British Journal of Applied Science & Technology
15(6): 1-6, 2016, Article no.BJAST.25600
ISSN: 2231-0843, NLM ID: 101664541

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Influence of Adhesive Application on the Bond Strength between Feldspathic Ceramic and Resin Cements

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Authors' contributions

This work was carried out in collaboration between all authors. Author AMS designed the study and wrote the first draft of the manuscript. Author BGD performed the experiments and managed the literature searches. Authors EDBJ, MNG and RNL contributed substantially to experimental design and discussion. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/BJAST/2016/25600
Editor(s):
(1) Nan Wu, Department of Mechanical and Manufacturing Engineering, University of Manitoba, Winnipeg, Canada.
Reviewers:
(1) Cesar Dos Reis Perez, State University of Rio de Janeiro, Brazil.
(2) Cornelis H. Pameijer, University of Connecticut, USA.
(3) Rama Krishna Alla, Vishnu Dental College, Bhimavaram, India.
Complete Peer review History: http://sciencedomain.org/review-history/14424

Original Research Article

Received 11th March 2016
Accepted 18th April 2016
Published 3rd May 2016

ABSTRACT

Aim: To evaluate the influence of an adhesive on the bond strength between feldspathic ceramic and two resin cements. In addition the interface between the resinous materials and the feldspathic ceramic was evaluated microscopically.

Methodology: Forty-eight feldspathic ceramic discs were imbedded in self-curing acrylic resin and randomly divided into four groups as follows (n=12 per group): 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 then light cured. After storage in distilled water at 37°C for 24 h, the specimens were subjected to tensile bond

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strength tests in a universal testing machine at a crosshead speed of 1 mm/minute. Analysis of Variance (ANOVA) and Tukey's test (α=0.05) were used to statistically analyse the results.

Results: The bond strength means (MPa) followed by the same letter were not significantly different: G1 (28.34 MPa)[a], G2 (28.05 MPa)[a], G3 (21.92 MPa)[b], G4 (18.19 MPa)[b]. Scanning Electron Microscopy images showed that the adhesive and the two resin cements infiltrated the surface irregularities created by the hydrofluoric acid.

Conclusion: Adhesive application on the ceramic that was etched with hydrofluoric 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 aesthetic restorative materials with optimal aesthetic properties that better simulate the appearance of natural dentition in comparison with composite resin. Other desirable characteristics include chemical stability, biocompatibility, high compressive strength, and a coefficient of thermal expansion similar to that of normal tooth structure. However, dental ceramics are brittle, which is 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 aluminium crystals [3]. Subsequently, the introduction of ceramics with different compositions combined with the use of improved laboratory techniques has resulted in better mechanical properties [4]. These strengthened all-ceramic restorations have been indicated for inlays, onlays, crowns, and fixed partial dentures [5].

The clinical success of ceramic restoration depends on a number of factors, one of which is the choice of a luting material and the luting procedure employed [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, etching with hydrofluoric acid (HF) and sandblasting with aluminium oxide particles are effective [11-13]. Additionally, the use of silane provides a chemical bond between the ceramic and the resinous material [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 adhesive application is not 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 HF 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. Additionally, this study microscopically evaluated the material 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. MATERIALS AND METHODS

Forty-eight feldspathic ceramic discs (Noritake, Aichi, Japão), with a diameter of 8 mm and 4 mm thick, were obtained from a dental laboratory. The ceramic discs were imbedded in self-curing acrylic resin using a metal cylindrical device. The surface of the ceramic to be bonded was polished with wet 400- and 600-grit silicon carbide abrasive paper. The samples were randomly divided into four groups (n=12 per group) according to the four luting procedures:

> Group 1 - hydrofluoric acid + silane + adhesive + RelyX ARC resin cement: (3M/Espe, St. Paul, MN, USA). The ceramic surface was etched with 10% hydrofluoric acid (Dentsply, York, PA, USA) for 2 minutes, followed by rinsing for 30 s, and then dried with compressed air. One coat of

