Original Article

Self-etching Ceramic Primer Protocol Provides Efficient Shear Bond Strength and Durability Between Lithium Disilicate Glass-ceramic and Resin Cement: A Potential Alternative to the Conventional Hydrofluoric Acid Protocol

Received: May 31, 2024
Revised: June 26, 2024
Accepted: September 23, 2024

Corresponding Author:
Terawat Tosiriwatanapong, Division of Prosthodontics, Faculty of Dentistry, Thammasat University, Pathum Thani 12120, Thailand
E-mail: terawatt@bu.edu

Top Chitkraisorn [1,2], Nontawat Chuinsiri[3], Chawin Aungkatawiwat[1], Weerachai Singhatanadgit[4], Terawat Tosiriwatanapong[1,2]

[1]Division of Prosthodontics, Faculty of Dentistry, Thammasat University, Thailand
[2]Research Unit in Remineralized Tissue Reconstruction, Thammasat University, Thailand
[3]Institute of Dentistry, Suranaree University of Technology, Thailand
[4]Division of Oral and Maxillofacial Surgery, Faculty of Dentistry, Thammasat University, Thailand

Abstract

Objectives: This study investigated the effects of two surface treatment protocols on the shear bond strength, bond durability, and failure mode at the interface between lithium disilicate glass-ceramic and resin cement. The protocols compared were a self-etching ceramic primer and the conventional hydrofluoric acid (HF) etching followed by silane.

Methods: Fifty lithium disilicate specimens were randomly divided into five surface treatment groups (n=10 each). A control group received no treatment. The remaining 4 groups included: 5% HF etch with Monobond Plus thermocycled and non-thermocycled, Monobond Etch & Prime (MEP), thermocycled and non-thermocycled. Microshear bond strength (microSBS) was assessed before and after thermocycling to evaluate bond durability. Failure modes (adhesive, mixed, cohesive in resin/ceramic) were recorded under a stereomicroscope.

Results: Both surface treatment protocols exhibited comparable microSBS for both pre- and post-thermocycling results. Moreover, bone durability obtained from the two treatment protocols seemed to be comparable. Most groups displayed adhesive/mixed failures. Notably, the self-etching ceramic primer group showed cohesive failure in half of the specimens initially, persisting in 20% after thermal aging.

Conclusions: Compared with the conventional HF protocol, the self-etching ceramic primer protocol provided similar microSBS and bond durability between lithium disilicate glass-ceramic and resin cement. The data suggest a self-etching ceramic primer is a viable option for the conventional HF protocol in bonding to glass-ceramic, minimizing the HF hazard and simplifying the clinical procedure.

Keywords: bond durability, bond strength, hydrofluoric acid, lithium disilicate, self-etching ceramic primer

Oral Sci Rep. 2024; 45(3): 45-51

Introduction

Current protocols for glass-ceramic surface treatment typically involve hydrofluoric acid (HF) etching followed by silane application. This multi-step process enhances bonding through a combination of chemical and micromechanical mechanisms. HF etching roughens the ceramic surface, increasing its surface area for mechanical interlocking with resin cements, and it also promotes the formation of hydroxyl groups on the ceramic surface, facilitating a chemical bond with silane coupling agents.[1] Silane molecules possess a bifunctional structure, with one end capable of covalently bonding to the hydroxyl groups on the ceramic and the other end designed to interact with the resin cement, effectively creating a chemical bridge at the interface.[2] Numerous studies have demonstrated the synergistic effect of this combined approach, with HF etching followed by silane application consistently yielding optimal bond strengths between ceramic and resin cements.[3,4]

