

Original Article

Effect of heat treatment and addition of 4-META to silane on microtensile bond strength of IPS e.max CAD ceramic to resin cement

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ABSTRACT

Background: Silane promotes bonding between ceramic and resin cement. Silane function could be improved by heat treatment and adding 4-Methacryloxyethyl trimellitate anhydride (4-META). The aim of this study was to assess the effect of heat treatment and adding 4-META to silanes on the microtensile bond strength of IPS e.max CAD ceramic and resin cement.

Materials and Methods: In this *in vitro* study, 32 IPS e.max CAD blocks (6 mm × 6 mm × 4 mm) were randomly divided into 8 groups of 4. The ceramic surfaces were etched by 10% hydrofluoric (HF) for 20 s, and Clearfil Ceramic Primer Plus, Clearfil Porcelain Bond Activator (CPA) +4-META, CPA + Clearfil SE Bond (CPA + SE), and Bis-Silane with and without Oven drying at 100°C for 5 min were applied on the ceramic surfaces as silanes, and then, the ceramics were bonded to composite disks by dual-cured resin cements. Ultimately, a total of 120 sticks (1 mm × 1 mm × 8 mm) were subjected to microtensile bond strength test (15 sticks from each group) and the failure types were assessed by stereomicroscope at 40x. Data were analyzed by two-sided analysis of variance test and Student's *t*-test the *P* values were considered to be < 0.05.

Results: The Bis Silane group (two bottles) showed maximum bond strength (*P* = 0.009). Heat treatment significantly reduced the bond strength in Bis Silane (*P* = 0.0001); but, in other groups, it did not show significant effect on the bond strength. Adding 4-META to CPA did not significantly affect the microtensile bond strength of IPS e.max CAD ceramic to resin cement, and there was no difference in mode of failure between groups.

Conclusion: The effect of silane type was significant on the microtensile bond strength of the ceramic to resin cement. The effect of 4-META on the bond strength was not significant. The interaction between silane type and heat treatment on the bond strength of ceramic to resin cement was also significant.

Key Words: IPS e.max CAD, resin cement, silane

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INTRODUCTION

Thanks to desirable translucence, fluorescence, abrasion resistance, biocompatibility, and chemical stability of ceramics, they have become the preferred

restorative material for esthetic dental works.^[1] IPS e.max CAD (Ivoclar vivadent, Schaan, Liechtenstein)

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is a monolithic lithium disilicate ceramic available in various colors and three translucencies.^[2] Depending on the chemical composition of ceramic, its bond strength to resin cement can be increased by a variety of surface conditioning methods.^[3]

In case of IPS e.max, surface conditioning must be carried out by etching with HF acid and applying silane-coupling agent.^[4] HF acid improves micromechanical retention by selective dissolving of glassy or crystalline matrix of ceramic to create a rough surface.^[3,5,6] Silane coupling agent improves the ceramic's surface energy and the cement's wettability and also enhances the bonding of ceramic's inorganic phase to the bonding agent's organic phase by creating a siloxane network over the ceramic surface.^[7] The bond between ceramic and composite resin is a factor of chemical bonding of silane-coupling agent and micromechanical retention resulting from grit blast or HF etching.^[8]

Reactions of silanes are in two ways: polymerization of organic functional component with organic matrix and reaction of alkoxy groups with inorganic matrix.^[9] The silane commonly used in dentistry is the gamma-methacryloxypropyltrimethoxysilane diluted in ethanol and water with pH of 4–5.^[10] Commercial silanes are available in two varieties: one bottle (prehydrolyzed) and two bottle (must be mixed for hydrolysis).^[8]

The silane compound 3-methacryloxypropyltrimethoxysilane is activated in low pH or by heat treatment.^[11] In two-bottle (nonhydrolyzed) silane products, the second bottle often contains acetic acid to reduce pH and facilitates hydrolysis.^[12] For one-bottle (hydrolyzed) silanes, the pH is reduced by a dentin-bonding agent containing a dentin-adhesive monomer. Kuraray has introduced a more convenient one-bottle silane that does not need to be mixed with dentin-bonding agent, but its bonding strength is lower than that of conventional one-bottle products because the acidic pH of containing 10-MDP has undesirable effects on polymerization process.^[13]

A study by Kitahara *et al.* (2013) on the bond strengths of one-bottle silane containing 10-MDP (Clearfil ceramic primer, Kuraray, Chiyoda, Tokyo, Japan) and one-bottle Clearfil Porcelain Bond Activator (CPA) (Chiyoda, Tokyo, Kuraray, Japan), reinforced with 5% and 10% 4-META, has reported a higher bond strength for 4-META reinforced silane and has attributed this to lower sensitivity of 4-META

to acidic pH.^[11] However, this study has not found any difference between varied concentrations of 4-META. This study has argued that once applied on ceramic surface, 4-META reacts with surface moisture and turns into 4-methacryloxyethyl trimellitate (4-MET); so, silane becomes activated and hydrolysis leads to formation of silanol groups that bind the ceramic by forming a siloxane structure.^[11]

It has been argued that heat treatment removes alcohol, water, and other byproducts from the ceramic surface and thus facilitates the completion of silicate silane condensation reaction and formation of covalent bond between silane and ceramic;^[7,14] this leads to a durable and reliable composite-ceramic bond without the need of micromechanical retention.^[15] Previous studies have tested the effect of different heat treatment methods and temperatures, such as heating in oven at 100°C for 2 min, at 100°C for 5 min, at 77°C for 1 min, rinsing with hot water and hot air on bond strength.^[6,14,16–18] Several studies have reported the effect of heat treatment on different silanes, but its effect on silanes containing 4-META is unknown.

