MESTRADO EM ODONTOLOGIA

ÁREA DE CONCENTRAÇÃO EM DENTÍSTICA

JOSÉ CARLOS ROMANINI JUNIOR

AVALIAÇÃO DE DIFERENTES PROTOCOLOS ADESIVOS E DE SILANIZAÇÃO NA DURABILIDADE DA UNIÃO À UMA CERÂMICA REFORÇADA POR DISSILICATO DE LÍTIO

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Orientador: Prof. Dr. André Figueiredo Reis
Co-orientador: Prof. Dr. José Augusto Rodrigues

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"Viva como se fosse morrer amanhã. Aprenda como se fosse viver para sempre."

Mahatma Gandhi

RESUMO

O objetivo deste estudo foi analisar os efeitos de diferentes protocolos adesivos e de silanização na durabilidade da resistência de união (RU) a uma cerâmica reforçada por Disilicato de Lítio (DSL) através do teste de microcisalhamento. Foram utilizados 40 discos de DSL com 13 mm de diâmetro (IPS E.max Press, Ivoclar Vivadent). Os discos cerâmicos foram polidos com lixas de SiC, condicionados com HF a 9,5% durante 20 s e lavados em ultrassom com álcool por 5 min. As amostras foram aleatoriamente divididas em 4 grupos de acordo com o protocolo: G1 – apenas aplicação de silano (ceramic primer, 3M ESPE); G2 – aplicação de silano seguido da aplicação do adesivo (XP Bond, Dentsply De Trey); G3 – apenas aplicação do adesivo universal que contém silano (Scotchbond Universal, 3M ESPE); G4 – aplicação do silano seguido da aplicação do adesivo universal (Scotchbond Universal, 3M ESPE). Em seguida, as amostras foram divididas em dois sub-grupos, de acordo com o tempo de armazenamento em água, 24h ou 12 meses (n=5). Uma resina composta de baixa viscosidade foi aplicada em 4 dispositivos cilíndricos de 1 mm de diâmetro e 3 mm de altura e fotoativados por 40 s. O teste de microcisalhamento foi realizado em uma máquina de ensaios universais (1mm/min). Os resultados foram analisados estatisticamente pela ANOVA a dois critérios e teste de Tukey ($p<0.05$). Após 24 horas de armazenamento em água, os maiores valores de RU foram obtidos quando foi aplicado um sistema adesivo após o silano (G2 e G4). No entanto, após 12 meses, os valores de RU nestes dois grupos (G2 e G4) apresentaram uma redução significativa nos valores de RU, enquanto o grupo em que se aplicou o silano apenas (G1), apresentou os maiores valores de RU. O grupo em que se aplicou apenas o adesivo Scotchbond Universal (G3) não apresentou redução significativa nos valores de RU, mas foi significativamente inferior às demais técnicas após 12 meses de armazenamento em água. Mesmo apresentando silano em sua composição, a silanização prévia não deve ser dispensada quando o adesivo Scotchbond Universal for utilizado sobre a superfície da porcelana condicionada.

Palavras-chave: Resistência de união, Cerâmica, Microcisalhamento, silano , sistema adesivo.

ABSTRACT

The aim of this study was to test the effects of different adhesive protocols and silane application on the durability of a composite to a Lithium Disilicate (LDS) reinforced glass ceramic using the microshear bond strength test (SBS). Ceramic disks were serially wet-polished with SiC paper, etched with 9.5% hydrofluoric acid for 20 s and cleaned in an ultrasonic bath with alcohol for 5 minutes. Disks were randomly assigned into 4 groups according to the adhesive/silane protocol: G1 – silane application only (ceramic primer, 3M Espe); G2 – silane application followed by adhesive application (XP Bond, Dentsply De Trey). G3 – silane-containing adhesive application (ScotchBond Universal, 3M Espe); G4 - silane application followed by adhesive application (ScotchBond Universal, 3M Espe). Afterwards, specimens were divided into 2 subgroups, according to the water storage time, 24 h or 12 months. Tygon tubes of 1-mm diameter and 3-mm height were used for application of a low-viscosity composite resin. Four resin composite cylinders were made on each ceramic disk and were light-cured for 40s. Microshear bond strenght test was carried out on a universal testing machine. Results were statistically analyzed by 2-way ANOVA and Tukey test ($p<0.05$). After 24 hours, the highest microshear bond strength (SBS) values were observed when silane application preceded adhesive application (G2 and G4). However, after 12 months, G2 and G4 presented a significant reduction in SBS values, while the highest SBS values were observed for the group in which only silane was applied (G1). When Scotchbond Universal was used without silane (G3) no significant reduction in SBS was observed, however it showed the lowest SBS after 12 months of storage in water. Regardless of the presence of silane on the composition of Scotchbond Universal, previous silane application is still recommended prior to cementation of lithium disilicate glass ceramic.

