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Evaluation of the Weakest Interface in the Adhesive Luting of a Computer-aided Design/ Computer-aided Manufacturing Composite Resin

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Abstract:

Background: The aim of the study was to evaluate, *in vitro*, the weakest bond interface of lava ultimate onlays luted with Scotchbond Universal and RelyX Ultimate.

Materials and Methods: A total of 56 Lava Ultimate onlays were milled and divided into eight groups according to the luting procedure to the human teeth (n = 7): G1 and G2 - Scotchbond Universal using the etch-and-rinse technique; G3 and G4 - Scotchbond Universal using the self-etch technique; G5 and G6 - Scotchbond Universal using the etch-and-rinse technique + adhesive application on the onlay; G7 and G8 - Scotchbond Universal using the self-etch mode + adhesive application on the onlay. The onlays were luted with RelyX Ultimate. Only the samples from G2, G4, G6 and G8 were submitted to cyclic loading. Beams with a cross section area of ~0.80 mm^2 were obtained and submitted to microtensile bond strength (μTBS) test in a universal testing machine. The types of failure were observed in optical microscope.

Results: According to three-way analysis of variance, there was a significant interaction between the three factors (P = 0.006). The μTBS means (MPa) followed by the same letter represent no statistical difference by Tukey's *post-hoc* test (P < 0.05): G6 = 34.64^{a}, G1 = 34.48^{a}, G2 = 31.94^{ab}, G5 = 30.88^{abc}, G7 = 26.66^{bc}, G8 = 24.03^{cd}, G4 = 18.81^{de}, G3 = 14.64^{e}. Most failures were at the resinous agent-restoration interface.

Conclusion: The resinous agent-restoration interface corresponded to the weakest interface. The application of Scotchbond Universal on the internal surface of the onlay was not a relevant procedure.

Key Words: Bond strength, computer-aided design/computer-aided manufacturing, composite resin, indirect restorations

Introduction

In clinical practice, indirect restorations have been preferred to direct restorations in extensive cavities.[1,2] These restorations have the advantage of better adaptation and marginal integrity, better anatomy and proximal contacts, and greater control of the stress generated during polymerization shrinkage in the case of composite resins.[1,3,4]

Indirect composite resin restorations have demonstrated good esthetic and functional results due to improvements in their properties and improvements in the adhesive luting agents.[5,6] These restorations also enable more conservative preparations, reinforce the tooth structure, cause low abrasion on the antagonist tooth and have a low elastic modulus, allowing greater absorption of functional loads.[1,3] Composite resin restorations represent a good alternative to ceramics. Currently, composite resins are available for use with CAD/CAM technology. This technology allows the production of indirect restorations in a single session, eliminating the steps of impression, and provisory restoration as in the conventional technique.[7]

Regardless of CAD/CAM technology, the choice of luting material has been considered one of the major factors that ensure the success of indirect restorations.[6] The professional must have knowledge of the techniques used with adhesive materials, as well as their ability to bond the dental substrate and indirect restorative materials because the function of adhesive materials is to ensure an effective link between restoration and tooth structure.[4,8-10]

The technique of conventional adhesive luting consists of the application of an adhesive system to the dental substrate before the resin cement. The adhesive system employed may be the etch-and-rinse technique, in which the dental substrate is etched with 35% phosphoric acid, or the self-etch technique, in which acidic monomers are used for etching. Another possibility is the use of self-adhesive resin cements, which dispense the application of the adhesive system. Regarding the different techniques, studies have shown that resin cements that employ adhesive systems promote increased bond strength to the dental substrate compared to the self-adhesives.[11-14]

Scotchbond Universal is a new one-bottle adhesive system recently launched on the market, and it is also indicated for luting indirect restorations. According to the manufacturer, this adhesive system is classified as "multi-mode" or "universal" because it can be applied using either the etch-and-rinse or the self-etch technique as well as on different materials. This

adhesive system should be associated with the resin cement RelyX Ultimate in adhesive luting procedures.

In addition to the treatment of the tooth surface with an adhesive agent, it is recommended to treat the internal surface of the restoration with sandblasting with aluminum oxide to increase the surface roughness.[6,15] However, whether the adhesive should be applied on the internal surface of the restoration is a controversial topic in the literature.[16]

Regardless of the technique used, different bond interfaces are formed in the adhesive luting of an indirect restoration, and it is important to assess which interface is less resistant and, consequently, could compromise the longevity of the bond and the success of an adhesive procedure.