This paper reports the results of an *in vitro* study on the effect of heat treatment and acidic monomer 4-META to silane on microtensile bond strength of dual-cured resin cement to IPS e.max CAD ceramic.

MATERIALS AND METHODS

Specimen preparation

This experimental *in vitro* study on the effect of heat treatment on the efficacy of silanes was carried out using three IPS e.max CAD ceramic blocks of size 14 (18 mm × 14 mm × 12 mm) (Ivoclar vivadent, Schaan, Liechtenstein). Each block was cut into 12 disk specimens (6 mm × 6 mm × 4 mm) by a cutting machine (Nemo Fanavar, Mashhad, Iran). The resulting 32 ceramic discs were randomly divided into 8 groups of 4. Specimens were heated in oven at 840°C for 13 min to ensure full crystallization. The bonding surface of every specimen was manually polished by 600–800 grit sandpaper under water spray and then subjected to ultrasonic cleaning (Biosonic UC50D, Whaledent, Altstätten, Switzerland) containing alcohol for 5 min. Each ceramic block was pushed into Polyvinyl siloxane impression material (Panasil Putty Fast, Kettenbach, Germany) to make a mold for composite. The composite Tetric N-Ceram A1 (Ivoclar vivadent, Schaan, Liechtenstein) was added to the mold incrementally. Each layer was

light cured for 40 s at 800 mW/cm² using Bluephase C8 (Ivoclar vivadent, Liechtenstein).

The silanes in the study were one-bottle Clearfil Ceramic Primer Plus (CCP) (Kuraray, Chiyoda, Tokyo, Japan), one-bottle CPA + Clearfil SE Bond (CPA + SE) (Kuraray, Chiyoda, Tokyo, Japan), and two-bottle Bis Silane (Bisco, Schaumburg, Illinois, USA). For the experimental specimens, CPA was mixed with 5% wt 4-META (Polyscience, Niles, Illinois, USA) (CPA + 4-META) to create an acidic environment, facilitate hydrolysis, and improve adhesion.

Ceramic and composite blocks were randomly divided into 8 groups:

- Group 1: In this group, ceramic specimens were etched with 10% HF for 20 s,^[19] rinsed with water spray (at room temperature) for 60 s, subjected to ultrasonic cleaning containing alcohol for 5 min, and dried with air spray (at room temperature) for 30 s. Bis Silane (Bisco, Schaumburg, Illinois, USA) was applied to the etched surface with a microbrush. After 1 min, the surface was dried by air spray for 15 s. The dual-cured resin cement Duo-Link (Bisco, Lasvevinu, USA) was applied to the conditioned ceramic surface as instructed in the product manual. Ceramic and composite blocks were placed (vertically) against each other and then subjected to a 1 kg vertical force for 10 s to remove the excess cement.^[18] Specimens were light cured at each side for 40 s using Bluephase C8 (Ivoclar, Vivadent, Schaan, Liechtenstein). Finally, ceramic and cement composite specimens were rinsed with water–air spray and stored for 24 h in distilled water at 37°C
- Group 2: After etching with 10% HF for 20 s and rinsing with water spray for 60 s, Bis Silane was applied to the specimens, and then, specimens were heated in oven at 100°C for 5 min. The rest of the procedure was followed as described for Group 1
- Group 3: After etching with 10% HF for 20 s and rinsing with water spray for 60 s, the CPA + 4-META silane was applied to the specimens, and the rest of the procedure was followed as described for Group 1
- Group 4: After etching with 10% HF for 20 s and rinsing with water spray for 60 s, CPA + 4-META silane was applied to the specimens, and then, specimens were heated in oven at 100°C for 5 min. The rest of the procedure was followed as described for Group 1

- Group 5: After etching with 10% HF for 20 s and rinsing with water spray for 60 s, a mixture of 1 drop of CPA with 1 drop of Clearfil SE primer (Kuraray, Chiyoda, Tokyo, Japan) was applied to the specimens. After 1 min, surface was dried by air spray for 15 s, and the rest of the procedure was followed as described for Group 1
- Group 6: After etching with 10% HF for 20 s and rinsing with water spray for 60 s, a mixture of 1 drop of CPA with 1 drop of silane Clearfil SE Bond primer was applied to the ceramics, and then, specimens were heated in oven at 100°C for 5 min. The rest of the procedure was followed as described for Group 1
- Group 7: After etching with 10% HF for 20 s and rinsing with water spray for 60 s, the CCP was applied to the specimens, and the rest of the procedure was followed as described for Group 1
- Group 8: After etching with 10% HF for 20 s and rinsing with water spray for 60 s, the silane CCP was applied to the specimens, and specimens were heated in oven at 100°C for 5 min. The rest of the procedure was followed as described for Group 1.

