

Original Research Article

Influence of adhesive application on the bond strength between feldspathic ceramic and resin cements

ABSTRACT

Aim: To evaluate the influence of adhesive application on the bond strength between feldspathic ceramic and two resin cements, as well as to evaluate microscopically the interface between the resinous materials and the feldspathic ceramic.

Methodology: Forty-eight feldspathic ceramic discs were included in self-cured acrylic resin and randomly divided into four groups (n=12): G1 – hydrofluoric acid + silane + adhesive + RelyX ARC; G2 – hydrofluoric acid + silane + adhesive + RelyX U100; G3 – hydrofluoric acid + silane + RelyX ARC; G4 – hydrofluoric acid + silane + RelyX U100. Cones of composite resin were bonded to the ceramic surface using the resin cements and light-cured. After storage in distilled water at 37° C for 24 h, the specimens were submitted to tensile bond strength in a universal testing machine at a crosshead speed of 1 mm/minute. The results were subjected to the Analysis of Variance and Tukey's test ($\alpha=0.05$).

Results: The bond strength means (MPa) followed by the same letter represent no statistical difference: G1 (28.34 MPa)^a, G2 (28.05 MPa)^a, G3 (21.92 MPa)^b, G4 (18.19 MPa)^b. The SEM images showed that the adhesive and the two resin cements infiltrated the surface irregularities created by the hydrofluoric acid.

Conclusion: The adhesive application on the ceramic etched with hydrochloric acid and silanized increased the bond strength between the resin cements and the feldspathic ceramic.

Keywords: adhesive; bond strength; feldspathic ceramic; resin cement.

1. INTRODUCTION

Dental ceramics are highly esthetic restorative materials with optimal esthetic properties that better simulate the appearance of natural dentition. Other desirable characteristics include chemical stability, biocompatibility, high compressive strength, and coefficient of thermal expansion similar to that of tooth structure. However, dental ceramics are brittle, a quality attributed to surface and bulk defects [1,2].

The first strengthened ceramic was developed in 1965 by changing the composition of the crystalline phase with the addition of aluminum crystals [3]. Subsequently, the introduction of ceramics with different compositions combined with the use of novel laboratory techniques has resulted in improved mechanical properties [4]. These strengthened all-ceramic restorations have been indicated for inlays, onlays, crowns, and fixed partial dentures [5].

The clinical success of the ceramic restoration depends on a number of factors, such as the luting procedure [6]. Strengthened all-ceramic restorations may be luted with zinc phosphate, glass ionomer, or resin cement [7]. However, feldspathic ceramic restorations have the lowest flexural strength [8], and adhesive luting systems are recommended to increase the resistance of the restoration [9,10].

In the case of adhesive luting, treatment of the internal surface of the restoration is recommended to improve the bond between the ceramic and the resinous material. For feldspathic ceramics, both the etching with hydrofluoric acid as the sandblasting with aluminum oxide particles are effective [11-13]. The use of silane that provides a chemical bond between the ceramic and the resinous material has also been recommended [14,15]. In clinical practice, some professionals use a hydrophobic adhesive on the internal surface of the ceramic restoration, while others dismiss this application and leave the resin cement directly in contact with the treated ceramic. However, whether the adhesive should be applied on the internal surface of the restoration is a controversial topic in the literature. Some studies show higher bond strength between the restoration and the resin cement with adhesive application [16], while others show that the adhesive application is not so important [17,18]. However, regardless of the luting technique, it is essential that the resinous material that is in direct contact with the ceramic is able to fill any irregularities created by the surface treatment.

Therefore, the aim of this study was to evaluate the influence of adhesive application on the bond strength between feldspathic ceramic and two resin cements. It also microscopically evaluated the bond interface between the resinous materials and the feldspathic ceramic. This study was conducted under the null hypothesis that the adhesive has no influence on bond strength values.