Scotchbond Ceramic Primer (3 M/Espe) was applied, followed by gentle air drying. One coat of Scotchbond Multipurpose adhesive (3 M/Espe) was applied, and the tip of the light-curing unit Radii-cal (SDI, Bayswater, Vic, Australia) was positioned close to the adhesive without touching it. The adhesive was light cured for 10 s with a light intensity of 1000 mW/cm^2. Equal quantities of base and catalyst pastes of RelyX ARC resin cement were mixed and applied on the ceramic surface. Previously, a split metal cylinder was used to build cones of composite resin (Z250, 3 M/Espe) measuring 3 by 5 mm with a height of 5 mm. The top of the split metal cylinder was positioned on a glass plate and the cone filled with the composite resin in two increments. Each increment was light cured for 20 s. The flat top of the composite resin cone was placed on the ceramic surface, and a load of 1 kg was applied with a metal tip. Excess cement was removed with a microbrush, followed by light curing for 40 s from three different angles for a total of 120s.

Group 2 - hydrofluoric acid + silane + adhesive + RelyX U100 resin cement: the luting procedure was the same as that described in group 1, using the RelyX U100 resin cement (3M/Espe).

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

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

The specimens were stored in distilled water at 37℃ for 24 h, and 12 specimens from each group were submitted to tensile bond strength testing in a universal testing machine, EMIC DL-2000 (EMIC, São José dos Pinhais, PR, Brazil). The specimen was attached to the base of the universal testing machine, while a clamp engaged the composite resin cone. The tensile test was carried out with 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. Analysis of variance (ANOVA) and Tukey's test (α=0.05) were used to statistically analyse the results.

After the tensile bond strength tests, the fractured surfaces of the specimens were visually examined with a stereomicroscope at x20 magnification to classify the type of failure that occurred during the debonding procedure. The failure types were classified as follows: a) adhesive (between the ceramic and the adhesive and/or resin cement), b) cohesive in ceramic, c) cohesive in composite resin cone and d) mixed (adhesive and cohesive failure in ceramic or composite resin cone).

The remaining two specimens in each group were sectioned in the middle with a diamond disc mounted on a low-speed laboratory cutting machine (Labcut 1010, Extec Corp., London, UK) under water cooling. The bond interface was polished with subsequently 600-, 800-, 1000-, 1200-, and 2000-grit wet silicon carbide abrasive papers using manual pressure and rotary movements. The interface was then polished with 6-, 3-, 1-, and 0.25-µm grit diamond pastes on a felt disk using manual pressure. The specimens were subsequently ultrasonically cleaned in distilled water for 10 min to remove the polishing residues. The specimens were dried at room temperature for 7 days, gold-sputter coated (Bal-Tec, Balzers, Liechtenstein), with gold-palladium and observed with a scanning electronic microscope (Philips XL 30, Philips Electronic Instruments Inc., Mahwah, NJ, USA) at 5000× magnification.

3. RESULTS

ANOVA revealed significant differences among the groups. Group 1 (28.34 MPa) and Group 2 (28.05 MPa) had the highest mean bond strengths; however, although these results were significantly stronger than the other groups, significant differences were not observed between Groups 1 and 2 (P>.05). Group 3 (21.92 MPa) had an intermediate mean and did not differ significantly from Group 4 (18.19 MPa) (Table 1). However, both Groups 3 and 4 demonstrated a significant statistical difference from Groups 1 and 2.

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

The SEM images showed that the adhesive had infiltrated into 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)) both infiltrated the surface irregularities created by the hydrofluoric acid.

4. DISCUSSION

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

In the present study, the ceramic surface was etched with 10% HF acid. This surface treatment is effective for feldspathic ceramics because it produces a microscopic irregular surface that enhances micromechanical retention between resinous materials and the ceramic surface [12]. The HF 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, which is ideal for micromechanical retention [19].

