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Laboratory Research

Effect of Different Adhesive Strategies and Time on Microtensile Bond Strength of a CAD/CAM Composite to Dentin

EM Meda • RN Rached • SA Ignácio • IA Fornazari • EM Souza

Clinical Relevance

The use of a dual-cure activator is recommended when a universal adhesive system in self-etching mode is associated with an amine-based resin cement.

SUMMARY

Purpose: The aim of this study was to evaluate the effect of adhesive strategy and time on the microtensile bond strength of a computer-aided design/computer-aided manufacturing (CAD/CAM) composite to dentin.

Methods and Materials: Sixty CAD/CAM composite blocks were bonded to human dentin with simplified bonding agents using etch-and-rinse and self-etching approaches and amine-based and amine-free resin cements, with and without the application of a dual-cure activator (DCA; n=10): SBP-ARC (Adper Single Bond Plus + RelyX ARC), SBP-RXU (Adper Single Bond Plus + RelyX Ultimate), SBP-DCA-RXU (Adper Single Bond Plus + DCA + RelyX Ultimate), SBU-ARC (Scotchbond Universal + RelyX ARC), SBU-RXU (Scotchbond Universal + RelyX Ultimate), and SBU-DCA-ARC (Scotchbond Universal + DCA + RelyX ARC). Each specimen was light cured for 40 seconds under load and stored in distilled water at 37°C for seven days. Stick-shaped specimens (1.0 mm^2) were obtained. Half of the specimens underwent microtensile bond strength testing, and the other half were subjected to the same tests after six months of storage. Failure mode was determined using an optical microscope (40×). The data were analyzed by a two-way analysis of variance followed by the Games-Howell test and Student *t*-test (preset alpha of 0.05).

Results: After seven days, SBU-RXU presented the highest mean bond strength, statistically

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different from only SBU-ARC ($p<0.05$). Most of the groups exhibited a statistically significant reduction in bond strength after 6 months ($p<0.05$), except SBP-RXU and SBU-ARC ($p>0.05$).

Conclusion: The adhesive strategy, with different associations between adhesive systems and resin cements, as well as the use of a DCA, affected the bond strength of both amine-free and amine-based resin cements to a CAD/CAM composite.

INTRODUCTION

Adhesive cementation is a technique-sensitive, complex procedure involving the use of a bonding agent in conjunction with a resin cement that requires various clinical steps to ensure adequate bond strength to dental structures.[1] These materials must not only adhere to dental structures but also have specific mechanical (flexural and compressive strength) and optical properties (shade and opacity), as well good handling characteristics and clinically acceptable working time.[2]

The light intensity from the curing device may be attenuated or totally blocked, depending on the thickness, color, and opacity of the indirect restorative material.[3-5] Furthermore, polymerization activation depends on the wavelength and intensity of the light that reaches the material.[6,7] Hence, use of dual-cure adhesive systems in conjunction with dual-cure resin cements may be necessary for cementation of indirect restorations in areas where it is difficult or impossible for light to penetrate.[8]

In dual-cure systems, polymerization is initiated partly by the formation of free radicals produced by the chemical reaction between benzoyl peroxide and the tertiary amine and partly by light-curing activation, which depends on photons to excite the photoinitiator.[9] Although resin-based materials also contain tertiary aliphatic amines in the initiator system to ensure that free radicals are formed over a more extended period, these amines are not inactivated by acid monomers in simplified adhesive systems.[10,11] Self-cure and dual-cure resin-based materials that contain basic amines are incompatible with the high concentration of acidic monomers in simplified self-etching approach.[12] The interaction between the monomers and tertiary amine results in the latter being consumed, reducing the availability of free radicals for the polymerization reaction.[13] It has also been reported that single-step self-etch adhesive systems act as permeable membranes that allow water to diffuse through the interface, one of the leading causes of premature failure of the resin cement/dentin bond.[12,14] To avoid this problem, some self-etching adhesive systems contain dual-cure activators in their composition or as separate solutions to be mixed with the bonding agent before it is applied in cementation procedures.

