

Effect of a Thermal Treatment of Two Silanes on the Bond Strength Between a Lithium Disilicate and a Resin Cement

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Abstract

Thermal drying of silanes is used for allows the evaporation of vehicles and accelerates condensation on the ceramic surface. The objective of this study was to determine the effect of a thermal drying treatment of two types of silane on the bond strength between a lithium disilicate and a resin cement.

Sheets were obtained from a lithium disilicate CAD-CAM block. All ceramic sheets were conditioned with 10% hydrofluoric acid and divided in four groups: The RXR and MNR groups, corresponding to a prehydrolyzed silane and a prehydrolyzed silane with 10-MDP respectively, dried at room temperature. Thermal drying was applied to the RXH and MNH groups, for the same silanes. The hot air was constantly fired for 15 seconds until it reached 100°C. Tubes of medical grade silicone were positioned on the ceramic surface and filled with dual resin cement, and them polymerized with light LED. A test micro-shear bond strength was used to determinate bond strength.

The highest value of bond strength was obtained by the MNH group and the lowest value was obtained by the SXR group.

Within the limitations of the present study, it can be concluded that the thermal drying treatment applied to both silanes demonstrates a positive influence on the adhesion values between a lithium disilicate-based ceramic and a dual resin cement.

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Introduction

Silane coupling agents play a very important role in the chemical interaction of silica-based surfaces with adhesive methacrylates¹. Creating surface roughness, preferably with hydrofluoric acid, followed by a silane agent, is the most usual and widely accepted method for the surface treatment of lithium disilicate-based glass ceramics². Silanes contain one or more alkoxy groups, which react with inorganic

substrates³. At the other extreme are organic functional groups such as: meth acryl, epoxy, amino, vinyl, styryl, ketimine, isocyanate⁴. In dentistry it is commonly expressed as 3-methacryloxypropyl-trimethoxysilane (MPS) to chemically refer to the composition of silanes. Some of them can be functionalized with the joint addition of 10-Methacryloyloxydecyl-dihydrogen-phosphate (MDP) and disulfide di-methacrylate⁵. Additionally, they have a pH that varies from 2.3 to 6, depending on their composition, and as vehicles they can include ethanol, acetone or water⁶.

Clinically, silanes reduce the contact angle leaving a film thickness between 10 to 50 nm approximately. Multiple layers or thick films result in internal cohesive tendencies of the silane, leading to bond failure. Because of this, an ultra-thin silane layer is considered as a

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requirement to promote a better bond strength⁷. A strategy to improve its performance includes thermal drying with temperatures between 50 and 100°C of silanes once applied. This allows the evaporation of vehicles and accelerates condensation on the surface, to promote the formation of covalent bonds more efficiently⁸.

The objective of this study was to determine the effect of a thermal drying treatment of two types of silane on the bond strength between a lithium disilicate and a resin cement. Two null hypotheses were established: 1- the average value of the bond strength of the groups without thermal drying and the groups with thermal drying would be the same. 2- the average value of the bond strength of the groups of both silanes after thermal drying would be the same.

Materials and methods

Sample elaboration:

Lithium Disilicate reinforced glass ceramic (e.max CAD[®] Ivoclar Vivadent, Schaan, Liechtenstein) was used. Using a precision saw at low speed and constant irrigation (Buehler IsoMet[®] Low Speed Saw), 8 ceramic sheets 1.5mm thick were cut from a CAD-CAM block. The ceramic sheets were crystallized at 850°C in an oven (Programat[®] EP 5000, Ivoclar Vivadent, Schaan, Liechtenstein), according to the program recommended by the manufacturer. The ceramic sheets were included in self-curing acrylic cylinders (self-curing Veracril, pink, New Stetic, Guarne, Colombia) keeping one surface free; these were sanded for one minute with # 400, 600, 800 and 1000 grit silicon carbide abrasive paper and manual pressure.

Surface treatment:

The ceramic sheets were conditioned with 10% hydrofluoric acid (Maquira, Maringá, Paraná, Brazil) for 20 seconds. The surface was washed with ultrasound and distilled water for 10 minutes, then these were dried with pressurized air. The ceramic sheets were randomly distributed (software-free random.org) in four groups. The RXR and MNR groups, corresponding to a prehydrolyzed silane based on 3-MPS (Relyx[™] Ceramic Primer 3M ESPE Saint Paul, MN, United States) and a prehydrolyzed silane with 10-MDP (Monobond[®] N, Ivoclar Vivadent, Schaan, Liechtenstein) respectively, dried at room temperature. Thermal drying was applied to

the RXH and MNH groups, for the same silanes. In all groups, the silanes were applied with a microbrush, brushing for 10 seconds and left to act for 60 seconds. For RXR and MNR groups an air-water syringe for drying with pressurized air was used. For thermal drying (RXH and MNH groups) the heat gun (RANGER[®] 2012004, 120V/60Hz) nozzle was positioned 3 cm from the ceramic surface in a perpendicular direction. The hot air was constantly fired for 15 seconds until it reached 100°C. An infrared thermometer (tekit[®] GM320) located 7 cm away from the ceramic surface recorded the temperature (passing from 0°C to 100°C in 15 seconds). Before applying thermal drying, it was standardized that the heat gun had to be previously operated for 3 minutes. This with the purpose that the air shot was hot.

