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Effect of Different Adhesive Systems Used for Immediate Dentin Sealing on Bond Strength of a Self-Adhesive Resin Cement to Dentin

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Clinical Relevance

Immediate dentin sealing (IDS) has been suggested in order to reduce postoperative sensitivity. Although IDS tends to enhance initial bond strength values, IDS does not prevent a decrease in bond strength values after three months of storage in water.

SUMMARY

Objective: The purpose of this study was to investigate the immediate and three-month water storage behavior of adhesives when used for immediate dentin sealing (IDS).

Methods and Materials: Four adhesive systems were used to perform IDS: a one-step self-etch (Xeno V), a two-step self-etch (Clearfil SE Bond), a two-step etch-and-rinse (XP Bond), and a three-step etch-and-rinse (Optibond FL). For the control group, IDS was not performed. The self-adhesive resin cement RelyX Unicem was used for the luting procedures. After seven days of water storage, specimens (n=6) were sectioned into beams (n=5) with an approximately 1-mm^2 cross-sectional area. Half of the specimens were tested in tension after seven days of water storage at 37°C, while the other half was stored for three months prior to testing in tension using a universal testing machine (1 mm/min). The failure pattern was determined using a stereomicroscope and scanning electron microscopy. Microtensile bond strength (μTBS) data were statistically analyzed by two-way analysis of variance and Tukey *post hoc* test (α=0.05).

Results: After seven days, the control group presented the lowest μTBS but did not differ from XP Bond and Clearfil SE Bond. After three months, there was no μTBS difference between the IDS groups and the control.

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Conclusions: After seven days of water storage, the groups with IDS presented higher μTBS values than the control group, although XP Bond and Clearfil SE Bond did not present significant differences. However, after three months of storage in water, IDS groups did not differ significantly from control group, which did not receive IDS.

INTRODUCTION

The multistep adhesive cementation technique is considered complex and sensitive.[1,2] Dentin hypersensitivity is a common symptom reported by patients after cementation procedures and can be associated with several factors, such as overheating and desiccation during tooth preparation, bacterial infiltration, and the fluid movement through dentinal tubules.[3]

Self-adhesive luting agents do not require any pretreatment of the tooth surface and were developed in an attempt to simplify bonding procedures and reduce the shortcomings of the conventional multistep adhesive cementation technique.[4,5] Their application on smear layer–covered substrates keep dentin permeability at very low levels,[6] contributing to reduced postoperative sensitivity and lower susceptibility to moisture degradation.[7]

Immediate dentin sealing (IDS) has been suggested to reduce postoperative sensitivity and bacterial infiltration while contributing to improved bond strength of indirect restorative procedures.[3,8-10] According to previous reports, patients treated with the IDS technique experienced improved comfort during the provisional restoration stage.[8,11]

In this technique, the dentin is hybridized using either a two-step self-etching or a three-step etch-and-rinse adhesive system (hydrophobic resin covering the primer layer) immediately after preparation and before impression taking, contributing to a reduction of dentin permeability.[12-14] Simplified etch-and-rinse and self-etching adhesive systems are widely available for clinicians; however, their efficacy in the IDS technique has not been reported so far.

The null hypotheses evaluated in this study were 1) that there is no difference in the bond strength produced by the different adhesives in the IDS technique and 2) that storage in water for three months does not affect the bond strength.

METHODS AND MATERIALS

Four adhesive systems were used in this study: two etch-and-rinse: the three-step Optibond FL (Kerr Corporation, Orange, CA, USA) and the two-step XP Bond (Dentsply De Trey, Konstanz, Germany); two self-etching: the two-step Clearfil SE Bond (Kuraray Medical, Okayama, Japan) and the one-step Xeno V (Dentsply De Trey). RelyX Unicem (3M ESPE, St Paul, MN, USA) was used as the self-adhesive resin luting agent. Materials, compositions, and directions for use are shown in Table 1.

Teeth were randomly assigned to five experimental groups: four according to the adhesive system used for IDS and one control group (without IDS).

Tooth Preparation

Sixty caries-free third molars were used after the protocol was approved by the review board. After disinfection and removal of soft tissues, flat middle-depth coronal dentin surfaces were exposed using 600-grit SiC paper (3M of Brazil Ltd, Sumare, Brazil) under running water to create a standardized smear layer. Teeth had their roots removed 2 mm below the cemento–enamel junction using a diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Teeth assigned to the control group were then stored in water for seven days before cementation procedures. All other groups received immediate dentin sealing as described below.