Keywords: **Bond strength, Ceramic, Microshear, Silane, Adhesive system.**

SUMÁRIO

1. INTRODUÇÃO

A Odontologia Adesiva é aplicada em diversos procedimentos clínicos. Nos últimos anos, tem-se observado uma crescente migração da utilização de próteses confeccionadas com infra-estruturas metálicas para o uso dos sistemas totalmente cerâmicos, os quais envolvem a confecção de peças protéticas como coroas, facetas e pequenas incrustações como *onlays* e *inlays* (Holand et al., 2008). Mudança essa que acompanha a alta procura dos pacientes por um sorriso natural, harmônico e estético. (Guess et al., 2012; Wang et al.,2015).

A diferença de composição das cerâmicas condicionáveis está na quantidade e tipo dos constituintes inorgânicos inseridos na matriz vítrea. A fase cristalina delimita o tipo e a resistência de cada tipo cerâmica, como as cerâmicas feldspáticas, reforçadas por leucita e as reforçadas por dissilicato de lítio que apresentam diferentes valores de resistência flexural (Kina & Bruguera et al., 2007).

Dentre os sistemas cerâmicos existente no mercado, um dos mais utilizados é a cerâmica reforçada por dissilicato de lítio, introduzida no mercado na década de 90 (Fabisnder et al., 2010; Pieger et al., 2014). O sistema é composto por pastilhas cerâmicas usualmente injetadas (IPS e.max Press, Ivoclar Vivadent) ou fresadas (IPS e.max/CAD, Ivoclar Vivadent), pelos inúmeros sistemas CAD/CAM existentes (Lise et al., 2015). O dissilicato de lítio é composto por uma matriz vítrea de sílica e por cristais de óxido de lítio (Li_2O) (Aboushelib et al., 2014). Possuem boas propriedades mecânicas, apresentando uma alta resistência flexural, entre 350 e 450 Mpa, além de uma boa estabilidade química e biocompatibilidade. (Guess et al., 2012; Fabisnder et al., 2010).

A cimentação de restaurações totalmente cerâmicas tornou-se possível através do desenvolvimento constante dos sistemas adesivos e cimentos resinosos, e das técnicas de tratamento de superfície cerâmica e dos substratos dentais (Braga et al., 1999; Piwowarczyk et al., 2004). Clinicamente e acompanhando a evolução da odontologia adesiva, as cerâmicas odontológicas, assim como a estrutura dentária necessitam de adequados métodos de tratamento de superfície, uma escolha correta do sistema adesivo e também de um agente de cimentação compatível é primordial para que a adesão do substrato cerâmico ao substrato dentário tenha um prognóstico favorável e uma ótima durabilidade com o passar do tempo. (Bailey et al., 1988).

Diferentes métodos de tratamento de superfícies de cerâmicas tem sido propostos e pesquisados (Steinhauser et al., 2014; De Carvalho et al., 2011). Dentre eles o tratamento mecânico, que envolve a utilização do jato de óxido de alumínio; o tratamento químico, que compreende a utilização do ácido fluorídrico em concentrações de 5% a 10%, ou também pela associação de ambos os tipos de tratamentos, são os mais indicados e utilizados atualmente (Steinhauser et al., 2014).

O tratamento da superfície interna das cerâmicas vítreas se tornam essenciais, passo esse indispensável no protocolo da cimentação adesiva. As microretenções mecânicas formadas após as técnicas de condicionamento permitem o embricamento do cimento resinoso junto a restauração cerâmica e o substrato dental (Steinhauser et al., 2014; De Carvalho et al., 2011; Cotes et al., 2013). Após o tratamento de superfície, resíduos internos são formados, causando a obstrução das microretenções, podendo afetar a resistência de união do complexo dente-cerâmica após a cimentação. Assim, a remoção destes resíduos deve ser realizada (Steinhauser et al., 2014), através da utilização da cuba ultrasônica (Cotes et al; 2013), a utilização de ácido fosfórico a 37% e/ou da combinação de ambos. (Steinhauser et al; 2014).

A não eliminação de tais resíduos pode causar uma queda de até 50% na resistência de união (Matinlinna et al; 2007). Após a utilização dos tratamentos de superfície interna da cerâmica, é indispensável também a aplicação de um agente mediador, como o silano, agente de união bifuncional constituído por braços que se ligam a compostos orgânicos e também aos inorgânicos (De Carvalho et al., 2011; Matinlinna et al., 2007; Kalavacharla et al., 2015). As moléculas presentes no silano interagem com a sílica presente na cerâmica de dissilicato de lítio, parte inorgânica e também com as moléculas de metacrilato presentes nos sistemas adesivos e cimentos resinosos, parte orgânica (Lise et al., 2015).

O silano forma três tipos de camadas diferentes após sua aplicação sobre a superfície cerâmica, sendo que apenas uma delas, a monocamada, é considerada de maior estabilidade e essencial para a resistência de união à cerâmica. As duas outras camadas formadas, maior parte do silano, não contribuem significativamente para a resistencia de união (Hooshmand et al., 2002). A camada mais externa possui inúmeros oligômeros, resultando em uma fraca união siloxana, podendo ser removida através de solvente orgânico ou com água; o mesmo acontece com a camada intermediária, podendo essa ser removida através da utilização de água fervente (Hooshmand et al; 2002).

