Dual-Cured Adhesive System Improves Adhesive Properties of Dentin Cavities Restored with a Bulk-Fill Resin Composite

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Aim: To evaluate the impact of a dual-cured adhesive system on the in situ degree of conversion (DC), bond strength (BS) and failure mode (FM) of adhesive interfaces in dentin cavities restored with a bulk-fill resin composite.

Methods: 4-mm-deep dentin cavities with a 3.1 C-factor were created in 68 bovine incisors (n = 17 per group). The light-cured (Scotchbond™ Universal) or the dual-cured (Adper™ Scotchbond™ Multi-purpose Plus) adhesive system was applied to the cavities, which were then restored with a bulk-fill resin composite (Filtek™ Bulk Fill). In situ DC analysis was performed by means of micro Raman spectroscopy at the top and bottom interfaces. Push-out BS was measured in a universal testing machine after 24-h or 6-month water storage. FM was determined with a stereomicroscope. Data of in situ DC and BS were analyzed by two-way analysis of variance (ANOVA) and Tukey test (p<0.05), while the FM was analyzed descriptively. Results: The groups that received the dual-cured adhesive system showed statistically higher in situ DC and BS than those that received the light-cured adhesive system. Cohesive failure mode was the most frequent in all conditions. Conclusion: In situ DC and BS were influenced by the curing strategies of the adhesive systems with better performance of the dual-cured material.

Keywords: Aging. Composite resins. Polymerization. Dentin-bonding agents. Physical Properties.
Introduction

The proper cure of the adhesive system is one of the factors needed to achieve the required mechanical properties\(^1\) and longevity\(^2,3\) of dental restorations. Inappropriate monomer conversion and the consequent residual monomers in the hybrid layer increase the local permeability and water sorption\(^2,4\), weakening the hybrid layer structure, and impairing the adhesive capacity\(^4\). Therefore, a higher degree of conversion of the adhesive system, would provide longer-lasting adhesive integrity to dental restorations.

The dual-curing strategy can promote the material self-cure when a proper light activation might be compromised. Therefore, its use is traditionally recommended in indirect restorations or fiber post bonding in the root canal\(^5\). The benzoyl peroxide and tertiary amine in the catalyst allow the cure reaction without light energy\(^6\), which can increase the degree of conversion and, consequently, promote long-lasting bonds.

Areas of the cavity preparation for direct resin composite restorations that are too far from the curing light tip may receive insufficient energy, negatively affecting the degree of conversion and bond stability\(^7\). To overcome this inconvenience, the use of dual-cured adhesive systems has been suggested in 2-mm-deep cavities resulting in greater dentin bonding durability\(^8\), probably due to their higher degree of conversion in the hybrid layer and faster cure. However, when such tooth preparations occur in posterior teeth, the tip of the curing light can be set even further than 2 mm in certain dentin sites, negatively affecting interfacial properties in such areas. Thus, regular viscosity bulk-fill resin composites, which allow light-curing single increments of up to 4 to 5 mm thickness\(^9\), could be used to fill deep preparations with a single resin composite increment, saving chair time. However, it is not well known if using a dual-cured adhesive system in 4-mm-depth would benefit the adhesive conversion and dentin bonding durability.

Thereby, the aim of this study was to evaluate the \textit{in situ} degree of conversion (DC) of light-cured and dual-cured adhesive systems and their impact on bond strength (BS) and failure modes (FM) in 4-mm-deep dentin cavities restored with a regular viscosity bulk-fill resin composite. The null hypothesis tested was that different curing strategies of adhesive systems and storage times would not affect the physical properties of the adhesive interface.

Materials and Methods

Experimental Design and Specimen Preparation

This \textit{in vitro} study involved the following response variables: \textit{in situ} DC, BS and FM. For \textit{in situ} DC (n=17), the factors studied were two adhesive systems with different curing strategies (light-cured: Scotchbond\textsuperscript{™} Universal, 3M ESPE, St. Paul, USA; and dual-cured: Adper\textsuperscript{™} Scotchbond\textsuperscript{™} Multi-purpose Plus, 3M ESPE, St. Paul, USA), and the regions of the specimen (top and bottom). For BS and FM (n=17), the factors studied were the two adhesive systems with different curing strategies and the time of water storage (24 h and 6 months aging). Materials used in this study are described in Table 1.
Table 1. Materials used in this study.

