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# Effects of Immediate Dentin Sealing and Pulpal Pressure on Resin Cement Bond Strength and Nanoleakage

VB Santana • RS de Alexandre • JA Rodrigues
C Ely • AF Reis

### Clinical Relevance

The immediate dentin sealing technique was able to prevent negative effects of pulpal pressure on interfaces produced by self-adhesive and conventional multistep resin cements.

## SUMMARY

**Objective: The object of this study was to evaluate the simulated pulpal pressure (SPP) and immediate dentin sealing technique (IDS) effects on the microtensile bond strength (μTBS) and nanoleakage of interfaces produced by different luting agents.**

**Methods and Materials: Two self-adhesive luting agents (RelyX Unicem [UC] and Clearfil SA Luting [SA]) and two conventional luting agents (Rely X ARC [RX] and Panavia F [PF]) were evaluated. Eighty human molars were divided in four groups according to luting agents. Each group was subdivided according to SPP (with or without) and dentin sealing (immediate or delayed) using Clearfil SE Bond (n=5). After IDS was performed, specimens were stored in water for seven days before luting procedures. Composite blocks were luted according to the manufacturers' instructions. One half of the specimens were subjected to 15 cm $H_2O$ of hydrostatic pressure for 24 hours before cementation procedures and continued for 24 hours afterward. Then, restored teeth were sectioned into beams and tested in tension. Two additional teeth per group were prepared for nanoleakage evaluation with scanning electron microscopy. Bond strength data were statistically analyzed by three-way analysis of variance and Tukey test.**

**Results: μTBS of RX decreased when it was subjected to SPP without IDS. However, in the same conditions, μTBS of UC increased. The IDS prevented negative influence of SPP on μTBS of RX and PF; however, a decrease in**

Veronica Batista Santana, DDS. MS, Department of Operative Dentistry, University of Guarulhos, Guarulhos, SP, Brazil

Rodrigo Sversut de Alexandre, DDS, MS, PhD, Department of Restorative Dentistry, Sao Paulo State University, Araçatuba Dental School, Araçatuba, SP, Araçatuba, Brazil

José Augusto Rodrigues, DDS, MS, PhD Department of Restorative Dentistry, University of Guarulhos, Guarulhos, SP, Brazil

Caroline Ely, DDS, MS, PhD, Department of Operative Dentistry, Guarulhos University, Guarulhos, SP, Brazil

*Andre F. Reis, Department of Operative Dentistry, Guarulhos University, Guarulhos, SP, Brazil

*Corresponding author: Praça Tereza Cristina, 229, Guarulhos, SP, Brazil 07023-070; e-mail: reisandre@yahoo.com

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**μTBS of SA and UC was observed. Except for RX, IDS increased μTBS for all resin cements.**

**Conclusion: Independent of SPP, the IDS technique obtained higher μTBS for PF, SA, and UC and did not influence RX μTBS.**

## INTRODUCTION

The multistep adhesive cementation technique is considered complex and sensitive.[1,2] Problems that can occur during application can account for higher incidence of postoperative sensitivity after bonding procedures,[3] pulpal damage,[4] and premature degradation of the resin-dentin interface.[5] 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.[6,7] Their application on smear layer-covered substrates maintains dentin permeability in very low levels,[8] contributing to reduced postoperative sensitivity and lower susceptibility to moisture degradation.[9] The interaction with subjacent dentin and enamel has been suggested to occur through formation of a hybridized complex and chemical interaction with hydroxyapatite.[10]

Dentin is the main substrate available for adhesion in prosthetic procedures, especially in vital teeth. Dentin is a hydrated hard tissue in the vital state, where there is an outward flow of dentinal fluid through the dentinal tubules with a positive pulpal pressure, estimated to be approximately 15 cm $H_2O$.[11] Water presents deleterious effects for adhesive procedures, such as the plasticization of the polymer chains, leading to compromised mechanical properties and hydrolytic degradation of resin and collagen fibrils.[12-14] Several studies have evaluated the microtensile bond strength (μTBS) using simulated pulpal pressure (SPP) during adhesive procedures.[15-20] These studies have demonstrated that the presence of positive pulpal pressure can decrease μTBS and dentin sealing ability.[16,21,22]

