ORIGINAL RESEARCH

Effect of Silanization on Microtensile Bond Strength of Different Resin Cements to a Lithium Disilicate Glass Ceramic

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ABSTRACT

Aim: This study evaluated the influence of a silane-coupling agent on the bond strength of a self-adhesive cement and a conventional resin cement to a lithium disilicate glass ceramic.

Materials and methods: A total of eight ceramic blocks were fabricated and divided into four groups (n = 2). In groups 1 and 3, ceramic surfaces were etched with hydrofluoric acid 10% for 20 seconds, rinsed for 30 seconds, and air-dried. One layer of a silane agent was applied onto all ceramic specimens and air-dried for 30 seconds. In groups 2 and 4, ceramic surfaces were etched with hydrofluoric acid, rinsed, and air-dried without application of the silane-coupling agent. The ceramic blocks were bonded to a block of composite with a self-adhesive resin cement or with a conventional resin cement, according to the manufacturer's instructions. After 24 hours in distilled water at 37°C, the specimens were sectioned perpendicular to the bonding interface area to obtain beams with a bonding area of 0.8 mm^2 and submitted to a microtensile bond strength test at a crosshead speed of 0.5 mm/min. Data were statistically analyzed with one-way analysis of variance and the Games–Howell *post hoc* test (p = 0.05). Fractured specimens were examined under optical microscopy at 40× magnification.

Results: Silanization resulted in higher microtensile bond strength compared to groups without silane. No significant differences were found between the conventional resin cement and the self-adhesive resin cement with silane agent (p = 0.983), and without silane agent (p = 0.877).

Conclusion: Silanization appears to be crucial for resin bonding to a lithium disilicate-based ceramic, regardless of the resin cement used. The self-adhesive resin cement performed as well as the conventional resin cement.

Clinical significance: Applying one layer of a silane-coupling agent after etching the ceramic surface with hydrofluoric acid 10% enhanced the bond strength between resin cements and a glass ceramic.

Keywords: Laboratory research, Microtensile bond strength, Resin cement, Silanization.

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INTRODUCTION

Success with resin-bonded all-ceramic restorations is highly dependent on obtaining a durable and trustworthy bond, which must integrate all parts of the system into one consistent structure.[1] Adhesive bonding depends on the surface energy and wettability of the adherent by the adhesive.[2,3] The adhesion between resin-based composites and dental ceramics is the result of a physicochemical interaction across the interface between the adhesive and the substrate.[2]

A newly introduced lithium disilicate glass ceramic (IPS e.max Press, Ivoclar Vivadent, Schaan, Liechtenstein) may be adhesively cemented. Bonding to this type of ceramic material is based in a mechanical and chemical interaction, promoted by hydrofluoric acid and the silane-coupling agent respectively.[4] Hydrofluoric acid etching removes the glassy matrix and the second crystalline phase, resulting in irregularities in the lithium disilicate crystals of the IPS e.max system, allowing a mechanical union with resin composites.[5-7] The silane-coupling

agent presents bifunctional characteristics, promoting a chemical interaction between the silica in the glass phase of ceramics and the methacrylate groups of the resin through siloxane bonds.[8,9]

Silane agents have been used since 1949 to improve the bonding among adhesive resins with organic components, ceramics, and metals, but in 1977 Eames et al[10] suggested the use of silane agent in dentistry. Previous studies report the effectiveness of the silane-coupling agent to improve the bond strength between resin composites and ceramics[11,12] and between resin cements and ceramics[9,13,14]; however, some studies[2,15-17] question the use of silane. Some authors concluded that the application of a silane agent did not show a significant effect in the bond strength between ceramic materials and resin cements.[2,15,17]

To simplify the clinical procedures and overcome the technique sensitivity of multistep luting systems, resin cements that combine an acid primer and cement in one application, called self-adhesive resin cements, have been introduced.[3,15] Self-adhesive resin cements do not require pretreatment and their application is accomplished using a single clinical step. Nevertheless, there are controversies in the literature about the bonding capacity of this type of adhesive agent to the ceramic surface treated or not treated with a silane-coupling agent.