Estudo propõe que o aquecimento da estrutura cerâmica após a aplicação, pode aumentar a eficiência do silano, fazendo com que seja eliminado a água, álcool e os demais subprodutos que se encontram nas camada mais externa e na intermediária após a silanização, promovendo assim uma melhor interação entre o silano e a cerâmica (Hooshmand et al., 2012).

O tratamento térmico após aplicação do silano pode otimizar a efetivação da resistência de união ao evaporar as duas camadas mais externas, isso poderia acabar suprindo a necessidade da utilização do ácido fluorídrico como etapa inicial ao tratamento de superfície (Hooshmand et al., 2004). Isso pode ser de grande vantagem, além das desvantagens já citadas, por ser altamente tóxico para a saúde podendo comprometer o sucesso da adesão final da restauração (Pollington et al., 2010).

Os tratamentos de superfície da cerâmica e a silanização tem sido os procedimentos mais recomendados para melhora da adesão, resistência e durabilidade do complexo cerâmica-cimento-estrutura dentinária, previamente à aplicação de um cimento resinoso. (Steinhauser et al., 2014; Cotes et al., 2013). Entretanto, se um dos passos não for corretamente aplicado ou negligenciado, o sucesso da cimentação final poderá ser comprometida assim como sua durabilidade. (Kalavacharla et al., 2015).

A procura pela otimização do tempo clínico e redução de passos clínicos sempre foi uma constante no meio de pesquisas de produtos odontológicos. Recentemente, foi introduzido no mercado, um sistema adesivo universal contendo em sua composição o agente silano, o que dispensaria na teoria o passo de aplicação do silano, previamente à aplicação do adesivo (Kalavacharla et al., 2015; Lawson et al., 2015). São comercializados através de frascos simplificados, contendo monômeros ácidos funcionais com a incorporação das moléculas de silano (Lawson et al., 2015). No entanto, existe pouca informação a respeito desse recente adesivo quando aplicado sobre estruturas cerâmicas a base de dissilicato de lítio (Amaral et al., 2013).

2. PROPOSIÇÃO

O objetivo desse estudo foi analisar os efeitos de diferentes protocolos adesivos e de silanização na durabilidade da união à uma cerâmica reforçada por Dissilicato de Lítio.

3. DESENVOLVIMENTO

Adhesive/Silane Application Effects on Bond Strength Durability to a Lithium Disilicate Ceramic.

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Abstract

The aim of this study was to test the effects of different adhesive protocols and silane application on the durability to a Lithium Disilicate (LDS) reinforced glass ceramic using the microshear bond strength test. Forty LDS disks of 13mm diameter (E.max Press, Ivoclar Vivadent) were used in the present study. They were wet-polished with SiC papers, etched with 9.5% HF for 20s and cleaned in an ultrasonic bath. Disks were randomly assigned into 4 groups according to the adhesive/silane protocol: G1 – silane application only (SIL); G2 – silane application followed by adhesive (SILXP - XP Bond, Dentsply De Trey). G3 – silane-containing adhesive (SBU - ScotchBond Universal, 3M Espe); G4 - silane application followed by silane-containing adhesive (SILSBU). Afterwards, specimens were divided into 2 subgroups, according to the water-storage time, 24 h or 12 months. Tygon tubes of 1-mm diameter and 3-mm height were used for application of a low-viscosity composite resin. Four resin composite cylinders were made on each ceramic disk and light-cured for 40s. Microshear bond strength (SBS) test was carried on a universal testing machine (1mm/min). Results were statistically analyzed by 2-way ANOVA and Tukey test ($p<0.05$). After 24 h, the highest SBS values were observed for SILXP and SILSBU. However, after 12 months, SILXP and SILSBU presented a significant reduction in SBS, while the highest SBS were observed for SIL. For SBU, no significant reduction in SBS was observed, however it showed the lowest SBS after 12 months of water-storage. Regardless of the presence of silane on the composition of SBU, previous silane application is still recommended prior to cementation of LDS.

Keywords: Adhesive, silane, lithium disilicate, shear strength bond.

Introduction

The search for a natural and esthetic smile has produced a crescent migration from prosthesis made with metallic substructures to the use of all-ceramic systems (Holand et al., 2008; Guess et al., 2012; Wang et al., 2015). Among the existing ceramic systems, one of the most used is the Lithium Disilicate reinforced glass ceramic (LDS) (Fabisnder et al., 2010; Pieger et al., 2014). The system is composed of ceramic ingots usually pressed (IPS and max Press, Ivoclar Vivadent, Schaan,Liechtenstein) or milled (IPS e.max/CAD, Ivoclar Vivadent) by CAD/CAM systems (Lise et al., 2015). It consists of a silica glass matrix and crystals of lithium oxide (Li_2O) (Aboushelib et al., 2014). LDS presents high flexural strength, chemical stability and biocompatibility (Guess et al., 2012; Fabisnder et al., 2010).