The immediate dentin sealing (IDS) technique has been suggested as an alternative to improve the quality of adhesion for indirect restorative procedures.[23-25] In this technique, dentin is hybridized using either a two-step self-etching or a three-step etch-and-rinse adhesive system immediately after preparation and prior to impression taking. Increased μTBS and reduced postoperative sensitivity have been reported for this technique.[24,26-28]

It is possible that use of the IDS technique can reduce the effects of positive pulpal pressure on resin-dentin interfaces, but it has not been evaluated to date. Thus, the aim of this study was to evaluate the effect of SPP and the IDS technique on the μTBS and nanoleakage of interfaces produced by different luting agents. The null hypotheses tested were: 1) IDS produces no difference in the bond strength and nanoleakage; 2) simulated pulpal pressure does not affect interfaces; and 3) there is no difference in the performance of the different luting materials.

## METHODS AND MATERIALS

Four resin luting agents were used in this study: two self-adhesive cements (RelyX Unicem [UC], 3M ESPE, St. Paul, MN, USA; Clearfil SA Luting [SA], Kuraray Medical, Okayama, Japan) and two conventional resin cements—one that uses a two-step etch-and-rinse adhesive (RelyX ARC [RX], 3M ESPE) and one that uses a one-step self-etching adhesive (Panavia F [PF], Kuraray Medical). Luting agents were mixed and placed according to the manufacturers' instructions (Table 1).

Teeth were randomly assigned to four experimental groups according to resin cement and then to four subgroups according to presence of SPP (with or without SPP) and dentin sealing condition (IDS or delayed dentin sealing [DDS]). This study design resulted in 16 experimental groups (n=5) according to luting agent, pulpal pressure, and dentin sealing.

### Tooth Preparation

One hundred twelve recently extracted caries-free third molars stored in 0.1% thymol (Symrise GmbH, Holzminden, Germany) solution at 4°C for no longer than three months were used in this study (80 for the μTBS test and 32 for nanoleakage evaluation). Teeth were obtained by protocols that were approved by the University's review board. After disinfection and removal of soft tissues, flat middle depth coronal dentin surfaces were exposed with 600-grit SiC paper (3M of Brazil Ltd, Sumare, Brazil) under running water to create a standardized smear layer. Teeth had their roots removed using a diamond saw (ISOMET, Buehler, Lake Bluff, IL, USA) 2 mm below the cemento-enamel junction. Pulpal tissue was gently removed to prevent damage of the predentin region.

### SPP

To simulate pulpal pressure on the dentin surface, each tooth was bonded to a Plexiglass platform ($3 \times 3 \times 0.3$ cm) penetrated by a 1-mm-diameter stainless steel tube and fixed with cyanoacrylate adhesive (Loctite Super Bonder Gel, Henkel, Düsseldorf,

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Table 1: *Materials, Lot Number, Manufacturers, Composition, and Application Technique*