The aim of this study was to evaluate the weakest bond interface of Lava Ultimate onlays luted with the adhesive system Scotchbond Universal and the resin cement RelyX Ultimate with the following variables: (a) Application of the adhesive system using the etch-and-rinse or the self-etch technique; (b) with and without the application of adhesive on the internal surface of the restoration; (c) with and without cyclic loading. The study was conducted under the hypothesis that the bond interface with the dentin is the weakest interface.

Materials and Methods

A total of 56 unerupted human third molars, extracted for therapeutic reasons, were obtained from the tooth bank after the approval of the Ethics Committee of the Pontifical Catholic University of Rio Grande do Sul. The teeth were cleaned of gross debris, disinfected with 0.5% chloramine for 24 h, and stored in distilled water at 4°C. The water was changed every week, and the teeth were used within 6 months.

Each tooth was mounted vertically in a plastic cylinder with self-cured acrylic resin (Jet Classico, São Paulo, SP, Brazil) up to 2 mm below the cement-enamel junction.

A single operator performed the mesio-distal-occlusal-lingual preparations using a no. 4138 diamond bur (KG Sorensen, Barueri, SP, Brazil) attached to a high-speed hand piece under constant water and air cooling, followed by a 4138F diamond bur (KG Sorensen, Cotia, SP, Brazil). The preparations presented rounded internal angles, expulsive walls and 2 mm deep at the occlusal surface. The diamond burs were replaced after every five preparations.

The onlays in composite resin Lava Ultimate (3M, St. Paul, MN, USA) were made by CAD-CAM using Cerec software (version 4.0.2, Sirona Dental Systems GmbH, Bensheim, Germany). The preparations received reflective spray titanium (VITA Zahnfabrik, Germany) to create an opaque surface needed for scanning by an optical 3D intraoral camera, creating a three-dimensional virtual model. The shape of the onlays was designed with an individual biogeneric copy from a lower right second molar. The thickness of the restoration in the occlusal face was 2.0 mm and 2.0 mm in the mesial-distal-lingual surfaces. The virtual die spacer was 50 μm without removal of retentions. The 56 teeth with the onlays were randomly divided into eight groups ($n = 7$, for each group) according to the luting procedure:

Group 1 and Group 2 - Scotchbond Universal was applied using the etch-and-rinse technique. The dentin was etched with 35% phosphoric acid for 15 s, followed by rinsing with air and a water spray for 15 s. The excess water was removed with cotton buds. The adhesive was applied with a microbrush and scrubbed for 20 s, followed by gentle air-drying for 5 s. The adhesive was light cured for 10 s with an LED light-curing unit Radii-cal (SDI, Bayswater, Vic, Australia).

Group 3 and Group 4 - Scotchbond Universal was applied using the self-etch technique. The adhesive was applied to the dentin with a microbrush and scrubbed for 20 s, followed by gentle air drying for 5 s and light-curing for 10 s.

Group 5 and Group 6 - Scotchbond Universal was applied using the etch-and-rinse technique with adhesive application on the onlay. The adhesive system was applied as described for Group 1, and a layer of the adhesive Scotchbond Universal was applied in the internal surface of the onlay and light cured for 10 s.

Group 7 and Group 8 - Scotchbond Universal was applied using the self-etch technique with adhesive application on the onlay. The adhesive system was applied as described for Group 3, and a layer of the adhesive Scotchbond Universal was applied in the internal surface of the onlay and light cured for 10 s.

The internal surfaces of the onlays were sandblasted with 50 μm aluminum oxide for 5 s. Equal quantities of base and catalyst pastes of RelyX Ultimate were mixed and applied on the onlay and preparation. The onlay was placed on the preparation, 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 60 s on each free surface. The specimens were stored in distilled water at 37°C for 24 h.

After storage, the specimens of Groups 2, 4, 6 and 8 were submitted to cyclic loading (Erios ER-11000, Erios, São Paulo, SP, Brazil) at 100 N using 1,000.000 cycles at 1 Hz in distilled water.

The teeth with the onlays were sectioned perpendicular to the occlusal surface using a Labcut 1010 laboratory cutting machine at a speed of 400 rpm with a diamond disk under water cooling. The specimens presented a transverse section approximately 0.80 mm × 0.80 mm, measured with a digital caliper (Mitutoyo Sul Americana Ltda., Suzano, SP, Brazil). Four beams were

used from the central region of each tooth, which corresponded with the occlusal region of the preparation. The beams were examined with an optical microscope (Telelupa, São Paulo, SP, Brazil) at 20x magnification to analyze the adhesive area. The specimens presenting defects such as bubbles, lack of material or irregular areas were discarded. 24 specimens were selected from each group.