The main components of the materials used in the present study are shown in Table 1. For microtensile bond strength test, the specimens were cut by water-cooled diamond wheel at low speed under force of 10 kgf, starting from the resin and into the ceramic, ultimately giving 1 mm × 1 mm × 8 mm sticks (adhesive area = 1 ± 0.05 mm²) and total number of specimens were 120. Before the test, the bonded area of all sticks (15 sticks per group) were measured by a digital caliper.^[20,21] Using the microtensile tester (Bisco, Schaumburg, Illinois, USA), distance between two parts of the machine was adjusted to a fixed value for all specimens. Each stick was mounted on the jig with cyanoacrylate glue in a way that the ceramic-composite interface was in the midway and parallel to the horizontal plates keeping the rod. Specimens were then subjected to tensile force at crosshead speed of 1 mm/min until failure. Bond strength was obtained from the following equation:

$$R = F/A$$

Where:

- R: Bond strength (MPa)
- F: Force applied at the time of failure (Newton)
- A: Cross-sectional area of specimen (mm²).

After the microtensile bond strength testing, failure pattern was examined by a stereo microscope (Dewinter, New Delhi, Delhi, India) at $\times 40$ magnification and classified into 4 categories: adhesive, cohesive in ceramic, cohesive in composite, and mixed.

Statistical analysis of data was performed using SPSS® 24 (IBM Corp, Armonk, N.Y., USA) for analysis of variances, and the *P* values were considered to be <0.05 .

Primarily normality of data was tested by Shapiro–Wilk test, which had normal distributions, and then, the effects of silane types and heat treatment on the microtensile bond strength of resin cement to ceramic were determined by two-way analysis of variance (ANOVA) of results. Furthermore, a one-way ANOVA was performed on the data of odd numbered groups (i.e., groups 1, 3, 5, and 7) and separately on the data of even numbered groups (i.e., groups 2, 4, 6, and 8) to determine the effect of silane type on the microtensile bond strength with and without interference from the effect of heat treatment. The Tukey's test was performed to detect statistical differences between each pair of groups. To determine the effect of heat treatment on the microtensile bond

strength without interference from the effect of silane type, the data of groups with the same silane type but different treatments were analyzed by Student's *t*-test. The Chi-square test was used to search for statistical difference between failure modes of different groups.

RESULTS

The microtensile bond strengths of IPS e.max CAD ceramic and resin cement bonded with Bis Silane, CPA + 4-META, CPA + SE, and CCP with and without heat treatment are presented in Table 2 and shown in Figure 1. The results of two-way ANOVA show that the effects of silane type on the microtensile bond strength of IPS e.max CAD to resin cement are significant ($P = 0.009$), but the effect of heat treatment on this strength is insignificant ($P = 0.16$). Furthermore, the mutual effect of silane type and heat treatment on the bond strength of ceramic to resin cement was found to be statistically significant ($P = 0.0001$).

One-way ANOVA found the effect of silane type on microtensile bond strengths of specimens in odd-numbered groups (without heat treatment) to be statistically significant ($P < 0.0001$). Pairwise

Table 1: Composition of the materials used in this study

| Product | Manufacturer | Main components |
|-----------------------------------|-------------------|--|
| Bis Silane | Bisco | Ethanol, silane-coupling agent |
| Clearfil Porcelain Bond Activator | Kuraray | Silane-coupling agent, methacrylate derivatives |
| Clearfil SE bond | Kuraray | Bond: MDP, HEMA, dimethacrylate monomer, micro filler, catalyst Primer: MDP, HEMA, dimethacrylate monomer, water, catalyst |
| Clearfil Ceramic Primer Plus | Kuraray | Silane-coupling agent, MDP $<5\%$, ethanol |
| 4-META | PolyScience | 4-META |
| Tetric N Ceram composite | Ivoclar, Vivadent | UDMA, bis-GMA, ethoxylated bis-EMA, TEGDMA barium glass, ytterbium trifluoride, silicon dioxide Additives, stabilizers, catalysts |
| Duo-Link cement | Bisco | Base: Bis-GMA; Triethylene glycol dimethacrylate; urethane dimethacrylate; fill the glass; catalyst; Bis-GMA. Triethylene glycol dimethacrylate; fiberglass |
| IPS Emax.CAD/CAM | Ivoclar, Vivadent | Lithium disilicate |