In addition to the above mentioned etching with HF acid, silane was applied to all samples. Silane coupling agents are monomeric in which silicon is linked to reactive organic radicals and hydrolysable ester groups. Hydrolysable monovalent groups bond chemically to silicon

Table 1. Tensile bond strength means (MPa), standard deviations (SDs) and coefficients of variation (CVs) 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%

Means followed by the same letter do not differ significantly according to Tukey's test (significance level of 5%)

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

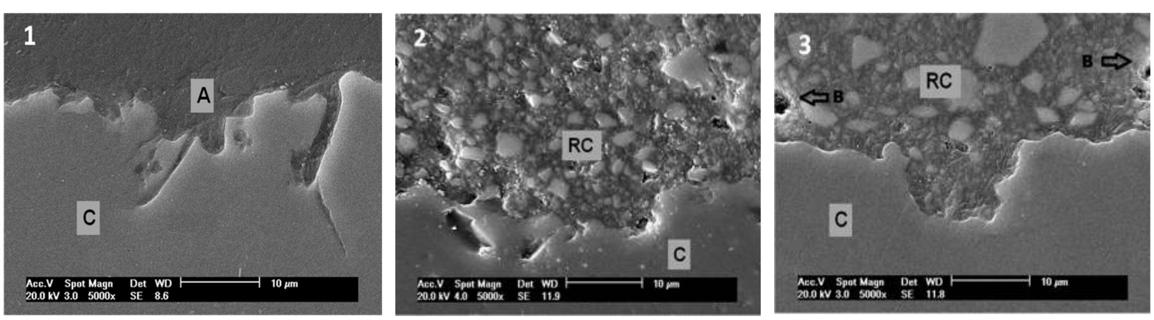

Fig. 1, 2 and 3. SEM images (5000x): (1) - interface between scotchbond multipurpose adhesive and feldspathic ceramic etched with 10% hydrofluoric acid (5000x): (A) adhesive; (C) ceramic. (2) - interface between relyx ARC resin cement and feldspathic ceramic etched with 10% hydrofluoric acid: (RC) resin cement; (C) ceramic. (3) - interface between RelyX U100 resin cement and feldspathic ceramic etched with 10% hydrofluoric acid: (RC) resin cement; (C) ceramic; (B) voids in cement, see arrows

contained in a glassy matrix. The reactive organic groups become chemically bonded to the resin molecules, such as the 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 have shown that the application of silane provides higher bond strength values between the feldspathic ceramic and the resinous materials [21,22]. A possible explanation for the higher bond strength values in groups in which the adhesive was applied may be related to silane application. The Scotchbond Multipurpose adhesive has no filler and therefore has a higher percentage of monomers available for reaction with silane than the resin cements that have fillers. Thus, the adhesive favoured a greater chemical interaction with silane, which resulted in higher bond strength values.

The RelyX ARC and RelyX U100 resin cements have fillers in their composition and therefore have higher viscosity than 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 presumption was not confirmed because SEM images showed that both resin cements were able to fill all of the surface irregularities in the same way as the adhesive. Two factors may have contributed to this positive result and are as follows: a) the 1-kg load applied during luting, which pushed the resin cements towards the surface of the ceramic, and b) the application of silane, which has the capacity to improve surface wettability, causing better contact and infiltration of the resinous material in the irregularities on the ceramic [20].

Analysis of failure 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), but not other regions, such as within the ceramic (cohesive ceramic) or within the composite resin cone (cohesive in composite resin). In this study, the majority of failures were adhesive or mixed. The mixed failures were a combination of 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, two cohesive failures occurred in the ceramic where the adhesive was applied; however, this type of failure was not observed in Groups 3 and 4, where the adhesive was not applied. Therefore, this type of failure has been associated with specimens in which bond strength values were higher. Additionally, a higher percentage of mixed failures occurred in Groups 1 and 2.

Although the results showed higher bond strength values with adhesive application, this does not necessarily mean that this application has to be used; the required minimum bond strength value between the ceramic and the resinous material for longevity and clinical success is unknown. However, the results of the present study showed that 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 whether there is a need for the adhesive application on the internal surface of the restoration.

5. CONCLUSIONS

Within the limitations of this study, the following can be concluded:

- 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 as the adhesive to penetrate the irregularities of the feldspathic ceramic surface after etching with hydrofluoric acid.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Peer-review history:
The peer review history for this paper can be accessed here:
http://sciencedomain.org/review-history/14424