ORIGINAL

Bonding performance of universal adhesives with concomitant use of silanes to CAD/CAM blocks

Associação de adesivos universais com silanos na adesão em blocos de CAD/CAM

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ABSTRACT

Objective: This study assessed whether the use of a silane coupling agent influence the bond strength of two universal adhesives to ceramic or resin CAD/CAM blocks. **Methods**: Forty-eight samples were obtained from each resin nano-ceramic hybrid block (RCBs) and lithium disilicate ceramic blocks (LD). Samples were treated with silane with MDP (Monobond - S-MDP), silane without MDP (Prosil - PS), and no silane application (Control - Ctr) followed by universal adhesive with silane (UAS) and without silane (UA) (n=8). Three polyurethane tubes (1.5 mm of internal diameter) were positioned in each sample treated surface and filled with a dual cured resin cement. Bond strength was assessed by microshear bond strength test and failure analysis was performed for all samples. **Results**: For the RCBs, UAS presented the highest bond strength values (p=0.004). Silane application was not significant in bond strength values (p=0.444). For LD, silane application was significant in bond strength values (p=0.066). Failure analysis showed high prevalence of adhesive failures for both substrates. **Conclusion**: A silane-containing universal adhesive promoted the best bond strength results to the resin nano-ceramic hybrid block. For bonding to a glass-ceramic CAD/CAM material, additional silane (without MDP) application presented the best results.

Indexing terms: Acid etching. Ceramics. Computer-aided design. Silanes.

RESUMO

Objetivo: Este estudo avaliou se o uso de um agente de união silano influencia na resistência de união de dois adesivos universais a blocos CAD/CAM cerâmicos ou resinosos. **Métodos**: Quarenta e oito amostras foram obtidas de blocos resinosos (RCBs) e cerâmicos de dissilicato de lítio (LD). As amostras foram tratadas com silano contendo: MDP (Monobond - S-MDP), silano sem MDP (Prosil - PS) ou sem aplicação de silano (Control - Ctr) seguido de adesivo universal com silano (UAS) ou sem silano (UA) (n=8). Três tubos de poliuretano (1,5 mm de diâmetro interno) foram posicionados em cada superfície tratada da amostra e preenchidos com um cimento

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resinoso dual. A resistência de união foi avaliada pelo teste de microcisalhamento e a análise de falha foi realizada para todas as amostras. Resultados: Para os RCBs, UAS apresentou os maiores valores de resistência de união (p=0,004). A aplicação de silano não foi significativa nos valores de resistência adesiva (p=0,444). Para LD, a aplicação de silano foi significativa nos valores de resistência de união (p<0,001), mas o adesivo não (p=0,066). A análise de falhas mostrou alta prevalência de falhas adesivas para ambos os substratos. Conclusão: O adesivo universal contendo silano promoveu os melhores valores de resistência adesiva ao bloco de resina. Para o bloco cerâmico, a aplicação adicional de silano (sem MDP) apresentou melhores resultados.

Termos de indexação: Cimentação. Cerâmica. Desenho assistido por computador. Silanos.

INTRODUCTION

Silane coupling agents are the gold standard adhesion promoters for indirect restorations [1]. They are imperative for bonding glass-rich ceramics and make de adhesion between filler particles (usually SiO2) and resin matrix in composite resins [2]. Silanes are called a bifunctional molecule since can bond to unpolymerized resin matrix (adhesives or resin cements) and to an inorganic compound (the surface of the restorative material). The strongest bond of silane molecules is made to silica, glass, and quartz, forming siloxane linkages (Si - O - Si). Besides that, silicon (Si) compounds present hydrophobicity, preventing the hydrolytic degradation of the bonding interface [1].

