

# Bonding of the silane containing multi-mode universal adhesive for lithium disilicate ceramics

Hyun-Young Lee<sup>1</sup>,  
Geum-Jun Han<sup>2</sup>, Juhea  
Chang<sup>3</sup>, Ho-Hyun Son<sup>1\*</sup>

<sup>1</sup>Department of Conservative Dentistry, <sup>2</sup>Department of Dental Biomaterials Science, Seoul National University School of Dentistry and Dental Research Institute, Seoul, Korea

<sup>3</sup>Special Care Clinic, Seoul National University Dental Hospital, Seoul, Korea

**Objectives:** This study evaluated the influence of a multi-mode universal adhesive (MUA) containing silane (Single Bond Universal, 3M EPSE) on the bonding of resin cement to lithium disilicate. **Materials and Methods:** Thirty IPS e.max CAD specimens (Ivoclar Vivadent) were fabricated. The surfaces were treated as follows: Group A, adhesive that did not contain silane (ANS, Porcelain Bonding Resin, Bisco); Group B, silane (S) and ANS; Group C, hydrofluoric acid (HF), S, and ANS; Group D, MUA; Group E, HF and MUA. Dual-cure resin cement (NX3, Kerr) was applied and composite resin cylinders of 0.8 mm in diameter were placed on it before light polymerization. Bonded specimens were stored in water for 24 hours or underwent a 10,000 thermocycling process prior to microshear bond strength testing. The data were analyzed using multivariate analysis of variance ( $p < 0.05$ ). **Results:** Bond strength varied significantly among the groups ( $p < 0.05$ ), except for Groups A and D. Group C showed the highest initial bond strength ( $27.1 \pm 6.9$  MPa), followed by Group E, Group B, Group D, and Group A. Thermocycling significantly reduced bond strength in Groups B, C, and E ( $p < 0.05$ ). Bond strength in Group C was the highest regardless of the storage conditions ( $p < 0.05$ ). **Conclusions:** Surface treatment of lithium disilicate using HF and silane increased the bond strength of resin cement. However, after thermocycling, the silane in MUA did not help achieve durable bond strength between lithium disilicate and resin cement, even when HF was applied. (*Restor Dent Endod* 2017;42(2):95-104)

**Key words:** Lithium disilicate; Microshear bond strength; Resin cement; Silane; Universal adhesive

Received July 11, 2016;  
Accepted December 6, 2016

Lee HY, Han GJ, Chang J, Son HH

\*Correspondence to

Ho-Hyun Son, DDS, MSD, PhD.  
Professor, Department of  
Conservative Dentistry, Seoul  
National University Dental Hospital,  
Seoul National University School  
of Dentistry and Dental Research  
Institute, 101 Daehak-ro, Jongro-  
gu, Seoul, Korea 03080  
TEL, +82-2-2072-2651; FAX, +82-  
2-2072-3859; E-mail, hhson@snu.  
ac.kr

## Introduction

All-ceramic restorations have gained popularity because of their biocompatibility and translucency as well as good esthetics.<sup>1,2</sup> The chosen material for all-ceramic restorations has shifted from pressed ceramic to monolithic ceramic to improve the mechanical properties. Monolithic ceramic, lithium disilicate, is popular because it provides good esthetics and better chipping fracture resistance relative to non-monolithic materials such as porcelain-veneered zirconia.<sup>3</sup> It also has greater strength than other ceramic materials such as leucite glass and metal ceramics.<sup>4</sup>

The clinical outcomes of ceramic restoration do not depend only on the properties of the material, but also on the resin-ceramic bond. Strong and durable resin bonding increases retention,<sup>5,6</sup> improves marginal adaptation,<sup>7,8</sup> reduces microleakage<sup>8,9</sup> and enhances fracture resistance.<sup>10</sup> This resin-ceramic bond is created through

micromechanical retention and chemical bonding to a silica-based ceramic surface.<sup>11,12</sup> To produce micromechanical retention, the surface is prepared by airborne-particle abrasion and/or etching with hydrofluoric acid (HF). However, airborne-particle abrasion is not recommended due to a significant reduction in the flexural strength of IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein).<sup>13</sup> HF etching dissolves the glass phase from the matrix, thus creating micro-undercuts and increasing the surface area.<sup>14</sup> Chemical bonding between the resin-ceramic surfaces can be achieved using a silane coupling agent. Silane is a bifunctional molecule that promotes adhesion via covalent bonds with hydroxyl (OH) groups on the ceramic surface.<sup>15</sup> One functional group can react with the inorganic ceramic surface and the other is a methacrylate group capable of reacting with an organic resin matrix.<sup>16</sup>

Recent trend in adhesive dentistry is to simplify bonding procedures by reducing the application steps.<sup>17</sup> Accordingly, many manufacturers have introduced new single-bottle adhesives called 'universal' or 'multi-mode universal' adhesives (MUAs). They contain many ingredients, such as bisphenol A glycidyl methacrylate (Bis-GMA), hydroxyethyl methacrylate (HEMA), 10-methacryloyloxydecyl dihydrogen phosphate (MDP), and/or silane. However, MDP and silane are usually not included in conventional ceramic adhesives. As MDP is a versatile amphiphilic functional monomer, it is the most important component in MUAs for practical use. MDP has the potential to bond chemically to metals,<sup>18</sup> zirconia,<sup>19</sup> and tooth tissue.<sup>20</sup> Furthermore, it possesses the 'ideal' bonding agent property, that the polar phosphate group of the functional monomer is initially hydrophilic, but MDP becomes more hydrophobic once polymerized.<sup>21</sup> Several studies investigated the bond strength of MUAs

applied on several materials such as enamel,<sup>22</sup> dentin,<sup>23</sup> zirconia,<sup>24,25</sup> and ceramics.<sup>10,26</sup> The manufacturer of MUAs containing silane proposes improved bonding to glass ceramics or resin composites without additional priming procedures. However, little is known regarding the bonding efficiency of MUAs to lithium disilicate ceramic with thermocycling compared to when silane and adhesive were used separately.

