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# ORIGINAL ARTICLE

# Interaction of a universal adhesive with different surface treatments with feldespathic ceramics



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# **KEYWORDS**

Adhesives; Universal bond; Surface properties; Methacryloxypropy-Itrimethoxysilane; 10-methacryloyloxydecyl dihydrogen phosphate; Dental restoration repairs (MeSH) **Abstract** *Introduction:* Universal adhesives provide the possibility of simplified protocols for using ceramics; however, determining the synergy of these adhesives with silanes and the possibility of replacing silanes requires extensive research.

*Objective:* To evaluate the influence of a universal adhesive, associated with airborne-particle abrasion, acid etching, and silane, on the bond strength of a feldspathic ceramic CAD-CAM to composite resin.

*Materials and methods:* CAD-CAM feldspathic ceramic blocks were cut into 2-mm slices and were randomly divided into six groups (n = 10): A1, Single Bond Universal (SBU); A2, Adper Single Bond 2 (SB2); B1, silane + SBU; B2, silane + SB2; C1, acid etching + silane + SBU; C2, and acid etching + silane + SB2. Tygon tubes were placed and filled with composite resin. All samples were stored in distilled water at 37 °C for 24 h and then subjected to micro-shear tests. The type of failure was analyzed using a stereomicroscope.

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*Results:* SBU demonstrated slightly higher adhesion values compared with SB2 in all groups; however, no significant differences were observed between the adhesives (p < 0.05).

*Conclusion:* SBU was not statistically superior to the control adhesive. The use of silane prior to a universal adhesive should not yet be eliminated.

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#### 1. Introduction

At present, the integration of CAD-CAM allows multiple indirect restorative treatments using pre-sintered feldspar blocks with alumina or leucite reinforcement (Basso et al., 2015). These ceramics have good optical and mechanical properties, which eliminates the need for a metal structure and reduces time dental practice (Van Noort, 2012). A 5-year success rate of 89% is reported for treatment with feldspathic ceramics crowns, and this is 96% with alumina- or leucite-reinforced ceramics (Sailer and Ha, 2007). As a result of different factors such as the impact of occlusal forces, low elastic modulus materials, inadequate designs, trauma, and micro-defects, ceramic restorations can develop fractures that required a change or repair (Loomans and Özcan, 2016). Changing a ceramic restoration involves renewed time, money, and possible complications. As consequence, alternatives such as direct repairs with composite resins are required to extend the longevity of existing restorations (Loomans and Özcan, 2016; Hickel et al., 2013).

The success of a direct repair primarily is associated the chemical and mechanical interactions of the adhesives and composite resin with the ceramic surface (Özcan and Vallittu, 2003; Isolan et al., 2014). Surface ceramics are subjected to varying treatments to increase the surface energy by mechanical or chemical action (Yoshida et al., 2015). Some treatments involving physical action include acid etching (Fabianelli et al., 2010; Kim et al., 2015), airborne-particle abrasion (Ritesh Gourav et al., 2013; Soares et al., 2016), diamond burs, and tribological silica (Neis et al., 2015). Chemical conditioning can be performed using bifunctional silane agents that facilitate the interaction of the -OH groups of ceramics with the -COOH group of composite resins (Lung and Matinlinna, 2012). Another frequently studied alternative is universal adhesive system, which is hypothesized to adhere to enamel, dentin, resins, ceramics, and metals (Feitosa et al., 2014). These adhesives develop their properties through the inclusion of phosphate monomers (10methacryloyloxydecyl dihydrogen phosphate, MDP) and silanized particles (Yoshihara et al., 2016).

The objective of this study was to evaluate the influence of a universal adhesive associated with airborne-particle abrasion, acid etching, and silane on the strength of a CAD-CAM feldspathic ceramic bond to composite resin. The null hypothesis was that the groups including universal adhesive would not demonstrate higher performance than the groups with a conventional adhesive or control.