Table 2: The mean of bond strength (MPa) of IPS e.max CAD ceramic to resin cement in the specimens prepared with different silanes with and without heat treatment ($n=15$)

| Group | Heat treatment | Mean \pm SD | Minimum | Maximum | Difference of means | <i>P</i> |
|--|----------------|---------------------------------|---------|---------|---------------------|-----------|
| Bis Silane (Bisco) | No | 32.5 \pm 4.05 ^A | 24.38 | 39.41 | 8.15 | <0.0001 |
| | Yes | 24.35 \pm 5.36 ^B | 13.37 | 30.92 | | |
| Clearfil Porcelain Bond Activator + 4-META | No | 25.13 \pm 6.03 ^B | 13.12 | 32.21 | 2.96 | 0.14 |
| | Yes | 28.09 \pm 4.67 ^{A,B} | 20.91 | 40.80 | | |
| Clearfil Porcelain Bond Activator + Clearfil SE Bond | No | 27.28 \pm 3.25 ^B | 22.53 | 31.81 | 0.93 | 0.42 |
| | Yes | 26.35 \pm 2.96 ^B | 21.99 | 30.26 | | |
| Clearfil Ceramic Primer Plus | No | 23.65 \pm 4.44 ^B | 15.14 | 35.93 | 1.47 | 0.39 |
| | Yes | 25.12 \pm 4.75 ^B | 18.47 | 33.17 | | |

Different uppercase letters show significant differences between various silanes in each column. SD: Standard deviation

comparison of silane groups with the Tukey test also found significant differences between the microtensile bond strengths of specimens prepared with Bis Silane and CPA + 4-META ($P < 0.0001$); Bis Silane and CPA + SE ($P < 0.01$); and Bis Silane and CCP ($P < 0.0001$), but not between CPA + 4-META and CPA + SE ($P = 0.57$); CPA + 4-META and CCP ($P = 0.81$); and CPA + SE and CCP ($P = 0.14$). The highest bond strength was observed in the specimen prepared with Bis Silane, and the lowest was observed in the one prepared with Clearfil Ceramic Primer.

One-way ANOVA found no statistically significant difference ($P = 0.13$) between the microtensile bond strengths of specimens in even numbered groups (with heat treatment). As shown in Figure 1 in these groups, the highest bond strength was observed in the specimen prepared with CPA + 4-META and the lowest in the specimen prepared with Bis Silane, but the difference between these two strength values was not great enough to be considered significant.

Student's *t*-test found the effect of heat treatment on the microtensile bond strength to be significant only when Bis Silane was used ($P < 0.0001$) and not when CPA + 4-META ($P = 0.14$); CPA + SE ($P = 0.42$); or CCP ($P = 0.39$) were used in preparation [Table 2].

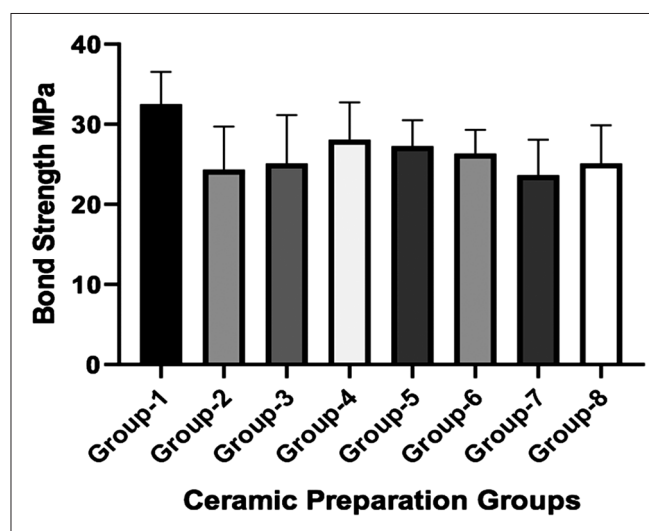


Figure 1: Bond strength of different surface preparation groups on ceramic (mean \pm standard deviationSD). Group- 1: Bis Silane, Group- 2: Bis Silane + Heat, Group- 3: Clearfil Porcelain Bond Activator + 4-META, Group- 4: Clearfil Porcelain Bond Activator + 4-METAeta + Heat, Group- 5: Clearfil Porcelain Bond Activator + Clearfil SE Bond Primer, Group- 6: Clearfil Porcelain Bond Activator + Clearfil SE Bond Primer + Heat, Group- 7: Clearfil Ceramic Primer Plus, Group- 8: Clearfil Ceramic Primer Plus + Heat.

And in general, adding 4-META did not significantly affect the microtensile bond strength of IPS e.max CAD ceramic to resin cement. Frequency and percentage of different failure modes in different groups are provided in Table 3. Chi-square test found no significant intergroup difference in the frequency of adhesive or mixed failures ($P = 0.9$). In other words, patterns of failure were found to be apparently unaffected by the silane type and whether or not specimen is heated.

DISCUSSION

Clinical success of ceramic restorations depends on quality and durability of the bond between ceramic and resin cement. Quality of this bond is a factor of bonding mechanisms which is determined in part by the method of surface conditioning aimed at creation of chemical or micromechanical retention to ceramic substrate. The present study investigated the effect of heat treatment and adding acidic monomer 4-META to silane on the microtensile bond strength of dual-cured resin cement to IPS e.max CAD ceramic.