The specimens were then fitted to the microtensile testing device for study. This device has two stainless steel grips with an area of 8 mm × 10 mm and sliding shafts that prevent torsion movements during the tests. These shafts have a fixing screw that prevents the specimen from moving during bonding. The specimens were fixed with cyanoacrylate glue (Loctite, São Paulo, SP, Brazil), associated with the Zip Kicker accelerator (Pacer Technology, Rancho Cucamonga, CA, USA), and stressed at a crosshead speed of 0.5 mm/min until failure in a universal testing machine (EMIC DL-2000, São José dos Pinhais, PR, Brazil) using a cell load of 50 N. The microtensile bond strength (μTBS) was expressed in MPa and derived by dividing the imposed force (N) at the time of fracture by the bond area (mm^2).

The fractured surfaces of all specimens were observed by an optical microscope at ×20 (Telelupa, São Paulo, SP, Brazil). The failures were classified as the following: (a) Cohesive in dentin, (b) dentin-resinous material interface, (c) cohesive in resin cement, (d) resinous material-restoration interface, and (e) cohesive in restoration. In the present study, resinous material corresponds to the adhesive system and resin cement.

The values of TBS were analyzed by three-way analysis of variance (ANOVA) (mode of adhesive system application X adhesive application on the internal surface of the onlay x cyclic loading) and *post-hoc* multiple comparisons using Tukey's test ($P < 0.05$).

Results

According to three-way ANOVA, there was an interaction between the three factors ($P = 0.006$).

Table 1 shows the mean bond strength values among the groups. Group 6 (34.64 MPa), Group 1 (34.48 MPa), Group 2 (31.94 MPa), and Group 5 (30.88 MPa) had the higher mean bond strengths without statistical differences between them ($P > 0.05$). Group 7 (26.66 MPa) had an intermediate mean and did not differ statistically from Groups 2, 5 and 8 (24.03 MPa). Group 8 did not differ statistically from group 4 (18.81 MPa). The lowest mean bond strength was in Group 3 (14.64 MPa), which was not statistically different from Group 4 ($P > 0.05$).

There was a predominance of failure in the resinous agent-restoration interface, especially for Groups 1, 3, 4 and 5. For Groups 2, 7 and 8, there was a distribution of failure in the resinous agent-restoration interface and cohesive in restoration. For Group 6, there was a distribution among the failures at the dentin-resinous agent interface, cohesive in resin cement, and resinous agent-restoration interface (Table 2).

Table 1: μTBS means and standard-deviations of the experimental groups.

Groups	μTBS means and standard-deviations (MPa)
G6 - Total-etch, with adhesive on onlay, with cyclic loading	34.64 (±8.85)[a]
G1 - Total-etch, without adhesive on onlay, without cyclic loading	34.48 (±12.58)[a]
G2 - Total-etch, without adhesive on onlay, with cyclic loading	31.94 (±9.18)[ab]
G5 - Total-etch, with adhesive on onlay, without cyclic loading	30.88 (±6.85)[abc]
G7 - Self-etch, with adhesive on onlay, without cyclic loading	26.66 (±8.25)[bc]
G8 - Self-etch, with adhesive on onlay, with cyclic loading	24.03 (±7.65)[cd]
G4 - Self-etch, without adhesive on onlay, with cyclic loading	18.81 (±4.14)[de]
G3 - Self-etch, without adhesive on onlay, without cyclic loading	14.64 (±5.35)[e]

Means followed by the same letter do not differ statistically according to Tukey's test at a significant level of 5%. μTBS: Microtensile bond strength

Discussion

This study evaluated the bond strength of specimens for the microtensile test obtained from onlays in Lava Ultimate composite resin made by CAD/CAM technology. These onlays were luted with the Scotchbond Universal adhesive system and RelyX Ultimate resin cement, with three factors of variation: (a) Etch-and-rinse or self-etch application of the adhesive system; (b) the application of the adhesive system on the internal surface of the onlay; (c) cyclic loading.

