

## Original Article

# Microshear bond strength of resin cement to a zirconia-reinforced lithium silicate glass ceramic using different surface treatments

Mohammadreza Nakhaei<sup>1</sup>, Hamideh-Sadat Mohammadipour<sup>2</sup>, Seyyed-Farzan Eslami<sup>2</sup>, Zahra Soroush<sup>3</sup>

<sup>1</sup>Department of Prosthodontics, Dental Materials Research Center, School of Dentistry, Mashhad University of Medical Sciences, <sup>2</sup>Department of Cosmetic and Restorative Dentistry, <sup>3</sup>Department of Prosthodontics, School of Dentistry, Mashhad University of Medical Sciences, Mashhad, Iran

## ABSTRACT

**Background:** The purpose of this study was to evaluate the effect of different surface treatments on the microshear bond strength ( $\mu$ SBS) of resin cement to zirconia-reinforced lithium silicate ceramic and to compare it with lithium disilicate ceramic.

**Materials and Methods:** In this *in vitro* study, 80 specimens containing two glass ceramics of IPS e.max press and VITA SUPRINITY were prepared and categorized into four groups according to the surface treatments ( $n = 10$ ) as Group 1 (C): no treatment (control); Group 2 (HF): etching with 9% hydrofluoric acid (HF) for 90 s followed by silane application; Group 3 (SPH): sandblasting with  $Al_2O_3$  particles (50  $\mu$ m), etching with 35% phosphoric acid for 40 s followed by application of silane and adhesive (Clearfil liner bond F); and Group 4 (SB): sandblasting with  $Al_2O_3$  followed by silanization. Then, a resin cement (Panavia F2) was applied to the prepared ceramic surfaces. All samples were subjected to thermal aging (5000 cycles, 5–55). The  $\mu$ SBS test was evaluated and failure modes were recorded. Data were analyzed using the Shapiro–Wilk, two-way analysis of variance and Tukey's Honest Significant Difference *post hoc* tests ( $P < 0.05$ ).

**Results:** IPS e.max press samples revealed significantly higher  $\mu$ SBS values compared to VITA SUPRINITY ( $P < 0.001$ ), in whole surface treatments. The HF group showed the highest  $\mu$ SBS value, followed by the SPH and SB groups, respectively ( $P < 0.001$ ). Adhesive failure was recorded as a predominant failure mode.

**Conclusion:** The adhesion performance of IPS e.max press was significantly higher than VITA SUPRINITY. The common surface treatment protocol including HF application followed by silanization was the most effective surface treatment for both glass ceramics.

**Key Words:** Glass ceramics, hydrofluoric acid, lithia disilicate, resin cements

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Address for correspondence:  
Dr. Zahra Soroush,  
Department of  
Prosthodontics, School  
of Dentistry, Mashhad  
University of Medical  
Sciences, Vakilabad  
Boulevard, Mashhad, Iran.  
E-mail: soroush.zahra@gmail.com

## INTRODUCTION

All-ceramic restorations are widely used in the dental field due to increased esthetic demands of patients.<sup>[1]</sup> These materials are biocompatible and, inert, and have a high degree of chemical stability.<sup>[2]</sup> However, the primary clinical problem is that ceramics are

brittle and subject to cracking and chipping.<sup>[3]</sup> Therefore, advances have been made in all-ceramic restorations to offer ceramic materials with optimized mechanical properties. Some examples of these new microstructures are highly translucent monolithic

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zirconia, glass ceramics reinforced with zirconia, and polymer-infiltrated ceramic networks.<sup>[4]</sup>

Zirconia-reinforced glass ceramics were designed to combine esthetic properties of the lithium disilicate system with the superior mechanical behavior of zirconia ceramics.<sup>[5]</sup> These new ceramics contain round submicrometric grains of lithium metasilicates and lithium orthophosphates in a vitreous matrix containing 10% zirconium oxide.<sup>[6]</sup> After the crystallization process, lithium silicate crystals achieve a mean size of 0.5–1  $\mu\text{m}$ , which is 4–8 times smaller than those observed in lithium disilicate glass ceramics. Indeed, the presence of zirconia particles prevents crystal growth and is responsible for favorable characteristics of these ceramics.<sup>[7]</sup> Previous studies have revealed superior or similar mechanical properties of zirconia-reinforced glass ceramics compared to lithium disilicate glass ceramics,<sup>[6–8]</sup> which is due to the smaller crystals or crack interruption mechanism that results from incorporation of zirconia fillers.<sup>[9,10]</sup> In spite of mechanical reinforcement, the esthetic properties of zirconia-reinforced glass ceramics are improved or maintained due to the fine-grained structure and the high glass content.<sup>[5]</sup>