IDS

The four adhesive systems evaluated in this study were applied according to the manufacturers' instructions to perform IDS. After initial light curing (Radii Cal Plus, SDI, Victoria, Australia) for 10 seconds (1500 mW/cm^2), the adhesive layer was covered by a layer of glycerin gel (KY gel, Johnson & Johnson do Brasil, Sao Paulo, Brazil) and light polymerized for 20 seconds to avoid the oxygen inhibition layer. After removing the gel using copious irrigation, the teeth were stored in water at 37°C for seven days before luting procedures.

Luting Procedures for Microtensile Bond Strength

Four-millimeter-thick composite resin discs with a 12-mm diameter were prepared by layering 2-mm-thick increments of a microhybrid composite resin (Filtek Z250, shade A1; 3M ESPE) into a silicone mold. Each increment was light activated for 40 seconds using a Radii Cal Plus (1500 mW/mm^2) light curing unit. One side of the composite resin discs was abraded with 600-grit SiC paper under water cooling to create a flat surface with standardized roughness for cementation. The composite surface

Table 1: *Classification, Manufacturer, Lot Number, and Composition of Adhesive Systems, Luting Agent, and Composite Resin Used in This Study*

Type	Manufacturer (Lot Number)	Composition
Self-adhesive resin cement	RelyX Unicem 3M ESPE (366321)	Base paste (white): methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers Catalyst paste (yellow): methacrylate monomers, alkaline (basic) fillers, initiators components, stabilizers, pigments
One-step self-etching adhesive	Xeno V Dentsply De Trey (0906000336)	Bifunctional acrylate, acidic acrylate, functionalized phosphoric acid ester, acrylic acid, water, tertiary butanol, initiator, stabilizer
Two-step self-etching adhesive	Clearfil SE Bond Kuraray Medical (primer, 01065A; bond, 01585A)	Primer: 10-MDP, HEMA, DMA, catalyst, water Bonding: 10-MDP, HEMA, DMA, Bis-GMA, filler, catalyst
Two-step etch-and-rinse adhesive	XP Bond Dentsply De Trey (1011000654)	34% phosphoric acid conditioner gel Adhesive: TCB resin, PENTA, UDMA, TEGDMA, HEMA, stabilizer, EDAB, CQ, functionalized amorphous silica, t-butanol
Three-step etch-and-rinse adhesive	Optibond FL Kerr Corp (primer: 3462545; bond: 3586282)	37.5% phosphoric acid conditioner gel Primer: HEMA, GPDM, MMEP, ethanol Bonding: HEMA, MPS, 2-hydroxy-1,3-propanediyl bismethacrylate, disodium hexafluorosilicate
Composite Resin	Filtek Z250 3M ESPE (N2580)	UDMA, Bis-EMA, Bis-GMA, silica/zirconia filler (average particle size of 0.6 μm)

Abbreviations: Bis-EMA, ethoxylated bisphenol A dimethacrylate; Bis-GMA, bisphenol-glycidyl methacrylate; CQ, camphorquinone; DMA, dimethacrylate; EDAB, ethyl-4-dimethylamino benzoate; GPDM, glycerol phosphate dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; MMEP, mon-2-methacryloxyethyl phthalate; MPS, 3-methacryloxypropyltrimethoxysilane; PENTA, phosphonated penta-acrylate ester; TCB resin, tetracarboxylic acid modified dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

was airborne-particle abraded with 50-μm aluminum oxide particles for 10 seconds. Before the luting procedures were performed, the composite resin discs were ultrasonically cleaned in distilled water for 10 minutes, rinsed with running water, air-dried, and silanated (RelyX Ceramic Primer, 3M ESPE).

After silanization, the luting procedures were performed according to the manufacturers' instructions. The composite resin discs were pressed on the cement using proper digital pressure, which was sustained until light curing was performed from the buccal and lingual sides. The cementation procedures were randomly processed. Specimens were light activated for 40 seconds from the buccal, lingual, and occlusal directions. Bonded specimens were stored in distilled water at 37°C for seven days.