The specimens submitted to μTBS test corresponded to a stick with a cross-sectional area of ~0.80 mm^2, and two main bond interfaces were formed at the time of luting the onlays on the tooth. The interfaces were a dentin-resinous agent interface and a resinous agent-restoration interface. Thus, the μTBS values should be analyzed together with the types of failures that occurred in the different groups because the bond strength may be related to different interfaces.

The failure analysis allows observation of the interface that has the lowest bond strength and, hence, is a more susceptible to failure. In this study, most failures occurred at the resinous agent-restoration interface and not in the dentin-resinous agent interface, as shown in Table 2. Thus, it can be assumed that the dentin-resinous agent interface was tougher and remained preserved, regardless of the application of the adhesive system in the etch-and-rinse or self-etch technique. Therefore, the adhesives materials employed in the luting procedure, more specifically the Scotchbond Universal adhesive system, showed a considerable bond to dentin because failures at the dentin interface were observed in 7 out of the 192 specimens

Table 2: Distribution of the failures in the experimental groups.

Failures	Cohesive in dentin	Dentin- resinous agent interface	Cohesive in resin cement	Resinous agent-restoration interface	Cohesive in restoration
Group 1	1	0	0	21	2
Group 2	0	0	3	13	8
Group 3	0	0	0	21	3
Group 4	0	0	0	17	7
Group 5	1	0	0	20	3
Group 6	0	6	11	6	1
Group 7	0	1	1	12	10
Group 8	0	0	2	11	11

tested. Thus, the hypothesis was rejected because the bonding interface with the dentin did not correspond to the weakest interface.

The Scotchbond Universal adhesive system includes the 10-methacryloyloxydecyl dihydrogen phosphate monomer (10-MDP) in its composition, which provides acidity to the adhesive and, consequently, the capability to etch the dentin surface.[17,18] Although the presence of this acidic monomer allows for the self-etch ability of the adhesive, the manufacturer claims that the Scotchbond Universal can be applied on dentin after 35% phosphoric acid etching. Therefore, the application of this adhesive system in the etch-and-rinse or self-etch mode depends on the professional's choice. According to a recent meta-analysis, Scotchbond Universal has demonstrated no significant difference in bond strength to dentin when used in a different mode of etching.[19]

Another important factor related to 10-MDP contained in the Scotchbond Universal is that this monomer has the ability to bond chemically to the hydroxyapatite present in dentin and enamel.[17,20,21] In the self-etch mode, the residual hydroxyapatite that remains around the collagen fibrils interacts with the 10-MDP monomer, improving the bond.[20] In addition, the bond of 10-MDP to calcium creates a salt (MDP-Ca) that protects against hydrolysis[20,22] because it is a hydrolytically stable salt.[23] Furthermore, Scotchbond Universal contains polyalkenoic acid copolymer, which provides chemical bonding through its spontaneous bonding to hydroxyapatite.[18,21] More than 50% of the carboxyl groups in the polyalkenoic acid copolymer are capable of bonding to hydroxyapatite. Carboxylic groups replace phosphate ions on the substrate and create ionic bonds with calcium.[24] Most likely, the presence of the polyalkenoic acid copolymer has led to higher bond stability between dentin and adhesive because the mechanical cycling did not cause a change in the failure pattern. Failures remained predominant in the resinous agent-restoration interface.

Currently, there is a greater preference for the application of self-etch adhesive systems on dentin due to the less deep dentin demineralization compared to 35% phosphoric acid.[25,26] Furthermore, there is no need to remove the excess moisture after rinsing the phosphoric acid. This clinical procedure is considered one of the most critical steps when applying the etch-and-rinse adhesives.[27] However, several morphological differences can be observed in the bond interface between the etch-and-rinse and self-etch techniques of Scotchbond Universal.[18] In the etch-and-rinse adhesives, the phosphoric acid at 35% is applied in dentin for 15 s, causing the removal of the smear layer and smear plugs and the opening of the dentin tubules, along with the demineralization of intertubular and peritubular dentin to a depth which can vary by approximately 5 μm.[12] Next, the adhesive contained in a single bottle is applied and polymerized with formation of a thick hybrid layer[18] with long resin tags.[12,28] In the self-etch adhesives, during the demineralization caused by the acidic monomer, the other components of the adhesive are infiltrated in the demineralized dentin. The residual hydroxyapatite crystals and the dissolved smear layer are embedded in the hybridized zone.[25] Scotchbond Universal contains the monomer 10-MDP and has moderate acidity, as the pH is 2.7. This pH causes partial demineralization of dentin and hybrid layer formation with a thickness of <1 μm.[12,19] Thus, the self-etch adhesive systems allow reduction in post-operative sensitivity.[19,29]