Different methods for treating ceramic surfaces have been proposed: mechanical, with the use of aluminum oxide particles; chemical, with application of hydrofluoric acid in concentrations between 5 to 10%; or the association of both treatments (Steinhauser et al., 2014). Etching with hydrofluoric acid promotes micro retentions in the ceramic surface, which is ideal for micromechanical interlocking of the resin cement (Steinhauser et al., 2014; De Carvalho et al., 2011; Cotes et al., 2013). Following etching and rinsing off residues, the application of a coupling agent is recommended. The silane, an agent of bifunctional bond constituted of organic and inorganic elements (De Carvalho et al., 2011; Matinlinna et al., 2007. Kalavacharla et al., 2015). Silane interacts with the silica present in the Lithium Disilicate ceramic and also with the methacrylate molecules present in the adhesive and resin cement (Lise et al., 2015).

Inner surface etching of glass ceramics and silanization has been the most recommended procedure to improve adhesion, resistance and durability of the complex ceramic-cement-tooth structure. (Steinhauser et al., 2014; Cotes et al., 2013). However, if one of the steps is not correctly performed, the long-term success of the restoration can be compromised (Kalavacharla et al., 2015). The effective cementation of all-ceramic restorations became possible through the constant development of adhesive systems and composite materials. (Braga et al., 1999; Peutzfeldt et al., 2011; Piwowarczyk et al., 2004).

The reduction of steps without compromising the quality of restorations is highly desired among practitioners and manufacturers. Recently, a silane-containing universal adhesive system was introduced in the market, making the silane application step unnecessary according to manufacturer (Kalavacharla et al., 2015; Lawson et al., 2015). However, there is little information with respect to the effectiveness and durability of the bond produced by this technique when applied on LDS glass ceramic.

Thus, the aim of this study was to test the effects of different adhesive protocols and silane application on the microshear bond strength durability to a lithium disilicate glass ceramic. The null hypotheses tested were: (1) there is no difference on the microshear bond strength produced by the different adhesive/silane protocols; (2) Water-storage time does not affect the bond strength produced by the different protocols.

Methods and Materials

Sample preparation

Materials, composition and manufacturers are listed in Table 1. Forty Lithium Disilicate-based ceramic disks (IPS e.max Press HT/A2, Ivoclar Vivadent, Schaan, Liechtenstein) measuring 13 mm diameter and 5 mm thickness were fabricated using the lost-wax and hot-pressing technique according to the manufacturer's instructions. After cooling, the disks were removed from the investment material and grit blasted with 50-µm aluminium oxide particles at a pressure of 2 bar. The ceramic disk surfaces were serially wet-polished with SiC papers to produce a standardized flat surface. Ceramic disks wer etched with 9.5% hydrofluoric acid for 20 seconds and cleaned in an ultrasonic bath with alcohol for 5 minutes.

Restorative procedures

Disks were randomly assigned into 4 groups according to the adhesive/silane protocol:

G1 – silane application only (**SIL** – ceramic primer, 3M Espe);

G2 – silane application followed by adhesive (**SILXP** - XP Bond, Dentsply De Trey);

G3 – silane-containing adhesive (**SBU** - ScotchBond Universal, 3M Espe);

G4 – silane application followed by silane-containing adhesive (**SILSBU -** ScotchBond Universal, 3M Espe).

Afterwards, specimens were divided into 2 subgroups, according to the water-storage time, 24 h or 12 months. For groups that received silane application, an air stream was used for 5 seconds after silane aplication (Ceramic Primer, 3M Espe). For groups that received adhesive application, an air-stream was applied for 10 s for solvent evaporation, and the adhesive layer was left uncured to simulate a veneer cementation procedure.

Table 1. Materials, manufactures, lot number and composition.

Materials, Manufacturer, Lot #	**Composition**
E.max Press Ingots, (Ivoclar Vivadent)	Lithium Disilicate reinforced glass ceramic: SiO_2, Li_2O, K_2O, MgO, ZnO_2, Al_2O, P_2O_5
XP Bond (Dentsply De Trey) 1310000932	Carboxylic acid modified dimethacrylate (TCB resin); PENTA; UDMA; TEGDMA; HEMA; Butylated benzenediol (stabilizer); Ethyl-4-dimethylaminobenzoate; Camphorquinone; Functionalized amorphous silica; t-butanol
Scotchbond Universal (3M ESPE) 520208	10-MDP, Dimethacrylate resins, HEMA, ethanol, Water, polyacrylic acid copolymer, Silane, Fillers, initiators
Low-viscosity composite resin, SureFil SDR Flow (Dentsply De Trey) 785961F	Barium-alumino-fluoro- borosilicate glass, Strontium alumino-fluoro-silicate glass, modified urethane dimethacrylate resin, EBPADMA, triethyleneglycol dimethacrylate, camphorquinone, butylated hydroxyl toluene, uv stabilizer, titanium oxide, iron oxide pigments. (68 wt%).
Relyx ceramic primer (3M ESPE) n548582	Ethyl Alcohol (70-80 wt%), water (20-30 wt%), methacryloxypropyltrimethoxysilane (<2 Trade Secret).
Hydrofluoric acid (Dentsply Brasil) 9759076	9.5% hydrofluoric acid

Legend: SiO_2, silicon dioxide; Li_2O, lithium oxide; K_2O, potassium oxide; P_2O_5, phosphorus pentoxide; ZrO_2, zirconium dioxide; ZnO, zinc oxide; PENTA, dipentaerythritol penta-acrylate monophosphate; UDMA, diurethane dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; HEMA, hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; EBPADMA, ethoxylated bisphenol A dimethacrylate.