Microtensile Bond Strength Evaluation

Seven days after the cementation procedures, the restored teeth were serially sectioned perpendicular to the adhesive–tooth interface into slabs and the slabs into beams (n=5) with a cross-sectional bonded area of approximately 1 mm^2 using a diamond saw (Isomet 1000, Buehler Ltd). Ten beams were selected for testing for each tooth. Half of the beams were fixed to the grips of a universal testing machine (EZ Test, Shimadzu Corp, Kyoto, Japan) using a cyanoacrylate adhesive (Loctite Super Bonder Gel, Henkel, Düsseldorf, Germany) and tested in tension at a crosshead speed of 1 mm/min until fracture occurred. The other half was stored in water at 37°C for three months prior to microtensile testing. Maximum tensile load was divided by the specimen cross-sectional area to obtain results in units of stress (MPa). Mean values were calculated for each tooth at each testing time.

Failure modes were determined by examining the fractured specimens using a stereomicroscope and classified into the following types: CD (cohesive in dentin), AD (adhesive between resin cement and dentin—sealed or unsealed), MI (mixed), ADR (adhesive between indirect restoration and resin cement), and CR (cohesive in composite resin).

Bond strength values were submitted to two-way analysis of variance, considering the factors "dentin sealing" and "water storage time" (5×2) and Tukey *post hoc* test (α=0.05) (SAS for Windows version 8, SAS Institute Inc, Cary, NC, USA).

Table 2: *Mean Bond Strength Values in MPa (Standard Deviation) for the Different Materials Applied to Dentin With Immediate Dentin Sealing and Without (Control Group)*[a]

	7 Days	3 Months
Xeno V	48.0 (14.1) Aa	18.6 (9.8) Ab
Clearfil SE Bond	33.0 (8.4) ABa	25.4 (3.9) Aa
XP Bond	30.8 (14.0) ABa	21.9 (2.5) Aa
OptiBond FL	45.1 (6.0) Aa	28.3 (9.2) Ab
RelyX Unicem (control)	22.8 (7.7) Ba	23.3 (17.3) Aa

[a] *Means followed by different uppercase letters (columns)and lowercase letters (rows) are significantly different by Tukey test at 5% confidence level.*

RESULTS

Microtensile Bond Strength

Mean microtensile bond strength (μTBS) values are presented in Table 2. Two-way analysis of variance revealed a significant difference for the factor "dentin sealing" (p=0.02380) and for the factor "water storage time" (p=0.00024). The interaction between factors was also statistically significant (p=0.03771).

After seven days, among the four groups subjected to IDS, the μTBS showed no significant differences between adhesive systems. However, Xeno V and Optibond FL presented significantly higher values when compared to the control group (no IDS). Clearfil SE Bond and XP Bond showed intermediate values and did not differ significantly from the control group.

The evaluation after three months of water storage demonstrated no significant differences among groups. However, when compared with seven days of storage, there was a significant reduction in bond strength values for the single-step self-etching system Xeno V and for the three-step etch-and-rinse system Optibond FL.

Failure mode analysis (Figure 1) showed a prevalence of adhesive failures between the resin cement and dentin. After three months, there was a slight