For all specimens, HF acid etching was considered as a fixed treatment modality. Acid etching of ceramics is known to lead to a surface morphology well suited for micromechanical retention. In addition, acid etching is expected to lower surface contact angle and higher surface free energy and better wettability of luting agent.^[22] In this study, IPS e.max CAD ceramic specimens were etched with HF for 20 s as instructed by the manufacturer.

The bond strength of IPS e.max CAD ceramic to resin cement was measured by microtensile bond strength testing machine. This method gives a more realistic adhesive bond strength than shear testing, which is focused on the strength of the base material

Table 3: The percent of mode of failure in different groups

| Group | Heat treatment | Mixed (%) | Adhesive (%) |
|--|----------------|-----------|--------------|
| Bis Silane (Bisco) | Yes | 12 (80) | 3 (20) |
| | No | 9 (60) | 6 (40) |
| Clearfil Porcelain Bond Activator + 4-META | Yes | 8 (53.3) | 7 (46.7) |
| | No | 9 (60) | 6 (40) |
| Clearfil Porcelain Bond Activator + Clearfil SE Bond | Yes | 10 (66.7) | 5 (33.3) |
| | No | 9 (60) | 6 (40) |
| Clearfil Ceramic Primer | Yes | 9 (60) | 6 (40) |
| | No | 10 (66.7) | 5 (33.3) |

4-META: 4-methacryloxyethyl trimellitate anhydride

and overestimates cohesive failure.^[23,24] In addition, this test allows more uniform stress distribution in interfaces than shear testing^[25] and is known to reduce interfacial defects and remove nonuniform stresses in this area. In view of these features, measurements of this study were made using the microtensile bond strength test on ceramic sticks.^[26,27]

Composite resin was used as the bonding substrate to minimize interfering variables such as human dentine, quality and structure of exposed dentine, and size and number of tubuli openings and surface treatment.^[18]

Silanization is the leading method of ceramic surface conditioning. It has been shown that silanes have a deep impact on the bonding of resin to lithium disilicate ceramics.^[28] Silanization improves resin ceramic adhesion and facilitates resin penetration into acid-etched ceramic by increasing the surface wettability. In addition, heat treatment of silane may increase the bond strength of ceramic to resin cement.^[18] When applied to the ceramic surface, silane tends to develop a layered structure.^[16] Heat treatment at 100°C can therefore be expected to increase the bond strength of composite to ceramic by integrating the surface layers and removing intermediate phase.^[29]

The results of this study, however, show that heat treatment has a very limited effect on the microtensile bond strength of IPS e.max CAD ceramic to dual-cured resin cement. In the case of (unhydrolyzed) Bis Silane, heat treatment showed a significant negative effect on microtensile bond strength ($P < 0.0001$) but for other tested silanes (CPA + 4-META, CPA + SE [unhydrolyzed]) and CCP (prehydrolyzed), the effect of heat treatment was overall insignificant.

In CPA + 4-META and prehydrolyzed CCP, heat treatment resulted in a statistically insignificant increase in the bond strength of IPS e.max CAD to resin cement. A study by Abduljabbar *et al.* (2016) reported an increase in the bond strength of lithium disilicate ceramic exposed to prehydrolyzed Calibra silane-coupling agent (Dentsply Caulk, Milford, Delaware, USA) after heat treatment at 100°C for 5 min, which is somewhat consistent with our results. Note that, etching time in that study was 90 s, but we etched the specimens for 20 s.^[18]

Carvalho *et al.* (2015) reported an increase in the microtensile bond strength of feldspathic ceramic prepared with Clearfil Ceramic Primer and heat treatment at 100°C for 2 min.^[30]

Several other studies also reported the positive effect of heat treatment and rinsing with hot water on bond strength of ceramic and resin cement even after storage of specimens and exposure to thermal cycles.^[14,31] This positive effect is possibly due to enhancement of cross-linking network and removal of physisorbed and chemisorbed areas of surface layer leading to better exposure of reactive areas near the ceramic surface.^[32]

In other words, the effect of heat treatment on the efficacy of silane could be due to improvement of cross-linking, prevention of water penetration in silane layer,^[16] oligomerization via bonding of glass matrix to silane agent,^[33] evaporation of solvent and residues of silanol hydrolysis, and finally, formation of extensive internal pressure gradients.^[34]

Corazza *et al.* (2013) have studied the effects of heat treatment after application of prehydrolyzed Monobond S (Ivoclar vivadent, Schaan, Liechtenstein) on feldspathic CAD/CAM ceramic and also rinsing with hot water on the microtensile bond strength of resin cement to this ceramic and have found that drying in oven at 77°C results in improved bond strength, but rinsing with hot water has the opposite effect.^[35] Rinsing of silanized surface with hot water reduces the thickness of silane coating and removes the outermost unreacted layer of silane agent.^[36] The thickness of silane also affects bond strength.^[32] Although application of too much coupling agent does not guarantee a reliable bonding,^[16] a thicker silane layer contributes to the strength of cross-linking network^[32,37] and pressed structures requires a thick layer of silane to exhibit better mechanical properties,^[38,39] so silane layer needs to be applied with an optimum thickness, that is, a thickness which results in a bond between organic and inorganic matter, with ability to absorb mechanical stress without cracking in the silane body.^[39] In the present study, the heat-treated Bis silane specimens showed significantly lower ceramic-cement bond strength, which may be due to the thinning of silane layer as a result of heating.