| Material                                      | Chemical composition (wt%)                                                                 | Lot number       |
|-----------------------------------------------|------------------------------------------------------------------------------------------|------------------|
| Filtek™ Bulk Fill (3M ESPE, St. Paul, MN, USA) | Silane treated ceramic (60 – 70)                                                          | 1717800606       |
|                                               | Aromatic urethane dimethacrylate (10 – 20)                                               |                  |
|                                               | Diurethane dimethacrylate (UDMA) (1 – 10)                                                 |                  |
|                                               | Ytterbium fluoride (YbF3) (1 – 10)                                                        |                  |
|                                               | Silane treated silica (1 – 10)                                                            |                  |
|                                               | 1,12-dodecane dimethacrylate (DDDMA) (<10)                                               |                  |
|                                               | Silane treated zirconia (<5)                                                              |                  |
|                                               | Water (<5)                                                                                |                  |
| Scotchbond™ Universal (3M ESPE, St. Paul, MN, USA) | 2-hydroxyethyl methacrylate (15 – 25)                                                     | 1718500452       |
|                                               | Bisphenol A diglycidyl ether dimethacrylate (BisGMA) (15 – 25)                            |                  |
|                                               | 2-propenoic acid, 2-methyl, reaction products with 1,10-decanediol and phosphorous oxide (P2OS) (10 – 20) |                  |
|                                               | Ethanol (10 – 15)                                                                         |                  |
|                                               | Water (10 – 15)                                                                           |                  |
|                                               | 2-propenoic acid, 2-methyl, 3-(trimethoxysilyl)propyl ester, reaction products with vitreous silica (7 – 13) |                  |
|                                               | Copolymer of acrylic and itaconic acid (1 – 5)                                            |                  |
|                                               | Camphorquinone (<2)                                                                       |                  |
|                                               | Dimethylaminobenzoato(4) (<2)                                                             |                  |
|                                               | (Dimethylamino)ethyl methacrylate (<1)                                                    |                  |
| Adper™ Scotchbond™ Multi-purpose Plus (3M ESPE, St. Paul, MN, USA) | Primer: Water (40 – 50)                                                                  | 1723700298       |
|                                               | 2-hydroxyethyl methacrylate (HEMA) (35 – 45)                                              |                  |
|                                               | Copolymer of acrylic and itaconic acids (10 – 20)                                         |                  |
|                                               | Activator: Ethyl alcohol (>95)                                                            | 1728600477       |
|                                               | Sodium benzenesulfinate (<5)                                                              |                  |
|                                               | Catalyst: Bisphenol A diglycidyl ether dimethacrylate (BisGMA) (60 – 70)                  | 1727600198       |
|                                               | 2-hydroxyethyl methacrylate (HEMA) (30 – 40)                                             |                  |
|                                               | Benzoyl peroxide (<2.5)                                                                   |                  |
|                                               | Triphenylantimony (<0.5)                                                                  |                  |
|                                               | Triphenylphosphine (<0.5)                                                                  |                  |
|                                               | Hydroquinone (<0.05)                                                                      |                  |

Source: Safety Data Sheet (SDS)

Figure 1 shows a schematic representation of the specimens’ preparation and analyses performed. Sixty-eight bovine incisors without enamel cracks or structural defects were selected for dental preparations according to a previously described method. Teeth were decontaminated in a water solution of thymol (0.1%) at 4°C for a week, and then the roots were removed at the cementoenamel junction (CEJ) with a diamond saw using a precision cutting machine (Isomet 1000; Buehler, Lake Forest, IL, USA) under irrigation. Such surface will be the future top of the sample. A parallel cut was made 4 mm from the CEJ, surface that will be the bottom of the sample, resulting in a 4-mm-high specimen. The top and bottom surfaces of the specimens were sanded with 400 and 600 grits sandpapers (Labopol-21, Struers, Copenhagen, Denmark). The central void of the specimen was prepared with Maxicut burs (Komet Inc, Lemgo, Germany) mounted in a handpiece under air-water cooling, resulting in standardized conical cavities (4.8 x 2.8 x 4 mm) with a 3.1 C-factor.
Curing strategies were as follow: Light-cured: No acid etching was done. The adhesive was applied and rubbed for 20 s, followed by gentle air drying for approximately 5 s to evaporate the solvent, and light cured for 10 s (Coltolux LED, Coltene/Whaledent, Altstätten, Switzerland - 1200 mW/cm²). Dual-cured: The surface was etched with 37% phosphoric acid for 15 s and rinsed for 30 s. Dentin was maintained moist after water excess removal with absorbent paper. The activator was applied and air-dried gently for 5 s. Then, the primer was applied and air dried gently for 5 s, leaving the surface with a shiny aspect. A coat of the catalyst was applied and light-cured for 10 s.
The adhesive system was applied according to the manufacturers’ instructions. The bond component was not applied because it is part of the Adper Scotchbond Multi-purpose Plus catalyst formulation.