It has been advocated to use resin cements for bonding glass ceramics to dental structures and materials, especially in situations where adequate retention is lacking.<sup>[11]</sup> In order to achieve durable bonding, it is necessary to select suitable surface treatments and resin cements based on the microstructure of the material.<sup>[12,13]</sup> For glass ceramics, the application of hydrofluoric acid (HF) followed by silane is the best-established procedure to be performed according to validated protocols.<sup>[12]</sup> However, HF is a highly hazardous chemical substance, and extreme care must be taken for intraoral porcelain repair.<sup>[14]</sup> Hence, several chemical conditioning agents and mechanical treatments have been introduced to substitute intraoral HF application.<sup>[13]</sup> Airborne particle abrasion is an alternative method to roughen the ceramic surface by blasting alumina ( $\text{Al}_2\text{O}_3$ ) particles.<sup>[13]</sup> For glass ceramics, alumina powder with a mean particle size of 25–50  $\mu\text{m}$  at a pressure of 0.28 MPa is commonly used.<sup>[15]</sup> Another surface treatment for these ceramics that is recommended by the manufacturer of Panavia F2.0 resin cement is combined application of a silane coupling agent and a functional monomer as a primer.<sup>[15,16]</sup> However, there is not adequate data on the effectiveness of this method.

Based on the microstructure of mentioned glass ceramics, the increased zirconia content in zirconia-reinforced glass ceramics may affect the adhesive performance of this ceramic. There are controversies about this issue in the literature. Some studies revealed lower or superior bonding strength of zirconia-reinforced glass ceramics in comparison to lithium disilicate glass ceramics,<sup>[11,12,17,18]</sup> whereas others reported no significant difference in their adhesion.<sup>[16,19]</sup> Therefore, the aim of this *in vitro* study was to evaluate the effect of different surface treatments on microshear bond strength ( $\mu\text{SBS}$ ) of zirconia-reinforced lithium silicate ceramic (VITA SUPRINITY) to a resin cement compared to lithium disilicate glass ceramic (intelligent porcelain system (IPS) e.max press). The null hypothesis was that none of the ceramics and surface treatment methods would affect the bond strength of the resin cement to these ceramics.

## MATERIALS AND METHODS

### Sample preparation and surface treatment

Eighty ceramic specimens (4 mm  $\times$  4 mm  $\times$  4 mm) including 40 zirconia-reinforced lithium silicate (VITA SUPRINITY HT, Vita Zahnfabrik, H. Rauter GmbH & Co., Bad Säckingen, Germany) and 40 lithium disilicate ceramics (IPS e.max press HT, Ivoclar Vivadent, Schaan, Liechtenstein) with A2 shade were used in this *in vitro* study. To prepare ceramic samples, VITA SUPRINITY blocks were mounted in a precision cutting device (Imes-Icore 650i, Coritec, Eiterfeld, Germany) and milled using a low-speed diamond saw under water cooling to make cubic specimens. Then, the prepared samples were crystallized in a ceramic furnace (Programat EP 5000, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions. In order to prepare IPS e.max press samples, 40 three-dimensional (3D) printed resin blocks were made by an in-office digital light processing (DLP) 3D printer (MAX, Asiga, Sydney, Australia) to prepare ceramic samples through the lost-wax technique. Resin patterns were invested in a phosphate-based material (IPS PressVEST Speed; Ivoclar Vivadent). The ceramic ingots were then pressed into the molds at 915 in a furnace press (Programat EP 5000, Ivoclar Vivadent, Schaan, Liechtenstein). The samples were not glazed. The dimensions of prepared samples were measured with a digital caliper (Pro-Max series, Fowler High Precision, Massachusetts). The specimens were then embedded in

an auto polymerizing acrylic resin (Acropars, Marlic Co., Tehran, Iran) with one surface exposed. Ceramic surfaces were carefully wet-ground for 15 s using 180 grit silicon-carbide abrasive papers (Starcke, Hoffman Co, Germany), ultrasonically cleaned in distilled water for 360 s and dried to remove surface debris and moisture. The materials used in the present study with their respective manufacturers and compositions are described in Table 1.