Therefore, the aim of the present study was to evaluate the influence of silane-coupling agent on the microtensile bond strength of a self-adhesive cement and a conventional resin cement to a glass ceramic reinforced with lithium disilicate, the IPS e.max system. The null hypotheses tested were as follows: (1) the silane agent does not influence the bond strength among resin cements and the ceramic reinforced with lithium disilicate; (2) the microtensile bond strength is not associated with the type of cement used.

MATERIALS AND METHODS

Ceramic Blocks

A total of eight rectangular blocks (11 mm long × 9 mm wide × 4 mm thick) of IPS e.max Press were fabricated in accordance with the manufacturer's instructions.

Rectangular wax patterns were created, sprued, and attached to a muffle base with a surrounding paper cylinder. The wax patterns were invested with phosphate-based material (IPS PressVest Speed, Ivoclar Vivadent), and the wax was eliminated in an automatic furnace (Vulcan A-550, Degussa-Ney, Yucaipa, California, USA) at 850°C for 1 hour. The IPS e.max Press ceramic ingots were pressed into the molds in an automatic press furnace (EP 500, Ivoclar Vivadent). After cooling, the specimens were divested and submitted to wet polishing with silicon carbide grinding paper (grits #220, #360, and #600) (Acqua Flex-Norton, São Paulo, Brazil) in a polishing machine (Panambra, São Paulo, Brazil). The blocks were cleaned in ultrasonic bath with distilled water for 5 minutes, air-dried, and divided into four groups (n = 2) according to the resin cement and the surface treatment performed.

Composite Resin Blocks

A total of eight rectangular blocks (11 mm long × 9 mm wide × 4 mm thick) of composite Filtek Z350, shade A2 (3M ESPE, St Paul, Minnesota, USA) were fabricated. To manufacture the composite resin blocks, molds from all the ceramic blocks were obtained with an elastomeric mold (Virtual, Ivoclar Vivadent, Schaan, Liechtenstein). The composite resin was inserted in the mold incrementally (2 mm thick). Each increment was light cured for 20 seconds using a light-emitting diode (LED) source (Translux Power Blue, Hareaus Kulzer, Germany) with an irradiance of 750 mW/cm^2.

Table 1 shows the materials used in the study, their composition, and mode of use.

Table 1: Materials, manufacturers, composition, and mode of use

Product name	Composition	Manufacturer	Batch number	Instructions for use
IPS e.max Press ingots	SiO$_2$; Li$_2$O; K$_2$O; MgO; ZnO; Al$_2$O$_3$; P$_2$O$_5$; other oxides	Ivoclar Vivadent, Schaan, Liechtenstein	R37944	x
Monobond S	3-methacryloxy-propyl-trimethoxysilane (1 wt%), water/ethanol solution containing acetic acid set to pH 4 (99 wt%)	Ivoclar Vivadent, Schaan, Liechtenstein	P70737	–Apply on the ceramic surface for 60 s –Gently air-dry for 30 s
SpeedCEM	Acidic monomers, dimethacrylates, barium glass, ytterbium trifluoride, co-polymer, silicon dioxides, catalysts, stabilizers, pigments	Ivoclar Vivadent, Schaan, Liechtenstein	S33619	–Apply on surface –Lute resin block using light pressure –Light polymerize for 20 s each side
Variolink II	BisGMA, TEGDMA, UDMA, benzoyl peroxide, inorganic fillers, ytterbium trifluoride, Ba-Al fluorosilicate glass, spheroid mixed oxide, initiator, stabilizers, pigments	Ivoclar Vivadent, Schaan, Liechtenstein	R69347	–Mix base and catalyst paste –Lute resin block using light pressure –Light cure for 40 s from each side

BisGMA: bisphenol A glycidylmethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate

Ceramic Surface Treatment

The eight ceramic blocks were randomly divided into four groups (n = 2). In groups 1 and 3, the test surface of the ceramic blocks was etched with hydrofluoric acid 10% (Condac Porcelana, FGM, Joinville/SC, Brazil) for 20 seconds, rinsed with water for 30 seconds, and air-dried for 30 seconds. One layer of a silane-coupling agent (Monobond S, Ivoclar Vivadent, Schaan, Liechtenstein) was applied onto all ceramic specimens and air-dried for 30 seconds. In groups 2 and 4, the ceramic surfaces were etched with hydrofluoric acid 10% for 20 seconds, rinsed with water for 30 seconds, and air-dried for 30 seconds without application of the silane-coupling agent.