Tygon tubes of 1-mm diameter and 3-mm height were used for application of a low-viscosity composite resin (Surefil SDR Flow, Dentsply Caulk). Four resin composite cylinders were made on each ceramic disk and were light-cured simultaneously using a light emitting diode (Radii Plus®, SDI, Victoria, Australia) with a radiant emittance of 1,500 mW/cm^2 for 40 s. Afterwards, the tygon tubes were carefully removed using a surgical blade. The restored samples were stored in distilled water at 37 °C for 24 hours or 12 months.

Microshear bond strength test

After each storage period specimens were mounted in a jig of a universal testing machine (EZ Test, Shimadzu Corp, Kyoto, Japan) equipped with an orthodontic wire placed parallel to the bonded interface and operated at a cross-head speed of 1 mm/min until specimens fracture occurred. Maximum tensile load was divided by specimen cross-sectional area to express results in units of stress (MPa). The mean value obtained from the four resin cylinders tested in each disk was considered for each ceramic disk (n=5).

Failure modes were determined by examination of fractured specimens with SEM (LEO 435 VP, LEO Electron Microscopy Ltd). Specimens were mounted on aluminum stubs and gold-sputter coated (MED 010, BAL-TEC AG, Balzers Union) prior to viewing at different magnifications. Failure modes at the fractured interface were classified into one of three types: CC (cohesive in ceramic), AD (adhesive failure between the ceramic and resin) or CR (cohesive failure in the composite resin). Instead of classifying failures as mixed, the area percentage of each type of failure in each specimen was recorded. (Kumagai et al., 2015).

Statistical analysis

Bond strength data were statistically analyzed by 2-way ANOVA, considering the factors "adhesive protocol" and "time", followed by Tukey test. In all analyses, a 95% confidence level was used.

Results

Mean bond strength values, standard deviation and significant differences are presented in Table 2. Two-way ANOVA revealed significant differences for the factor “adhesive protocol” (p=0.00001) and for the interaction between factors “adhesive protocol*time” (p=0.00005)

Table 2. Microshear bond strength values in MPa (SD) produced by the different groups after 24 hours and 12 months of storage in water.

Group	24h		12 months	
SBU - Scotchbond Universal	29.1 (3.9)	Ba	26.9 (3.0)	Ca
SIL - Silane	33.9 (3.7)	Bb	43.8 (2.2)	Aa
SILSBU - Silane+Scotchbond Universal	41.4 (1.7)	Aa	34.6 (2.9)	Bb
SILXP - Silane + XP Bond	42.7 (3.8)	Aa	38.5 (4.0)	ABb

Means followed by different letters (upper case - column, lower case - line) are significantly different by Tukey test at the 95% confidence level.

After 24 hours of storage in water the highest bond strength values were obtained when the adhesive systems were used in combination with the silane (SILSBU and SILXP). Significantly lower bond strength values were obtained for SIL and SBU, which were not significantly different. After 12 months, the highest bond strength values were obtained for SIL, which did not differ significantly from SILXP ($p>0.05$). After 12 months, the lowest bond strength values were observed for SBU ($p<0.05$).

Water-storage for 12 months produced a significant reduction in bond strength values for both groups that used adhesive systems in combination with the silane (SILSBU and SILXP) ($p<0.05$). SBU did not present a significant reduction in bond strength after 12 months ($p>0.05$). On the other hand, when no adhesive system was used after silane application (SIL), significantly higher bond strength values were observed after 12 months ($p<0.05$).

Results for the failure mode analysis are presented in Figure 1. Representative images for each type of failure are presented in Figures 2 to 4. In all groups, the majority of failures occurred adhesively, between resin and ceramic, followed by cohesive failures in resin. Cohesive failures in ceramic were the least predominant type of failure.