With the increasing demand for a simpler and more versatile adhesive, a new type of adhesive system has emerged on the dental market that allows the clinician to choose the adhesive strategy and the number of steps used to treat the dental substrates. These so-called "universal" or "multi-mode" adhesive systems can be used in a conventional approach called "total etch" or "etch-and-rinse," in which both enamel and dentin are previously acid etched; in a "selective etching" approach, in which only the enamel is acid etched but the dentin is etched by the acidic monomers in the adhesive system; and in a "self-etch" approach, in which the acidic monomers etch and prime both enamel and dentin at the same time.[15,16] In addition to dimethacrylate monomers and acidic functional monomers, universal adhesive systems usually contain solvents, filler particles, and initiators. Some commercial brands include silane, allowing them to be used in indirect restorative procedures, such as cementation of ceramics, zirconia, indirect resins, and metal restorations.[17,18]

Scotchbond Universal Adhesive must be mixed with a dual-cure activator (Scotchbond Universal DCA, Dual-Cure Activator, 3M ESPE, St Paul, MN, USA) containing sodium p-toluenesulfinate and ethanol, when an amine-based resin cement is used. However, the manufacturer does not recommend its use with RelyX Ultimate cement, because it was developed with a redox system that uses sodium persulfate and tert-butyl peroxy-3,5,5-trimethylhexanoate to suppress adverse interactions between the adhesive system and dual-cure or chemically activated resin cements. As the cement already contains an activator that copolymerizes when it comes into contact with the adhesive layer, there is no need for additional activators (information provided by the supplier, 3M ESPE).

The effectiveness of dual-cure adhesive systems to bond posts and indirect restorations to dentin has been extensively studied in the literature, but the outcomes are controversial. One study showed that the bond strength of light-cure was superior to that of dual-cure adhesive systems (when a dual-cure activator was mixed) because the concentration of photoinitiator and functional monomers is reduced,

Table 1: *Experimental Groups Used in the Study*

Group	Adhesive Strategy		
	Bonding Agent	*DCA*	*Resin Cement*
SBP-ARC	Adper Single Bond Plus	No	RelyX ARC
SBP-RXU	Adper Single Bond Plus	No	RelyX Ultimate
SBP-DCA-RXU	Adper Single Bond Plus	Yes	RelyX Ultimate
SBU - RXU	Scotchbond Universal	No	RelyX Ultimate
SBU - ARC	Scotchbond Universal	No	RelyX ARC
SBU - DCA - ARC	Scotchbond Universal	Yes	RelyX ARC

adversely affecting the degree of conversion and bond strength to dentin.[8] However, the bond strength of dual-cure adhesive systems with dual-cure activators can be more uniform in different parts of the dental structure where the light is attenuated.[19] Further studies are therefore required to clarify the impact of a dual-cure activator on the effectiveness of simplified adhesive systems used in conjunction with dual-cure resin cements.

The aim of this study was to evaluate the effect of different adhesive strategies, including the addition of a dual-cure activator, and time on the microtensile bond strength of a computer-aided design/computer-aided manufacturing (CAD/CAM) composite to dentin using simplified total-etch and self-etching adhesive systems. The hypotheses to be tested were that 1) there would be no differences in bond strength of CAD/CAM composite to dentin when different adhesive strategies were used and 2) there would be no differences in bond strength between the groups after a seven-day or a six-month water storage.

METHODS AND MATERIALS

Sixty healthy human third molars were obtained from the tooth bank after the research protocol had been approved by the local Committee for Ethics in Research (No. 759.419). The teeth had been stored in 0.5% chloramine-T at 4°C for up to six months after extraction.

Preparation of the Teeth

The occlusal third of the crowns was removed with a precision sectioning cutter (Isomet 1000, Buehler, Lake Bluff, IL, USA) and a wafering diamond blade (Extec Corp, Enfield, CT, USA) under water cooling to expose the midcoronal dentin. The surfaces of the exposed dentin were wet polished with 600-grit SiC paper for 30 seconds and rinsed under running water for 60 seconds. The specimens were then gently air dried for three seconds so that the surface was slightly shiny. The teeth were randomly allocated to six groups (n=10) according to the adhesive system/resin cement combination used (Table 1). The materials used, their composition, and the procedures for application are shown in Table 2.

Pretreatment of the Indirect Restorative Material

CAD/CAM composite blocks (Lava Ultimate A2-HT, 3M ESPE) were sectioned with a diamond blade in a precision cutter to produce 3-mm-thick specimens with a 12-mm × 12-mm cross section. The upper surface of each slice was sandblasted with 50 µm alumina particles under a pressure of 2 bar for 10 seconds and then cleaned with distilled water in the ultrasonic bath for 10 minutes. A silane coating (RelyX Ceramic Primer, 3M ESPE) was applied for 60 seconds and then air dried for 5 seconds.