The prepared specimens were then randomly divided into four groups of 10 specimens depending on the surface treatments. The study design and preparation steps are summarized and presented in Table 2.

Group 1 (C): No surface treatment was performed on samples in this group as the control group.

Group 2 (HF): The ceramic surfaces were etched with 9.5% HF (Bisco's porcelain etchant; Bisco inc., Schaumburg, IL USA) for 90 s, washed under copious distilled water and air-dried. Then, a thin layer of silane coupling agent (Bis-Silane; Bisco inc., Schaumburg, IL, USA) was applied using a micro brush and allowed to dry for 30 s.

Group 3 (SPH; based on Panavia F2.0 procedure for cementation of silanated porcelain): The ceramic

surfaces were abraded with an intraoral airborne particle abrasion device (Kolo, Multi-functional Micro blaster, Sun Ring Dental Medical Instrument Co., Japan) using 50- $\mu$ m aluminum oxide particles (2.8 bar, 10 s blasting time at 10 cm distance perpendicular to the ceramic surface). Then, they were etched with 35% phosphoric acid etchant (Ultra-Etch; Ultradent Products Inc., USA) to clean the surface for 5 s, rinsed, and air-dried. The adhesive (Clearfil liner bond F; Kuraray Noritake dental Inc., Japan) and silane were mixed in a 1:1 ratio and applied with a micro brush, and allowed to react for 30 s.

Group 4 (SB): The ceramic surfaces were air-abraded by a similar method to the previous group. Next, a silane coat was applied using a micro brush and left in place for 30 s to evaporate.

After surface treatment in the three study groups (except for the control group), one drop of bottle B of ED Primer II was applied to the ceramic surfaces for 30 s and air-dried gently. Then, equal amounts of paste A and B of the Panavia F2.0 cement (Kuraray Noritake Dental Inc., Japan) were mixed with a plastic spatula on a paper pad and pushed into the plastic molds with a height of 3 mm and internal diameter of 1 mm, held perpendicular over the ceramic surfaces. Great care was

**Table 1: Description of materials, their manufacturers, and composition of the materials used in this study**

Material	Commercial name/manufacturer	Composition
Lithium disilicate glass ceramic	IPS e.max press; Ivoclar Vivadent, Schaan, Liechtenstein	SiO <sub>2</sub> , Li <sub>2</sub> O, K <sub>2</sub> O, P <sub>2</sub> O <sub>5</sub> , ZrO <sub>2</sub> , ZnO, and other coloring oxides
Zirconia-reinforced lithium silicate glass ceramic	VITA SUPRINITY; Vita Zahnfabrik, H. Rauter GmbH and Co., Bad Säckingen, Germany	SiO <sub>2</sub> ; Li <sub>2</sub> O; K <sub>2</sub> O; P <sub>2</sub> O <sub>5</sub> ; ZrO <sub>2</sub> ; Al <sub>2</sub> O <sub>3</sub> ; CeO <sub>2</sub> ; pigments
Hydrofluoric acid	Bisco's porcelain etchant; Bisco inc. Schaumburg, Illinois, USA	9.5% concentration of hydrofluoric acid (main component)
Phosphoric acid	Ultra-Etch; Ultradent products Inc., South Jordan, USA	35% concentration phosphoric acid (main component)
Aluminum oxide	Siladent; Siladent Dr. Böhme and Schöps GmbH, Germany	50 $\mu$ m Al <sub>2</sub> O <sub>3</sub> particles (main component)
Silane	Bis-Silane; Bisco inc. Schaumburg, Illinois, USA	Part A: Ethanol, 3-(Trimethoxysilyl) propyl-2-Methyl-2-propenoic acid Part B: Ethanol, Phosphoric acid
Adhesive	Clearfil liner bond F; Kuraray Noritake dental inc., Sakazu, Kurashiki, Okayama, Japan	bisphenol A diglycidyl methacrylate, 2-hydroxyethyl methacrylate, sodium fluoride, 10-Methacryloyloxydecyl dihydrogen phosphate, hydrophobic aliphatic dimethacrylate, colloidal silica, dl-Camphorquinone, initiators, accelerators
Resin-based luting cement	Panavia F 2.0; Kuraray Noritake dental Inc., Sakazu, Kurashiki, Okayama, Japan	Paste A: 10-Methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-Camphorquinone, catalysts, initiators Paste B: Sodium fluoride, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments ED primer II Liquid A: 2-hydroxyethyl methacrylate, 10-Methacryloyloxydecyl dihydrogen phosphate, N-Methacryloyl-5-aminosalicylic acid, water, accelerators ED primer II Liquid B: N-Methacryloyl-5-aminosalicylic acid, water, catalysts, accelerators