The purpose of our current study was to investigate the effects of silane containing MUA on the bonding of resin cement to lithium disilicate ceramic using the microshear bond strength ( $\mu$ SBS) test. The null hypotheses tested were: (1) silane containing MUA does not increase the bond strength between lithium disilicate and resin cement compared to when silane and adhesive are used separately and (2) thermocycling does not affect  $\mu$ SBS between lithium silicate ceramic and resin cement.

## Materials and Methods

### Specimen preparation

Thirty 12 mm × 14 mm × 5 mm IPS e.max CAD blocks were fabricated (Table 1). The blocks were sintered in a furnace (Horizon Press, ShenPaz Dental Ltd., Migdal Haemek, Israel) according to the manufacturer's instructions. After cooling, each specimen was embedded into an acrylic resin block. The lithium disilicate surfaces were sequentially polished with 120, 220, and 500 grit silicon carbide paper using an automatic polishing machine (Rotapol-V, Struers, Ballerup, Denmark) under water cooling. The specimens were treated with 10% citric acid<sup>27</sup> and cleaned in an ultrasonic water bath for 10 minutes to remove the smear layer, and then dried under vacuum for 24 hours.

**Table 1.** Materials used in the study

Product	Manufacturer	Main components*
IPS e.max CAD	Ivoclar Vivadent	Lithium disilicate
Ceramic etching gel	Ivoclar Vivadent	5% hydrofluoric acid
Bis-Silane	Bisco	Ethanol, silane
Porcelain Bonding Resin	Bisco	Bis-GMA, UDMA, TEGDMA
Single Bond Universal	3M ESPE	Organophosphate monomer (MDP), Bis-GMA, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane
NX3	Kerr	7,7,9-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate, TEGDMA, HEMA
Filtek Z250	3M EPSE	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA, silane-treated ceramic

Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; Bis-EMA, bisphenol A ethoxylated dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate.

\*As provided by the manufacturers.

**Surface treatment of lithium disilicate blocks**

The specimens were randomly divided into five groups (Figure 1). The lithium disilicate surface of each specimen was treated as follows:

Group A (ANS, control): an adhesive that did not contain silane (ANS, Porcelain Bonding Resin, Bisco Inc., Schaumburg, IL, USA) was applied with a microbrush.

Group B (S + ANS): silane (S, Bis-Silane, Bisco Inc.) was applied and air-dried, followed by application of ANS as above.

Group C (HF + S + ANS): 5% HF (Ceramic Etching Gel, Ivoclar Vivadent) was applied for 20 seconds, rinsed with distilled water for 1 minute, and air-dried. Then, S and ANS were applied in the same manner as in Group B.

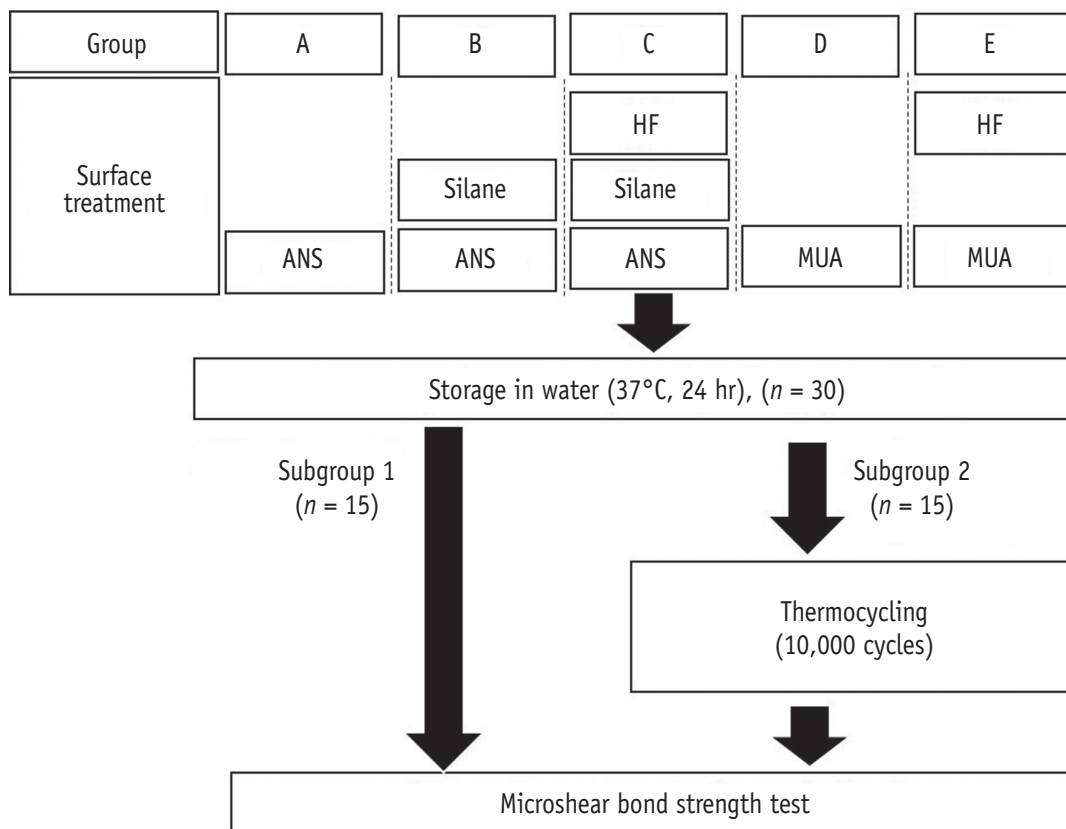
Group D (MUA): The surface was treated with MUA (Single Bond Universal, 3M EPSE, St. Paul, MN, USA) and agitated via scrubbing with a microbrush for 20 seconds. It was