Bonding Procedures

The enamel and dentin surfaces were etched with 32% phosphoric acid (Scotchbond Universal Etchant, 3M ESPE) for 30 seconds and 15 seconds, respectively. The etchant gel was rinsed with water spray for 30 seconds, and excess moisture was removed by blotting with tissue paper. The total-etch bonding agent (Adper Single Bond Plus, 3M ESPE) was applied to the etched enamel and dentin surfaces with gentle agitation for 15 seconds using a fully saturated applicator and gently air dried for five seconds to evaporate the solvent. The universal adhesive system (Scotchbond Universal Adhesive, 3M ESPE) was applied using the selective enamel etch mode that relies on separate enamel etching and dentin self-etching. A 32% phosphoric acid was applied to the enamel for 30 seconds and rinsed with water spray. Any excess moisture was removed by blotting with tissue paper, keeping the dentin moist. A single coat of the bonding agent was applied to the dentin (and enamel) by rubbing it onto the surface for 20 seconds with a fully saturated disposable

Table 2: *Description of the Materials Used in the Study With Trade Names, Manufacturer, Composition, and Application Procedures*

Material Trade Name, Manufacturer (Batch No.)	Composition[a]	Application Mode[a]
Adper Single Bond Plus, 3M ESPE (N456049)	Bis-GMA, UDMA, HEMA, copolymer of acrylic acid and itaconic acid, silanized colloidal silica particles, ethanol, water, and photoinitiator	Etch enamel and dentin with 32% phosphoric acid for 30 s and 15 s, respectively, and rinse with water. Dry by blotting, keeping dentin moist. Rub one coat of adhesive onto the dentin surface for 15 s and air-dry gently for 5 s.
Scotchbond Universal, 3M ESPE (595105)	Bis-GMA, 10-MDP, dimethacrylate resins, HEMA, copolymer of acrylic and itaconic acids, silane-treated silica, ethanol, water, initiators, and silane	Etch enamel with phosphoric acid for 30 s and rinse with water, leaving the dentin slightly moist. Rub one coat of adhesive onto the dentin surface for 20 s and air-dry gently for 5 s.
DCA, Dual-Cure Activator, 3M ESPE (509461)	Sodium p-toluenesulfinate and ethanol	Mix a drop of co-initiator with a drop of Scotchbond Universal for 5 s and apply to the surface of the tooth for 20 s by rubbing; air-dry gently for 5 s to evaporate the solvent.
RelyX Ceramic Primer, 3M ESPE (N561569)	Methacryloxypropyltrimethoxysilane, ethanol, and water	Apply to resin nanoceramic for 1 min and air-dry for 5 s.
RelyX ARC, 3M ESPE (N545532)	Bis-GMA, TEGDMA, pigments, initiators, silica, and zirconia	Mix A and B pastes in equal quantities (two clicks) with a spatula for 20 s.
RelyX Ultimate, 3M ESPE (601450)	Base paste: silane-treated glass powder; 2-propenoic acid; 2-methyl-, 1,1′-[1-(hydroxymethyl)-1,2- ethanediyl] ester; reaction products with 2-hydroxy-1,3-propanediyl dimethacrylate and phosphorous oxide; TEGDMA; silane-treated silica; oxide glass chemicals; sodium persulfate; tert-butyl peroxy-3,5,5-trimethylhexanoate; and copper (II) acetate monohydrate Catalyst paste: silane-treated glass powder; dimethacrylate; silane-treated silica; 1-benzyl-5-phenyl-barbic-acid; calcium salt; sodium p-toluenesulfinate;1,12-dodecane dimethacrylate; calcium hydroxide; 2-propenoic acid, 2-methyl-,[3-methoxypropyl)imino]di-2,1-ethanediyl ester; and titanium dioxide	Mix A and B pastes in equal quantities (two clicks) with a spatula for 20 s.
Lava Ultimate 3M ESPE (N538333)	Inorganic phase: silica and zirconia nanoparticles (approximately 80% by weight) Organic phase: UDMA and Bis-EMA (approximately 20% by weight)	Sandblast with aluminum oxide for 10 s and clean in ultrasonic bath for 10 s. Apply RelyX Ceramic Primer for 1 min and air-dry for 5 s.

Abbreviations: 10MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-EMA, bisphenol A polyethylene glycol dimethacrylate; Bis-GMA, bisphenol A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA urethane dimethacrylate.
[a] Data supplied by the manufacturer.

applicator and then gently air dried for five seconds or until the liquid no longer moved on the surface.