In groups 1 and 2, the self-adhesive resin cement SpeedCEM (Ivoclar Vivadent, Schaan, Liechtenstein) was applied directly to the previously treated ceramic surface. In groups 3 and 4, the resin cement Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein) was applied following the manufacturer's instructions.

Cementing the Composite to the Ceramic

After the resin cement was applied in the ceramic surface, the composite resin block was positioned over the resin cement under a 500-gm static load for 2 minutes.[4] The excess cement was removed with a disposable microbrush. Light activation was performed for 40 seconds at right angles to each of the IPS e.max Press/Filtek Z350 margins using an LED source (Translux Power Blue, Hareaus Kulzer, Germany) with a final 40-second light exposure from the top surface.

Microtensile Bond Strength Testing

After 24 hours of storage in distilled water at 37°C, the specimens were sectioned perpendicular to the bonding interface area to obtain beams with a bonding area of 0.8 mm^2 using a water-cooled diamond blade (Buehler Wafering Blades, Buehler Ltd., Illinois, USA) in a low-speed saw machine (Isomet 1000, Buehler, Lake Bluff, Illinois, USA). The cross-sectional area of the bond interface of each beam was measured using a digital caliper (KingTools, São Paulo, Brazil). Thirty specimens from each group were randomly selected for the microtensile bond strength test.

Each beam was fixed to the grips of a microtensile device using a cyanoacrylate adhesive, and the microtensile bond test was conducted in a testing machine (Instron 4444, Instron Corp., Canton, Massachusetts, USA) at 0.5 mm/min crosshead speed until failure.

The fractured specimens were observed under optical microscopy (Olympus Corp., Tokyo, Japan) at 40× magnification.

Table 2: Descriptive statistics (in MPa) of microtensile bond strengths of the four groups tested

Groups	Adhesive	Silane	Mean	SD	Min	Max
1	SpeedCEM	Yes	22,162a	10.32	5.75	42.87
2	SpeedCEM	No	14,434b	5.08	6.02	23.98
3	Variolink	Yes	21,485a	4.66	7.78	27.89
4	Variolink	No	15,819b	4.08	5.79	23.14

Means with the same letters are not significantly different by Games–Howell *post hoc* test at 5%; SD: standard deviation

Failure modes were classified as adhesive, mixed, or cohesive. In the event of spontaneous debonding, specimens were excluded from the statistical analysis. Bond strength data were statistically analyzed with one-way analysis of variance (ANOVA) and the Games–Howell *post hoc* test (p = 0.05).

RESULTS

Descriptive statistics of the microtensile bond strength (MPa) of the different subgroups are illustrated in Table 2. Normality of the distribution was attested with Shapiro–Wilk test (p > 0.05). One-way ANOVA showed the presence of significant differences among the groups (p < 0.0001). Games–Howell *post hoc* test showed that silane coupling resulted in higher microtensile bond strength compared to groups without silane. No significant differences were found between Variolink and SpeedCEM with silane coupling (p = 0.983) and between Variolink and SpeedCEM without silane coupling (p = 0.877).

DISCUSSION

The results of the present study revealed that the treatment of the glass-ceramic surface with the silane-coupling agent increased the microtensile bond strength for both evaluated resin cements; thus, the first null hypothesis must be rejected.

The type of cement did not influence the bond strength values. The self-adhesive and the conventional resin-based cements presented similar performance; therefore, the second hypothesis must be accepted.

Applying silane-coupling agent on the ceramic surface after etching with hydrofluoric acid is a well-established protocol.[11,18] Due to its bifunctional characteristics, application of silane on the etched ceramic surface may increase the chemical bonding between the ceramic and resin materials once the silane agents, by means of siloxanic bonds, couple the silica oxides present in ceramics to the organic matrix of resin cements.[8]

Our results reveal that application of silane was fundamental in order to achieve durable adhesion between both resin cements tested and a glass ceramic

reinforced with lithium disilicate, suggesting that beyond the micromechanical bond promoted by the etching with hydrofluoric acid, the siloxanic bond obtained with the silane-coupling agent application was important.[19] These results are in agreement with other studies[9,13,14] that reported the effectiveness of the silane-coupling agent in improving the bond strength of ceramic materials and resin cements.