2. MATERIAL AND METHODS

Forty-eight feldspathic ceramic discs (Noritake, Aichi, Japão), with diameter of 8 mm, and a thickness of 4 mm, were obtained in a specialized laboratory. The ceramic discs were included in self-cured

acrylic resin using a metallic cylindrical device. The exposure surface of the ceramic was polished with wet 400 and 600 grit silicon carbide abrasive paper using a polishing under water cooling. The samples were randomly divided into four groups (n=12) according to the luting procedure:

Group 1 - hydrofluoric acid + silane + adhesive + RelyX ARC resin cement: the ceramic surface was etched with 10% hydrofluoric acid (Dentsply, York, PA, USA) for 2 minutes, followed by rinsing for 30 s, and dried with compressed air. One coat of Scotchbond Ceramic Primer (3M/Espe, St. Paul, MN, EUA) was applied, followed by gentle air drying. One coat of Scotchbond Multipurpose adhesive (3M/Espe, St. Paul, MN, USA) was applied, and light cured for 10 s with a LED light-curing unit Radium-cal (SDI, Bayswater, Vic, Australia) with light intensity of 1.000 mW/cm². Equal quantities of base and catalyst pastes of RelyX ARC resin cement (3M/Espe, St. Paul, MN, USA) were mixed and applied on the ceramic surface. Previously, a metallic split cylinder was used to build cones of composite resin (Z250, 3M/Espe, St. Paul, MN, USA) with inferior diameter of 3mm, superior diameter of 5 mm and height of 5 mm. The smallest diameter of the composite cone was placed on the ceramic surface, and a load of 1 kg was applied by means of a metallic tool. The excess cement was removed with a microbrush, followed by light curing for 40 s on three different areas of the sample, totaling 120 s of light curing.

Group 2 - hydrofluoric acid + silane + adhesive + RelyX U100 resin cement: the luting procedure was the same as described in group 1, being used the RelyX U100 resin cement (3M/Espe, St. Paul, MN, USA) .

Group 3 – hydrofluoric acid + silane + RelyX ARC resin cement: the same procedure as applied in group 1, but without the adhesive application.

Group 4 - hydrofluoric acid + silane + RelyX U100 resin cement: the same procedure as applied in group 2, but without the adhesive application.

The specimens were stored in distilled water at 37 °C for 24 h, and 12 specimens of each group were submitted to the tensile bond strength in a universal testing machine EMIC DL-2000 (EMIC, São José dos Pinhais, PR, Brazil) at a crosshead speed of 1 mm/minute. Tensile bond strength values in MPa were calculated from the peak load at failure divided by the specimen surface area. The results were subjected to the Analysis of Variance and Tukey's test ($\alpha=0.05$).

After the tensile bond strength tests, the fractured surfaces of the specimens were visually examined with a stereomicroscope at x20 to classify the type of failure that occurred during the

92 debonding procedure. They could be: a) adhesive (between the ceramic and the adhesive and/or
93 resin cement); b) cohesive in ceramic; c) cohesive in composite resin cone; d) mixed (adhesive and
94 cohesive failure in ceramic or composite resin cone).

95 The other two specimens in each group were sectioned in the center with a diamond disc
96 mounted on a low speed laboratory cutting machine (Labcut 1010, Extec Corp., London, UK) under
97 water cooling. The bond interfaces were polished with 600-, 800-, 1000-, 1200-, and 2000-grit silicone
98 carbide abrasive papers and wetted with water using manual pressure and rotary movements. The
99 interfaces were then polished with 6-, 3-, 1- and 0.25- μ m grit diamond pastes on a felt disk, also with
100 manual pressure. All of the specimens were ultrasonically cleaned in distilled water for 10 min to
101 remove the residues from polishing. The specimens were dried at room temperature for 7 days, gold-
102 sputter coated (Bal-Tec, Balzers, Liechtenstein) and observed by scanning electronic microscope
103 (Philips XL 30, Philips Electronic Instruments Inc., Mahwah, NJ, USA) at 5000 $\times$ magnification.