A single drop of the dual-cure activator was mixed during five seconds with a drop of the adhesive system for the groups SBP-DCA-RXU and SBU-DCA-ARC. The mixture was then applied to the dentin as described previously. The resin cements were handled according to the manufacturer's instructions and applied to the surface of the CAD/CAM composite blocks, which were then placed on the treated dentin under a constant seating force of 1 kg for one minute.[20,21] A light-emitting diode curing unit (Elipar FreeLight 2, 3M ESPE) with approximately 700 mW/cm^2 irradiance was activated for 40 seconds on the top and four sides of the specimen once the load had been removed, giving 200 seconds total activation time. The specimens were then stored in distilled water at 37°C for seven days.

Microtensile Bond Strength Testing

After storage, the specimens were sectioned in the x- and y-direction using a high-precision diamond saw (Extec Corp) in a precision cutter (Isomet 1000, Buehler) to obtain stick-shaped micro-specimens with a cross-sectional area of approximately 1 mm^2. Half of the micro-specimens underwent microtensile bond strength tests immediately after cutting, and the other half were stored in distilled water

Table 3: *Mean Values (±Standard Deviation) of Microtensile Bond Strength in MPa, Number of Specimens Tested (n), and Number of Pretesting Failures (ptf) in Each Group After Storage for Seven Days and Six Months*[a]

Group	7 d		6 mo	
	Mean ± SD	n/ptf	Mean ± SD	n/ptf
SBP-ARC	19.07 ± 8.25 aA	50/0	14.28 ± 6.51 aB	43/2
SBP-RXU	18.52 ± 11.06 abA	50/0	16.46 ± 8.17 aA	45/3
SBP-DCA-RXU	19.42 ± 10.37 aA	50/0	12.78 ± 7.33 aB	50/0
SBU-RXU	23.12 ± 10.94 aA	50/0	16.76 ± 7.20 aB	42/1
SBU-ARC	13.35 ± 6.32 bA	50/0	12.98 ± 8.28 aA	41/6
SBU-DCA-ARC	19.67 ± 10.19 aA	50/0	15.4 ± 8.90 aB	43/2

Abbreviations: SBP, Scotchbond Multipurpose; SBU, Scotchbond Universal; ARC, RelyX ARC; RXU, RelyX Utlimate; DCA, Dual-cure activator.
[a] *Different lowercase letters indicate significant differences in column (p<0.05). Different uppercase letters indicate significant differences in row (p<0.05).*

at 37°C and tested after six months. The thickness of the adhesive interface was measured with a digital caliper (Absolute Digimatic Caliper, Mitutoyo Corp, Kawasaki, Japan) to calculate individual areas of the specimens' interface. The specimens were fixed in a microtensile jig with a cyanoacrylate gel (Loctite 454 Gel, Henkel North America, Rocky Hill, CT, USA). Microtensile bond strength testing was performed in a universal test machine (Instron DL2000, Grove City, PA, USA) with a crosshead speed of 0.5 mm/min. The results in kgf were converted to MPa based on the cross-sectional area of each specimen.

Failure Mode Analysis

The failure mode for each specimen was determined using an optical microscope at 40× magnification (BX60, Olympus Corp, Tokyo, Japan) and classified according to the structures involved as follows: cohesive failure in the dentin, cohesive failure in the CAD/CAM composite, cohesive failure in the cement, adhesive failure between the dentin and cement, adhesive failure between the cement and the CAD/CAM composite, and mixed failure (two or more structures involved).

Scanning Electron Microscopy

The most representative failures of each group were selected for analysis using scanning electron microscopy (SEM). The fractured specimens were cleaned in an ultrasonic bath with distilled water for 15 minutes and kept in a vacuum desiccator with silica for seven days. They were then coated with Au-Pd alloy and examined under SEM at 300× and 1500× magnification (Vega 3, Tescan Orsay Holding, Brno, Czech Republic).

Statistical Analysis

Each tooth was considered a sampling unit, and the mean values for specimens from the same tooth were used to calculate the mean microtensile bond strength for the group in both storage times. The data were analyzed for normality with the Kolmogorov-Smirnov test and homogeneity of variance with Levene's test. Two-way analysis of variance ("adhesive strategy" and "storage time") was used followed by the Games-Howell post hoc test and for each possible comparison. Student *t*-test was performed to compare differences between group means at seven-day and six-month storage times. A significance level of 5% was used for all the tests. The data were analyzed in SPSS 24.0 (IBM Software, New York, NY, USA).