| Type | Manufacturers (lot number) | Composition | Application technique |
|---|---|---|---|
| Dual-polymerizing resin cement + two-step etch-and-rinse adhesive | RelyX ARC (N097266) + Adper Single Bond 2 (7MY) (3M ESPE) | Cement: Bis-GMA, TEGDMA polymer, zirconia/silica filler<br>Etchant: 35% $H_3PO_4$<br>Adhesive: bis-GMA, HEMA, UDMA, dimethacrylates,ethanol, water, camphorquinone , photoinitiators, polyalkenoic acid copolymer, 5-nm silica particles | a (15 s); b (15 s); c; d; e; i (10 s); mix cement; apply mixture |
| Dual-polymerizing resin cement + one-step self-etching adhesive | Panavia F (paste A, 00249D; paste B, 0027A) + ED Primer (primer A, 00262A; primer B, 00137A) (Kuraray Medical) | Primer A: HEMA, 10-MDP, 5-NMSA, water, accelerator<br>Primer B: 5-NMSA, accelerator, water, sodium benzene sulphinate<br>Paste A: 10-MDP, silanated silica, hydrophobic aromatic and aliphatic dimethacrylate, hydrophilic dimethacrylate photoinitiator, dibenzoyl peroxide<br>Paste B: silanated barium glass, sodium fluoride, sodium aromatic sulfinate, dimethacrylate monomer, BPO | h (A + B) (leave undisturbed for 60 s); mix cement; apply mixture; i (40 s) |
| Dual-polymerizing self-adhesive resin cement | RelyX Unicem (366321) (3M ESPE) | Base: glass fiber, methacrylated phosphoric acid esters, dimethacrylates, silanated sílica, sodium persulfate<br>Catalyst: glass fiber, dimethacrylates, silanated sílica, p-toluene sodium sulfate, calcium hydroxide | Mix cement; apply mixture; i (40 s) |
| Dual-polymerizing self-adhesive resin cement | Clearfil SA luting (00081A) (Kuraray Medical) | Paste A: MDP, Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate DL-camphorquinone, benzoyl peroxide, initiator, silanated barium glass filler, silanated colloidal silica<br>Paste B: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, accelerators, pigments, surface-treated sodium fluoride, silanated barium glass filler, silanated colloidal silica | Automix cement; apply mixture i (40 s) |
| Two-step self-etching adhesive system | Clearfil SE Bond (00788A) (Kuraray Medical) | Primer: MDP, HEMA, hydrophilic dimethacrylate, DL-camphorquinone, *N,N*-diethanol p-toluidine, water<br>Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, DL-camphorquinone, *N,N*-diethanol p-toluidine, silanated colloidal silica<br>Paste A and paste B: as described above | f (20 s); e; g; i (10 s) |

*Application technique: a, acid etch; b, rinse surface; c, dry with cotton pellet; d, apply one-bottle adhesive; e, gently air dry; f, apply primer; g, apply adhesive; h, apply mixture; i, light polymerize; j, autopolymerize. Abbreviations: Bis-GMA, bisphenol A diglycidyl ether methacrylate; HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloxydecyl dihydrogen phosphate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; 5-NMSA, N-methacryloxyl-5-aminosalicylic acid; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.*

Germany). The pulp chamber was filled with distilled water via a polyethylene tube connected to a syringe barrel with 10 mL distilled water and suspended 15 cm from the tooth crown. Thus, each specimen was connected to a hydraulic pressure device that delivered 15 cm of water pressure.[11,16] For the group not subjected to SPP, the apparatus was not assembled.

## IDS

For IDS, the two-step self-etching adhesive Clearfil SE Bond was applied according to the manufacturer's instructions. After initial light curing (Optilux 501, Demetron/Kerr, Danbury, CT, USA) for 10 seconds, the adhesive layer was covered with a layer of glycerin gel (KY gel, Johnson & Johnson, Sao Paulo, Brazil) and light polymerized for 20 seconds to avoid the oxygen inhibition layer. After rinsing off the gel, the teeth were stored in water at 37°C for seven days before the luting procedures. Following this storage time period, sealed dentin was cleaned by airborne particle abrasion with 50-µm aluminum oxide particles (Asfer Indústria Química Ltda, São Caetano do Sul, Sao Paulo, Brazil).

The teeth submitted to IDS and SPP were kept under hydrostatic pressure for eight days, starting 24 hours before IDS.

Other groups, without IDS and with SPP, were kept under hydrostatic pressure for 48 hours, starting 24 hours before the luting procedure.

## Luting Procedures for µTBS

Four-millimeter-thick composite resin discs 12 mm in diameter were prepared by layering 2-mm-thick

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increments of a microhybrid composite resin (Filtek Z250, shade A1, 3M ESPE) into a silicone mold. Each increment was light activated for 40 seconds with the Optilux 501 (700 mW/mm$^2$) halogen light curing unit (LCU). 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. The composite surface was airborne particle abraded with 50-μm aluminum oxide particles for 10 seconds. Before 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).

Excess water was removed with cotton pellets. Care was taken not to dehydrate dentin surfaces. In all groups (with or without IDS and SPP), the adhesive system (when necessary) and luting agents were applied according to the manufacturer's instructions. Composite resin discs were pressed on the cement using 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 for 24 hours, and the specimens submitted to SPP were kept under constant pulpal pressure during the same storage time.