HF etching is a process known to be hazardous due to its potential for oral mucosa and cutaneous burns.[5] Prolonged etching time with hydrofluoric acid could jeopardize the flexural strength of the restorative material.[6,7] To address these safety concerns, self-etching ceramic primers have been introduced as possible alternatives.[4] The composition of one currently available commercial self-etching ceramic primer product contains ammonium polyfluoride, which reacts with ceramic surface to create a rough etching pattern, and also contains trimethoxypropylmethacrylate -functionalized silane, according to the manufacturer's specifications.[8] A self-etching ceramic primer chemically modifies the ceramic surface to enhance bonding, eliminating the need for separate HF etching and silanization steps.[4,8] This approach not only improves safety but also reduces treatment time and potentially reduces patient discomfort during the bonding procedure. However, studies have yielded mixed results regarding the bond strength achieved with silane-containing self-etching primers compared to conventional HF-etching and silanization. Some investigations report comparable bond strengths[9-11], while others observe a slight decrease[3] or a statistically significant reduction.[12-15] Therefore, this *in vitro* study investigated the effects of two surface treatment. protocols on the microshear bond strength and durability of resin cement to lithium disilicate glass-ceramics. The protocols compared were: (1) conventional HF etching followed by silane application protocol and (2) self-etching ceramic primer application protocol. Thermal aging simulation was used to determine their bond durability. The null hypothesis postulated no significant differences in microshear bond strength and durability between the two surface treatment protocols for bonding resin cement to lithium disilicate glass-ceramics.

Materials and Methods

Specimen preparation

A total of four CAD/CAM blocks (12×14×18 mm) of lithium disilicate glass ceramic (IPS E.max® CAD; Ivoclar Vivadent, Schaan, Liechtenstein) were used in this study. The blocks were cut into 50 rectangular sections (12×7×2 mm) using a diamond wheel saw (Accutom-50, Struers) under water irrigation. Afterwards, the ceramic specimens were fired according to the crystallization program (P161 Crystallization LT/MT/HT) in a furnace (Programmat P310, Ivoclar Vivadent) as recommended by the manufacturer. After cooling, the specimens were cleaned ultrasonically and mounted on polyvinyl chloride (PVC) rings filled with acrylic resin, displaying the specimen surface on the top of the cylinder, with a height of 1 mm. The mounted specimens were cleaned ultrasonically in isopropyl alcohol for 60 seconds.

Surface treatment & storage conditions

The specimens were randomly assigned into 5 groups, each consisting of 10 specimens based on surface treatment and storage conditions. Material compositions are shown in Table 1. The five surface treatment groups were listed as follows:

Group 1. Control: no treatment (NT)

Group 2. Conventional HF & silane protocol: etched with <5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent) for 20 seconds, thoroughly rinsed with water spray until the red color was removed, dried with oil-free air for 20 seconds, followed by the application of Monobond Plus for 60 seconds using a microbrush, and dispersed remaining excess with oil-free air for 10 seconds, specimens were stored in distilled water at 37°C for 24 hours

Group 3. Conventional HF & silane protocol +TC: same as Group 2 but stored under thermal cycling conditions, cycled between 5°C and 55°C for 10,000 cycles with 5-second dwell times

Group 4. Self-etching ceramic primer protocol: treated with self-etching ceramic primer (Monobond Etch & Prime, MEP; Ivoclar Vivadent) by agitating onto the ceramic surface for 20 seconds using a microbrush, left the agent for another 40 seconds to react, thoroughly rinsed with water spray until the green color was removed, and dried with oil-free air for 10 seconds, specimens were stored in distilled water at 37°C for 24 hours

Group 5. Self-etching ceramic primer protocol +TC: same as Group 4 but stored under thermal cycling conditions, cycled between 5°C and 55°C for 10,000 cycles with 5-second dwell times.

Resin cement application

After the surface treatment (NT, HF, or MEP), Tygon® tubes with an internal diameter of 0.8 mm and a height of 1 mm were positioned over the ceramic surface using perforated adhesive tape as a stabilizer to prevent movement during resin cement application. One operator positioned two tubes on each ceramic surface followed by the application of resin cement (Multilink N, Ivoclar Vivadent, Schaan, Liechtenstein). The resin cements were light-cured for 40 seconds using a LED light-curing unit with an output of 1,200 mW/cm². After each mode of storage, the tubes were carefully removed with a sharp scalpel blade. Each specimen was examined for any defects in the resin cylinders, and no porosities or gaps at the interface were observed. The procedures were carried out under 3.5X magnifying loupes.