Bonding process:

Tubes of medical grade silicone of 0.75mm internal diameter and 0.8mm high were obtained from a hose. Each mold was positioned on the ceramic surface and filled with dual resin cement (Variolink N[®], Ivoclar Vivadent, Schaan, Liechtenstein) with the help of a dental explorer and tweezers. Each of the cylinders was polymerized for 65s with an LED light cure unit (Radii plus, SDI Limited, Bayswater, Australia). All samples were stored at 37°C for 24 h in distilled water (HYGROBATH[®], Whip Mix Louisville, KY, United States) before performing micro-shear bond strength test (ISO / TS 11405: 2015).

Micro-shear bond strength:

Silicone tubes were then carefully removed using #12 scalpel blades. Only one sample in the MNR group was compromised during the removal, therefore it was decided not to use it (n=14). The micro-shear bond strength test (μ SBS) was executed in the universal mechanical testing machine (Shimadzu AG-IS, Shimadzu Corporation, Kyoto, Japan), a steel wire loop (0.22 mm) was placed embracing the resin cement tubes with a 50 N load cell and 1 mm/min crosshead speed. All tests were performed by same experienced operator, along with a prior standardization of the test design. The bond strength (MPa) was calculated using the equation:

$$1. \text{ MPa} = N/\text{mm}^2$$

N = corresponded to the fracture force.
 mm^2 = corresponded to the adhesive area determined by the equation $\pi.r.^2$. The adhesive area was a constant (0.442mm²).

Failure classification:

The type of failure was classified according to the separation at the interface: adhesive failure, described by an exposed ceramic surface without visible alterations, separation of cement and ceramic; cohesive cement failure, when great part of the adhesive area was covered by cement remains; cohesive ceramic failure when any ceramic depression was observed involving almost the entire adhesive area; and mixed failure, which involved the previous failure patterns without a clear visual predominance. For the analysis of the type of failure, a stereomicroscope (Nikon SMZ800, Chicago, IL, United States) with 50X magnification was used.

Statistical analysis:

Shapiro-Wilk normality tests, Kruskal-Wallis analysis, Levene's test and the function of the nparcomp package were used⁹. The R-projet version R-3.6.3 for Windows was used (The R Project for Statistical Computing, St. Louis, Missouri, USA).

Results

A normality test was performed, and the normality assumption was rejected for each group (Shapiro-Wilk = 0.55; 0.28; 0.57; 0.23). The highest value of bond strength was obtained by the MNH group and the lowest value was obtained by the SXR group (Fig 1). To check the equality of variances of the groups, a Levene's test was performed (p = 0.012), followed by a non-parametric analysis. To establish statistical differences between groups, a Kruskal-Wallis analysis (p = 0.002) and nparcomp package were used (p <0.05). This comparison was statistically significant for the MNR and MNH groups (Table 1).

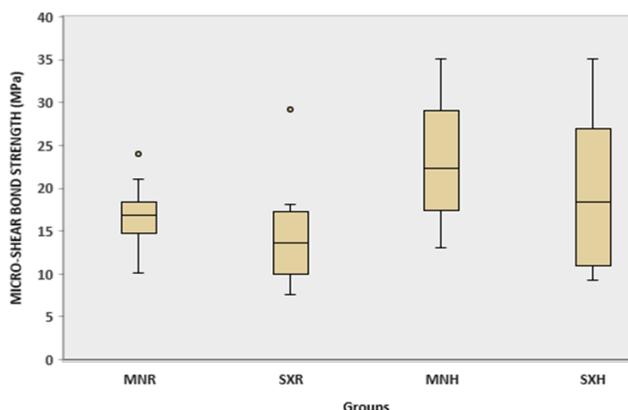


Figure 1. Box-plot Micro-shear bond strength.

Groups	n	Mean	standard deviation	Median	Maximum value	minimum value
MNR	14	16.61	3.81	16.79 [†]	24.00	10.10
SXR	15	14.43	5.30	13.56	29.20	7.64
MNH	15	23.26	6.65	22.26 [†]	35.15	13.00
SXH	15	19.53	8.40	18.38	35.12	9.24

Table 1. Micro-shear bond strength.