In this study, there was a predominance of failures in the resinous agent-restoration interface, showing that this was the weakest interface. One possible explanation for this finding is that the Lava Ultimate is a composite resin with a high polymerization rate. As a consequence, there are few monomers in the material for chemical interaction between the resinous agent and internal surface of the restoration.[30] Thus, the bond of the resinous agent with the surface of the restoration should occur primarily by micromechanical retention obtained through sandblasting with aluminum oxide.[4,6,15,31]

The application of silane also favors the chemical bond of the resinous agent to the exposed fillers of composite resin,[7,32] as well as an increase in surface energy, allowing a greater wettability of the resinous agent on the surface of the restoration.[10] According to the manufacturer, Scotchbond Universal has silane in its composition, which would eliminate the isolated application of the silane on the surface of ceramic and polymeric materials. Therefore, in this study, half of the groups received the application of one coat of Scotchbond Universal on the internal surface of the restoration. The only difference between Groups 1 and 5, Groups 2 and 6,

Groups 3 and 7, and Groups 4 and 8 was the application of the adhesive on the internal surface of the restoration. There was significant difference in bond strength values only between Groups 3 and 7 because the application of the adhesive in the restoration (Group 7) provided higher bond strength. For the other groups, the adhesive application did not influence the bond strength. The adhesive application on the internal surface of the restorations is a controversial topic in the literature. Some studies show higher bond strength between the restoration and the resin cement with adhesive application,[4] while others show that the adhesive application is not so important.[6,7,16] The results of the present study suggest that the adhesive application in the internal surface of the restoration is not an important factor to ensure or to obtain higher bond strength between the restoration and resinous agent.

Cyclic loading is an *in vitro* methodology that tries to simulate the masticatory loads applied on the restorations. In this study, the onlays luted on the tooth were submitted to cyclic loading at 100 N using 1,000,000 cycles, simulating 4 years in function.[33-35] For the four groups, the cyclic loading did not cause failure, fractures, chips or cracks on the restoration.

Comparing solely the groups in which the only difference was the application of cyclic loading (Groups 1 and 2, Groups 3 and 4, Groups 5 and 6, and Groups 7 and 8), the bond strength values were not significantly different from each other. The main difference was a change in failure pattern between the Groups 5 and 6. The failures were predominantly in the resinous agent-restoration interface without cyclic loading (Group 5), changing to a distribution of resinous agent-restoration and dentin-resinous agent interfaces, and cohesive failures in resin cement with cyclic loading (Group 6).

Regardless of the distribution of failures, bond strength values did not change significantly. The cyclic loading was not significant for the bond strength values and did not change the failure pattern significantly, i.e., failures continued to be predominantly in the resinous agent-restoration interface. The cyclic loading was conducted with samples immersed in water at 37°C. Marchesi *et al*. found signs of degradation after 12 months of storage in water when the universal adhesives were applied using both the etch-and-rinse and self-etch techniques.[29] In this study, the onlay preparations had the presence of surrounding enamel, the absence of intrapulpal pressure and the inclusion of the roots in acrylic resin. These factors probably favored the protection of the central area of the onlay preparation (the place where the specimens for the microtensile test were obtained) for the accelerated hydrolysis that may occur when samples are submitted to cyclic loading.[14,18]

In June 2015, the manufacturer announced a recall of Lava Ultimate. This recall aimed to change the indications of this material because it was found that approximately 10% of customers reported that the crowns made with Lava Ultimate decemented at a higher rate than expected for this procedure. Due to this finding, the manufacturer contraindicated Lava Ultimate for crowns including crowns on implants. However, the manufacturer did not contraindicate Lava Ultimate for veneers, inlays and onlays. Therefore, the failures observed clinically demonstrate a problem in adhesively luting the Lava Ultimate restorations, which requires further laboratory and clinical studies regarding the bond capacity of this composite resin to resinous agents.

Conclusions

According to the results of this study, it was concluded that:

- The resinous agent-restoration interface corresponds to the weakest interface in Lava ultimate onlays luted with the Scotchbond Universal adhesive system and RelyX Ultimate resin cement.
- The application of Scotchbond Universal on the internal surface of the onlay was not a relevant procedure.
- The 1,000,000 load cycles at 100 N were not a relevant factor in the modification of the bond strength values and the failure patterns.

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