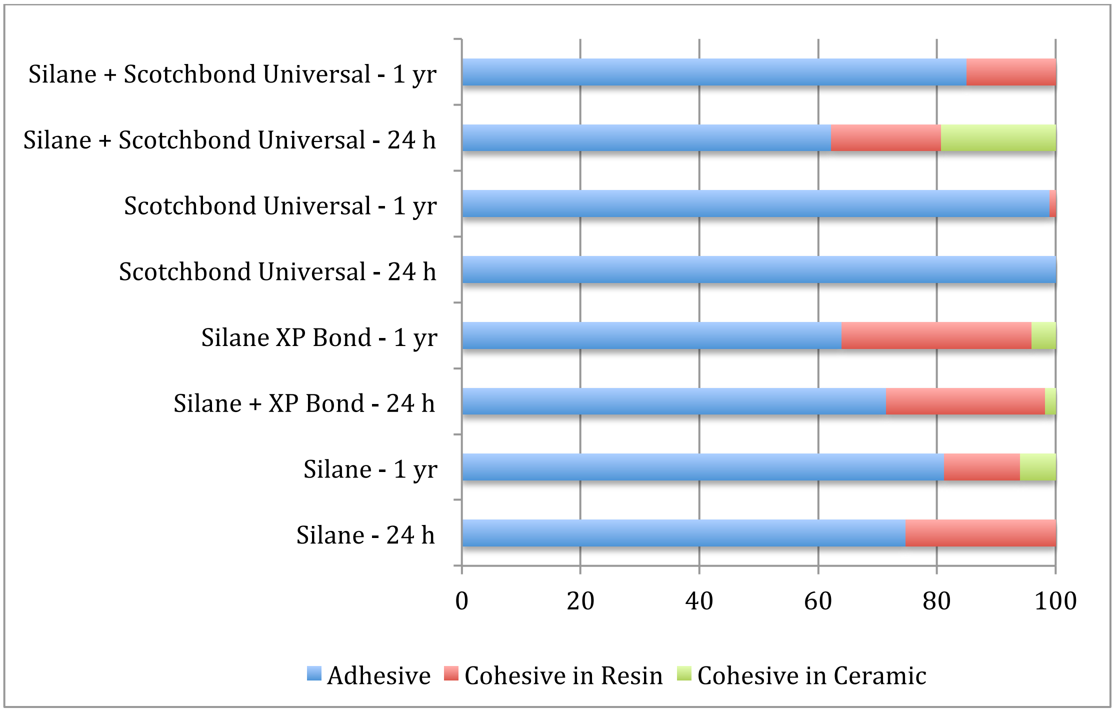

Figure 1. Distribution of failure modes within groups.

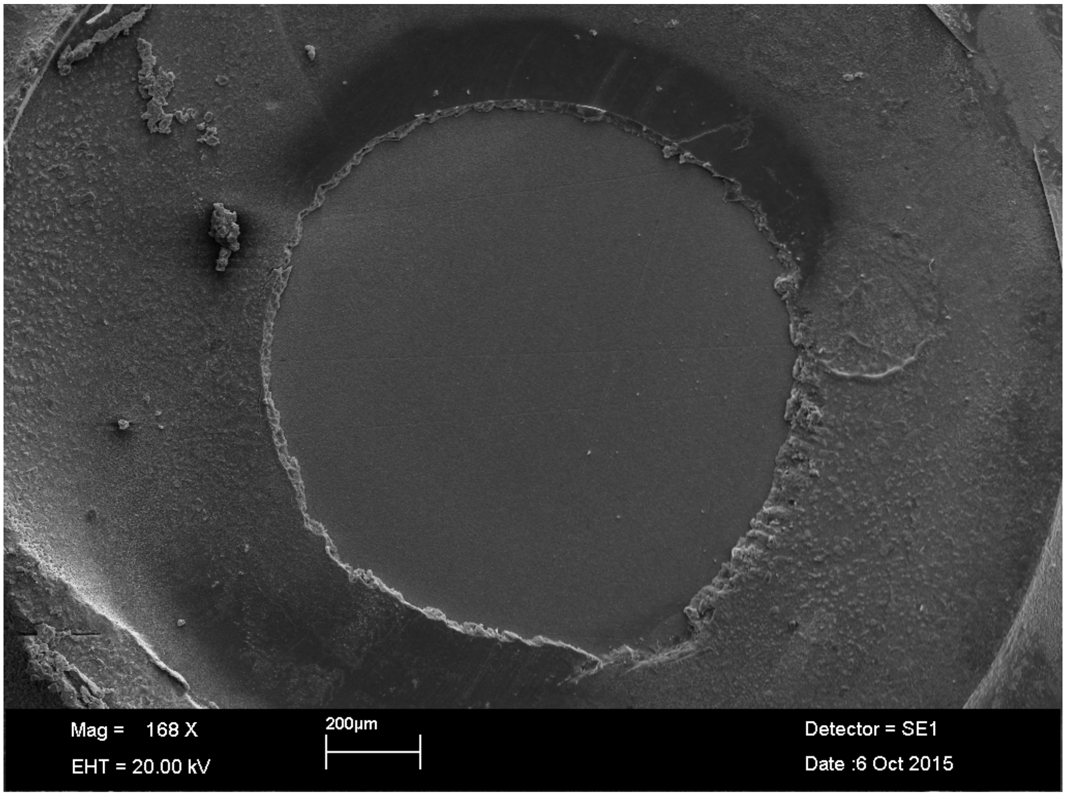

Figure 2. Representative SEM of an adhesive failure between composite resin and ceramic observed for the group in which the silane-containing adhesive Scotchbond Universal was applied and stored for 24 hours.