The data in the table was expressed in megapascals (MPa). Levene test: p = 0.012. Kruskal Wallis: (chi2 = 14.99; gl = 3; p = 0.002). Equal symbol in superscript in the medians indicate statistically significant differences (p <0.05) according to the nparcomp package.

Discussion

In the present investigation, the evaluated methodology of thermal drying of two types of silane with continuous air current at 100°C for 15 seconds had a positive effect on the adhesion between lithium disilicate and resin cement. Due to this, the first null hypothesis was not accepted. The theoretical objective of these treatments is to promote an increase in bond strength, accelerating the removal of vehicles and other by-products, decreasing the film thickness, facilitating the condensation and formation of a greater number of covalent bonds on the ceramic surface¹⁰. However, post-silanization heat treatments in the literature show conflicting results¹¹. Air flows with a temperature range between 38 and 120°C report improving adhesion values compared to drying at room temperature¹². Drying in an oven at 100°C for 5 minutes reports increases in the adhesion values between a lithium disilicate-based ceramic and composite resin, compared to drying at room temperature¹³. This is consistent with the information obtained in this study, where an increase in bond strength was observed after heat treatment. In contrast, oven drying at 77°C for 1 minute in leucite-reinforced feldspathic ceramics does not report such benefit¹⁴. An investigation evaluated a treatment with an oven at 100°C for 1 min of various silanes, including a silane with MDP in its composition, and found that there was not benefit for the bond strength values between lithium disilicate and resin cement¹⁵. The different silane compositions, the interaction of different combinations of materials involved in the protocols (adhesives and/or resin cements), the heat source and its parameters (duration, distance, air flow, oven or hot water) can be the responsible variables for the contradictory results in this research topic¹⁵⁻¹⁸. Another possible explanation for the positive result this work could have to do with the use of a

direct and constant heat source at 100°C from the start of the heat treatment, and additionally, the use of a thermometer that guaranteed temperature control of the ceramic surface.

The second hypothesis of this investigation was not accepted. Although an increase in the bond strength values was observed in both silane groups, this difference was only statistical in the silane group with MDP (MBH). This is contrary to evidence that suggests that silanes or primers with phosphate monomers (10-MDP) have a lower affinity for glass ceramics compared to silane monomers¹⁹. However, the higher MBH result could be due to the fact that this silane contains 3-MPS, 10-MDP, disulfide ester monomers, phosphoric and acetic acids, water and ethanol, which can be benefited by the temperature increase to accelerate its volatilization and ability to infiltrate porosities in the ceramic. A possible example of this is a report of the addition of 4-META to a silane with MDP in its composition that seems to improve the silane's response to heat drying²⁰. A limitation of this investigation was that only one resin cement was tested. The affinity of any of the two silanes for said cement could create an uncontrolled variable. It is recommended to carry out future investigations where multiple silanes associated with more than one resin cement are evaluated.

Regarding the failure pattern, the micro-shear test can reveal predominantly adhesive and mixed patterns, both on dental and ceramic substrates^{11,21}. Consistent with the present study where adhesive failure predominated, followed by mixed and the less common cohesive (Table 2), contrary to some reports of silane heat treatments evaluated by micro-shear, with prevalence of cohesive and mixed failures^{15,16}. Trying to establish a correlation between the adhesive values and the type of failure can be somewhat controversial. Another type of information can be analyzed from this descriptive test, a predominance of the same failure pattern in the groups with and without thermal treatment was observed. Due to this, with the limitations of this study, the drying of silanes at high temperature seems to have no influence on the type of failure obtained¹⁵.

Groups	Failure classification n (%)				Total
	adhesive	cohesive cement	cohesive ceramic	mixed	
FSR	7 (50)	3 (21.4)	2 (14.3)	2 (14.3)	14(100)
NSR	11 (73.3)	2 (13.3)	0 (0)	2 (13.3)	15(100)
FSH	10 (66.6)	0 (0)	2 (13.3)	3 (20)	15(100)
NSH	10 (66.6)	0 (0)	2 (13.3)	3 (20)	15(100)

Table 2. Failure classification.

Conclusions

Within the limitations of the present study, it can be concluded that the thermal drying treatment applied to both silanes, with 10-MDP and silanes based on 3-MPS, demonstrates a positive influence on the adhesion values between a lithium disilicate-based ceramic and a dual resin cement. Being of greater impact in silanes with 10-MDP in its structure, by promoting statistically significant increases in the bond strength.

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Declaration of Interest

The authors declare no potential conflicts of interest with the materials involved in the present investigation.

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