Aiming to simplify the adhesive procedures, some universal multimode adhesives contain silane and functional monomers (as phosphate monomers, MDP) for bonding to indirect materials, besides offering to bond to tooth structures [3]. For bonding to tooth structures, universal adhesives may be used in self-etch or total-etch modes [3,4], requiring low pH in the solution for tooth surface etching combined with monomer penetration. Adhesives also contain water as a solvent, but silane hydrolysis is caused by water and low pH [1,2]. Thus, the stability of silane from universal adhesives is questionable [5].

The use of universal adhesives shows to promote high and stable bond strength to the ceramic surface [6,7]. But the additional silane application associated with universal adhesives showed an increase in bond strength to lithium disilicate [6,8]. For indirect resin blocks, the literature is not conclusive, since the presence or absence of silane in universal adhesives, or additional silane + MDP primer was reported to not influence the bond strength to a resin cement [9,10], but the silane application was significant for bond strength improvement in other studies [11,12]. Silane would bond to the exposed filler on the composite surface, besides increasing surface energy and enhancing the cement/adhesive wettability [1,13].

Besides that, the application of silane in different materials may present different results in bond strength. Clinically, lithium disilicate ceramics are extensively used with high survival rates [14]. But the difference of glass ceramics to dentin, mainly regarding elastic modulus may represent a mechanical challenge in tooth survival [15]. Thus, hybrid resin ceramic material was introduced, aiming to represent an alternative material more similar to the modulus of elasticity of dentin than traditional ceramics [16].

Therefore, the aim of this study was to evaluate the influence of the additional application of silane over the bond strength of universal adhesives to lithium disilicate and resin nano-ceramic hybrid CAD/CAM blocks. The null hypothesis is that the silane application does not improve bond strength values between glass ceramic or resin blocks to resin cement bonded with universal adhesive.

METHODS

Specimen preparation

Forty-eight rectangular samples with dimensions (3 mm width × 7 mm length × 1 mm thickness) were obtained from each resin nano-ceramic hybrid block (RCBs, Grandio Blocs, Voco, Cuxhaven, Germany) and lithium disilicate

ceramic blocks (LD, IPS e.max, Ivoclar Vivadent, Schaan, Liechtenstein), using an automatic cutting machine (IsoMet 1000, Buehler, Illinois, USA).

Samples were embedded into epoxy resin (2001 Resin, Valglass, Sao Jose dos Campos, SP, Brazil) using a PVC matrix and were polished with silicon carbide paper (SiC paper - grit #600, #800 and #1200, Struers, Ballerup, Denmark), for 30 seconds each, under irrigation. Samples were subjected to an ultrasonic bath for 10 min with distilled water between each SiC paper grit to remove abrasive grains and at the end of the polishing procedure.

Then, samples from each material (RCBs and LD) were randomly divided in three groups, according to the silane application (n=8) [6,8,10]: S-MDP: silane with MDP in the composition (Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein); PS: silane without MDP in the composition (Prosil, FGM, Joinville, Brazil), Ctr: no silane application. Samples were finally divided according to the adhesive applied: UAS: universal adhesive with silane in the composition (Scothbond Universal, 3M ESPE, St Paul, USA) and UA: universal adhesive without silane in the composition (Ambar Universal, FGM, Joinville, Brazil), resulting into 6 evaluation groups per material tested (n = 8). Table 1 shows the composition of materials used.

Material (manufacturer)	Composition		
Monobond N (S-MDP) (Ivoclar Vivadent)	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulfide methacrylate.		
Prosil (PS) (FGM, Brazil)	3-methacryloxypropyltrimethoxysilane; Ethanol, water		
Scothbond Universal (UAS) (<i>3M ESPE, USA</i>)	Phosphate monomer (MDP), dimethacrylate resins, BisGMA, HEMA, methacrylate modified polyalkenoic a copolymer, camphorquinone, filler, ethanol, water, initiators, silane.		
Ambar Universal (UA) (FGM, Brazil)	MDP, methacrylic monomers, photoinitiators, coinitiators, silica nanoparticles, ethanol stabilizers.		
Rebilda DC resin cement (VOCO, Germany)	Bis-GMA, UDMA, DDDMA, inorganic fillers (69% weight)		
Resin nano-ceramic hybrid block (RCBs) (Grandio Blocs, Voco, Cuxhaven, Germany)	86% w/w inorganic filler (n.i.) in a methacrylate polymer matrix.		
Lithium disilicate ceramic block (LD) (IPS e.max, Ivoclar Vivadent)	58 - 80% SiO ₂ , 11 - 19% Li ₂ O, 0 - 13% K ₂ O, 0 - 8% ZrO ₂ , 0 - 5% Al ₂ O ₃		