IPS: Intelligent Porcelain System

**Table 2: Experimental design of study groups**

Groups	Ceramic materials	Step 1: Mechanical etching	Step 2: Chemical etching	Step 3: Silane/ Adhesive application	Step 4: Primers	Step 5: Resin cement insertion	Step 6: Aging	Step 7: Bond strength test
C	IPS e.max press VITA SUPRINITY	-	-	-	ED Primer II B bottle	Self-etch resin cement (Panavia F.2)	Thermocycling	μSBS test Stereomicroscopy
HF	IPS e.max press VITA SUPRINITY	-	9.5% HF (Bisco's porcelain etchant)	Silane (Bis-Silane)	ED Primer II B bottle	Self-etch resin cement (Panavia F.2)	Thermocycling	μSBS test Stereomicroscopy
SPH	IPS e.max press VITA SUPRINITY	Air born particle abrasion +35% Phosphoric acid (Ultra-Etch) for surface cleansing	-	Adhesive (Clearfil liner bond F) + Silane	ED Primer II B bottle	Self-etch resin cement (Panavia F.2)	Thermocycling	μSBS test Stereomicroscopy
SB	IPS e.max press VITA SUPRINITY	Air born particle abrasion	-	Silane	ED Primer II B bottle	Self-etch resin cement (Panavia F.2)	Thermocycling	μSBS test Stereomicroscopy

IPS: Intelligent Porcelain System

taken to avoid any cement flow out of the plastic mold. Cylindrical buildups light-polymerized with at least intensity of 650 mW/cm<sup>2</sup> using a LED unit (Bluephase C8, Ivoclar Vivadent, Schaan, Liechtenstein) for 40 s. The intensity of the light-curing device was measured after every five exposures.

All samples were then stored in distilled water for 24 h in an incubator at 37°C with 100% humidity. Then, the plastic molds were carefully separated from the periphery of cylindrical buildups with a scalpel blade (Moris, China). Each specimen was examined to verify that the buildup had no interfacial defects, such as bubbles, gaps, and composite cement flow beyond the limits of the bonding area. Afterward, all samples were aged by thermocycling machine (Nemo Co., Mashhad, Iran) for 5000 cycles at 5°C–55°C with 20 s dwell time and 5 s transfer time.

### Microshear bond strength and failure mode evaluation

The samples were placed into the universal testing machine (Santam, model STM, Tehran, Iran) to apply a shear load using a knife-edge chisel parallel to the ceramic-resin interface at a 1 mm/min crosshead speed. The shear load was applied until debonding occurred, and the value was recorded in Newtons (N). The obtained μSBS values were expressed in megapascals by dividing the recorded peak load at failure (N) by the adhesive surface area (mm<sup>2</sup>).

Next, the fractured areas were examined with a stereomicroscope (Dino lite Pro, Anmo Electronics Corp., Taiwan) at ×20. For high quality images, digital single-lens reflex camera (Canon EOS 1200D) with Canon EF-S lens mount was used

to capture images. The failure type was classified as either adhesive (failure at the adhesive layer), cohesive (failure within ceramic or cement), or mixed (a mixture of both failures).

### Statistical analysis

Data were analyzed using the SPSS software version 19.0 (Chicago, IL, USA). The normality of data distribution among all tested groups was verified using the Shapiro–Wilk test. Two way analysis of variance (ANOVA) was used to analyze the difference between two evaluated ceramics and different surface treatments. Tukey's Honest Significant Difference (HSD) and *t*-test *post hoc* tests were applied for pairwise comparisons. The significance level was set at 5% for all statistical analyses.