Microhear bond strength test

The PVC rings were mounted in a universal testing machine (EZ-test-50N, Shimadzu Co, Japan). The shear blade was positioned close to the resin cylinder and the load was applied at a constant crosshead speed of 1 mm/minute until failure occurred. The microSBS values (MPa) were calculated by dividing the load at failure by the surface area (mm²).

Failure mode analysis

After the microSBS test, the ceramic surfaces of all debonded specimens were examined under a stereomicroscope (SMZ1000, Nikon, Japan) at x40 magnification to determine the failure modes. Failure modes were classified into four types: adhesive failure (AD), characterized by separation at the interface between the resin cement and ceramic; mixed failure (M), combining a combination of adhesive and cohesive failure; cohesive failure within the resin cement (CR); and cohesive failure in ceramic (CC).

Table 1: The products used in this study.

Product	Composition	Manufacturer
1. Ceramic		
IPS e.max CAD	SiO_2, Li_2O, K_2O, MgO, ZnO_2, Al_2O_3, P_2O_5 and other oxides	Ivoclar Vivadent, Schaan, Liechtenstein
2. Etchant		
IPS Ceramic Etching Gel	4.9% HF acid, water, colorant (pH=2)	Ivoclar Vivadent, Schaan, Liechtenstein
3. Silane		
Monobond Plus	Silane methacrylate, phosphoric acid methacrylate, sulphide methacrylate, ethanol	Ivoclar Vivadent, Schaan, Liechtenstein
4. Self-etching ceramic primer		
Monobond Etch and Prime (MEP)	TADF, silane methacrylate, BTSE, methacrylated phosphoric acid ester, butanol, water, colorant (pH=3.7)	Ivoclar Vivadent, Schaan, Liechtenstein
5. Resin cement		
Multilink N	Monomer matrix: HEMA, Dimethacrylate Inorganic fillers: 0.25–3.0 µm of barium glass, ytterbium trifluoride, spheroid mixed oxide	Ivoclar Vivadent, Schaan, Liechtenstein

** According to the manufacturers' information. TADF: Tetrabutyl ammonium dihydrogen trifluoride, BTSE: Bis(triethoxysilyl)ethane, HEMA: 2-Hydroxyethyl Methacrylate.*

Statistical analyses

The microSBS values were expressed as the mean ± standard deviation (SD) (N=10 per group). Inter-group comparisons of microSBS were performed using a one-way analysis of variance (ANOVA) with Tukey's post hoc comparison. Failure modes were analyzed descriptively, with percentages used to quantify their prevalence. Fisher's exact test has been used to find an association between type of primer type and failure mode. A significance level of α=0.05 was employed. Statistical analyses were conducted using the statistical software package SPSS version 28.0 (SPSS Inc.).

Results

Mean microSBS values are depicted in Table 2. For the control no surface treatment group, resin cements debonded in six specimens before the microSBS test. The mean microSBS was significantly lower than 1 MPa. After storage in distilled water at 37°C for 24 hours, both surface treatment protocols exhibited comparable initial non-aged microSBS (12.39 MPa for the conventional HF & silane group vs 11.92 MPa for the self-etching ceramic primer group). After 10,000 cycles of thermal aging, the microSBS values obtained from both protocols were still statistically similar (13.48 MPa for the conventional HF & silane group vs 10.36 MPa for the self-etching ceramic primer group). Considering the thermal aging process, no significant differences in microSBS were observed between the groups with and without TC within the same surface treatment protocol (Table 2).

There were 3 types of failure mode found as shown in Figure1. The frequency distribution of the resulting failure modes following microSBS testing is shown in Table 3. The control group, with no surface treatment, exhibited 100% adhesive failure. Adhesive or mixed modes of failure were common in all groups except for the self-etching ceramic protocol group. Half of the self-etching ceramic primer-treated specimens stored in distilled water at 37°C for 24 h exhibited cohesive failure in the resin cement, which was still observed in 20% of the specimens after thermal aging. Fisher's exact test revealed a correlation between types of failure mode and the groups tested.