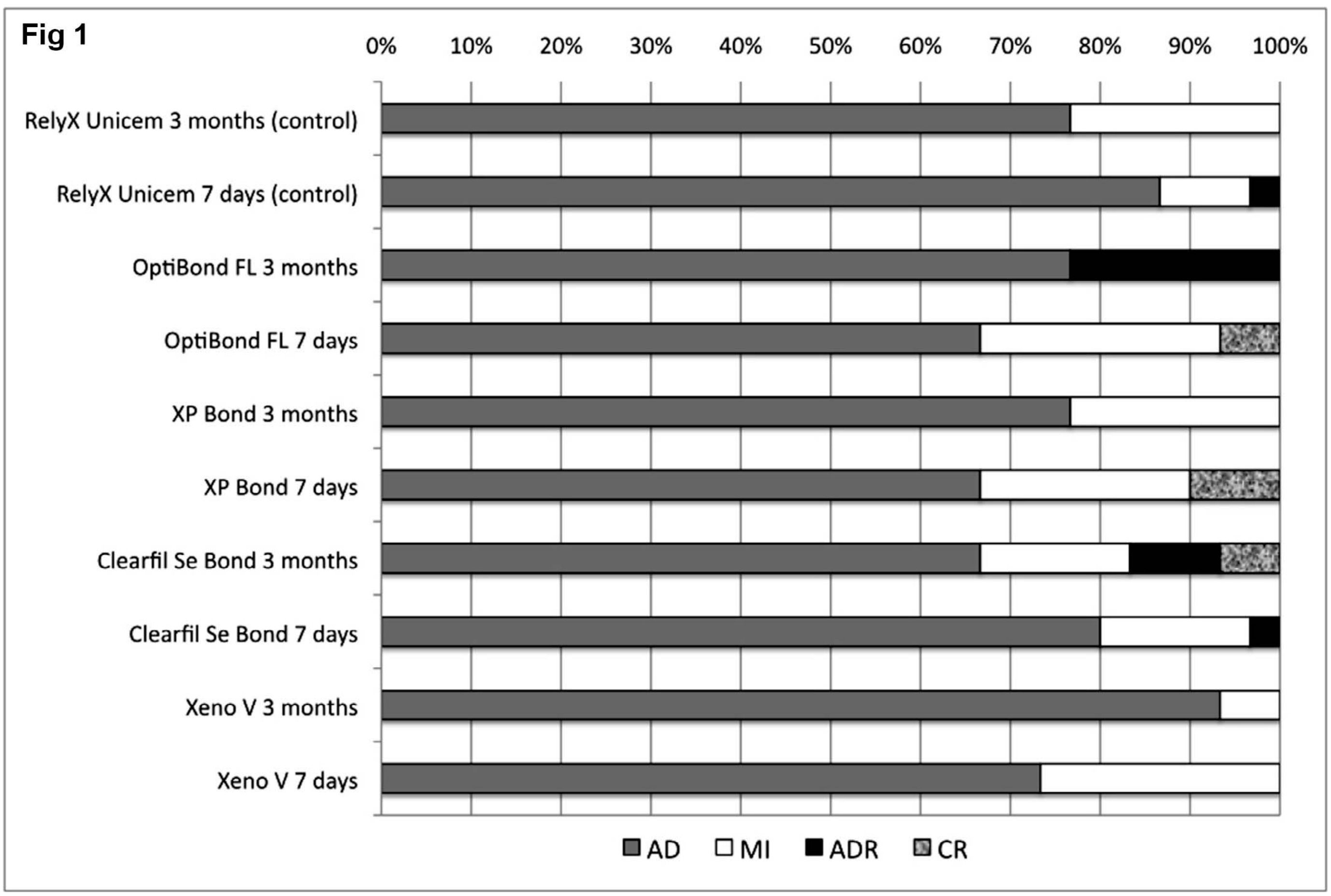

Figure 1. *Distribution of failure modes within groups. CD, cohesive in dentin; AD, adhesive between resin cement and dentin (sealed or unsealed), MI: mixed; ADR, adhesive between indirect restoration and resin cement; CR, cohesive in composite resin.*

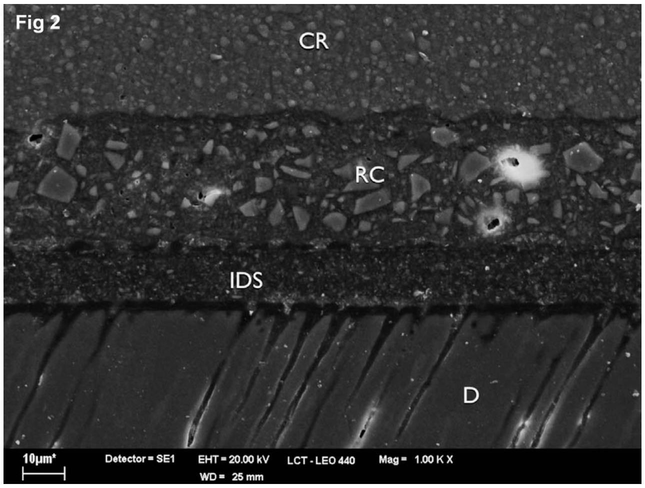

Figure 2. *Representative SEM image of the interface when IDS was performed with Optibond FL adhesive system. D: dentin; IDS: immediate dentin sealing; RC: resin cement; CR: indirect composite resin.*

increase in the amount of adhesive failures for Xeno V, XP Bond, and Optibond FL. No cohesive failures in dentin were observed.