## 2. Materials and methods

#### 2.1. Preparation of sample

Twelve plates (2 mm thickness) were obtained from  $10 \times 12 \times 15$  mm CAD-CAM blocks of feldspathic ceramic

(Vitablocs TriLuxe, VITA Zahnfabrik, Germany) using a precision diamond saw with constant water cooling (Isomet 1000 Precision Saw, Buehler). The plates were sintered and glazed in a VITA VACUMAT 40 furnace using program number 58 of the VITA for VITABLOCS guide, at 900 °C for 10 min. The ceramic plates were then mounted in a self-curing acrylic (Veracril pink color, New Stetic, Medellín, Colombia), and one surface was left exposed for adhesion testing. The ceramicfree surfaces were ground using 400- and 600-grit sandpaper with abundant water. Cleaned using a steam gun (Triton-BEGO, USA Inc.) for 10 s at 20 mm in a perpendicular direction.

#### 2.2. Surface treatments

Airborne-particle abrasion (50  $\mu$ m Al<sub>2</sub>O<sub>3</sub>) was performed at 10 mm from the ceramic surface in a perpendicular direction using continuous firing (pressure, 2.5 bar) with an intraoral blaster (Microjato, Bio-Art, São Carlos – SP, Brazil) for 10 s. All samples were washed for 10 min with 97% ethanol. Two plates were randomly assigned in each group as follows (n = 10): ol (Monobond N, Ivoclar Vivadent Schaan, Liechtenstein) + SBU; B2, silane + SB2; C1, acid etching (9.6% hydrofluoric acid, Porcelain Etch, Ultradent Products Inc.) + silane + SBU; and C2, acid etching (9.6% hydrofluoric acid) + silane + SB2.

Silane (Monobond N) was applied with a microbrush, allowed to stand for 60 s, and then dried using high-pressure oil-free air for 5 s. SBU was applied with a microbrush and vigorously rubbed (20 s), dried with high-pressure, oil-free air (5 s), and was then polymerized (10 s) using LED lamp (Blue-phase C8, Ivoclar Vivadent Schaan, Liechtenstein) in the low program. SB2 was applied and vigorously rubbed (10 s), dried with high-pressure, oil-free air (5 s), and was polymerized for 10 s using LED unit (Bluephase C8, Ivoclar Vivadent, Schaan, Liechtenstein) in the low program. Hydrofluoric acid (9.6%, Porcelain Etch, Ultradent Products Inc.) was allowed to act for 90 s, jet-washed with water (20 s), and then dried with high-pressure, oil-free air.

#### 2.3. Adhesion

The adhesive area was limited using a double-sided tape (Scotch®-3M) and was previously drilled (diameter, 0.56 mm) using a rubber cloth perforator (Premium). Then, silane and/or adhesive was applied based on each group. Tygon tubes with 0.76 mm internal diameter and 2 mm height were installed on the exposed ceramic area, filled with resin (Filtek Z350 xt, 3 M ESPE, Saint Paul, MN, USA), and light cured using LED lamp (Bluephase C8, Ivoclar Vivadent, Schaan, Liechtenstein) in the soft program for 20 s for each 1-mm layer. All samples were stored in distilled water at

37 °C for 24 h (HYGROBATH) before performing microshear bond strength test (ISO/TS 11405:2015). Tygon tubes were then carefully removed using scalpel blades #12 and #15. The micro-shear bond strength test ( $\mu$ SBS) was executed using a universal mechanical testing machine (Shimadzu® AG-IS) a steel wire handle (0.22 mm) to pull the resin tubes, with a 50 N load cell and a 1 mm/min crosshead speed. All tests were performed by the same experienced operator, along with a prior standardization of the test design. The bond strength (MPa) was calculated using the equation MPa = N/ mm<sup>2</sup>, where N corresponded to the fracture force and mm<sup>2</sup> corresponded to the adhesive area determined by the equation  $\pi$ .r<sup>2</sup>. The adhesive area in the present study was 0.246 mm<sup>2</sup>.

