aerosol. It is also highly reactive. Research found that accidental exposure of soft tissues to solutions containing more than 0.2% HF could be harmful⁹. Although no studies have been conducted on the incidence of the hazardous effects of HF in dentistry, caution must be taken when handling and storing it.

A newly launched self-etching ceramic primer named Monobond Etch & Prime (MEP, Ivoclar Vivadent, Schaan, Liechtenstein) aims to simplify the procedure while providing equivalent bond strength compared to the conventional HF and S (HFS) method. Reviewing the available publications, limited evidence of MEP performance can be found¹⁰⁻¹⁸ and, in different experimental setups, the results may be contradicting. In vitro investigations found that the difference in bond strength with lithium disilicate glass ceramic between MEP and the conventional method was not statistically significant^{12,15-16}, but in two papers MEP was less effective than HFS^{10,17}. A more recent study showed that the HFS method was significantly better than MEP even after artificial ageing¹¹. At present, the reaction mechanism of MEP is not very clear. The aim of this study was to investigate the microshear bond strength (uSBS) of MEP to a lithium disilicate glass ceramic. The null

hypothesis was that there was no difference in μ SBS between different surface treatment procedures.

Materials and methods

A lithium disilicate glass ceramic (IPS e.max CAD, Ivoclar Vivadent) was used. According to the manufacturer, HF etching gel (IPS Ceramic Etching Gel, Ivoclar Vivadent) contains 4.5% HF. The S coupling agent used was Monobond N (Ivoclar Vivadent). A one-step self-etching primer (Monobond Etch & Prime) was also used. Resin cement (Multilink N, Ivoclar Vivadent) was dual cured. Further details of the materials used in this study are listed in Table 1.

Specimen preparation and surface treatment

The IPS e. max CAD blocks were cut into 2-mm-thick ceramic slices using a low-speed sectioning machine (SYJ-150, Shenyang Kejing Auto-Instrument, Shenyang, China) under water cooling. The slices were then crystallised (Programat CS2, Ivoclar Vivadent) according to the manufacturer's recommendations. All slices were embedded with epoxy resin and the ceramic was wet polished with up to 600-grit silicon carbide paper discs in a grinder (AutoMet 250, Buehler, Lake Bluff, IL, USA). After being cleaned in an ultrasonic bath (Bio-Sonic UC100, Coltène/Whaledent, Altstatten, Switzerland) with 99.5% isopropyl alcohol for 5 minutes, the specimens were randomly allocated into five groups (n = 10) according to the surface treatments as follows:

• Group 1: No surface treatment (NT) group as the negative control group.

- Group 2: Hydrofluoric acid (HF) group. IPS Ceramic Etching Gel was applied to the surfaces to be etched using a plastic brush and the gel was allowed to react for 20 seconds. It ws then rinsed off the ceramic surfaces under running water and air-dried.
- Group 3: Silane (S) group. A thin layer of Monobond N was applied to the ceramic surfaces using a plastic brush and allowed to react for 60 seconds before any excess was removed with a strong air blow force.
- Group 4: Hydrofluoric acid and silane (HFS) group. IPS Ceramic Etching Gel was applied to the surfaces to be etched using a plastic brush and allowed to react for 20 seconds, before being rinsed from the ceramic surfaces under running water and air-dried. A thin layer of Monobond N was applied to the ceramic surfaces using a plastic brush and allowed to react for 60 seconds before any excess was removed with a strong air blow force.
- Group 5: Monobond Etch & Prime (MEP) group. MEP was applied to the surfaces to be etched using a plastic brush and brushed for 20 seconds. The MEP was allowed to react for 40 seconds, before being rinsed from the ceramic surfaces under running water and air-dried for 10 seconds.

Surface roughness

The surface roughness value (Ra) of each specimen was measured using a surface profilometer (Surftest SJ-401, Mitutoyo, Kanagawa, Japan) with a stress force of 0.75 mN, standard cutoff of 1.0 mm, transverse length of 0.8 mm, amplitude height of 2.5 mm and stylus speed of 0.5 mm/second. Two perpendicular Ra measurements for each specimen, and the numerical mean of these values was reported.