Table 1. Composition of the materials used in the study.

Note: MDP - 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA - Bisphenolglycidil methacrylate; HEMA - 2-hydroxyethyl methacrylate; n.i. - non-informed.

Treatments and resin cement application

The surface treatments described were recommended by manufacturers. The RCBs samples were air abraded with 50 µm-particles aluminum oxide (2.8 psi) for 30 s, at a 50 mm distance. Samples were water-washed and air-dried for 60 s and placed in an incubator at 37°C for 24 h for complete drying. After, samples were divided into 3 groups (16 samples each) for silane/primer application: S-MDP, PS, and Ctr. Primers were actively applied to RCBs surface with a disposable microbrush for 60 s, and gently air dried for 20 s. After that, each group was divided into two subgroups (n = 8), for the application of adhesives: UAS and UA. One coat of UAS was applied to the silanized material surface with a disposable microbrush and left undisturbed for 20 s, gently air dried for 5 s, and light-activated for 10 s (Bluephase N, lvoclar Vivadent, Schaan, Liechtenstein). Two coats of UA were actively applied for 10 s each, gently air dried for 10 s, and light-activated during 10 s (Bluephase N, lvoclar Vivadent, Schaan, Liechtenstein).

The LD samples were acid etched with 10% hydrofluoric acid (Condac 10, FGM, Joinville, Brazil) for 20 s, samples were abundantly washed and dried. After, the samples were divided into 3 groups (16 samples each) for silane/primer application: S-MDP, PS, and Ctr (no silane application), as already described, and each group of silanized samples was divided in half (n = 8), for application of adhesives: UAS and UA, as already described.

Three polyurethane tubes (1.5 mm of internal diameter, 2.5 mm high) were positioned in each sample treated surface and filled with dual-cured resin cement (Rebilda DC, VOCO, Cuxhaven, Germany), totaling 24 cylinders per group. Each resin cement/tube was light activated during 40 s (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein). Samples were stored immersed in distilled water, at 37°C for 7 days until the test.

Bond strength analysis

Bond strength was assessed by microshear bond strength test. The samples were attached to a universal testing machine (MBio, BioPDI, Sao Carlos, Brazil) by a metal jig allowing the positioning of the specimens with the adhesive interface parallel to the load application.

The load was applied by a metallic chisel device and a shear force with a speed of 0.5 mm/min was applied parallel to the adhesive interface until the system failed. The micro shear bond strength (, MPa) was calculated by: $\sigma = L / A$, in which "L" is the load (N) at the moment of failure, and "A" is the area of the adhesive interface (mm2), considering the diameter of the cylinder 1.5 mm. Each sample contained 3 cylinders and the average bond strength of each sample was considered for statistical purposes. Pre-test failures (PTF) occurred in a few samples, mostly during the set of the samples in the machine, therefore they were disregarded for statistical analysis. The total number of PTF for each group are shown in figure 1. Failure analysis was performed for all samples and classified as adhesive (Adh), mixed (M), cohesive in the restorative material (ceramic or resin - CB), and cohesive in the resin cement (CC).