RESULTS

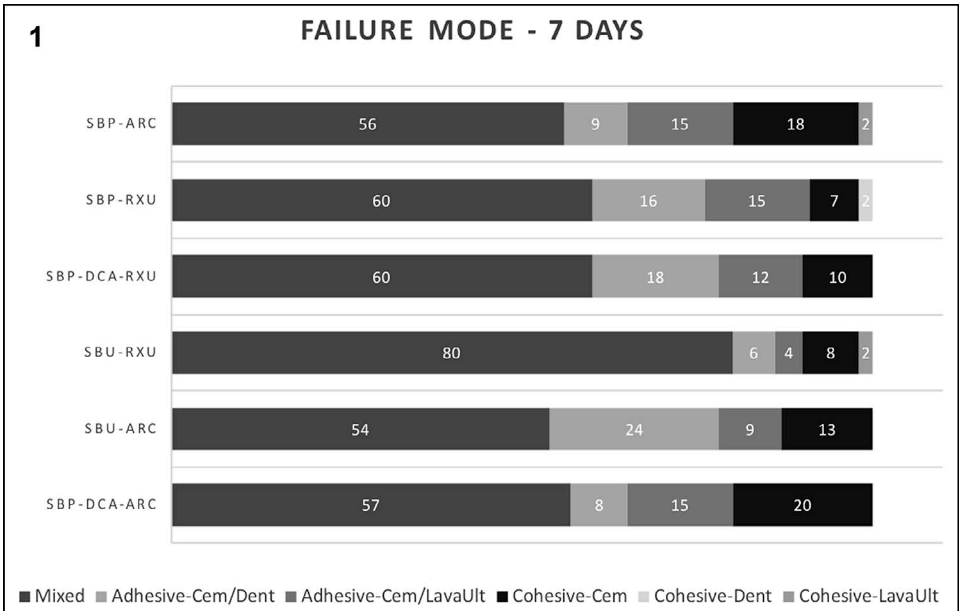
Statistically significant differences were observed for the factors "adhesive strategy" and "storage time" ($p<0.05$), but no significant interaction was found between the factors ($p= 0.096$).

The mean microtensile bond strength, number of specimens tested, and number of pretest failures for each group are shown in Table 3.

After seven days, the lowest bond strength was observed for the SBU-ARC group, which did not differ statistically from SBP-RXU ($p>0.05$). After six months, there was no statistically significant difference in bond strength between any of the groups ($p>0.05$). Most of the groups exhibited a statistically significant reduction in mean bond strength between the seven-day and six-month assessments ($p<0.05$), except SBP-RXU and SBU-ARC ($p>0.05$).

Although there were no pretest failures after seven days, after six months all the groups except SBP-DCA-RXU exhibited this type of failure. When a pretest failure occurred, a value of zero was assigned to the specimen when the mean for the tooth in question was calculated.

The frequency distributions of the failure modes for each group expressed as a percentage of the total number of specimens in the group after seven days