SpeedCem is a self-adhesive resin cement with adhesive monomers with long methacrylate chains and a phosphoric acid group which can establish chemical bonds with the dental structure and with ceramic surfaces, so no additional adhesive agents are needed.[20] In the present study, when the silane-coupling agent was applied prior to the application of the self-adhesive cement, statistically higher bond strength was obtained. This result could be explained by the fact that the surface treatment of the ceramic surface with the silane-coupling agent promotes a chemical bond between the ceramic and the resin cement and increases the cement wettability of the ceramic surface, improving the contact of the resin cement with the ceramic surface and enhancing the infiltration of the resin cement in the irregularities obtained with the acid etching.[21]

The conventional resin cement Variolink II is an etch-and-rinse, dual-curing luting composite resin with the presence of bisphenol A glycidylmethacrylate (BisGMA), urethane dimethacrylate, triethylene glycol dimethacrylate (TEGDMA), and filler particles. The treatment of the ceramic surface with the silane-coupling agent previously to the resin cement application provides a better wettability of the ceramic surface and also a chemical bond between the reactive organic groups of the silane-coupling agent and the resin molecules of the resin cement, like BisGMA and TEGDMA.[5] In addition, the hydrolyzable monovalent groups, present in the silane-coupling agent composition, chemically bond the silica present in the glass ceramic.[7]

Unlike other studies that questioned the use of silane-coupling agents in the etched ceramic surface,[2,15,16,22] in this study, the application of the silane-coupling agent was fundamental for the union of resin cements with the ceramic.

Monobond S ceramic primer is a single-phase preactivated solution based on the 3-methacryloxypropyltrimethoxysilane molecules. These solutions are composed of bifunctional molecules that bond silicon dioxide with the –OH groups on the ceramic surface,[23] and normally contain a silane coupler and a weak acid, which enhances the formation of siloxane bonds.[24] They also have a degradable functional group that copolymerizes with the organic matrix of the resin.[25]

Various methods are available for assessment of the bond strength. In this study, the microtensile bond strength test, preconized by Sano et al,[26] but without additional wear to obtain an hourglass specimen, was performed in an attempt to eliminate the nonuniform stress distribution within the adhesive zone.[27] The bonded interfaces of the specimens used in this study are approximately 0.8 mm^2, allowing a more uniform stress distribution during loading and failure predominantly at the adhesive interface.

In the present study, three specimens failed cohesively in the ceramic substrate, which is in line with Della Bonna et al[11] who found that most of the failures in microtensile bond strength tests that evaluate the bond strength between composite resin and ceramic occurred within the adhesion zone.

CONCLUSION

Based on the current results, the following can be concluded:

- Applying a silane-coupling agent in the etched ceramic surface enhanced the microtensile bond strength between the evaluated resin cements and the glass-ceramic surface.
- The self-adhesive resin cement performed as well as the conventional resin cement when bonded to a glass ceramic, regardless of the ceramic surface treatment.

CLINICAL SIGNIFICANCES

The current results indicate that applying one layer of a silane-coupling agent after etching the ceramic surface with hydrofluoric acid 10% enhanced the bond strength between resin cements and a glass-ceramic surface.

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REFERENCES

1. Pollington S, Fabianelli A, Van Noort R. Microtensile bond strength of a resin cement to a novel fluorcanasite glass-ceramic following different surface treatments. Dent Mater 2010 Sep;26(9):864-872.
2. Della Bonna A, Shen C, Anusavice KJ. Work of adhesion of resin on treated lithia disilicate-based ceramic. Dent Mater 2004 May;20(4):338-344.
3. Pisani-Proenca J, Erhardt MC, Valandro LF, Gutierrez-Aceves G, Bolanos-Carmona MV, Del Castillo-Salmeron R, Bottino MA. Influence of ceramic surface conditioning and resin cements on microtensile bond strength to a glass ceramic. J Prosthet Dent 2006 Dec;96(6):412-417.

4. 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 Mar-Apr;38(2):208-17. Epub 2012 Aug.

5. Salvio LA, Correr-Sobrinho L, Consani S, Sinhoreti MAC, De Goes MF, Knowles JC. Effect of water storage and surface treatments in the tensile bond strength of IPS Empress 2 ceramic. J Prosthodont 2007 May-Jun;16(3):192-199.