104

105 3. RESULTS

106 ANOVA revealed significant differences among the groups. Group 1 (28.34 MPa) and group 2 (28.05
107 MPa) had the highest mean bond strengths, without statistical differences between them ($P>.05$) and
108 were significantly greater than the other groups. Group 3 (21.92 MPa) had an intermediate mean and
109 did not differ statistically from groups 4 (18.19 MPa) (Table 1).

110

111 **Table 1. Tensile bond strength means (MPa), standard-deviations (SD) and coefficient of**
112 **variation (CV) of the groups.**

Groups	n	Mean (MPa)	SD	CV
Group 1 – With adhesive + RelyX ARC	10	28.34 ^a	7.39	26%
Group 2 – With adhesive + RelyX U100	10	28.05 ^a	7.42	26%
Group 3 – Without adhesive + RelyX ARC	10	21.92 ^b	9.47	43%
Group 4 – Without adhesive + RelyX U100	10	18.19 ^b	6.11	33%

113 Means followed by the same letter do not differ statistically according to Tukey's test at a significant
114 level of 5%

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116

The most common failure pattern in all groups was adhesive failure (Table 2), which is characterized by the failure between the ceramic and the adhesive and/or resin cement. There was cohesive failure in the ceramic in Groups 1 and 2. Mixed failures (adhesive and cohesive in composite resin cone) occurred in 3 specimens of group 1, and in 2 specimens of group 2 and group 4.

Table 2. Analysis of the failure mode.

Groups	Adhesive	Cohesive in ceramic	Mixed (adhesive and cohesive in ceramic)	Mixed (adhesive and cohesive in composite resin)
Group 1 – With adhesive + RelyX ARC	5	1	1	3
Group 2 – With adhesive + RelyX U100	7	1	0	2
Group 3 – Without adhesive + RelyX ARC	9	1	0	0
Group 4 – Without adhesive + RelyX U100	8	0	0	2

The SEM image show that the adhesive has infiltrated the ceramic surface irregularities that were formed by the hydrofluoric acid etching (Fig. 1(i)). The resin cements RelyX ARC (Fig. 1(ii)) and RelyX U100 (Fig. 1(iii)) also infiltrated the surface irregularities created by the hydrofluoric acid.

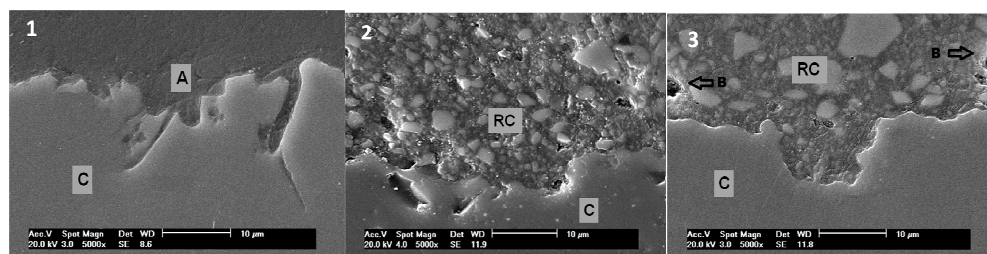


Fig. 1 – SEM images (5000x): (i) - Interface between Scotchbond MultiPurpose adhesive and feldspathic ceramic etched with 10% hydrofluoric acid (5000x): (A) adhesive; (C) ceramic. (ii) - Interface between RelyX ARC resin cement and feldspathic ceramic etched with 10% hydrofluoric: (RC) resin cement (C) ceramic. (iii) - Interface between RelyX U100 resin cement and feldspathic ceramic etched with 10% hydrofluoric: (RC) resin cement; (C) ceramic (B) bubbles.

4. DISCUSSION

This study analyzed the tensile bond strength between two resin cements and a feldspathic ceramic with and without application of the adhesive in the ceramic surface. The RelyX ARC conventional resin cement, the RelyX U100 self-adhesive resin cement, and the Scotchbond Multipurpose adhesive were used. The application of the adhesive on the ceramic surface favored the higher bond strength values on the ceramic etched with hydrofluoric acid and silanized. Therefore, the null hypothesis was rejected.