Surface topography examination and elemental analysis

One additional specimen from each group was selected for scanning electron microscope (SEM) analysis. The specimens were sputter-coated with gold and observed with an SEM (EVO 18; Zeiss, Wetzlar, Germany).

Elemental analysis of the untreated and treated surfaces using SEM-coupled energy dispersive X-ray (EDX) analyser (X-act, Oxford Instruments, Abingdon, UK) operated at a take-off angle of 35 degrees with both map and point modes at the same operating voltage. Each ceramic surface was analysed in the centre of the specimen. Compositional element concentrations above 1.0 wt% were recorded.

Cementation procedure

A cylinder transparent polyethylene mould (height 4.0 mm, internal diameter 2.0 mm) was placed on the ceramic surface and Multilink N was injected and packed against the surface using a composite modelling instrument. The cement was light cured (1200 mW/cm², Bluephase, Ivoclar Vivadent) from the top and around the outside, both for 20 seconds. All procedures were performed by the same calibrated operator. All specimens were stored in distilled water at 37°C for 24 hours¹⁹.

μSBS

For the μ SBS test, each specimen was mounted on a μ SBS machine (Shear Bond Tester, Bisco, Schaumburg, IL, USA) at a crosshead speed of 1.0 mm/minute. Bond strength was calculated according to the formula R = F/A, where R is the bond strength in MPa, F is the load at failure in N and A is the cross-sectional interfacial area. After debonding, each specimen was examined under 2.5× magnification to make sure there were no bubbles or defects at the interface.

Failure analysis

The fracture surfaces of specimens were examined by a blinded evaluator using optical microscopy (SZ61, Olympus, Tokyo, Japan) at 25× magnification. Failures were classified as adhesive failure between resin cement and ceramic (A), mixed failure (M), cohesive failure in resin cement (CR) or cohesive failure in ceramic (CC).

Statistical analysis

Statistical analysis was carried out using SPSS (version 20.0, IBM, Armonk, NY, USA). Data for surface roughness and μ SBS were normally distributed and subjected to a one-way analysis of variance (ANOVA) and Tukey honestly significant difference test, with the level of significance set at 0.05.

Results

The mean Ra values are listed in Table 2. No statistical significance was found between the different surface treatment groups. Representative surface morphologies of all groups are presented in Fig 1. The HF group had a porous surface, whereas the MEP group showed a less notable etching pattern. The S group seemed to show no effect on surface roughness, but crystal substances could be observed on the ceramic surface.





 Table 2
 Surface roughness value (Ra, mean ± standard deviation), microshear bond strength (µSBS, mean ± standard deviation) and failure analysis (%) of the tested ceramic with different surface treatments.

Different lowercase letters in each row indicate significant differences within different surface treatment groups (P < 0.05; Tukey honestly significant difference test).



Fig 1 SEM micrograph of IPS e.max CAD surface after (a) no treatment (NT), (b) application of hydrofluoric acid for 20 seconds (HF), (c) application of ceramic primer Monobond N (S), (d) application of HF for 20 seconds followed by Monobond N (HFS), and (e) application of self-etching ceramic primer Monobond Etch & Prime for 60 seconds (MEP).

 μ SBS data and failure analysis are also presented in Table 2. All four surface treatment groups showed significantly higher μ SBS than the NT group. The mean μ SBS for the MEP group was comparable to that of the HFS group, but significantly higher than the HF and S groups. Without surface treatments, adhesive failure accounted for 100%. When HF was applied, the percentage dropped to 70%. The S, HFS and MEP groups had the most mixed failures, and then cohesive failure in resin cement. No cohesive failure in ceramic was found in any of the groups.

The element analysis of surface composition (wt%) of all groups is presented in Table 3. Increased proportions of carbon in the S, HFS and MEP groups revealed silane coupling agent in the surfaces. Minor amounts of residual fluorine were identified in the HF (0.99%) and MEP groups (4.19%).

Discussion

The study evaluated the μ SBS of different surface treatment procedures with lithium disilicate glass ceramic using a μ SBS test. Surface roughness testing, SEM and EDX were also performed to investigate the surface morphology. MEP presented a μ SBS value similar to the conventional HFS group, but a significantly higher value than the HF and S groups; thus, the null hypothesis was partially rejected.