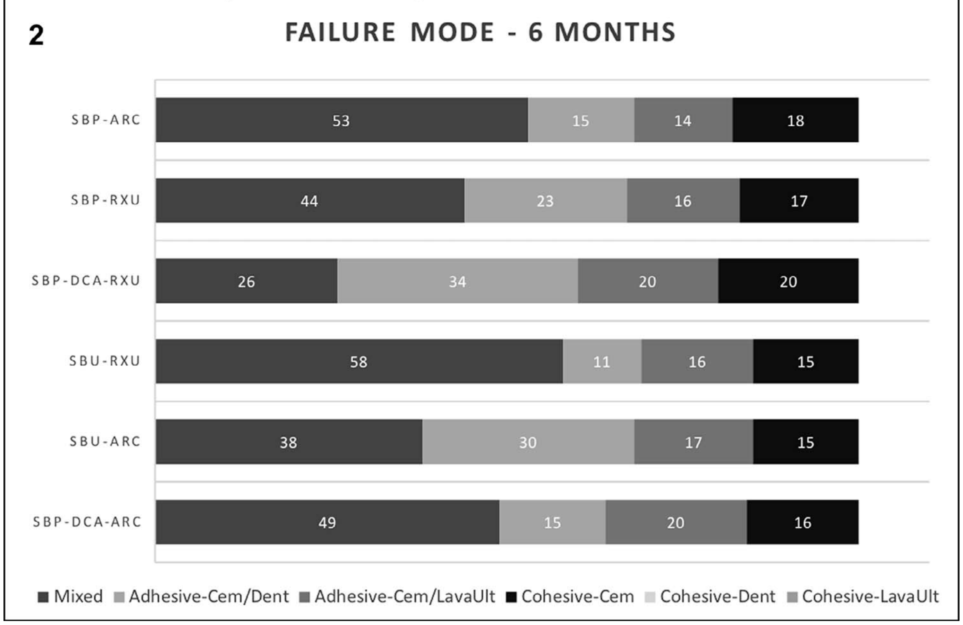


Figure 1. *Failure mode distribution (%) for the groups after seven days.*

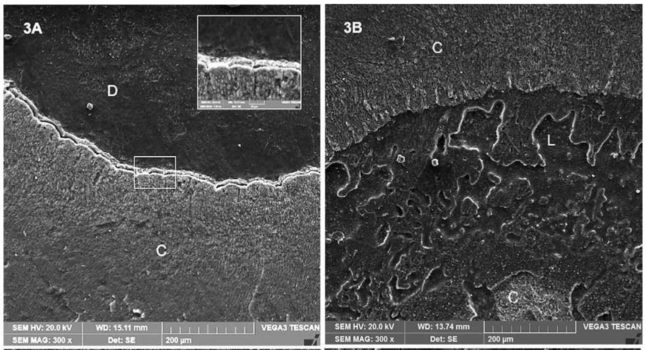
Figure 2. *Failure mode distribution (%) for the groups after six months.*

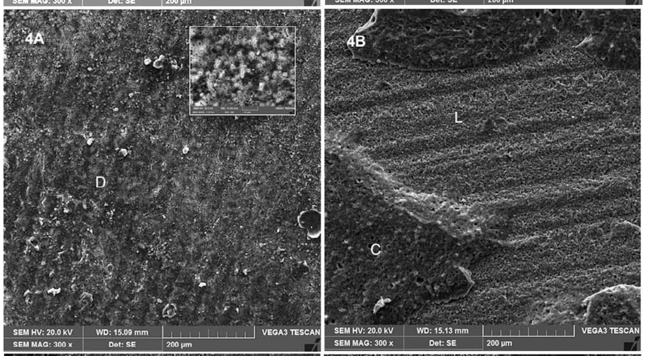
and six months are shown in Figures 1 and 2. The most common type of failure in all the groups at both time points was the mixed failure when more than two structures were involved. Adhesive failures between the cement and dentin and cohesive failures in the cement were more common after six-month water storage. Figures 3 to 8 demonstrate the most frequently type of failures found in the tested groups with detailed images of the adhesive interfaces.

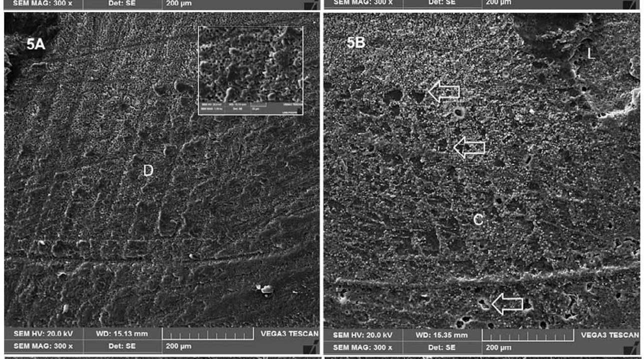
DISCUSSION

This study aimed to assess the effectiveness of a dual-cure activator used with a conventional total-etch or a universal adhesive system with self-etching approach and two different resin cements, one of which was based on a new redox system and amine free. An unbalanced factorial model was used for the analysis. In two groups (SBP-ARC, SBU-RXU), the manufacturer's standard recommendations were followed, and in the remaining groups, the adhesive system and resin cement were used with and without a dual-cure activator. The classic combinations were not subjected to the use of dual-cure activator because there is no recommendation for this additional step.

Both study hypotheses were rejected as there were differences in bond strength to dentin between the







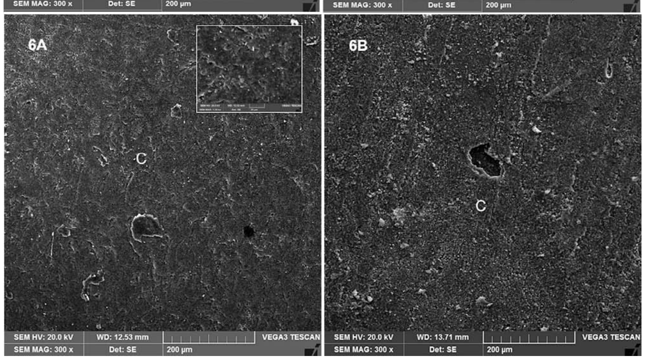


Figure 3. *(A*): Specimen from group SBP-ARC showing mixed failure with dentin (D) and resin cement (C) exposure after seven days. (B): Counterpart of the same specimen showing resin cement (C) remaining on the CAD/CAM composite surface (L).*