Our results support the results of the study of de Carvalho *et al.* (2011) on the effect of Clearfil Ceramic Primer on the microtensile bond strength of resin cement silane to feldspathic ceramic, which has found no such effect. However, heat treatment of that study was 2 min long but ours was 5 min.

Although the aim of heat treatment is to reinforce the bond between ceramic and resin cement, there is

no general consensus regarding the efficacy of this approach. Our results also bring up this question, as the effect of heat treatment on bond strength between IPS e.max CAD ceramic and resin cement was found to be dependent on the type of silane although insignificantly. It has been argued that heating at higher temperatures cleans the ceramic surface from water, alcohol, and products of silane reaction and leads to greater condensation of silicate silane and thereby improves formation of siloxane.^[17] Evaporation of alcohol and acetic acid may also lead to increased local density of agents available for reaction with ceramic.^[17] This divergence of results concerning the effect of heat treatment on bond strength between ceramic and resin cement could be due to difference in the silane type, composition, solvents and reactions and their stability, and also the details of application, such as air temperature, method and duration of heating, and whether specimen is rinsed with hot water. Therefore, there seems to be a desperate need for further research on standardization of heating techniques to be utilized after application of silane agent.

Quality of the bond of ceramic to resin cement after silanization depend not only on the silane properties such as pH, concentration, solvent, and hydrolysis duration but also on application protocol such as drying condition, the time between silanization and bonding, temperature, and humidity.^[40] Silane adhesion decreases with the increase in air humidity, which affects the extent of absorption.^[41] In this study, silanization was performed at room temperature (about 20°C) and was followed by 5 min of heat treatment at 100°C.

Silanization is crucial for bonding of ceramic to resin cement.^[42] For silane agent to be activated, it needs to be mixed with an acid agent.^[43] After this mixing, hydrolysis of silane leads to formation of silanol groups, which facilitates the bonding by forming the siloxane structure. Common silane agents must be mixed with a dentin-bonding agent containing an acidic-bonding monomer. But more convenient one-bottle products already mixed with dentin bonding monomer are also available.^[44]

The result of this study showed that heat treatment has a limited effect on improvement of microtensile bond strength in prehydrolyzed silanes which is similar to studies by Carvalho *et al.* (2015) and Abduljabbar *et al.* The study of Kitahara *et al.* reported a higher

bond strength for 4-META-reinforced silane but has not found any difference between bond strengths resulting from different concentrations of 4-META,^[11] which is in contradiction with our results. It has been argued that surface moisture turns 4-META into 4-MET, and this leads to activation of silane, and then hydrolysis, formation of silanol groups, formation of siloxane structure, and ultimately the bonding. This mechanism is expected to strengthen the bond, but we found no increase in bond strength due to addition of 4-META to CPA (without heat treatment). This inconsistency could be due to differences in the type of bond strength test, resin cement, or ceramic since Kitahara *et al.* have used the shear bond strength test and composite flow as resin cement and have focused on feldspathic ceramic whereas we used microtensile bond strength test and dual-cured resin cement and concentrated on lithium disilicate CAD/CAM ceramic.

Quality of ceramic-cement bond cannot be examined exclusively by estimation of bond strength; it also requires close examination of failure modes. In the present study, regardless of heat treatment, the specimens prepared with different silanes exhibited either mixed or adhesive failure. Furthermore, no significant difference was found in terms of failure mode between different groups. In all groups, however, the frequency of mixed failure was higher than adhesive failure.

None of the specimens tested in this study failed in cohesive mode. Similarly, previous studies have reported a higher frequency of adhesive and mixed failures than cohesive ones.^[45] In the case of our study, the higher frequency of adhesive and mixed failures can be attributed to the fact that microtensile test evaluates a small cross-section and also resin cement bond is stronger to composite than to ceramic, which bond is chemical. Hence, the mechanism and pattern of failure at microscopic level seem to require further research.

The effect of addition of 4-META to CPA silane on microtensile bond strength was the same as silane containing 10-MDP (CCP) and CPA + SE which can be used in making one-bottle silane and simplifying the method. *In vitro* studies provide important information when assessing biomaterials; however, they have limitations and do not replace clinical trials. Furthermore, *in vitro* studies are required to evaluate the durability of the formed bond after thermo cycling and load cycling tests. Future studies

are recommended to assess the shelf life of silanes containing 4-META and 10-MDP, the effect of longer heating times, higher concentration of 4-META, and to evaluate the bond strength of ceramic to resin cement after aging.

CONCLUSION

The findings of this study showed that heat treatment of Bis-Silane decreased the microtensile bond strength but had no significant effect on the other three silanes. By adding 4-META to CPA, one-bottle silane with a bond strength as high as CCP (10-MDP) and CPA + SE but lower than Bis-Silane was made. Heat treatment of the silane containing 4-META increased the microtensile bond strength which has no significant difference with Bis-Silane without heat treatment (maximum bond strength).