then gently air-dried for 5 seconds.

Group E (HF + MUA): 5% HF was applied for 20 seconds, rinsed with distilled water for 1 minute, and air-dried. Then, the surface was treated with MUA in the same manner as in Group D.

**Cementation of the composite to the ceramic**

Dual-cure resin cement that did not contain amine (NX3, shade clear, Kerr Corp., Orange, CA, USA) was applied and pre-cured composite resin cylinders (Filtek Z250, 3M EPSE) with diameters of 0.8 mm were placed on six treated ceramic surfaces in each group under a fixed load of 0.4 N. After excess cement was removed with microbrushes, the resin cement was light-cured for 40 seconds with an LED light-curing machine at wavelengths of 430 - 490 nm (Be Lite, B&L Biotech, Ansan, Korea).



**Figure 1.** Experimental design of the study. HF, Hydrofluoric acid; ANS, adhesive that does not contain silane (Porcelain Bonding Resin, Bisco); MUA, Multi-mode universal adhesive (Single Bond Universal, 3M EPSE).

### Storage of the specimens

All specimens were stored in distilled water at 37°C for 24 hours and were divided randomly into two subgroups. Half were subjected to  $\mu$ SBS testing ( $n = 15$ ), and the remainder were underwent for 10,000 thermal cycles with a dwell time of 24 seconds and transfer time of 6 seconds between 5 and 55°C water baths and subjected to  $\mu$ SBS testing ( $n = 15$ ).

### Microshear bond strength testing

The specimens were mounted in the jig of a universal testing machine (UTM, LF Plus, Lloyd Instruments Ltd., Fareham Hampshire, UK). A wire of 0.2 mm in diameter was looped around the resin composite cylinder as closely as possible to the bonded interface. The shear force was applied at a cross-head speed of 0.5 mm/min until failure occurred. If premature failure occurred before bond strength testing, the bond strength was recorded as 0 MPa.

### Failure analysis

After  $\mu$ SBS testing, the fractured interfaces of the specimens were observed using a stereomicroscope (SZ-PT, Olympus Co., Tokyo, Japan) at  $\times 40$  magnification to determine the failure mode. The failure mode was classified as 'adhesive failure' when it occurred between the ceramic and the resin cement, 'cohesive failure' when it occurred within the ceramic or resin, and 'mixed failure' when a combination of adhesive and cohesive failures occurred. The representative fractured specimens were examined in a field-emission scanning electron microscope (FE-SEM, S-4700, Hitachi High technologies Co., Tokyo, Japan) operated at 15 kV.

### Microscopic observation of bonded interfaces

To observe the bonded interfaces among the lithium disilicate ceramic, adhesive, and resin cement, 3 mm  $\times$  5 mm  $\times$  14 mm IPS e.max stick specimens were prepared. The ceramic surfaces were treated according to the procedures for each group. The resin cement was applied and light-cured for 40 seconds. All specimens were stored in water for 24 hours, and half were subjected to 10,000 cycles of thermocycling. To observe the bonding quality, the middle point of each stick specimen was fractured in compression mode with a UTM. The fractured surfaces of the sticks were observed by the same FE-SEM.

### Statistical analysis

Bond strength data were statistically analyzed using multivariate analysis of variance with statistical software (SPSS version 18.0, SPSS Inc., Chicago, IL, USA). Multiple comparisons were performed using the Tukey's *post hoc* test, where a  $p$  value less than 0.05 was considered statistically significant.

### Results

The mean bond strength values and standard deviations are presented in Table 2. Before thermocycling, the bond strength of Group A ( $1.35 \pm 1.12$  MPa) was the lowest. The bond strength of Group D ( $1.53 \pm 0.61$  MPa) was similar to that of Group A. Groups B ( $8.66 \pm 2.83$  MPa), C ( $27.14 \pm 6.85$  MPa), and E ( $21.37 \pm 5.08$  MPa), which were treated with either HF or silane, showed higher bond strength of the resin cement to the ceramic surface than Group A ( $p < 0.05$ ).

During the thermocycling procedure, all specimens of Groups A and D were spontaneously debonded. After

**Table 2.** Microshear bond strength after different surface treatments on lithium disilicate

Group	Treatment	Bond strength (MPa)		Reduction rate of bond strength (%)
		24 hr	Thermocycling	
A	ANS	$1.35 \pm 1.12^{aA}$	$0.00 \pm 0.00^{aB}$	100.0
B	S + ANS	$8.66 \pm 2.83^{bA}$	$2.68 \pm 1.43^{bB}$	69.1
C	HF + S + ANS	$27.14 \pm 6.85^{cA}$	$13.08 \pm 3.80^{cB}$	51.8
D	MUA	$1.53 \pm 0.61^{aA}$	$0.00 \pm 0.00^{aB}$	100.0
E	HF + MUA	$21.37 \pm 5.08^{dA}$	$3.13 \pm 1.82^{dB}$	85.4

Different superscript lowercase letters in the same column indicate significant differences; different superscript capitalized letters in the same row indicate a significant difference.