6. Borges GA, Spohr AM, Goes MF, Sobrinho LC, Chan CN. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. J Prosthet Dent 2003 May;89(5):479-488.

7. Spohr AM, Correr-Sobrinho L, Consani S, Sinhoreti MAC, Knowles JC. Influence of surface conditions and silane agent on the bond of resin to IPS Empress 2 ceramic. Int J Prosthodont 2003 May-Jun;16(3):277-282.

8. Matinlinna JP, Lassila LV, Ozcan M, Yli-Urpo A, Vallittu PK. An introduction to silanes and their clinical applications in dentistry. Int J Prosthodont 2004 Mar-Apr;17(2):155-164.

9. Kamada K, Yoshida K, Atsuta M. Effect of ceramic surface treatments on the bond of four resin luting agents to a ceramic material. J Prosthet Dent 1998 May;79(5):508-513.

10. Eames WB, Rogers LB, Feller PR, Price WR. Bonding agents for repairing porcelain and gold: an evaluation. Oper Dent 1977 Summer;2(3):118-124.

11. Della Bonna A, Anusavice KJ, Shen C. Microtensile strength of composite bonded to hot-pressed ceramic. J Adhes Dent 2000 Winter;2(4):305-313.

12. Kupiec KA, Wuertz KM. Evaluation of porcelain surface treatments and agents for composite-to-porcelain repair. J Prosthet Dent 1996 Aug;76(2):119-124.

13. Brentel AS, Ozcan M, Valandro LF, Alarca LG, Amaral R, Bottino MA. Microtensile bond strength of a resin cement to feldpathic ceramic after different etching and silanization regimens in dry and aged conditions. Dent Mater 2007 Nov;23(11):1323-1331.

14. Sato K, Matsumara H, Atsuta M. Effect of three-liquid bonding agents on bond strength to a machine-milled ceramic material. J Oral Rehabil 1999 Jul;26(7):570-574.

15. Dos Santos VH, Griza S, de Moraes RR, Faria-E-Silva AL. Bond strength of self-adhesive resin cements to composite submitted to different surface pretreatments. Restor Dent Endod 2014 Feb;39(1):12-16.

16. Oliveira AS, Ramalho ES, Ogliari FA, Moraes RR. Bonding self-adhesive resin cements to glass fibre posts: to silanate or not silanate? Int Endod J 2011 Aug;44(8):759-763.

17. Sorensen A, Engelman J, Torres J, Avera P. Shear bond strength of composite resin to porcelain. Int J Prosthodont 1991 Jan-Feb; 4(1):17-23.

18. Anagnostopoulos T, Eliades G, Palaghias G. Composition, reactivity and surface interactions of three dental silane primers. Dent Mater 1993 May;9(3):182-190.

19. Pleuddemann, EP. Nature of adhesion through silane coupling agents. New York: Plenum Press; 1982. p. 111.

20. Goracci C, Cury AH, Cantoro A, Papacchini F, Tay FR, Ferrari M. Microtensile bond strength and interfacial properties of self-etching and self-adhesive resin cements used to lute composite onlays under different seating forces. J Adhes Dent 2006 Oct;8(5):327-335.

21. Söderholm KJ, Reetz EA. Factors affecting reliability of a resin-based cement joint. Gen Dent 1996 Jul-Aug;44(4):296-302.

22. Sorensen A, Engelman J, Torres J, Avera P. Shear bond strength of composite resin to porcelain. Int J Prosthodont 1991 Jan-Feb;4(1):17-23.

23. Bailey JH. Porcelain-to-composite bond strengths using four organosilane materials. J Prosthet Dent 1989 Feb;61(2): 174-177.

24. Barghi N. To silanate or not to silanate: making a clinical decision. Compend Contin Educ Dent 2000 Aug;21(8):659-662.

25. Söderholm KJ, Shang SW. Molecular orientation of silane at the surface of colloidal silica. J Dent Res 1993 Jun;72(6):1050-1054.

26. Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R, Pashley DH. Relationship between surface area for adhesion and tensile bond strength: evaluation of a micro-tensile bond test. Dent Mater 1994 Jul;10(4):236-240.

27. Shono Y, Ogawa T, Terashita M, Carvalho RM, Pashley EL, Pashley DH. Regional measurement of resin-dentin bonding as an array. J Dent Res 1999 Feb;78(2):699-705.