## RESULTS

The mean values and standard deviations of μSBS for each group are presented in Table 3. The results of Shapiro–Wilk analysis showed the normal distribution of the data ( $P < 0.05$ ). Two-way ANOVA showed that both surface treatments and ceramic types had significant effects on the bond strength values ( $P < 0.001$ ). Tukey's HSD test showed a significant difference between different surface preparations protocols used for each ceramic ( $P < 0.001$ ). Based on *t*-test analysis, there was a significant difference between different ceramics in each surface preparation protocol ( $P < 0.001$ ). For both tested ceramics, the highest and lowest mean values of μSBS were found in HF and SB subgroups, respectively. The mean μSBS of IPS e.max press was significantly higher than that of VITA SUPRINITY for all surface treatment methods [Table 3]. Since,

**Table 3: Microshear bond strength mean±Standard deviation of two tested ceramics after different surface treatments**

Surface treatments (mean $\mu$ SBS [MPa]±SD) Ceramics	Number	HF	SPH	SB
IPS e.max press	10	21.84 (1.72) <sup>Aa</sup>	14.97 (1.25) <sup>Ba</sup>	10.78 (0.70) <sup>Ca</sup>
VITA SUPRINITY	10	14.02 (1.05) <sup>Ab</sup>	10.71 (1.54) <sup>Bb</sup>	7.05 (1.06) <sup>Cb</sup>

Different uppercase letters in the rows and lowercase letters in the columns indicate statistically significant differences (Tukey HSD and t-test,  $P < 0.001$ ).  $\mu$ SBS: Microshear bond strength, SD: Standard deviation, HF: Hydrofluoric acid, IPS: Intelligent Porcelain System

all control samples with no surface treatment showed pretest failure, their data were excluded from statistical analysis.

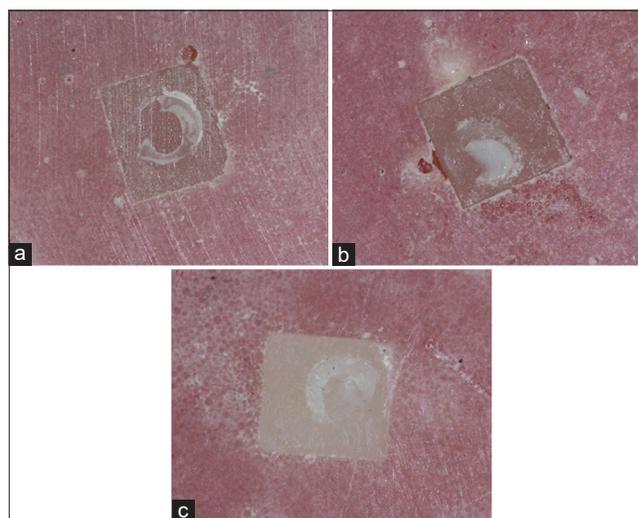
The obtained results of the failure mode assessment are presented in Table 4. The adhesive failure was the predominant failure mode in all experimental groups. Cohesive failures were observed only in the SB group of IPS e.max press ceramic, while mixed failure modes were observed in all experimental groups [Figure 1].

## DISCUSSION

This study evaluated the effect of different surface treatment protocols on  $\mu$ SBS of a resin cement to lithium disilicate (IPS.e.max press) and zirconia-reinforced lithium silicate (VITA SUPRINITY) glass ceramics. Since the results showed that both ceramic types and different surface treatment protocols significantly affected the bonding strength, the null hypothesis was rejected.

In the present study, the  $\mu$ SBS of IPS.e.max press was significantly higher than that of VITA SUPRINITY, which was independent of surface treatments. However, there is no consensus in this respect in the literature. While some studies have found the superior bond strength of zirconia-reinforced glass ceramics in comparison with lithium disilicate glass ceramics,<sup>[11,12,17,18]</sup> others reported no significant difference in the bonding strength after similar surface treatments.<sup>[16,19]</sup> These contradictions may be related to the different surface treatments, aging protocols, and different chemical compositions of adhesive materials such as resin cements or adhesives.<sup>[20]</sup>

The application of HF as a common surface treatment for glass ceramics resulted in favorable adhesion. Based on the results of this study, both ceramic samples treated with HF showed the highest  $\mu$ SBS values among the study groups. However, this treatment could not produce a bond strength value for VITA SUPRINITY that was as



**Figure 1:** The three images which obtained under digital single-lens reflex camera (Canon EOS 1200D) with Canon EF-S lens mount showed; (a): the adhesive, (b): cohesive and (c): mixed failure modes.