Discussion

This study investigated the efficacy of a user-friendly and less hazardous self-etching ceramic primer for surface pretreatment of lithium disilicate, a commonly used glass-ceramic material. The aim was to compare its bonding effectiveness to the traditional HF etching method. The microSBS testing was employed to assess bonding performance. Results indicated that the self-etching ceramic primer protocol achieved microSBS values, both

Table 2: Mean microSBS values (MPa) of the five experimental groups.

Experimental groups	microSBS (mean ± SD)
1. Control (untreated surface)	0.96±1.02A
2. Conventional HF & silane protocol	12.39±5.55B
3. Conventional HF & silane protocol + TC	13.48±5.18B
4. Self-etching ceramic primer protocol	11.92±5.17B
5. Self-etching ceramic primer protocol + TC	10.36±3.6B

Different superscript letters indicate statistically significant difference. TC: thermocycling.

Table 3: Frequency distribution of failure modes (%) of the five experimental groups.

Experimental groups	Failure modes (%)			
	AD	M	CR	CC
1. Control (untreated surface)	100	0	0	0
2. Conventional HF & silane protocol	20	70	10	0
3. Conventional HF & silane protocol + TC	70	30	0	0
4. Self-etching ceramic primer protocol	40	10	50	0
5. Self-etching ceramic primer protocol + TC	40	40	20	0

AD, adhesive failure; M, mixed failure; CR, cohesive failure in resin cement; CC, cohesive failure in ceramic.

initial and aged, comparable to the conventional HF and silane protocol for bonding to lithium disilicate glass-ceramic. Consequently, the null hypothesis, which posited that the self-etching primer would provide microSBS and bond durability similar to the recommended HF and silane protocol, cannot be rejected.

Established literature demonstrates a positive correlation between adhesion and the reinforcement of esthetic indirect restorations achieved through adhesive techniques. Conversely, insufficient bonding between the restoration and resin cement may lead to an uneven distribution of stresses. This non-uniform stress distribution can culminate in the resin cement layer and weaken the unsupported restoration under occlusal forces, ultimately leading to restoration failure.(4) Although HF etching followed by silane application has long been used with great success for bonding to glass-ceramic substrates, this surface treatment protocol using HF, which is highly hazardous and toxic(5), did not provide significantly higher bond performance, at least in terms of microshear bond strength and durability, as demonstrated in the present study. The present results provide evidence supporting the potential clinical success of the self-etching ceramic primer comparable to the recommended HF-containing procedure, as previously reported.(9-11) According to the manufacturer's documentation(8), tensile bond strength, shear bond strength and aging resistance obtained from the application of self-etching ceramic primer are comparable to the conventional combination of hydrofluoric acid etching and Monobond Plus application but superior to other universal adhesives. From El-Damanhoury's study(3), the etching pattern created by self-etching ceramic primer had less surface roughness and a less dominant etching pattern compared to conventional hydrofluoric acid, while the surface morphology was more uniform. Despite this reduced roughness, it seems to be sufficient for retention, and the ceramic surface does not become over-etched after prolonged exposure. Several studies(3,4), evaluate the effect of self-etching ceramic primer at different durations (60, 100 and 140 seconds). The result shown that there were no significant changes on surface morphology on glass ceramic and no impact on shear bond strength. Evaluating the impact of thermal aging, no statistically significant differences in microSBS bond strengths were observed between groups with and without thermocycling within the same surface treatment protocol. This finding suggests that both surface treatment protocols achieve comparable bond durability following *in vitro* simulation of thermal aging. These encouraging results justify further clinical investigations to confirm the clinical relevance of the self-etching ceramic primer.