Reduction rate of bond strength (%) =  $([\text{bond strength after water storage for 24 hours} - \text{bond strength after thermocycling}] / \text{bond strength after water storage for 24 hours}) \times 100$

ANS, adhesive that did not contain silane; S, silane; HF, hydrofluoric acid; MUA, Multi-mode universal adhesive.

thermocycling, the bond strengths decreased in Groups B, C, and E ( $p < 0.05$ ). Group C showed the highest bond strengths regardless of the storage conditions ( $p < 0.05$ ).

The distribution of failure modes after  $\mu$ SBS testing is presented in Figure 2. The mode of failure was all adhesive failure in Groups A and D. However, cohesive failures occurred in the HF-treated groups (33.3 and 26.7% in Groups C and E, respectively). After thermocycling, 100% of the failures were adhesive in Groups B and E, whereas mixed failures (33.3%) occurred in Group C. The mean shear bond strength for Group C was significantly higher than

that of Group E ( $p < 0.05$ ), and Group C had fewer adhesive failures than Group E did. Figure 3 shows representative SEM images of e.max surface after  $\mu$ SBS tests.

While the specimens were being processed for microscopic observation of the bonded interfaces, all specimens spontaneously debonded in Groups A and D before thermocycling, and in Groups B and E during thermocycling. There was no gap between the ceramic and adhesive in Group C before or after thermocycling (Figures 4b and 4d) compared to Groups B and E before thermocycling (Figures 4a and 4c).

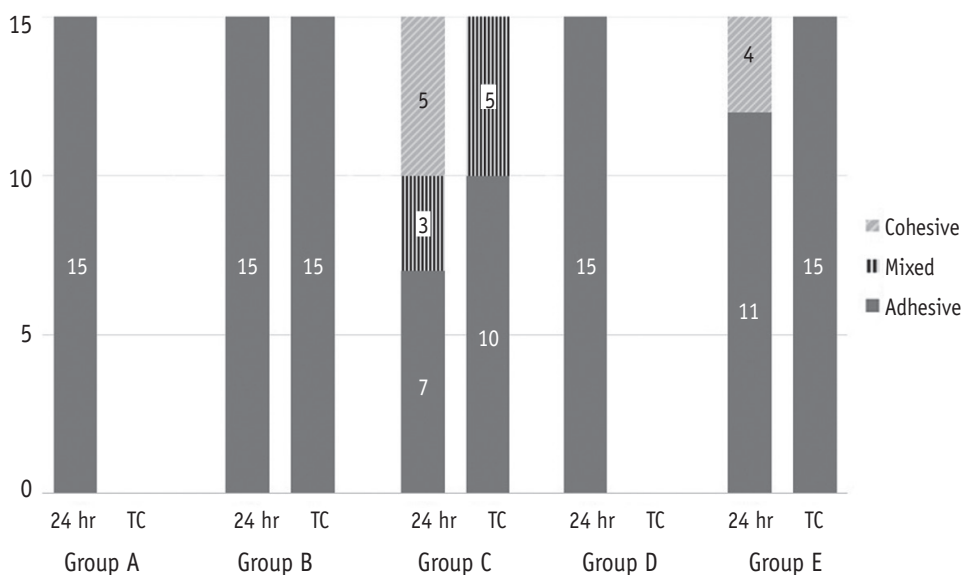


Figure 2. Failure mode distribution after microshear bond strength testing. TC, thermocycling.