### Luting Procedures for Nanoleakage Analysis and Scanning Electron Microscopy Evaluation

For each experimental group, two additional teeth were similarly prepared as previously described, with the exception that after luting agents were mixed and applied onto the dentin surfaces, a polyester strip was placed over the luting agent, and a glass slide was used to apply digital pressure while the luting agent was light activated for 40 seconds with the LCU. Afterward, a thin layer of a low-viscosity resin composite (Surefil SDR flow, Dentsply Caulk, Milford, DE, USA) was applied and light activated for 40 seconds. After storage in similar conditions to those described above, teeth were sectioned perpendicular to the adhesive-tooth interface into 0.9-mm-thick slabs using a diamond saw (Isomet 1000, Buehler).

Bonded slabs were coated with two layers of nail varnish applied up to within 1 mm of the bonded interfaces. To rehydrate specimens and avoid desiccation artifacts, they were immersed in distilled water for 20 minutes prior to immersion in the tracer ammoniacal silver nitrate solution for 24 hours. Tooth slabs were placed in the tracer solution in total darkness for 24 hours, rinsed thoroughly in distilled water, and immersed in a photodeveloping solution for eight hours under a fluorescent light to reduce silver ions into metallic silver grains within voids along the interface.

For scanning electron microscopy (SEM) analysis, two slices of each tooth were fixed in Karnovsky's solution and embedded in epoxy resin (Epoxycure, Buehler). Afterward, they were polished with 400-, 600-, 1200-, and 2400-grit SiC paper and 6-, 3-, 1-, and 0.25-μm diamond paste (Arotec, Cotia, Sao Paulo, Brazil). Then, specimens were dehydrated in ascending ethanol series and coated with carbon (MED 010, Balzers Union, Balzers, Liechtenstein). Resin-dentin interfaces were observed with a SEM (LEO 435 VP, LEO Electron Microscopy Ltd, Cambridge, United Kingdom) operated in the backscattered electron mode. After SEM analysis, representative leakage patterns at the cement-dentin interfaces produced by each system were photographed at 500× magnification.

### μTBS Evaluation

Twenty-four hours after cementation procedures, the restored teeth were serially sectioned perpendicular to the adhesive-tooth interface into slabs and then the slabs into beams with a cross-sectional bonded area of approximately 1 mm$^2$ (±0.2 mm$^2$) using a diamond saw (Isomet 1000, Buehler). 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. Maximum tensile load was divided by specimen cross-sectional area, measured with a digital caliper (Mitutoyo Co, Tokyo, Japan) to express results in units of stress (MPa). Five beams were selected from each restored tooth, and the average value for each tooth was used in the calculations.

Failure modes were determined by examination of fractured specimens with SEM (LEO 435 VP, LEO Electron Microscopy Ltd). Specimens were mounted on aluminum stubs and gold-sputter coated (MED 010, BAL-TEC AG, Balzers Union) prior to viewing at different magnifications. Failure modes at the fractured interface were classified into one of four types: CD (cohesive failure in dentin), AD (adhesive failure between hybrid layer and dentin), CC (cohesive failure in the cement), or ADR (adhesive failure between the luting agent and composite

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Table 2: Dentin Bond Strength Values in MPa (SD) for the Different Resin Cements and Different Dentin Conditions (Delayed Dentin Sealing or Immediate Dentin Sealing and With Simulated Pulpal Pressure or No Pulpal Pressure)

| Product type | Resin cement | Delayed dentin sealing | | Immediate dentin sealing | |
|---|---|---|---|---|---|
| | | No pressure | Hydrostatic pressure | No pressure | Hydrostatic pressure |
| Two-step etch-and-rinse adhesive/resin cement | RelyX ARC/Single Bond | 53.0 (8.6)Aa | 34.8 (11.3)Ab | 44.8 (3.1)Aa | 42.3 (8.8)Aa |
| One-step self-etching adhesive/resin cement | Panavia F/ED Primer | 14.1 (4.6)Ba | 7.8 (1.4)Ca | 26.2 (15.5)Ba* | 22.1 (5.5)Ba* |
| Self-adhesive cement | Clearfil SA Luting | 13.1 (11.1)Ba | 14.3 (5.3)BCa | 50.5 (5.6)Aa* | 33.2 (8.5)ABb* |
| Self-adhesive cement | RelyX Unicem | 14.2 (5.6)Bb | 24.2 (2.3)ABa | 52.2 (5.4)Aa* | 38.7 (8.0)Ab* |

*Means followed by different uppercase letters (columns), lowercase (rows within each sealing condition), and asterisks (rows within each hydrostatic pressure condition) are significantly different by Tukey test at the 5% confidence level.*

resin). Instead of classifying failures as mixed, the area percentage of each type of failure in each specimen was recorded.