Although HF reacts with the matrix, such as glass-ceramics, that contains the silica, generating a micro-mechanically retentive surface and promoting hydroxyl group formation on the ceramic surface for enhanced bonding(16), well-controlled HF etching technique is crucial to achieving optimal bonding performance. Previous studies suggested that prolonged HF etching times negatively affect the flexural strength of the materials(6,7) and that neither an increase in etching time nor concentration resulted in a statistically significant enhancement of bond strength.(17) The observed similarity in bond performance between the traditional HF-etching protocol and the single-step self-etching ceramic primer protocol, despite the latter's less technique-sensitive nature, may be attributed to the technique sensitivity of the former. Moreover, thermocycling conditions may play an important role in the bond performance obtained from a previous study(3) that reported conflicting results with the present study. That study employed long dwell times of about 30 seconds in each temperature, while the present study used only 5-second dwell times in each temperature, which has been

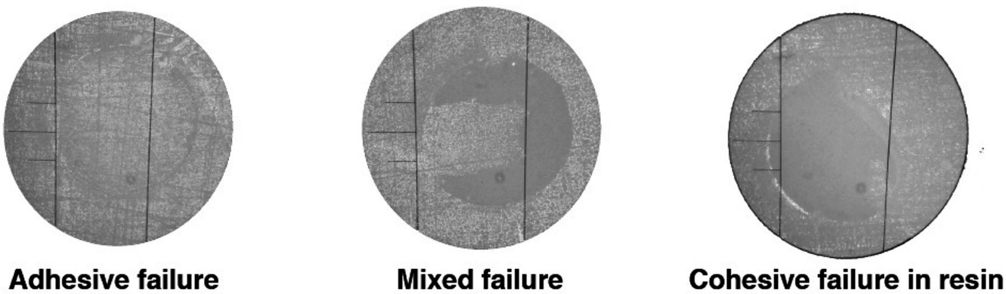

Figure 1: Representative sterio microscopic images of three fracture modes: adhesive failure (AD), mixed failure (M) and cohesive failure within the resin cement (CR).

proposed to simulate more accurately the actual changes of temperature occurring in the oral cavity since no patients would be able to tolerate an extreme temperature for an extended period of time.[18,19] The number 10,000 cycle can represent approximately one year of intraoral aging.[19] This could provide us with more information on the long term performance of self-etching ceramic primers. Future investigation into the underlying bond mechanisms of both protocols would undoubtedly shed light on achieving strong and durable resin-ceramic bonds.

Interestingly, the self-etching ceramic primer protocol exhibited a lower incidence of adhesive failure compared to the conventional HF & silane protocol. This observation held true for both freshly prepared and aged specimens. These failure pattern results obtained from both initial and thermally aged specimens suggest that the self-etching ceramic primer-treated surface provides a strong and durable resin-ceramic bond. Further studies focusing on the analysis of failure patterns obtained in the present study are important to gain insights into the potential success or failure of this alternative surface treatment approach in clinical settings. Although a cohesive failure within resin cement generally signifies that the resin cement's adhesion to the ceramic surpasses the resin cement's cohesive shear strength, further investigations are warranted to elucidate the mechanisms underlying the observations presented in this study.

The limitations of this study included the use of only one type of artificial aging and ceramic material. Future research should incorperate additional types of artificial aging, beyond thermal cycling, and conduct more clinical study to evaluate the long-term performance of this seft-etching ceramic primer protocal.

Conclusions

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

1. For bonding to lithium disilicate glass-ceramic, the self-etching ceramic primer protocol performed similarly to the HF & silane conventional protocol in terms of initial and aged microSBS.

2. Thermal aging did not affect microSBS obtained from both self-etching ceramic primer and HF & silane conventional protocols.

3. Compared to the conventional HF & silane protocol, the self-etching ceramic primer protocol resulted in lower prevalence of adhesive failure for both initial and aged specimens after thermal aging.

Acknowledgments

The authors gratefully acknowledge the financial support from the Faculty of Dentistry, Thammasat University, and the Thammasat University Research Unit in Remineralized Tissue Reconstruction. We also express our sincere gratitude to Dr. Nichamon Chaianant for her advice on statistical analysis.

Conflicts of Interest

The authors declare no conflict of interest.

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