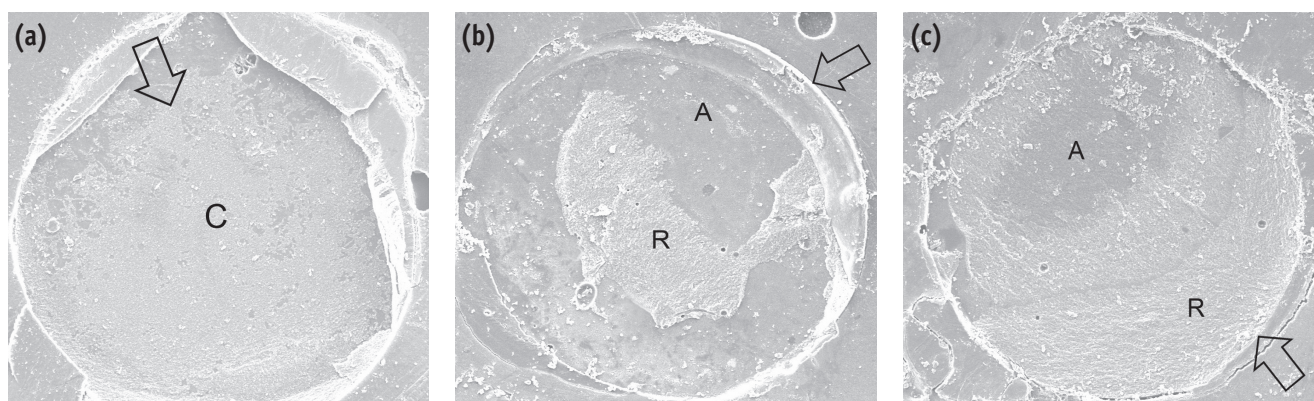
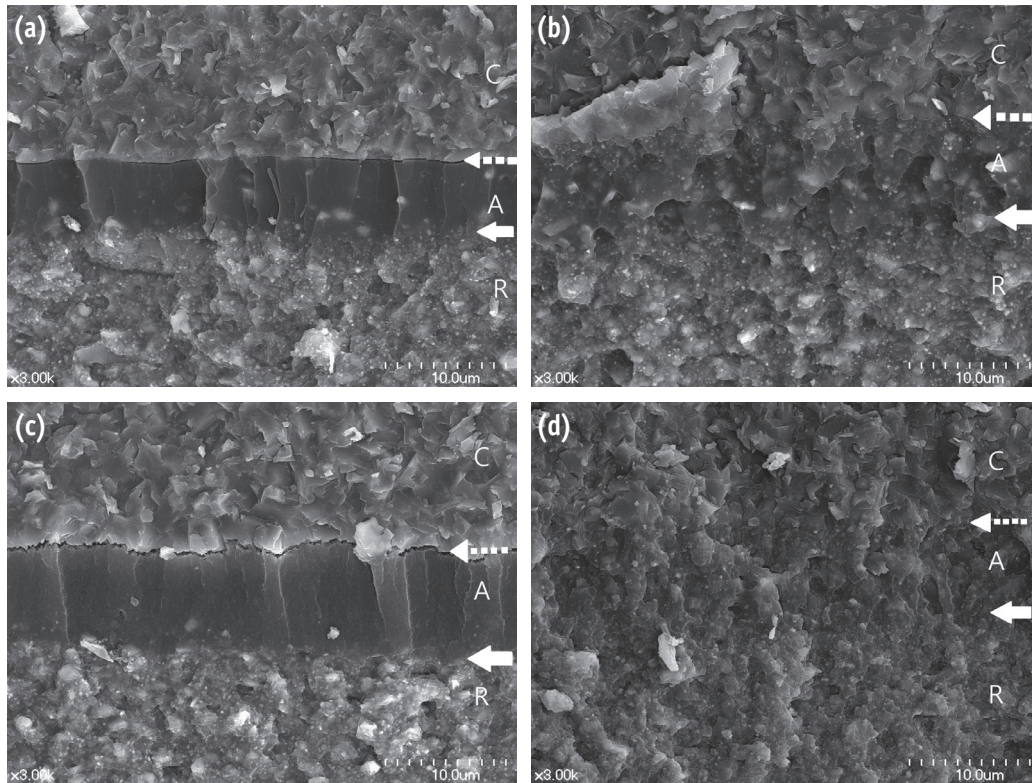


Figure 3. Representative SEM photomicrographs of fractured ceramic surfaces after microshear bond strength testing showing (a) adhesive failure; (b) mixed failure; (c) cohesive failure at  $\times 100$  magnification. The arrow shows the fracture origin and the direction of the arrow represents that of shear force. In Figure (c), the resin cement remained on the loading point side. C, ceramic; A, adhesive; R, resin cement.





**Figure 4.** SEM micrographs of the fractured surfaces comparing the adaptation between the adhesive and the ceramic surfaces treated with different procedures: (a) Group B (silane, adhesive that did not contain silane [ANS], and resin cement) before thermocycling. The surface of the lithium disilicate ceramic was flat, and there was no micro-undercut, because hydrofluoric acid (HF) had not been applied. The adhesive and resin cement layers can be discriminated. There were some filler particles in the adhesive layer; (b) Group C (HF, silane, ANS, and resin cement) before thermocycling. The borders of each material were not easily distinguishable because the adhesive had infiltrated the micro-undercut and the fillers were distributed throughout the full thickness of the adhesive; (c) Group E (HF, multi-mode universal adhesive [MUA], and resin cement) before thermocycling. The etched ceramic surface had micro-undercuts and MUA had infiltrated the undercuts. However, there was a gap between the adhesive and the ceramic surface; (d) Group C (HF, silane, ANS, and resin cement) after thermocycling. This had a similar morphology to Figure 4b. Dashed arrow, the interface of the ceramic and adhesive; hollow arrow, the interface of the adhesive and resin cement. C, ceramic; A, adhesive; R, resin cement.

## Discussion

We sought to examine the efficacy of MUA on the bond strength of resin cement to lithium disilicate ceramic using  $\mu$ SBS testing. Several testing methods are used to evaluate the bond strength between different materials, such as shear bond strength (SBS), tensile bond strength (TBS),  $\mu$ SBS, and microtensile bond strength ( $\mu$ TBS) tests. The larger the bonding area is, the higher the likelihood of a flaw being present and the lower the bond strength.<sup>28</sup> Either  $\mu$ SBS or  $\mu$ TBS tests are the most common approach. The  $\mu$ TBS test requires a uniform stress distribution during loading.<sup>29</sup> However, it is difficult to fabricate

microbeam specimens with sintered IPS e.max CAD blocks without damaging the bonded interface. Conversely,  $\mu$ SBS specimens are pre-stressed prior to testing only by mold removal.<sup>30</sup> Therefore, in this study, the  $\mu$ SBS test method was used, because it is not only a simple and reproducible procedure,<sup>31</sup> but it also permits efficient screening of adhesive systems.<sup>28</sup>

Single Bond Universal has a low pH of 2.7 due to a MDP. When it is mixed with self-cured resin cement, an acid-base reaction occurs between MDP and an aromatic tertiary amine which is the activator of chemical polymerization. The consequence of this reaction is lack of polymerization at the adhesive-cement interface. Therefore,

the manufacturer recommends mixing it with a separate activator if another manufacturer's self- or dual-cure resin cement is used,<sup>21</sup> or to use it with amine-free dual-cured resin cement. When Single Bond Universal is used with its activator, it is obtained in two different bottles. This does not have any advantages compared to a separate silane and adhesive system, like Group C. To simplify the process, we chose to use an amine-free dual-cured resin cement, NX3.