high as the bond strength value for the IPS e.max press. It can be related to the viscosity of the HF previously considered as a main factor negatively affecting the adhesion of resin cements to ceramic substrates.<sup>[20]</sup> IPS.e.max press contains large, needle-shaped fine-grained crystals embedded in a glassy matrix; however, VITA SUPRINITY has a very fine-grained structure.<sup>[8]</sup> Therefore, viscous etchants cannot possibly penetrate deep between the fine VITA SUPRINITY crystallites.<sup>[11]</sup> Studies that used more diluted etchants (4.7%–5%) found a higher bonding strength for zirconia-reinforced glass ceramics compared to lithium disilicate glass ceramics.<sup>[11,17,18]</sup> Fabian Fonzar *et al.* evaluated the effect of HF concentration and etching time on the  $\mu$ SBS of RelyX Unicem 2 to VITA SUPRINITY and IPS e.max CAD and, found that these ceramics showed different bonding abilities after application of 4.9% and 9.5% HF. Unlike IPS e.max CAD, VITA SUPRINITY achieved significantly higher  $\mu$ SBS after conditioning with 4.9% HF. This finding might be due to the lower viscosity of 4.9% HF compared to 9.5% HF. The higher wettability of 4.9% HF and the smaller

**Table 4: Failure modes of the study groups**

Surface treatments	Ceramics	Failure modes (%)		
		Adhesive	Cohesive	Mixed
HF	VITA SUPRINITY	90	0	10
	IPS e.max press	80	0	20
SPH	VITA SUPRINITY	90	0	10
	IPS e.max press	90	0	10
SB	VITA SUPRINITY	80	0	20
	IPS e.max press	80	10	10

HF: Hydrofluoric acid, IPS: Intelligent Porcelain System

crystals in VITA SUPRINITY (0.5  $\mu\text{m}$  compared to 1.5  $\mu\text{m}$  in IPS e.max CAD) may have produced a deeper etching pattern on the zirconia-reinforced glass ceramic surface, which resulted in greater adhesion.<sup>[11]</sup> similarly, Belli *et al.* used a highly diluted etchant (0.5% HF) for microstructural characterization of ceramics with fine grains like VITA SUPRINITY and used 5% HF for IPS e.max press ceramics.<sup>[6]</sup>

The viscosity of resin cement is another factor that can affect adhesion to ceramics. High-viscosity resin-based luting agents cannot easily flow into the minor pores of the sandblasted or etched zirconia-reinforced glass ceramic surface compared to lithium disilicate glass ceramic surfaces resulting in a decrease in the final micromechanical locking effect of the zirconia-reinforced glass ceramic.<sup>[11,21]</sup> Incorporating zirconia fillers into lithium disilicate glass ceramics inhibits metasilicate crystal growth and leads to a microstructure similar to zirconia ceramics.<sup>[22,23]</sup> In a study by Moon *et al.*, Superbond C and B and Multilink showed the highest and lowest mean values of SBS of the resin cement to zirconia ceramic, respectively. The authors attributed this finding to the viscosity of the resin cement. Resin cement with lower viscosity can easily flow into the microporosities of the sandblasted zirconia surface and produce greater adhesion to ceramic substrate. Fracture photographs have shown a higher cohesive failure rate in Superbond C and B, which is consistent with the above statements.<sup>[21]</sup> In agreement with aforementioned statements, the low penetration of high viscosity HF gel and resin cement in VITA SUPRINITY surface, which was used in the present study, resulted in a lower  $\mu\text{SBS}$  value compared to IPS e.max press. Hence, besides common parameters such as acid etching time and concentration in ceramic surface conditioning, the clinician should consider the effect of acid gel viscosity and choose the compatible one with ceramic microstructure. In particular, cement viscosity is another factor that is less noted in ceramic bonding and seems to be another

important parameter that one should consider when bonding different ceramics clinically.