Lithium disilicate glass ceramics are frequently used in conservative restorations like inlays, onlays and veneers. As bonding techniques have improved, these restorations have long-lasting functions²⁰. Bonding performance includes complicated interactions between ceramic, luting cement and tooth substrates. Without pretreatment, bond strength with ceramic can be very low, and this was confirmed in the present study (negative control group 1.0 MPa) and others^{12,15}. Multiple pretreatment methods were explored, for example air abrasion with alumina or silica-coated alumina particles, acid etching with different solutions and treatment with monomers and silane coupling agents, but none presented satisfactory bonding²¹⁻²³. Currently, a combination of HF and S is considered the gold standard⁷. By selectively dissolving the glass phase in silicate ceramics, HF etching leads to a micromorphological 3D porous surface. SEM showed that the HF group had a rougher surface than any other groups. These pores allow silane coupling agents and/or luting cements to infiltrate and obtain micromechanical retention²⁴. HF is efficient and easy to use chairside²¹; however, its hazardous effect on human health⁸ and potential impairment of bond strength and mechanical properties need be considered²⁵⁻²⁷.

Unlike HF, a milder acid composition named ammonium polyfluoride is added in MEP. A previous study showed that ammonium polyfluoride acted like HF for a shorter time or in a lower concentration²⁸; thus, concerns arose about MEP with a latent weaker etching pattern. Although there was no significant difference, the Ra for the MEP group was lower than the

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Table 3 EDX analysis of major components of element surface composition (wt%) of the tested ceramic materials with ceramic with different surface treatments.

Surface treatment	Element composition (wt%)							
	С	0	Si	F	Al	К		
NT	2.50	53.13	36.93	0.00	1.80	3.51		
HF	2.82	53.03	35.30	0.99	1.79	3.55		
S	30.33	39.78	24.54	0.00	1.09	2.14		
HFS	22.59	42.78	28.42	0.00	1.33	2.66		
MEP	15.41	43.98	31.53	4.19	1.97	2.91		

HF group. This result was coincident with a previous study¹². Another study employed atomic force microscopy (AFM) to evaluate surface roughness and found that HF and MEP resulted in significantly different Ra values, but the sampling was rather limited, usually a 20- \times 20-µm region¹⁰. Thus, AFM is regarded more as a qualitative evaluation method²⁹. In our study, SEM micrographs also confirmed a smoother surface in the MEP group than the HF group.

With ammonium polyfluoride, an S system based on trimethoxypropyl methacrylate is incorporated into MEP. EDX data showed an increase of carbon in the MEP group, which confirmed a silanised ceramic surface. Unlike the S group with a crystal substance shown in Fig 1, MEP left a thin, uniform silane layer on the surface demonstrated by SEM micrographs, and this was coincident with other research¹⁰. µSBS values were not influenced by surface morphology and were comparable between the HFS and MEP groups. Meanwhile, a minor proportion of fluorine was noticed after the surface was treated with MEP. As no other cleaning methods such as an ultrasonic bath were used but running water wash-off, the residual fluorine was speculated to be insoluble silica-fluoride salts produced by ammonium polyfluoride having reacted with the glass matrix in the ceramic and trapped in the silane layer³⁰. Although the clinical relevance of residual fluorine is not clear, the µSBS value was not affected within the limitations of this study, and similar conclusions have been drawn by others³⁰⁻³¹.

This study has several limitations. The procedures used did not completely simulate a clinical setting. Occlusal forces are more complicated in vivo, and μ SBS investigations in vitro can deviate from true shear force measurements due to a nonuniform distribution of stress³². Further in vitro and in vivo studies should be performed on MEP.

Conclusion

Within the limitations of this study, it can be concluded that the μ SBS of resin cement to lithium disilicate glass

ceramic etched with MEP is as efficient as that treated with HF and silane.

Conflicts of interest

The authors declare no conflicts of interest related to this study.

Author contribution

Dr Peng YU designed the study, conducted the experiments, collected the data, undertook the statistical analysis and prepared the manuscript; Dr Xiao Yan WANG designed the study and revised the manuscript.

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