The application of MUA alone showed similar bond strength compared to Group A. All specimens in Group D were debonded during thermocycling (Table 2). The first hypothesis was accepted. One possible explanation for this interesting finding is impairment of silane stability in the acidic environment.<sup>32</sup> Because the pH of MUAs is 2.2 to 3.2 for self-etching capability, a self-condensation reaction occurs in the silanol groups of hydrolyzed silane.<sup>33,34</sup> A second possible explanation is that Bis-GMA in MUAs significantly inhibits the condensation reaction between the hydroxyl groups of lithium disilicate ceramic and the silanol groups of silane.<sup>35</sup> Moreover, extra resin could inhibit the condensation reaction that releases water molecules according to the Le Chatelier principle.<sup>36</sup> Furthermore, Uncured HEMA lowers the vapor pressure of water and make it difficult to remove water by air-drying.<sup>37</sup> Another explanation is that the concentration of silane in MUA might not be sufficient to react with the hydroxyl groups of the ceramic surface. This was confirmed by the studies by Zaghoul *et al.*<sup>35</sup> and Kalavacharla *et al.*<sup>26</sup> According to these authors, treatment with silane followed by MUA significantly improved the bond strength between the ceramic and the composite resin. The additional silanization step enhanced chemical bonding to the exposed hydroxyl groups and surface wettability with resin impregnation.

The results of the current study supported the importance of HF etching prior to ceramic surface bonding. Lower bond strength was obtained if the cement was applied without HF etching of the ceramic surface (Table 2), which confirmed the findings reported in earlier studies.<sup>13,38</sup> The large difference in bond strength contingent on HF etching is explained by the difference in surface texture (Figures 4a and 4c). HF etching of a ceramic surface dissolves the glass phase and forms soluble hexafluorosilicates, which can be rinsed out with water. In addition, HF etching creates surface irregularities, thus increasing surface area.<sup>39</sup> It also exposes OH groups, consequently improving the wettability of the ceramic by silane agents.

In the present study, the application of HF etching, silane, and adhesive showed the highest bond strength in comparison to other experimental groups. This procedure achieved durable bonds for silica-based ceramics.<sup>11,40</sup> Conversely, Isolan *et al.*<sup>41</sup> reported that the  $\mu$ SBS achieved with MUA was higher than that obtained with HF, silane,

and Single Bond 2 (SB2, 3M ESPE) treatment. Although we used Porcelain Bonding Resin, which is HEMA-free, Isolan *et al.*<sup>41</sup> used SB2, which is not. According to El Zohairy *et al.*,<sup>42</sup> bonding agents containing hydrophilic monomers have a negative effect on resin-ceramic bonds. Therefore, SB2 might influence the bond strength during water storage. In addition, different experimental settings, such as the use of different ceramic blocks, could have influenced these results.

Our results showed that durable resin-ceramic bond cannot be obtained by silane application without HF acid etching of the ceramic surface (Table 2). The bond strength in Group B, which was treated with silane and adhesive, was slightly higher compared to that in Group A. This indicated that silane contributes to the resin-ceramic bond, but showed that silane alone is not sufficient to produce durable ceramic bonding. This was corroborated by failure mode data, that is, adhesive failure was more common in Group B than in Group C.

Dental restorations are exposed to a harsh environment, such as repeated occlusal force, moisture and the thermal variation in the oral cavity. These can cause the failure of restorations. In the present study, thermal cycling was used to simulate clinical conditions. The results indicated that we could reject the second hypothesis. The post-thermocycling bond strength decreased significantly in Groups B, C, and E (Table 2). Several studies have proposed that thermocycling might have a negative effect on the bond strength between resins and ceramics.<sup>43-45</sup> As mentioned above, an adhesive layer including HEMA could influence the resin-ceramic bond durability.<sup>42</sup> MUA contains hydrophilic monomers, such as HEMA, that vary from the monomers present in ANS. The deterioration of the bond observed when MUA was used was correlated with the hydrophilic characteristics of the adhesive. Previous studies have proposed that water uptake will diminish the siloxane bond by hydrolysis and water swelling. HEMA has a low partition coefficient ( $p = 0.26$ ). The more HEMA resin present, the more water is absorbed.<sup>46,47</sup> The effect of swelling will stress the bond at the adhesive interface and will significantly weaken adhesive bonds.<sup>42</sup>

In current study, there were gaps in the resin-ceramic interfaces in Group E before thermocycling (Figure 4c), and spontaneous failure occurred in Group E during thermocycling for microscopic observation. Considering the composition of MUA and the gaps, the bond strength of Group E was significantly decreased after thermocycling. In contrast, the ceramic-adhesive-cement interface could not be detected and intimate bonding was observed in Group C before and after thermocycling (Figures 4b and 4d). This was consistent with the results of bond strength testing in Group C.