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Conflicts of interest

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REFERENCES

- Silva LH, Lima E, Miranda RB, Favero SS, Lohbauer U, Cesar PF. Dental ceramics: A review of new materials and processing methods. *Braz Oral Res* 2017;31:e58.
- Daryakenari G, Alaghehmand H, Bijani A. Effect of simulated mastication on the surface roughness and wear of machinable ceramics and opposing dental enamel. *Oper Dent* 2019;44:88-95.
- Petrauskas A, Novaes Olivieri KA, Pupo YM, Berger G, Gonçalves Betiol EÁ. Influence of different resin cements and surface treatments on microshear bond strength of zirconia-based ceramics. *J Conserv Dent* 2018;21:198-204.
- Dünder M, Ozcan M, Gökçe B, Cömlekoğlu E, Leite F, Valandro LF. Comparison of two bond strength testing methodologies for bilayered all-ceramics. *Dent Mater* 2007;23:630-6.
- Dilber E, Yavuz T, Kara HB, Ozturk AN. Comparison of the effects of surface treatments on roughness of two ceramic systems. *Photomed Laser Surg* 2012;30:308-14.
- Aboushelib MN, Sleem D. Microtensile bond strength of lithium disilicate ceramics to resin adhesives. *J Adhes Dent* 2014;16:547-52.
- de Carvalho RF, Martins ME, de Queiroz JR, Leite FP, Ozcan M. Influence of silane heat treatment on bond strength of resin cement to a feldspathic ceramic. *Dent Mater J* 2011;30:392-7.
- Bruzi G, Carvalho AO, Giannini M, Maia HP, Magne P. Silane application can influence the bond strength to lithium disilicate. *Dent Mater* 2015;31 Suppl 1:e9.
- D'Arcangelo C, D'Amario M, Vadini M, De Angelis F, Caputi S. Influence of surface treatments on the flexural properties of fiber posts. *J Endod* 2007;33:864-7.
- Perdigao J, Gomes G, Lee IK. The effect of silane on the bond strengths of fiber posts. *Dent Mater* 2006;22:752-8.
- Kitahara N, Itoh K, Kusunoki M, Oikawa M, Miyazaki T. One-bottle silane coupling agent containing 4-META. *Dent Mater J* 2013;32:409-12.
- Baratto SS, Spina DR, Gonzaga CC, Cunha LF, Furuse AY, Baratto Filho F, *et al.* Silanated surface treatment: Effects on the bond strength to lithium disilicate glass-ceramic. *Braz Dent J* 2015;26:474-7.
- Ikemura K, Tanaka H, Fujii T, Deguchi M, Endo T, Kadoma Y. Development of a new single-bottle multi-purpose primer for bonding to dental porcelain, alumina, zirconia, and dental gold alloy. *Dent Mater J* 2011;30:478-84.
- Fabianelli A, Pollington S, Papacchini F, Goracci C, Cantoro A, Ferrari M, *et al.* The effect of different surface treatments on bond strength between leucite reinforced feldspathic ceramic and composite resin. *J Dent* 2010;38:39-43.
- Cotes C, de Carvalho RF, Kimpura ET, Leite FP, Ozcan M. Can heat treatment procedures of pre-hydrolyzed silane replace hydrofluoric acid in the adhesion of resin cement to feldspathic ceramic? *J Adhes Dent* 2013;15:569-74.
- Pereira CN, Buono VT, Mota JM. The influence of silane evaporation procedures on microtensile bond strength between a dental ceramic and a resin cement. *Indian J Dent Res* 2010;21:238-43.
- Shen C, Oh WS, Williams JR. Effect of post-silanization drying on the bond strength of composite to ceramic. *J Prosthet Dent* 2004;91:453-8.
- Abduljabbar T, AlQahtani MA, Jaaidi ZA, Vohra F. Influence of silane and heated silane on the bond strength of lithium disilicate ceramics mics *in vitro* study. *Pak J Med Sci* 2016;32:550-4.
- Mokhtarpour F, Alaghehmand H, Khafri S. Effect of hydrofluoric acid surface treatments on micro-shear bond strength of CAD/CAM ceramics. *Electron Physician* 2017;9:5487-93.
- Mousavinasab SM, Atai M, Barekaini M, Fattahi P, Fattahi A, Rakhshan V. Effects of ethanol concentrations of acrylate-based dental adhesives on microtensile composite-dentin bond strength and hybrid layer structure of a 10 wt% polyhedral oligomeric silsesquioxane (POSS)-incorporated bonding agent. *Dent Res J (Isfahan)* 2018;15:25-32.
- Nassoohi N, Kazemi H, Sadaghiani M, Mansouri M, Rakhshan V. Effects of three surface conditioning techniques on repair bond strength of nanohybrid and nanofilled composites. *Dent Res J (Isfahan)* 2015;12:554-61.
- Addison O, Marquis PM, Fleming GJ. The impact of hydrofluoric acid surface treatments on the performance of a porcelain laminate restorative material. *Dent Mater* 2007;23:461-8.
- Fernandes VV Jr., Oliani MG, Nogueira L Jr., Silva JM, Araújo RM. Analysis and Comparison of Different Bond Strength Tests; 2016;4:1076.W
- Della Bona A, Borba M, Benetti P, Cecchetti D. Effect of surface