After adhesive procedures, the teeth were set on a glass slab and filled with a single increment of Filtek™ Bulk Fill (3M ESPE, St. Paul, MN, USA) resin composite. A glass slide was pressed onto the specimen in order to standardize the smooth surface and the distance of 1.0 mm between the light source and the resin composite during photoactivation. The resin composite was cured for 20 s (Coltolux LED - 1200 mW/cm²). All the adhesive, restorative and curing procedures were performed by a single operator. Samples were polished with 400, 500, and 800 grit sandpapers and cleaned in an ultrasonic bath for 20 min. Half of samples were stored in distilled water at 37 ºC for 24 hours. Another thirty four samples were stored at the same conditions for aging in distilled water changed weekly for 6 months.

“In Situ” Degree of Conversion (DC)

The degree of conversion (DC) of each material tested in this study was evaluated through micro-Raman spectroscopy (Xplora micro-Raman, Horiba, Paris, France). Spectroscope calibration was done using a Silicon sample. Raman spectra were collected using in the range between 1590 and 1670 cm⁻¹ using the 638 nm laser emission wavelength with 10 s acquisition time and 3 accumulations. The diameter of the laser beam used over the specimen was 1 µm and the analysis was performed with a 100x magnification lens (Olympus UK, London, UK). The spectrum was obtained in the middle of hybrid layer. Six random spots of each sample were analyzed (three at the top and three at the bottom). A non-cured amount of each adhesive system was used as reference. Processing was performed with the Opus Spectroscopy Software version 6.5 (Bruker Optik GmbH, Ettlingen, Baden-Wurttemberg, Germany).

The % DC was calculated using the monomers to polymer double bonds ratio in the adhesive, according the formula (I) below, in which “R” is the ratio between aliphatic and aromatic bond peaks at 1635 and 1605 cm⁻¹ in the adhesive layer, as performed in a previous study12.

\[
\text{DC} (\%) = 100 \times (1 - \frac{R_{\text{cured}}}{R_{\text{uncured}}})
\]

Bond Strength (BS) Evaluation

BS was evaluated by the push-out method in a universal testing machine (EMIC DL 2000, São José dos Pinhais, PR, Brazil) as previously reported10,11. An acrylic device with a central hole was adapted on the machine base where specimens were placed with its larger surface facing down. The smaller surface was pushed by a cylindrical plunger (1-mm diameter) with a compressive force (0.5 mm/min) until failure. Data were obtained in N and transformed in MPa using the following equation (II), in which “N” is the bond strength in Newton, “R” is the larger surface radius, and “r” is the smaller surface radius and “h” is the sample height10,11.

\[
\text{MPa} = \frac{N}{\pi (R + r)} \left(\frac{h^2 + (R - r)^2}{2}\right)
\]
Failure Mode (FM) Analysis

After BS test, the fractured specimens were examined on the surface of the dentin and on surface of resin composite using a stereomicroscope (Stereo Zoom, Bausch & Lomb, New York, NY, USA) at 30x magnification. The failure modes were categorized into cohesive (dentin or resin composite failure), adhesive (dentin/resin composite interface failure), or mixed (cohesive failure in resin composite and adhesive in interface), as previously described13.

Statistical Analysis

Data normality and homoscedasticity were analyzed through, respectively, D’Agostino & Pearson test (p>0.05) and Bartlett’s test in GraphPad Prism 7 software (San Diego, CA, USA). In situ DC and BS data were analyzed using two-way ANOVA and Tukey’s post hoc tests (p<0.05) in GraphPad Prism 7 software. Failure modes patterns were descriptively analyzed.

Results

“In Situ” Degree of Conversion

Significant differences in DC were found between curing strategies (p<0.01). Multiple comparisons are shown in Table 2. Top and bottom interfaces showed similar in situ DC, while the dual-cured adhesive system showed higher DC than light-cured adhesive system in both regions.

Table 2. Means (standard deviations) of in situ DC (%) according to the curing strategy of the adhesive system and specimen region.

| Region | Curing strategy | Light-cured | Dual-cured |
|--------|----------------|-------------|------------|
| Top    |                | 53.4 (8.6)  | 62.6 (6.9) |
|        |                | AA          | AA         |
| Bottom |                | 54.4 (8.9)  | 62.2 (10.1)|

Distinct uppercase letters indicate statistically significant differences between curing strategies for the same region (p<0.05). Distinct lowercase letters indicate statistically significant differences between regions for the curing strategy (p<0.05).