Figure 4. *(A*): Specimen from group SBP-RXU showing mixed failure with dentin (D) exposure after seven days. (B): Counterpart of the same specimen showing resin cement (C) remaining on the CAD/CAM composite surface (L).*

Figure 5. *(A*): Specimen from group SBP-DCA-RXU showing adhesive failure with dentin (D) exposure after six months. (B): Counterpart of the same specimen showing voids and porosities (arrows) in the resin cement (C) and areas of CAD/CAM composite exposure (L).*

Figure 6. *(A*): Specimen from group SBU-RXU with a cohesive failure in the resin cement layer (C) after six months. (B): Counterpart of the same specimen showing the resin cement (C) covering the CAD/CAM composite surface.*

** Inset is 1500x.*

groups after seven days, and in one group, the mean bond strength after six months of storage in water was significantly lower than that after seven days.

Bonding using a self-etching technique is based on two mechanisms: micromechanical interlocking (monomer penetration in interfibrillar spaces) and chemical interaction between acidic functional monomers and hydroxyapatite.[22] With this mode, the bonding procedure is faster and less critical because it does not involve moisture control, unlike techniques that require the etching to be performed beforehand.[23] Scotchbond Universal Adhesive contains the functional monomer 10-MDP, which promotes chemical interaction with the calcium in the dental structure,[24,25] making bonding more hydrolytic stable.[26,27] Recent studies have shown that this adhesive provides higher bond strength when used in self-etch mode on dentin.[13,28-30]

In the present study, the bond strength of the self-etch universal adhesive system used with the amine-free dual cement (SBU-RXU) was not statistically different from that obtained with the conventional adhesive system used in total-etch mode with the amine-containing resin cement (SBP-ARC). Both groups represented classic approaches recommended by the manufacturer. However, when the materials were switched (SBU-ARC and SBP-RXU), without the use of a dual-cure activator, the bond strength was reduced. The lower mean bond strength observed for the SBU-ARC group after seven days may be a result of the interaction between the acidic monomers in the self-etch mode adhesive system and the tertiary amine in the conventional resin cement.[12,31] This interaction may lead to amine consumption reducing the extent and rate of polymerization and increasing premature bonding failures to dentin, as reported in previous studies.[14,32]

Although the recommended combinations of adhesive systems and resin cements tested in groups SBP-ARC and SBU-RXU were effective after seven days, the bond strength was reduced after six months of water storage. This finding may be a result of increased water sorption and the following

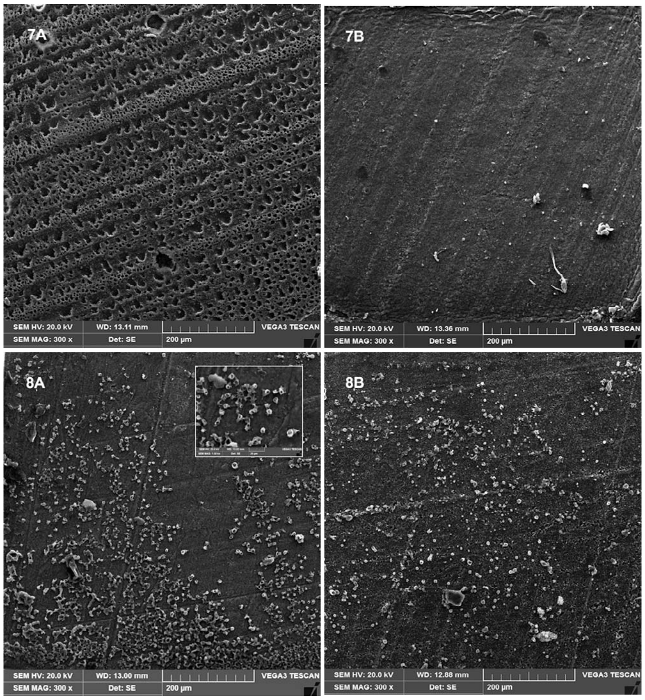


Figure 7. *(A): Specimen from group SBU-ARC showing adhesive failure with voids and porosities in the adhesive interface after seven days. (B): Counterpart of the same specimen with the CAD/CAM composite surface free of resin cement.*