The difference in bonding strength of zirconia-reinforced and lithium disilicate glass ceramics may be related to the surface energy of the substrates.<sup>[24]</sup> The surface energy of glass ceramics increases after performing HF etching due to the removal of the low-energy contaminants, increasing surface roughness, and density of hydroxyl groups.<sup>[25]</sup> Silanization reduces the surface energy because silane molecules will bond to Si-OH on the surface, which forms a branched hydrophobic layer and reduces the ceramic surface energy.<sup>[20]</sup> The formation of this cross-linked structure enhances the penetration of the hydrophobic luting cement into the microporosities of the etched ceramic surface, which facilitates mechanical interlocking and also increases the hydrolytic stability of the bonding interface. Ramakrishnaiah *et al.* evaluated the effect of etching duration on the micromorphology, surface roughness, and wettability of ceramic surfaces. The contact angle and mean surface roughness of VITA SUPRINITY were higher than those of IPS e.max CAD in longer etching times with 5% HF for 80 s and 160 s; however, the difference was not significant.<sup>[25]</sup> Strasser *et al.* found comparable surface energy and lower surface roughness for VITA SUPRINITY in comparison with IPS e.max CAD after HF etching with IPS e.max press, which may be more similar to the results of a study by Ramakrishnaiah *et al.* that confirmed the higher contact angle of VITA SUPRINITY.

In contrast to the present study, Aboushelib and Sleem showed superior bond strength for Celtra-duo, a zirconia-reinforced lithium silicate, compared to IPS Empress 2 and IPS e.max CAD after HF application or airborne particle abrasion. The authors attributed the improved Celtra duo bond strength to the greater mechanical properties such as elastic modulus and flexural strength.<sup>[12]</sup> There is a direct correlation between the elastic modulus of the ceramic and its bonding performance,<sup>[21,22]</sup> and ceramics with smaller and denser crystals have increased stiffness, flexural strength, and higher bonding strength.<sup>[12]</sup> However, the results of this study failed to confirm the effective role of mechanical properties in adhesion performance of zirconia-reinforced glass ceramics. It seems that several factors interact with each other for optimal

adhesion. Further investigations are needed to clarify this tentative explanation.

All control specimens with no surface treatment showed pretest failure, indicating the importance of proper surface treatment for bonding resin materials to glass ceramics. In this study, HF-etching followed by silane coupling agent application produced the highest  $\mu$ SBS values after thermocycling while sandblasting resulted in lower  $\mu$ SBS values, regardless of the type of glass ceramic. This finding could be explained by different topographic patterns created by chemical and mechanical etching by HF-etching and sandblasting, respectively.<sup>[16,24,26,27]</sup> It is known that HF-etching selectively dissolves the glass matrix phase and exposes the crystalline portion of the ceramics creating a uniform microporous surface, i.e., quite different from that of sandblasted ceramic surfaces. Sandblasting creates irregular surfaces that form wedge-shaped fissures without creating uniform micro-retentive features and may produce microscopic cracks in glass ceramics.<sup>[12,16,26,27]</sup>

Another factor that may affect the difference in SBS values is the adhesive system and its composition. Clearfil liner bond F, an adhesive agent recommended in the Panavia F2.0 adhesive protocol has distinct hydrophilicity due to the incorporation of hydrophilic phosphate monomers,<sup>[28]</sup> which could weaken the adhesive interface through water uptake and hydrolysis of the Si–O-bonds,<sup>[17,28,29]</sup> especially after thermal aging. Since all of the specimens were subjected to 5000 cycles of thermal aging in the present study, the adverse effect of hydrophilic monomers and hydrolysis of the bond should be considered.

In order to simulate the thermal cycles of the oral cavity in the laboratory setting, thermocycling was used in the present study for all study groups. There is no agreement on a standardized thermocycling protocol in the literature, but specimens are usually subjected to 1000–100,000 cycles at 5°C–55°C.<sup>[30]</sup> It is estimated that approximately 10,000 thermal cycles correspond to 1 year of clinical function.<sup>[31]</sup> Therefore, the 5000 cycles of thermal aging applied in this study was equivalent to 6 months' clinical service.

Similar to other *in vitro* studies, this study had several limitations. The authors of the present study evaluated the bonding performance of resin-ceramic using one resin cement, one HF concentration, and one etching time; however, each parameter significantly affects the bonding strength, based on previous studies.<sup>[11,25,32]</sup>

Despite thermal aging, changes of pH levels and dynamic fatigue loading are other factors that might influence the durability of the resin bonds, which were not evaluated in the present study. Thus, further studies are needed to evaluate the effects of these parameters on bond strength and to investigate the clinical performance of different surface treatments.

## CONCLUSION

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

- VITA SUPRINITY showed lower bond strength to resin cement compared to IPS e.max press in all studied surface treatments
- HF application followed by silanization was the most effective surface treatment for both evaluated glass ceramics.

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## Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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