The main limitation of this study is that only one brand of MUA was tested. There are many MUAs that have

**Table 3.** Composition of adhesives according to the material safety data sheets provided by the manufacturers

	Porcelain Bonding Resin (wt%)	Single Bond Universal (wt%)	All-Bond Universal (%)	Clearfil Universal Bond (%)	Adhese Universal (%)
Bis-GMA	< 40	15 - 20	20 - 50	15 - 35	20 - < 25 <sup>†</sup>
Urethane dimethacrylate	< 40				
TEGDMA	< 30				
HEMA		15 - 20	5 - 25	10 - 35	20 - < 25 <sup>†</sup>
DGDMA		5 - 15			
Ethanol		10 - 15	30 - 50	< 20	10 - 13
Water		10 - 15		*	
MDP		*	5 - 25	*	
Silane-treated silica		5 - 15			
2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorous oxide		1 - 10			
Copolymer of acrylic and itaconic acid		1 - 5			
Dimethylaminobenzoate		< 2			
(Dimethylamino) ethyl methacrylate		< 2			
Methyl ethyl ketone		< 0.5			
				Colloidal silica, silane etc.	

Bis-GMA, bisphenol A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; DGDMA, decamethylene glycol demethacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

\*present, but composition information was not provided.

<sup>†</sup>from 20 or more to less than 25.

different compositions, as well as different ingredients (Table 3). For example, Single Bond Universal and Clearfil Universal Bond (Kuraray Noritake Dental, Tokyo, Japan) include silane, while other MUAs did not incorporate silane. Some adhesives include special ingredients, such as 3 - 7% methacrylated carboxylic acid polymer in Adhese Universal (Ivoclar Vivadent), or 1 - 5% polyacrylic acid copolymer in Single Bond Universal. Each MUA may have different bonding interactions according to the surface treatments used and their ingredients. The resin cement used was NX3, which is not manufactured by the supplier of the MUA. The manufacturer of the MUA recommends using it in combination with Rely X Ultimate cement. Further studies are needed to compare the bond strength of MUA between these two cements.

## Conclusions

Micro-undercuts formed by HF etching on lithium disilicate played an important role in the bond strength between lithium disilicate ceramic and resin cement. Silane also contributed to the formation of a durable bond to lithium

disilicate. Silane contained in the tested MUA (Single Bond Universal) did not seem to enhance the bonding strength between lithium disilicate ceramic and resin cement.

Conflict of Interest: No potential conflict of interest relevant to this article was reported.

## References

1. Bergmann CP, Stumpf A. Dental ceramics: microstructure, properties and degradation. Berlin Heidelberg: Springer Science & Business Media; 2013. Chapter 1.
2. Rosenblum MA, Schulman A. A review of all-ceramic restorations. *J Am Dent Assoc* 1997;128:297-307.
3. Zhang Y, Lee JJ, Srikanth R, Lawn BR. Edge chipping and flexural resistance of monolithic ceramics. *Dent Mater* 2013;29:1201-1208.
4. Tysowsky GW. The science behind lithium disilicate: a metal-free alternative. *Dent Today* 2009;28:112-113.
5. Gorodovsky S, Zidan O. Retentive strength, disintegration, and marginal quality of luting cements. *J Prosthet Dent* 1992;68:269-274.