DISCUSSION

The IDS technique can contribute to stability and reduction in permeability of the adhesive interface in indirect restorations.[12,14] This technique consists of dentin hybridization with an adhesive system (Figure 2) after preparation and contributes to increased bond strength and reduced dentin sensitivity during the provisional phase.[8,9]

Adhesive systems that have a hydrophobic resin coating the primer layer play an important role in reducing dentin permeability.[12] On the other hand, simplified adhesive systems, which have the hydrophilic primer and the hydrophobic resin in the same bottle, have been increasingly used because of their user-friendly characteristics. Single-step self-etching adhesives are considered the most susceptible to degradation when stored in water due to the increased hydrophilicity of the interface.[4] Thus, in this study, the first hypothesis was validated since there was no significant difference when simplified adhesive systems were compared with the three-step etch-and-rinse and the two-step self-etch adhesives.

Although XP Bond and Clearfil SE Bond did not demonstrate a significant difference from the control after seven days, all groups in which IDS was performed presented higher µTBS values when compared to the control group. This is probably due to the etch-and-rinse and self-etch adhesives having a better demineralization capacity and hybrid layer formation than the self-adhesive resin cement.[15] The hybrid layer produced by the self-etching adhesive systems used in this study was approximately 0.5 µm, while for the etch-and-rinse, it was around 5 µm.[5,15-18] Moreover, recent studies demonstrated that the demineralization depth produced by RelyX Unicem is approximately 200 nm,[15] and the initial bond strength is higher for multistep resin cements that are associated with some type of adhesive system.[5]

The second hypothesis was partially rejected because two of the materials used for IDS were not able to eliminate the interface degradation effects produced by storage in water for three months. IDS performed with Xeno V and Optibond FL had a significant µTBS reduction after three months of water storage. In addition, there was no significant difference between the IDS groups and the control after three months. A previous study showed a µTBS increase when IDS was performed with Clearfil SE Bond and used RelyX Unicem as a luting agent in the absence of simulated pulpal pressure.[14] These data corroborate the µTBS stability observed after three months of water storage in the present study when IDS was performed using Clearfil SE Bond.

In this study, specimens were sectioned into beams with a 1-mm^2 cross-sectional area and stored in water for a period of three months. Despite the relatively short storage time, the beam shape accelerates the interface degradation.[19] Interestingly, the µTBS of the control group did not decrease after water storage. Reports show that, even after one year of storage in water, there was no µTBS reduction for this material. It is believed that the change of the self-adhesive cement from hydrophilic to hydrophobic improves the interface stability.[15] After three months, IDS with Clearfil SE Bond and XP Bond demonstrated no reduction in µTBS values, remaining stable during the study period.

As previously indicated, the IDS technique is recommended in order to increase µTBS and reduce dentin sensitivity during the provisional phase.[8,9] According to the limitations of this study, the one-step self-etching adhesive (Xeno V) and conventional three-step adhesive (Optibond FL) provided significantly higher µTBS values after seven days when compared to the control group. Although after three months this performance was not maintained, there was no difference when compared to the control group. Therefore, a decisive factor for clinically indicating this technique would be its ability to reduce dentin hypersensitivity.

The failure pattern SEM images showed that the failure normally occurred between the resin cement and the sealing, which suggests that the dentin would still remain sealed even if interface debonding occurred. According to Magne and others,[20,21] the adhesion of the sealed dentin to resin cement may occur due to the presence of residual free radicals, van der Waals–type interactions, and micromechanical retention.

CONCLUSIONS

Based on the findings and limitations of this study, we conclude the following:

- The μTBS values produced by the different materials used for IDS were not significantly different from each other.
- After seven days of water storage, the μTBS of groups sealed with XP Bond and Clearfil SE Bond did not differ significantly from the control group.
- Storage in water for three months produced a significant reduction in μTBS for groups in which IDS was performed with Xeno V and Optibond FL.
- After three months of water storage, the μTBS values for groups with IDS were not significantly different from the control group (without IDS).

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Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of the SISNEP. The approval code for this study is 94/2008.

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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