Bond strength values were submitted to three-way analysis of variance (ANOVA) considering the factors cement, pulpal pressure, and dentin sealing ($4 \times 2 \times 2$) and Tukey *post hoc* test ($\alpha$=0.05; SAS for Windows V8, SAS Institute, Cary, NC, USA).

## RESULTS

### μTBS

Mean μTBS values are presented in Table 2. Three-way ANOVA revealed significant differences for the factors cement ($p$=0.00001), pulpal pressure ($p$=0.00084), and dentin sealing ($p$=0.00001). The double interaction for factors cement × dentin sealing was also significant ($p$=0.00001). However, the double interactions cement × pulpal pressure ($p$=0.33868) and pulpal pressure × dentin sealing ($p$=0.08434) were not significant. The triple interaction cement × pulpal pressure × dentin sealing was also found to be significant ($p$=0.00064).

Among the four tested groups that were not subjected to SPP or IDS, RX showed significantly higher μTBS than the other materials, whereas PF, SA, and UC presented similar μTBS values when exposed to this same condition (no SPP/DDS). In the presence of SPP and DDS, RX and UC presented the highest μTBS values and were not significantly different. SA self-adhesive cement had intermediate values and did not differ from UC and PF, which presented the lowest μTBS.

Comparing the groups in which DDS was performed (with or without SPP), a significant reduction in μTBS was observed for RX when SPP was applied. The opposite occurred for UC, which presented significantly higher μTBS values in the presence of SPP. However, SPP did not affect the μTBS values of PF and SA.

When IDS was evaluated without SPP, the highest μTBS values were observed for RX, SA, and UC, with no differences among them. PF presented the lowest μTBS compared with the other cements for this condition (IDS, no SPP). When IDS was performed in the presence of SPP, RX presented the highest μTBS values and was not significantly different from SA and UC. PF showed values significantly lower than RX and UC but was not different from SA. When IDS was performed, SPP did not affect μTBS for RX and PF, whereas a significant reduction was observed for SA and UC.

When groups with and without SPP were compared, RX demonstrated no changes in μTBS when IDS or DDS was performed. However, significantly higher μTBS values were recorded for PF, SA, and UC when IDS was performed, either with or without SPP.

Failure mode analysis (Figure 1) showed a prevalence of adhesive failures between resin cement and dentin (sealed or not). The only exception was for RX applied with DDS, which showed a high percentage of cohesive failures in the resin cement. It was observed that failure modes were mainly classified as adhesive (between resin cement and dentin) and cohesive within resin cement. No cohesive failure in dentin or adhesive (between resin cement and indirect composite restoration) was detected. For RX, there was an increase in the percentage of adhesive failures when IDS was performed. For PF, except for the group with IDS and without SPP, where there was a small percentage of cohesive failure within the resin cement, failure mode was 100% adhesive between resin cement and dentin.

### Nanoleakage

Figures 2-5 show representative SEMs for the nanoleakage patterns observed for the different tested groups. For all materials tested, whenever IDS was performed, the resin-dentin interfaces