## 2.4. Failure classification

For the analysis of the type of failure, a stereomicroscope with 50X of magnification was used (Nikon SMZ800, Nikon Instruments Inc. New York, United States). Failures were classified as follows: adhesive, when separation occurred between the two interfaces (ceramic and resin); cohesive, when separation occurred in a single interface, leaving a depressed ceramic surface or composite resin; and mixed, which included the above two types of failures in the same sample

## 2.5. Statistical analysis

The bond strength values of each group were recorded in a Microsoft® Office Excel database directly from the universal mechanical testing machine. Shapiro–Wilk normality test and Wilcoxon test (for pairwise comparison), were used for statistical analysis (R-Project for Statistical Computing).

#### 3. Results

Using Shapiro–Wilk test, a result of W = 0.87278(p =  $1.026 \times 10^{-6}$ ) was obtained, which meant the hypothesis of normality was rejected. Fig. 1 shows the distribution of values where the median was chosen as a measure of centrality due to the asymmetrical distribution. The lowest bond strength values were obtained for the groups in which only adhesives were used (A groups). For statistically comparing medians, there were no significant differences between both the adhesives in all the groups (Table 1). The result of the analysis of failure with stereomicroscope reported a higher tendency for cohesive failure (40%), followed by adhesive failure (36%), and finally mixed failure (24%) (Table 2). (See Fig. 2).

Stereomicroscope images at 50X (Nikon SMZ800 and Nikon NI-150 light fibers). A: adhesive failure with possible delimited exposed ceramic surface; B: cohesive failure with composite resin surfaces in almost the entire extension of the adhesive area; C: mixed failure with exposed ceramic surface and one area of the composite resin. The letters C and R correspond to ceramic and composite resin, respectively.

#### 4. Discussion

This study evaluates the interaction between universal adhesive and different surface treatments, comparing this performance with a conventional adhesive. Considering the results, the null hypothesis that the bond strength between a CAD-CAM feldspathic ceramic with different surface treatments and universal adhesive is equivalent to the bond strength of a conventional adhesive was not rejected. Thus, the groups with two adhesives were considered statistically equivalent in the three comparisons. Similarly, Ito et al. compared five universal adhesive systems and a conventional system on a feldspathic ceramic reinforced with leucite using tension test. Significant differences between the universal systems of a bottle and the conventional were not observed (Ito et al., 2015).

Adhesives after silane application, generally during a repair with composite resin, are used to improve the infiltration of adhesive molecules into roughness previously induced by some physical action treatment (Guarda et al., 2013). A significant increase in the bond strength in a lithium disilicatereinforcement ceramic along with the association of nonfunctional silanes and multipurpose adhesives compared to the only silane has been reported in the literature (Sundfeld et al., 2015). In contrast, when a functional silane is associated with a multipurpose adhesive on lithium disilicate reinforcement ceramics the previous statement is not exactly reproducible (Lise et al., 2015). This information can help interpret the results in the present study, where the association of the universal adhesive with a functional silane does not produce significant increases in the adhesion in comparison with the control adhesive. Regarding this interpretation, other study compared the synergy of a fifth-generation adhesive and universal adhesive with a functional silane and two surface treatments (diamond burs and tribological silica) under a micro-shear bond strength test in a lithium disilicate reinforcement ceramic. They reported that the synergy with universal adhesive was significantly lower than with the control adhesive (Wahsh and Ghallab, 2015).