Bond Strength

Significant differences were found between curing strategies (p<0.05), storage times (p<0.01), and the interaction between the two factors was significant (p<0.01). Multiple comparisons are shown in Table 3. Dual-cured adhesive system provided higher BS then light-cured adhesive system in both storage times. For light-cured material, 24-h and 6-month storage times provided similar BS. Specimens bonded with dual-cured adhesive system stored for 6 months provided lower BS than those stored for 24 h, even though they showed higher BS than the light-cured specimens.
Table 3. Means (standard deviations) of BS (MPa) according to the curing strategy of the adhesive system and storage time in water.

| Storage time | Curing strategy | Light-cured | Dual-cured |
|--------------|----------------|-------------|------------|
| 24 h         | Light-cured    | 7.7 (1.4)   | 12.2 (1.5) |
|              | Dual-cured     | 8.0 (0.7)   | 9.7 (0.7)  |

Distinct uppercase letters indicate statistically significant differences between curing strategies for the same storage time (p<0.05). Distinct lowercase letters indicate statistically significant differences between storage times for the curing strategy (p<0.05).

Failure mode

Most failures were of cohesive mode regardless of the adhesive and storage time. The light-cured adhesive system showed more adhesive failures than the dual-cured (Table 4).

Table 4. Number of adhesive, cohesive, and mixed failures according to curing strategy of adhesive systems and storage times.

| Curing strategy | Light-cured | Dual-cured |
|-----------------|-------------|------------|
| Storage time    | Adhesive    | Cohesive   | Mixed | Adhesive | Cohesive | Mixed |
| 24 h            | 6           | 8          | 3     | -        | 13        | 4     |
| 6 months        | 3           | 13         | 1     | 2        | 9         | 6     |
| Total           | 9           | 21         | 4     | 2        | 22        | 10    |

Discussion/Conclusion

The null hypothesis tested – that different curing strategies of adhesive systems and storage times do not affect physical properties of adhesive interfaces – was rejected, as specimens in which dual-cured adhesive system was used had significant differences in the analyzed variables.

An universal adhesive system was chosen as the light-cured material since it represents the newest eighth-generation adhesives that can be applied according to the dentist’s preference, with or without dentin etching (etch-and-rinse or self-etch application)⁴. Previous studies showed that Scotchbond™ Universal adhesive presents similar dentin bond strength¹⁵ and bond stability¹⁶ for etch-and-rinse and self-etch techniques, or higher bond strength for self-etch technique¹⁷, which was used in this investigation.

Although in situ DC was similar for top and bottom regions with both curing strategies, the dual-cured adhesive system provided higher DC and BS than the light-cured. The dual-cured adhesive used in this study requires the application of an activator and a catalyst before and after the primer, respectively. The activator contains components derived from sulfinate salts, such as Sodium benzenesulfinate, that react with acidic monomers of the primer to produce phenyl or benzenesulfonyl free radicals and initiate polymerization¹⁸. The catalyst contains benzoyl peroxide that can chemically acti-
vate the polymerization reaction without light energy by reacting with tertiary amine to produce free radicals. These characteristics can contribute to increase the degree of conversion of the adhesive system in order to promote long-lasting bonds, which could justify the better results obtained with the dual-cured adhesive system to in situ DC and BS.

In addition, higher DC values were reported for multi-step adhesives compared to simplified adhesive systems due to the relatively hydrophobic and un-solvated bond layer from the primer of multi-step adhesive systems. On the other hand, the higher percentage of hydrophilic monomers and water presence in simplified adhesives (as Scotchbond™ Universal) impair the curing reaction, decreasing DC.

Although differences regarding bond strength were found among groups, the low occurrence of adhesive failures may reflect an adequate interaction between materials and dentin. The decreased BS values after 6-month aging for dual-cured strategy is possibly associated with the degradation of collagen fibers exposed to acid etching that were not completely covered by the primer, enabling the enzymatic action on matrix metalloproteinases (MMPs) and cysteine cathepsins. On the other hand, as the light-cured strategy had a universal adhesive system applied in self-etching mode, it is likely that fewer collagen fibrils were left uncovered after adhesive application, favoring bond stability. Although presenting lower BS values after water storage, the dual-cured adhesive system provided higher BS means than the light-cured material, which is an important finding. This might have been due to the better mechanical strength of multi-step adhesive systems, which is in part related to their higher DC. Further studies should be conducted to evaluate bonding stability of dual-cured adhesives after longer aging times.

The results of this laboratorial investigation indicated that the use of a dual-cured adhesive system may result in higher DC within the hybrid layer and better bonding performance, increasing the longevity of tooth restorations.

Concerning the findings of this study, the dual-cured adhesive system influenced the bond strength and degree of conversion in 4-mm deep dentin cavities filled with a bulk-fill resin composite. Although the dual-cured adhesive bond strength had reduced after 6 months, it was higher compared to the light-cured independently of the storage time. The degree of conversion was greater in both regions for the dual-cured adhesive system. These results suggest a positive effect on bond strength, and better performance of the dual-cured adhesive system, which might contribute to the success and longevity of resin composite restoration in deep cavities filled with a bulk-fill resin composite.
Data availability
Datasets related to this article will be available upon request to the corresponding author.

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