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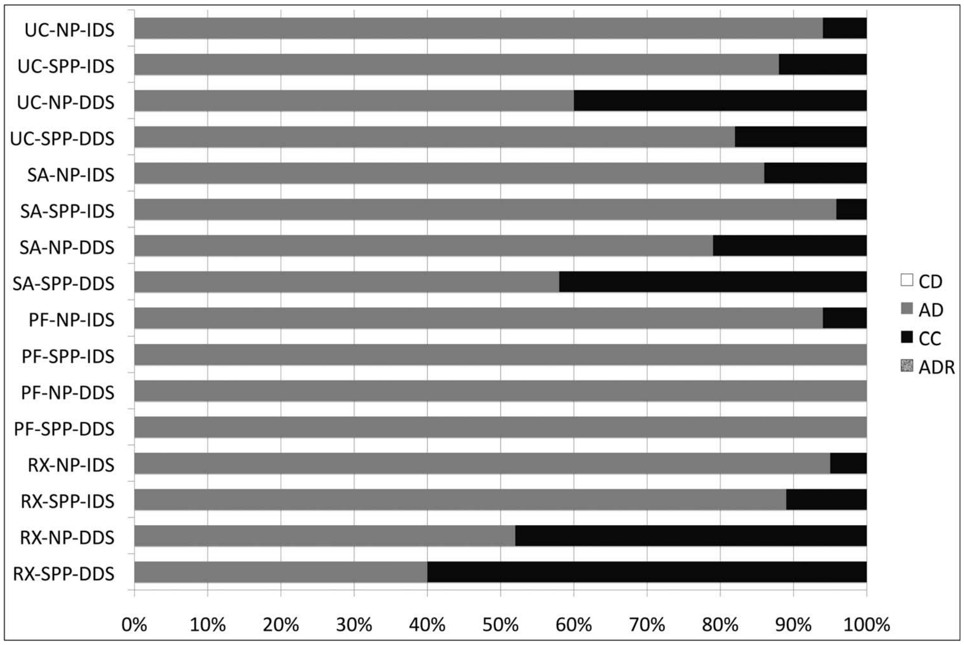


Figure 1. *Distribution of failure modes within groups. CD - cohesive failure within dentin; AD - adhesive failure between hybrid layer and dentin; CC - cohesive failure within resin cement; ADR - adhesive failure between the luting agent and composite resin; NP, no simulated pressure; SPP - simulated pulpal pressure; IDS - immediate dentin sealing; DDS - delayed dentin sealing. UC - Unicem; SA - Clearfil SA Luting; PF - Panavia F; RX - Rely X ARC.*

presented a different characteristic, because there was always the hybrid and adhesive layer produced by Clearfil SE Bond underneath the resin cements.

For groups restored with RX with DDS, an increase in silver deposition was observed in the presence of SPP (Figure 2A,B). However, when IDS was performed, lower silver deposition occurred, which was not affected by the presence of SPP (Figure 2C,D). For PF, silver deposition and gap formation was observed, which increased in the presence of SPP (Figure 3A,B). When IDS was performed, there was a reduction in silver deposition and no gaps were observed (Figure 3C,D).

For the self-adhesive cement SA, little silver deposition was observed when DDS was applied with no SPP (Figure 4A). However, in the presence of SPP,