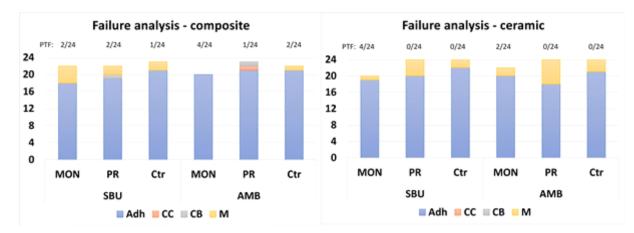


Figure 1. Failure distribution among groups: adhesive (Adh), mixed (M), cohesive in the restorative material (ceramic or resin - CB) and cohesive in the resin cement (CC).

Statistical analysis

Data were checked for normality assumption and then were submitted to analysis of variance in two factors (silane and adhesive) (ANOVA Two way) separately for ceramic and composite resin, followed by Tukey post hoc test ($\alpha = 0.05$).

RESULTS

For RCBs, silane application was not significant in bond strength values (p=0.444). The interaction of factors (silane x adhesive) was also not significant (p=0.668), but the adhesive was (p=0.004). The universal adhesive with silane (UAS) presented the highest bond strength values (table 2).

For LD, silane application was significant in bond strength values (p<0.001), but the interaction of factors (silane x adhesive) (p=0.201) and adhesive were not (p=0.066). The silane without MDP (PS) promoted the highest bond strength values, despite the adhesive used (table 2).

Failure analysis showed a high prevalence of adhesive failures for both substrates and is presented in Figure 1. Mixed failures were found for all groups regardless of the substrate, with the exception of composite blocks restored with UA and UAS. Cohesive failures were minor and happened only for resin composite blocks. Also, they were excluded from the statistical analysis.

Nano-ceramic hybrid block			
Silane	Adhesive		T. 4.1
	UAS	UA	– Total
S-MDP	15.98 (3.21) ^{Aa}	12.65 (2.63) ^{Aa}	14.31 (2.92) ^a
PS	15.09 (4.51) ^{Aa}	13.34 (3.07) ^{Aa}	14.21 (3.79) ^a
Ctr	17.45 (3.65) ^{Aa}	13.69 (2.37) ^{Aa}	15.57 (3.01) ^a
Total	16.17 (3.79) ^A	13.23 (2.69) ^B	
Lithium disilicate ceramic			
S-MDP	10.90 (2.75) ^{Aa}	12.83 (3.32) ^{Aa}	11.86 (3.03) ^a
PS	19.17 (2.40) ^{Ab}	18.74 (2.71) ^{Ab}	18.95 (2.55) ^b
Ctr	12.09 (2.41) ^{Aa}	15.43 (3.89) ^{Aab}	13.76 (3.15) ^a
Total	14.05 (2.52) ^A	15.67 (3.30) ^A	

 Table 2. Micro shear bond strength values (MPa) for the resin nano-ceramic hybrid block according to adhesive and silane application, respective standard deviation, and significance.

Note: Different uppercase letters indicate statistical difference in the same line. Different lowercase letters indicate statistical difference in the same column.

DISCUSSION

The additional application of silane associated with universal adhesives was relevant only for bonding to LD. Thus, the null hypothesis was rejected. But the universal adhesive with silane in composition presented higher bond strength values than the universal adhesive without silane for bonding to RCBs. A previous study reported that the use of materials containing methyl methacrylate (i.e. adhesives) improved the bond strength values to a polymeric CAD/CAM block [17].

The additional application of silane was not relevant for bonding to RCBs. The findings in the literature are controversial, since silane may be presented as relevant or not for bonding to polymeric CAD/CAM blocks [18,19]. As a bifunctional molecule, silane reacts both with the resin cement by the methacrylate group and with the filler particles (usually a glass, such as silica) of the nano-ceramic hybrid block or with the alumina from air abrasion by the silanol group [2]. The hypothesis is that the alumina/silica available on the RCBs surface was not be able to establish siloxane bonds to silane, and then, silane was not effective.