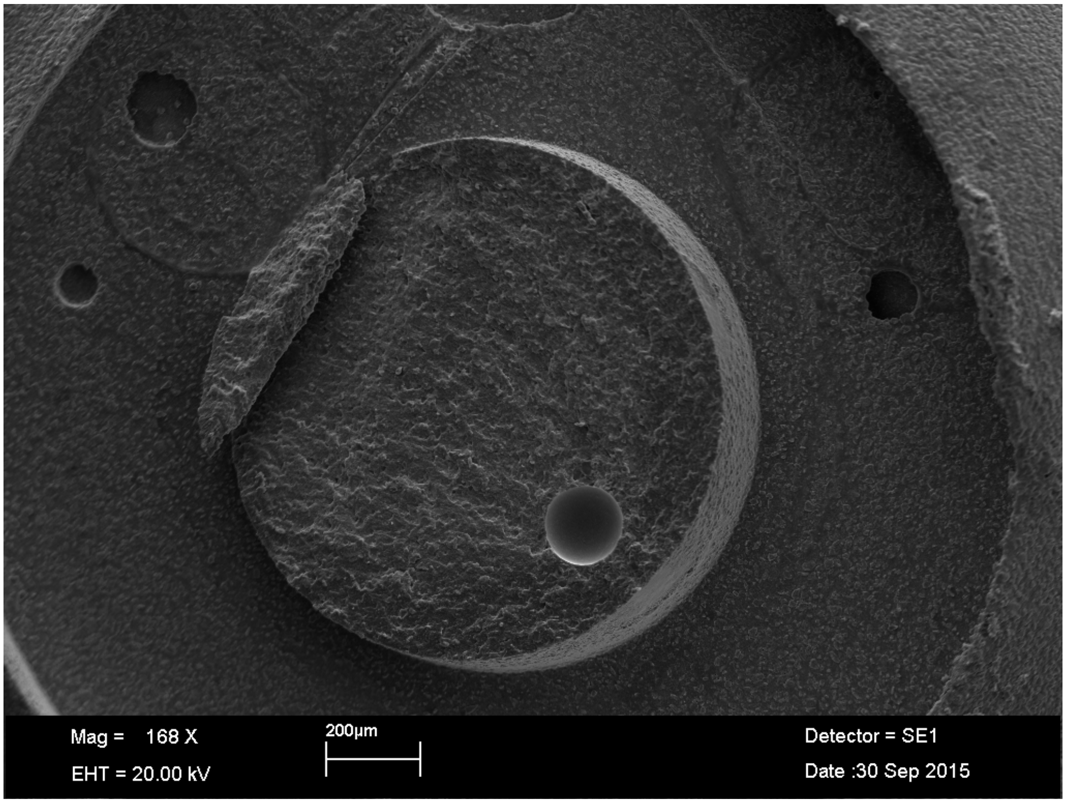

Figure 3. Representative SEM of a cohesive failure in composite resin observed for the group in which the silane was applied without any adhesive system and stored for 24 hours.