6. El-Mowafy O. The use of resin cements in restorative dentistry to overcome retention problems. *J Can Dent Assoc* 2001;67:97-102.
7. Rosentritt M, Behr M, Lang R, Handel G. Influence of cement type on the marginal adaptation of all-ceramic MOD inlays. *Dent Mater* 2004;20:463-469.
8. Alber FE, El-Mowafy OM. Marginal adaptation and microleakage of Procera AllCeram crowns with four cements. *Int J Prosthodont* 2004;17:529-535.
9. Sorensen JA, Kang SK, Avera SP. Porcelain-composite interface microleakage with various porcelain surface treatments. *Dent Mater* 1991;7:118-123.
10. Jensen ME, Sheth JJ, Tolliver D. Etched-porcelain resin-bonded full veneer crowns: *in vitro* fracture resistance. *Compendium* 1989;10:336-347.
11. Nagai T, Kawamoto Y, Kakehashi Y, Matsumura H. Adhesive bonding of a lithium disilicate ceramic material with resin-based luting agents. *J Oral Rehabil* 2005;32:598-605.
12. Filho AM, Vieira LC, Araújo E, Monteiro Júnior S. Effect of different ceramic surface treatments on resin microtensile bond strength. *J Prosthodont* 2004;13:28-35.
13. Menees TS, Lawson NC, Beck PR, Burgess JO. Influence of particle abrasion or hydrofluoric acid etching on lithium disilicate flexural strength. *J Prosthet Dent* 2014;112:1164-1170.
14. Kang SH, Chang J, Son HH. Flexural strength and microstructure of two lithium disilicate glass ceramics for CAD/CAM restoration in the dental clinic. *Restor Dent Endod* 2013;38:134-140.
15. Della Bona A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. *Dent Mater* 2004;20:338-344.
16. Alex G. Preparing porcelain surfaces for optimal bonding. *Compend Contin Educ Dent* 2008;29:324-335.
17. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, Vanherle G. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent* 2003;28:215-235.
18. Kadoma Y. Surface treatment agent for dental metals using a thiirane monomer and a phosphoric acid monomer. *Dent Mater J* 2002;21:156-169.
19. Chen L, Suh BI, Brown D, Chen X. Bonding of primed zirconia ceramics: evidence of chemical bonding and improved bond strengths. *Am J Dent* 2012;25:103-108.
20. Fukegawa D, Hayakawa S, Yoshida Y, Suzuki K, Osaka A, Van Meerbeek B. Chemical interaction of phosphoric acid ester with hydroxyapatite. *J Dent Res* 2006;85:941-944.
21. Alex G. Universal adhesives: the next evolution in adhesive dentistry? *Compend Contin Educ Dent* 2015;36:15-26.
22. Suzuki T, Takamizawa T, Barkmeier WW, Tsujimoto A, Endo H, Erickson RL, Latta MA, Miyazaki M. Influence of etching mode on enamel bond durability of universal adhesive systems. *Oper Dent* 2016;41:520-530.
23. Sai K, Shimamura Y, Takamizawa T, Tsujimoto A, Imai A, Endo H, Barkmeier WW, Latta MA, Miyazaki M. Influence of degradation conditions on dentin bonding durability of three universal adhesives. *J Dent* 2016;54:56-61.
24. Passia N, Mitsias M, Lehmann F, Kern M. Bond strength of a new generation of universal bonding systems to zirconia ceramic. *J Mech Behav Biomed Mater* 2016;62:268-274.
25. Kim JH, Chae SY, Lee Y, Han GJ, Cho BH. Effects of multipurpose, universal adhesives on resin bonding to zirconia ceramic. *Oper Dent* 2015;40:55-62.
26. Kalavacharla VK, Lawson NC, Ramp LC, Burgess JO. Influence of etching protocol and silane treatment with a universal adhesive on lithium disilicate bond strength. *Oper Dent* 2015;40:372-378.
27. Han GJ, Chung SN, Chun BH, Kim CK, Oh KH, Cho BH. Effect of the applied power of atmospheric pressure plasma on the adhesion of composite resin to dental ceramic. *J Adhes Dent* 2012;14:461-469.
28. Braga RR, Meira JB, Boaro LC, Xavier TA. Adhesion to tooth structure: a critical review of 'macro' test methods. *Dent Mater* 2010;26:e38-e49.
29. Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA, Tay F. The microtensile bond test: a review. *J Adhes Dent* 1999;1:299-309.
30. Armstrong S, Geraldeli S, Maia R, Raposo LH, Soares CJ, Yamagawa J. Adhesion to tooth structure: a critical review of 'micro' bond strength test methods. *Dent Mater* 2010;26:e50-e62.
31. Kim JH, Chae S, Lee Y, Han GJ, Cho BH. Comparison of shear test methods for evaluating the bond strength of resin cement to zirconia ceramic. *Acta Odontol Scand* 2014;72:745-752.
32. Arkles B, Steinmetz JR, Zazyczny J, Mehta P. Factors contributing to the stability of alkoxysilanes in aqueous solution. *J Adhes Sci Technol* 1992;6:193-206.
33. Brochier Salon MC, Bayle PA, Abdelmouleh M, Boufi S, Belgacem MN. Kinetics of hydrolysis and self condensation reactions of silanes by NMR spectroscopy. *Coll Surf Physicochem Eng Asp* 2008;312:83-91.
34. Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. *Dent Mater* 2012;28:467-477.
35. Zaghoul H, Elkassas DW, Haridy MF. Effect of incorporation of silane in the bonding agent on the repair potential of machinable esthetic blocks. *Eur J Dent* 2014;8:44-52.
36. Fox MA, Whitesell JK. Organic chemistry. 3rd ed. Sudbury: Jones and Bartlett Publishers; 2004. Chapter 16.

37. Pashley EL, Zhang Y, Lockwood PE, Rueggeberg FA, Pashley DH. Effects of HEMA on water evaporation from water-HEMA mixtures. *Dent Mater* 1998;14:6-10.
38. Guarda GB, Correr AB, Gonçalves LS, Costa AR, Borges GA, Sinhoreti MA, Correr-Sobrinho L. Effects of surface treatments, thermocycling, and cyclic loading on the bond strength of a resin cement bonded to a lithium disilicate glass ceramic. *Oper Dent* 2013;38:208-217.
39. Borges GA, Sophr AM, De Goes MF, Sobrinho LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. *J Prosthet Dent* 2003;89:479-488.
40. Lise DP, Perdigão J, Van Ende A, Zidan O, Lopes GC. Microshear bond strength of resin cements to lithium disilicate substrates as a function of surface preparation. *Oper Dent* 2015;40:524-532.
41. Isolan CP, Valente LL, Münchow EA, Basso GR, Pimentel AH, Schwantz JK, da Silva AV, Moraes RR. Bond strength of a universal bonding agent and other contemporary dental adhesives applied on enamel, dentin, composite, and porcelain. *Appl Adhes Sci* 2014;2:1-10.
42. El Zohairy AA, De Gee AJ, Hassan FM, Feilzer AJ. The effect of adhesives with various degrees of hydrophilicity on resin ceramic bond durability. *Dent Mater* 2004;20:778-787.
43. Kato H, Matsumura H, Tanaka T, Atsuta M. Bond strength and durability of porcelain bonding systems. *J Prosthet Dent* 1996;75:163-168.
44. Wolf DM, Powers JM, O'keefe KL. Bond strength of composite to porcelain treated with new porcelain repair agents. *Dent Mater* 1992;8:158-161.
45. Diaz-Arnold AM, Aquilino SA. An evaluation of the bond strengths of four organosilane materials in response to thermal stress. *J Prosthet Dent* 1989;62:257-260.
46. Seo B. Principles of adhesion dentistry. 1st ed. Newtown: AEGIS Publications; 2013. Chapter 1.
47. Takahashi M, Nakajima M, Hosaka K, Ikeda M, Foxton RM, Tagami J. Long-term evaluation of water sorption and ultimate tensile strength of HEMA-containing/-free one-step self-etch adhesives. *J Dent* 2011;39:506-512.