Polymerized composites, such as the prefabricated RCBs used in the present study, presents a high degree of monomer conversion, therefore, with few unreacted C=C bonds. Thus, the adhesion to them depends on physical (abrasion) treatment over the surface to create microretentions that improve bonding [17] Besides the abrasion with aluminum oxide particles performed over the RCB blocks, the UAS adhesive presents in its composition methacrylate-modified polyalkenoic acids [10] and 10-MDP, a monomer with known wetting ability and recognized chemical bond to the tooth mineral structures, with an indication that can benefit the composite-composite adhesion [20], but further analysis shall be performed to confirm this.

For polymeric CAD/CAM blocks, which had their behavior compared more to feldspathic ceramic than to composites (Vita Enamic, Ivoclar Vivadent), the use of silane, associated or not to adhesive is imperative [21]. The so-called 'resin

matrix ceramics' are defined as "materials with an organic matrix highly filled (> 50% by weight) with ceramic particles" and present a wide range of indications. Their composition may vary substantially in terms of organic and inorganic composition, and consequently, the surface treatment changes from one material to another, more similar to ceramics or more similar to composite resins [22].

The results obtained in the present study confirm that silane is imperative for bonding to glass-rich ceramic and may not be replaced by universal adhesive application, as also reported by Souza et al. [23] Therefore, the separate application of silane, or silane freshly mixed with the adhesive is recommended [5]. For this substrate, the acid etching procedure with HF aims to create micro-retentions and exposes hydroxyl groups that chemically bond to silane coupling agents, improving the overall bond strength to the resin cement.[23] Multimode adhesives, such as the universal ones, contain silane and MDP monomers for the purpose of simplifying the clinical process, however, silane suffers hydrolyzation depending on acidic pH and solvent system, among other factors [1]. Thus, the presence of water and acids on the composition of UAS (pH 2.7) impair the silane stability, hydrolysis, and performance of the double functional monomers,[10,23] justifying its inefficiency on bonding to LD and requiring additional silane application. The same situation may be the reason why S-MDP (MDP + Silane, Monobond N, Ivoclar Vivadent) presented an inferior performance on bonding ability when compared to PS (only silane, Prosil, FGM): the present of a solvent monomer (MDP), inside the same primer bottle, may have contributed to formation of siloxane oligomers/polymers that are inactive [1].

In the present study, the absence of adhesive was not simulated. The application of an adhesive layer after silane increases the wettability of the composite to be applied thereafter [24]. However, literature reports that the application of an adhesive layer after silanization of glass ceramic surface did not improve bond strength values to resin cement [25], and that silane treatment was the main factor responsible for resin bonding to ceramic [26]. Each material must be investigated regarding the additional application of a silane layer before the universal adhesive, and results may vary according to the material's composition[17].

The microshear bond strength test was chosen for this study because is cost-effective, easy to implement, and often used in studies to assess adhesion between two interfaces. The small area adopted (< 2 mm²) reduces the non-uniform stress distribution in the shear test [23]. Since the standard deviation in data was below 25% (most values below 20%) and failures were mainly adhesive (at least 70%), shear bond strength test results may be considered reliable in the present investigation.

Hence, in vitro analysis helps to better understand the behavior of materials before clinical applications. Our results indicated that the presence of silane in a universal adhesive system was not as efficient as the use of both agents (silane and adhesive) separately for ceramic materials. Future analysis shall consider the evaluation including the aging of the samples.

CONCLUSIONS

The additional application of silane is not required for bonding resin nano-ceramic hybrid CAD/CAM block; the use of a silane-containing universal adhesive presented the highest adhesion to resin nano-ceramic hybrid CAD/CAM block. However, for glass-ceramic CAD/CAM material, the isolated application of silane is essential, regardless of its presence in universal adhesives.

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Collaborators

M Amaral, data acquisition, statistical analysis, manuscript preparation. JMB Rizzato and VCS Almeida, literature search, data acquisition. PCS Liporoni, concepts, design, manuscript review. RF Zanatta, concepts, design, definition of intellectual content, statistical analysis, manuscript editing and review.

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