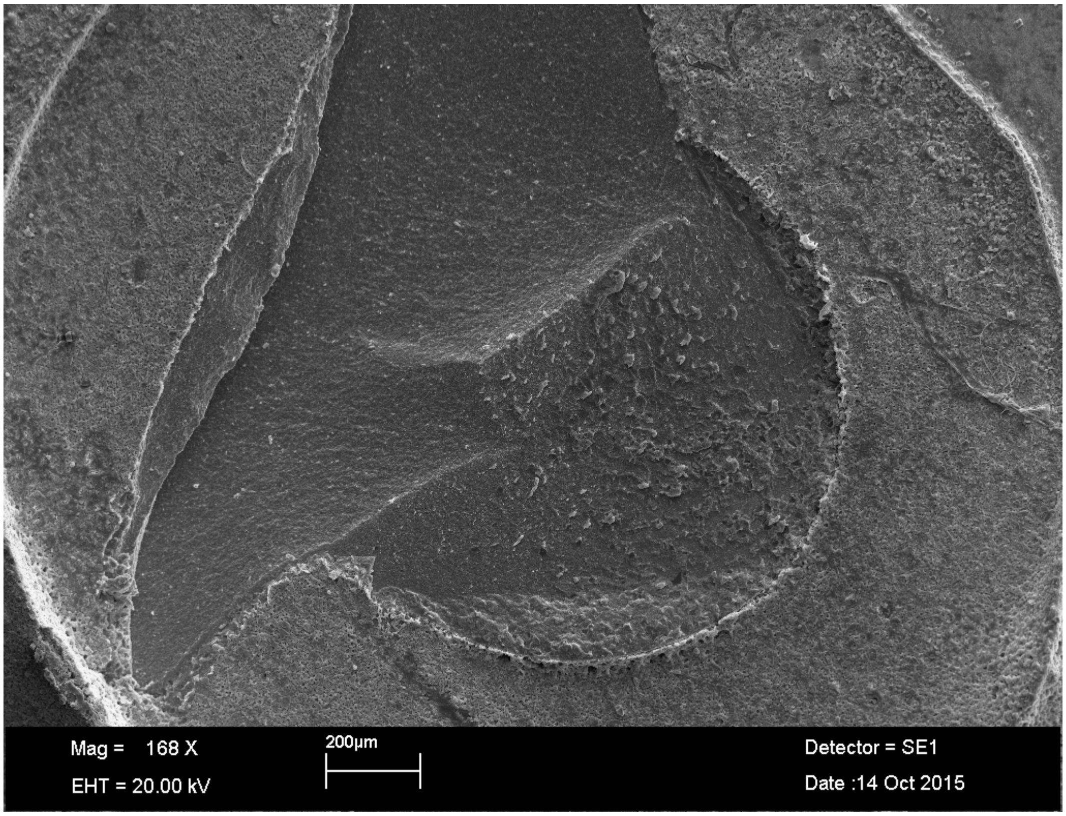

Figure 4. Representative SEM of a cohesive failure in ceramic observed for the group in which the adhesive system XP Bond was used after silane application and stored for 12 months.

Discussion

The microshear test is the most suitable to evaluate bond strength when using brittle materials such as glass ceramics and enamel, where the specimens do not require sectioning. Sectioning of brittle materials for bond strength tests can result in a large number of cohesive failures (Zohairy et al., 2010; Armstrong et al., 2010).

Interface bond strength tests using lithium disilicate glass ceramic have shown that surface treatment protocols, as well as the type of adhesive protocol used for cementation can affect the performance of the final restoration (Hooshmand et al., 2012). In the present investigation, the different adhesive/silanization protocols used produced significantly different bond strength values, which leads us to reject the first null hypothesis. After 24 hours, the association of the separate steps of silane and adhesive application resulted in the highest bond strength values.

Silane promotes the formation of three different layers after application on the ceramic surface, and only one monolayer is considered the most critical for stability and bond strength to ceramic. The other two layers formed, holds no value for the bond strength (Hooshmand et al., 2002). The external and intermediate layers have loose oligomers, resulting in a weak siloxane bond and can be removed using organic solvent or water; the most external layer and the intermediate can be removed by using hot water (Hooshmand et al., 2012).

The amount of silane present in the universal adhesive (Scotchbond Universal) may therefore not be enough to provide the same effect of when it is applied prior to the universal adhesive, showing that the bond strength is higher when applied in separate steps. Suggesting application of silane independent of the presence of silane within the universal adhesive solution. Similar results were observed in the study of Kavalacharla et al., (2015), where it was emphasized that the application of silane prior to universal adhesive produced higher bond strength. That study suggested that if the silane is not to be used, etching time should be increased from 20 to 60 seconds, because adhesion to the ceramic surface becomes totally dependent on the micromechanical retention promoted by etching with hydrofluoric acid (Kavalacharla et al., 2015).

Amaral et al. (2014) showed that application of the universal adhesive without silane is effective only when applied to zirconia restorations, and the effectiveness in the adhesion to disilicate restorations had not been fully investigated until recently.

Except for the group in which Scotchbond Universal was applied alone, 12-month water-storage resulted in significantly different bond strength values for the other groups. Both groups in which silane was used in association with an adhesive system demonstrated significantly reduced bond strength values after 12 months. In addition, the group in which silane was used without any adhesive, presented significantly higher bond strength after 12 months. These observations lead us to reject the second null hypothesis.

The high bond strength values observed in the group in which silane agent was applied without adhesive, in the specimens tested after 12 months, may be due to the ability of hydrofluoric acid to create micro-retentions on the internal surface, ideal situation for the flow of the resin cement to the ceramic surface (Steinhauser et al., 2014; Cotes et al., 2013; Lise et al., 2015). As well as the effectiveness of the silane agent in the formation of three-dimensional chains through siloxane bonds formed between the hydroxyl groups (-OH) present in the silica-based ceramic, and the methacrylate group present in both, as the adhesive as the resin cement (Kavalacharla et al., 2015).

The high bond strength values observed after 12 months for the group in which silane was applied alone, may be explained by the flow characteristics of the resin cements used in adhesive cementation combined with a mechanical interlocking in the etched ceramic surface. The resin cements present hydrophobic characteristics, preventing degradation by water absorption (Osorio et al., 2012).

The decrease in bond strength values for the groups in which the adhesives were applied in association with the silane (Scothbond Universal and XP Bond), may be justified by the degradation suffered during the 12 months of water-storage. The adhesives contain hydrophilic monomers and solvents, being more susceptible to water sorption and hydrolytic degradation (Reis et al., 2007a; Reis et al. 2007b; Reis 2007c).

Conclusion

Regardless of the presence of silane on the composition of Scotchbond Universal, previous silane application is still recommended prior to cementation of Lithium Disilicate reinforced glass-ceramic.

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4. CONCLUSÃO

O protocolo de silanização mostrou-se indispensável na cimentação adesiva de cerâmicas à base de Dissilicato de Lítio. A presença do silano na composição do adesivo universal não foi capaz de produzir os mesmos valores de resistência de união que a aplicação separada do silano e adesivo. Após o armazenamento em água por 1 ano a aplicação de silano apenas, sem posterior aplicação de adesivo apresentou os maiores valores de resistência de união.

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