Reportedly, MDP and silane present in the universal adhesive are not effective on their own. Some additional treatments, such as hydrofluoric acid and prior silane treatment, can significantly improve the union of the composite resin to vitreous ceramics (Kalavacharla et al., 2015). In 2017, a study compared the association of a universal adhesive, a fifthgeneration adhesive, and dual-cure adhesive and with a nonfunctional silane, in a lithium disilicate reinforcement ceramic. Using micro-shear test, they determined that the universal and dual-cured adhesives were not superior to the control. Additionally, silane can improve the values of bond strength when applied first in conventional and universal adhesive systems (Vasconcelos et al., 2017). In another study, this compared two universal adhesives and their association with a nonfunctional silane on lithium disilicate ceramics. They concluded that using silane prior to universal adhesive significantly optimized the values of bond strength compared with using the universal adhesive alone (Alrabiah et al., 2018). These results were consistent with the present study findings, where the groups that used silane prior to the application of both adhesives demonstrated a considerable increase in bond strength values. A limitation of this investigation is that only one type of silane is used. It is probable that different chemistry of another non-functional silane could change the results.

With respect to acid etching, an increase in bond strength values of composite resin on vitreous ceramics has been reported with airborne-particle abrasion and etching with hydrofluoric acid compared with the airborne-particle abrasion alone (Kim et al., 2005; Yucel et al., 2012). However,



Fig. 1 Descriptive bond strength (MPa).

Table 1	Bond strength	results (MPa	) and com	parison	by W	/ilcoxon	test.

	, <u>1</u>		
Groups $(n = 10)$	Median (MPa)	Mean (standard deviation) (MPa)	p-value
$\overline{A1}(AA + SBU)$	5.72 <sup>A</sup>	5.61 (1.29)	0.105
A2 $(AA + SB2)$	3.83 <sup>A</sup>	4.57 (1.68)	
B1 $(AA + S + SBU)$	11.82 <sup>B</sup>	10.94 (4.71)	0.853
B2 (AA + S + SB2)	9.37 <sup>B</sup>	10.64 (4.84)	
C1 (AA + HF + S + SBU)	12.89 <sup>B</sup>	15.38 (7.03)	0.063
C2 (AA + HF + S + SB2)	8.31 <sup>B</sup>	10.18 (4.33)	

Bond strength was measured in megapascals (MPa). The p-value column indicates comparison within each group between the two adhesives. P < 0.05 indicated statistically significant differences. Different superscript capital letters indicate a statistically significant difference. AA: airborne-particle abrasion, S: silane, SB2: Adper Single Bond 2, SBU: single bond universal.

Table 2	Classification of the type of failure.					
Groups	Adhesive failure	Cohesive failure	Mixed failure			
	%	%	%			
A1	80	10	10			
A2	60	0	40			
B1	0	60	40			
B2	30	30	40			
C1	20	80	0			
C2	70	0	30			
Total	36	40	24			

the current study show that the hydrofluoric acid doesn't produce a relevant positive effect on the bond strength with both the adhesives. Although the mechanism for this is unclear, it may be related to the excessive action of hydrofluoric acid on the vitreous matrix and crystals of the ceramic surface. Therefore, these results need to be interpreted with caution. Evaluation using hydrofluoric acid at different times and concentrations is warranted, along with that for acid etching without airborne-particle abrasion.

The largest number of cohesive failures in the study was observed in group C1. It is thus possible that a better bond strength between the ceramic and universal adhesive is a function of acid etching. However, this hypothesis is not applicable to the control adhesive. For future investigations, it is recommended that a cohesive failure pattern of the ceramic and resin be separately considered. Alternatively, it is interesting to change the pattern of failure from adhesive to cohesive and mixed, when silane is applied prior to both adhesives. This is consistent with the bond strength values reported in this investigation.

#### 5. Conclusions

Considering the limitations of the present investigation, the bond strength values of both adhesives was considered statistically the same. Therefore, a universal adhesive cannot be recommended to replace a conventional or fifth generation adhesive. Further studies evaluating the possible superiority



Fig. 2 Types of failure.

of these universal systems over other adhesives are recommended.

Also, silane present in the universal adhesive cannot yet replace a prior application of silane on the ceramic surface. A prior application of silane to the universal adhesive is required to improve the adhesion of composite resins to feldspathic ceramics.

## **Conflicts of interest**

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

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