In the present study, the ceramic surface was etched with 10% hydrofluoric acid. This surface treatment is effective for feldspathic ceramics, because it produces an irregular surface that enhances micromechanical retention between resinous material and ceramic surface [12]. The hydrofluoric acid reacts with the silica phase of the feldspathic ceramic to form hexafluorosilicates. These silicates are removed by rinsing with water, and the final result is a honeycomb-like surface, ideal for micromechanical retention [19].

Besides the etching with hydrofluoric acid, the silane was applied in all samples. Silane coupling agents are monomeric species in which silicon is linked to reactive organic radicals and hydrolyzable ester groups. Hydrolyzable monovalent groups bond chemically to silicon contained in the glassy matrix. The reactive organic groups become chemically bonded to the resin molecules, such as Bis-GMA and HEMA monomers found in the Scotchbond Multipurpose adhesive as well as in the RelyX ARC and RelyX U100 resin cements [17,20]. Studies show that the application of silane provides higher bond strength values between the feldspathic ceramic and resinous materials [21,22]. A possible explanation for higher bond strength values in groups in which the adhesive was applied may be related to the silane application. The Scotchbond Multipurpose adhesive has no filler and, therefore, has a higher percentage of monomers available for reaction with the silane in comparison with the resin cements that have fillers. Thus, the adhesive must have favored greater chemical interaction with the silane and higher bond strength values.

The RelyX ARC and RelyX U100 resin cements have fillers in their composition and, therefore, higher viscosity in comparison with the Scotchbond Multipurpose adhesive that has no filler. Therefore, the expectation of this study was that both resin cements would not have the same ability to fill the irregularities of the ceramic surface. However, this expectation was not confirmed because the SEM images showed that both resin cements were able to fill all the surface irregularities in the

same way as the adhesive. Two factors may have contributed to this positive result: a) the 1 kg load applied during the luting, which pushed the resin cements toward the surface of the ceramic; b) the application of silane which has the capacity to improve surface wettability, causing better contact and infiltration of the resinous material in irregularities on the ceramic [20].

Analysis of failures is mandatory when bond strength tests are performed. This analysis aims to determine where the rupture (failure) occurs because the location of the failure corresponds to the value obtained in megapascals. When failure occurs cohesively in the composite resin cone, the value obtained corresponds to the cohesive strength of the composite resin. However, in bond strength tests of resinous materials, the aim is to evaluate the adhesive interface with the substrate (feldspathic ceramic), and not in other regions, such as inside the ceramic (cohesive ceramic) or inside the composite resin cone (cohesive in composite resin). In this study, the majority of failures were adhesive or mixed. The mixed failures were the association between adhesive and cohesive failure in the composite resin cone. Therefore, in most specimens, the adhesive interface, which corresponds to the most important region, was evaluated. In groups 1 and 2, where the adhesive was applied, there were two cohesive failures in ceramic, and this type of failure was not observed in groups 3 and group 4 where the adhesive was not applied. Therefore, this type of failure has been associated with the specimens in which the bond strength values were higher. There were also a higher percentage of mixed failures in groups 1 and 2.

Although the results showed higher bond strength values with adhesive application, this does not necessarily mean that it has to be used. This is because no one knows the minimum bond strength value required between the ceramic and the resinous material in order to have longevity and clinical success. However, the results of the present study show that the adhesive is necessary to optimize the bond between the ceramic and the resinous material. Longitudinal clinical trials evaluating feldspathic ceramic restorations, with and without adhesive application, are important to show the real need of the adhesive application on the internal surface of the restoration.

5. CONCLUSIONS

According to the results of the study, it was possible to conclude that:

- The adhesive application on the ceramic, etched with hydrochloric acid and silanized, increased the bond strength between the resin cements and the feldspathic ceramic.

- The RelyX ARC and RelyX U100 resin cements had the same ability of the adhesive to fill the irregularities of the feldspathic ceramic surface etched with hydrofluoric acid.

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