- treatments on the bond strength of a zirconia-reinforced ceramic to composite. *Braz Oral Res* 2007;21:10-5.
25. Braga RR, Meira JB, Boaro LC, Xavier TA. Adhesion to tooth structure: A critical review of “macro” test methods. *Dent Mater* 2010;26:e38-49.
 26. Beloica M, Goracci C, Carvalho CA, Radovic I, Margvelashvili M, Vulicevic ZR, *et al.* Microtensile vs. microshear bond strength of all-in-one adhesives to unground enamel. *J Adhes Dent* 2010;12:427-33.
 27. Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, *et al.* Relationship between bond-strength tests and clinical outcomes. *Dent Mater* 2010;26:e100-21.
 28. Alfredo MH, Luiz CC, Alito A, Sylvio MJ. Effect of different ceramic surface treatments on resin microtensile bond strength. *J Prosthodont* 2004;13:28-35.
 29. Roulet JF, SJF, S JF KJ, Longmate J. Effects of treatment and storage conditions on ceramic/composite bond strength. *J Dent Res* 1995;74:381-7.
 30. de Carvalho RF, Cotes C, Kimpara ET, Leite FP, Özcan M. Heat treatment of pre-hydrolyzed silane increases adhesion of phosphate monomer-based resin cement to glass ceramic. *Braz Dent J* 2015;26:44-9.
 31. Hooshmand T, van Noort R, Keshvad A. Bond durability of the resin-bonded and silane treated ceramic surface. *Dent Mater* 2002;18:179-88.
 32. Mansura AA, Nascimentob OL, Vasconcelos WL, Mansura HS. Chemical functionalization of ceramic. Tile surfaces by silane coupling agents: Polymer modified mortar adhesion mechanism implications. *Mater Res* 2008;11:293-302.
 33. Bertelsen CM, Boerio FJ. Linking mechanical properties of silanes to their chemical structure: An analytical study of dy of y of study of ar adheProg Org Coat 2001;41:239-46.
 34. El-Nahhal IM, El-Ashgar NM. A review on polysiloxane-immobilized ligand systems: Synthesis, characterization and application. *J Organometal Chem* 2007;692:2861-86.
 35. Corazza PH, Cavalcanti SC, Queiroz JR, Bottino MA, Valandro LF. Effect of post-silanization heat treatments of silanized feldspathic ceramic on adhesion to resin cement. *J Adhes Dent* 2013;15:473-9.
 36. Vechiato Filho AJ, dos Santos DM, Goiato MC, de Medeiros RA, Moreno A, Bonatto Lda R, *et al.* Surface characterization of lithium disilicate ceramic after nonthermal plasma treatment. *J Prosthet Dent* 2014;112:1156-63.
 37. Hoikkanen M, Honkanen M, Vippola M, Lepisto T, Vuorinen J. Effect of silane treatment parameters on the silane layer formation and bonding to thermoplastic urethane. *Prog Org Coat* 2011;72:716-23.
 38. Della Bona A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramics. *J Adhes Dent* 2000;2:305-13.
 39. Queiroz JR, Benetti P, Ozcan M, de Oliveira LF, Della Bona A, Takahashi FE, *et al.* Surface characterization of feldspathic ceramic using ATR FT-IR and ellipsometry after various silanization protocols. *Dent Mater* 2012;28:189-96.
 40. Abel ML, Allington LD, Digby RP, Porritt N, Shaw SJ, Watts JF. Understanding the relationship between silane application conditions, bond durability and locus of failure. *Int J Adhesi Adhes* 2006;26:2-15.
 41. Underhill RP, Goring G, Du Quesnay DL. The effect of humidity on the curing of 3-glycidoxypropyltrimethoxy silane. *Int J Adhesi Adhes* 2000;20:195-9.
 42. Shayegh SS, Amini P, Yaghmai K, Massoumi F, Abbasi K. Comparison of the effect of three methods of porcelain surface treatment on shear bond strength of composite to porcelain. *JIDAI* 2016;28:72-7.
 43. Matos NR, Costa AR, Valdrighi HC, Correr AB, Vedovello SA, Santamaria M Jr, *et al.* Effect of acid etching, silane and thermal cycling on the bond strength of metallic brackets to ceramic. *Braz Dent J* 2016;27:734-8.
 44. Alex G. Preparing porcelain surfaces for optimal bonding. *Compend Contin Educ Dent* 2008;29:324-35.
 45. Moharamzadeh K, Hooshmand T, Keshvad A, Van Noort R. Fracture toughness of a ceramic-resin interface. *Dent Mater* 2008;24:172-7.