Figure 8. *(A*): Specimen from group SBU-DCA-ARC with adhesive failure showing voids on the dentin surface (D) after seven days. (B): Counterpart of the same specimen showing the CAD/CAM composite surface with remnants of resin cement.*

** Inset is 1500x.*

solubilization of resin monomers causing breakdown of the polymer chains.[33,34] This failure could also occur because sometimes adhesive systems do not fully infiltrate the exposed collagen mesh and the solvent (in this case ethanol) fails to volatilize completely, adversely affecting dentin bond strength.[35]

In the present study, all of the groups with the amine-free resin cement had similar results independently of the adhesive system and whether or not a dual-cure activator was used. The association between Scotchbond Universal Adhesive in the self-etch mode and RelyX Ultimate (SBU-RXU) achieved the highest short-term bond strength. This association combines the low technique sensitivity of the bonding approach with the convenience of dispensing the use of a dual-cure activator to improve polymerization. When the activator inside RelyX Ultimate comes into contact with the Scotchbond Universal adhesive layer, it ensures adequate polymerization of the adhesive, even without light. Recent studies have shown the superiority of this combination over other resin cement systems.[20,21,36-40]

The effect of dual-cure activators on polymerization of dual-cure resin cements has been the subject of much research in the past decade.[3,11,13,19,41-45] Some studies[13,42,46,47] have shown that dual-cure activators have a limited effect on the degree of conversion of chemically activated adhesive systems and argue that the effectiveness of polymerization in dual-cure systems is highly dependent on the adhesive system used.[42,43] Moreover, the higher acidic monomer content in the partially polymerized adhesive layer can interfere with the amine in the chemically cure systems, potentially resulting in less amine being available for the polymerization process even when dual-cure activators are used.[42]

Scotchbond Universal is considered a mild self-etch adhesive with a pH of 2.7,[25,48] which reaches 2.9 after the dual-cure activator is added.[49] According to the manufacturer, the dual-cure activator (pH=7) was developed to optimize copolymerization with self-cure and dual-cure resin cements, other than RelyX Ultimate. Indeed, the results of this study show a significant increase in bond strength when the dual-cure activator was used with the universal adhesive and the amine-containing resin cement compared with that of the same combination but without the dual-cure activator (SBU-ARC). The group SBP-DCA-RXU was tested to evaluate whether the dual-cure activator would improve the performance of the two-step total-etch adhesive with the amine-free resin cement. However, although the results for this group were similar to those for the group with the same adhesive and resin cement without the dual-cure activator (SBP-RXU) after seven days, the results for that group were significantly worse after six months. This was confirmed by the SEM analysis, having disclosed porosities in the cement surface (Figure 5B) probably as a result of an increase in the amount of residual solvent at the interface, which can dilute the functional monomers and affect the long-term bonding performance.[34]

In the present study, although the CAD/CAM composite blocks with a mean thickness of 3 mm undoubtedly attenuated the light, this attenuation was probably made up for by the longer light-curing time (200 seconds) and the five different positions in which the light-curing unit was held for each specimen. Also, polymerization of the adhesive system and the resin cement was carried out simultaneously to avoid problems with poor seating of indirect restorations due to a thicker adhesive film.[50,51] Although recent studies have shown that light-curing of the adhesive system before application of the resin cement can be decisive in determining the dentin bond strength of indirect restorations,[20,21] it should be pointed out that this procedure can be used only when the adhesive is very thin, so the fit of the restoration is not affected.

Observation of the failure modes after the microtensile bond strength tests revealed a large number of mixed failures in all the tested groups. After six months of water storage, there was an increase in the number of adhesive failures possibly as a result of degradation of the exposed adhesive interface by water inflow, leading to hydrolysis of collagen fibers. The observation of voids and porosities (Figures 7A, 8A) appeared to indicate that water reached the interface and inhibited the polymerization of resin components, affecting the bond strength.[33,34,52]

Adequate selection of materials in adhesive cementation is fundamental to ensure clinical success of indirect restorations and should be based on their chemical composition and compatibility, as well as on their mechanical and optical characteristics. The manufacturers' recommendations should be followed strictly by the clinician since some associations between adhesive systems and resin cements are not compatible and could jeopardize the effectiveness of the cementation procedure.

CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that the adhesive strategy, with different associations between adhesive systems and resin cements, as well as the use of a dual-cure activator, affected the bond strength of both amine-free and amine-based resin cements to a CAD/CAM composite.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the Research Ethical Committee of PUCPR. The approval code for this study is 759.419.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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