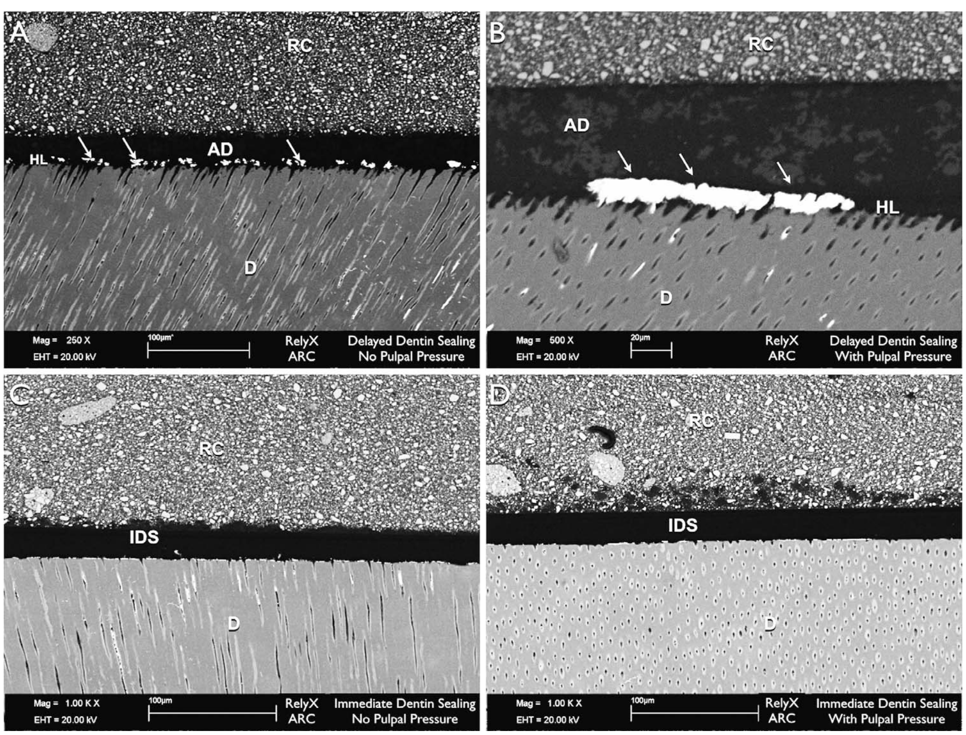


Figure 2. *Representative SEMs of specimens luted with the etch-and-rinse cement RelyX ARC. (A and B): DDS. (A): No simulated pulpal pressure (NPP); silver deposits are indicated by arrows. (B): With SPP, higher amounts of silver deposits were observed. (C and D): IDS. (C): NPP. (D): SPP. RC, resin cement; D, dentin; HL, hybrid layer. Arrows point silver deposits within the interface.*

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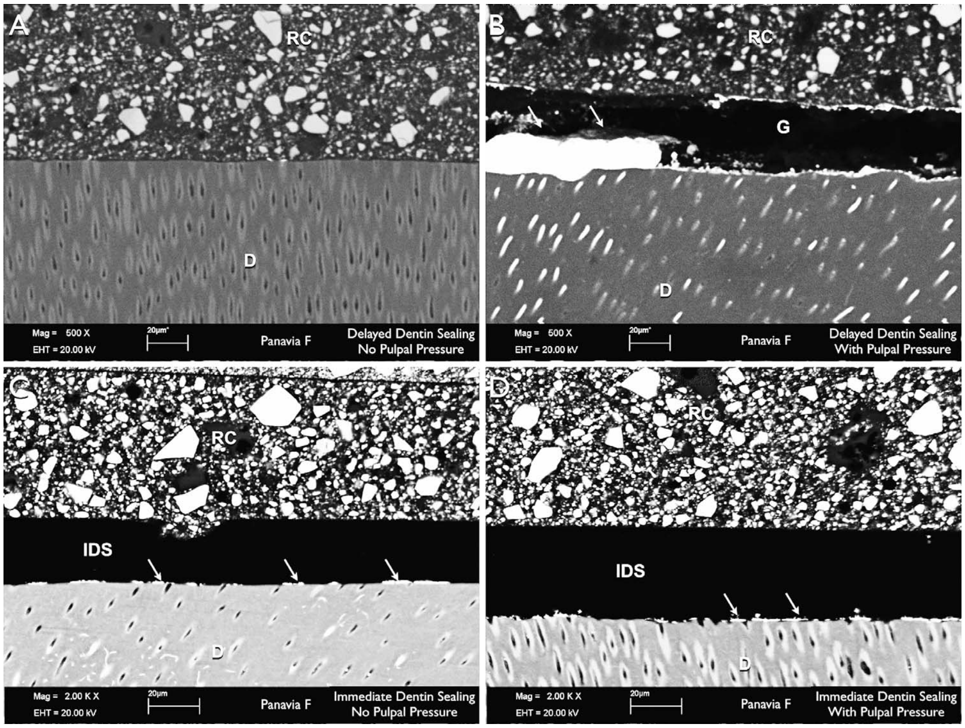


Figure 3. *Representative SEMs of specimens luted with the self-etching cement Panavia F. (A and B): DDS. Silver deposition was observed without (A) and with (B) pulpal pressure. (C and D): IDS. (C): NPP. (D): SPP. The arrows indicate a small amount of silver deposition. DE, dentin.*

silver deposition increased and gaps were present in the interfaces (Figure 4B). When IDS was performed, silver deposition was greatly reduced both in the absence and presence of SPP (Figure 4C,D).

Very little silver deposition was observed for UC self-adhesive cement, even when DDS was applied in the presence of SPP (Figure 5A,B). When IDS was performed, the bonded interface was similar to that

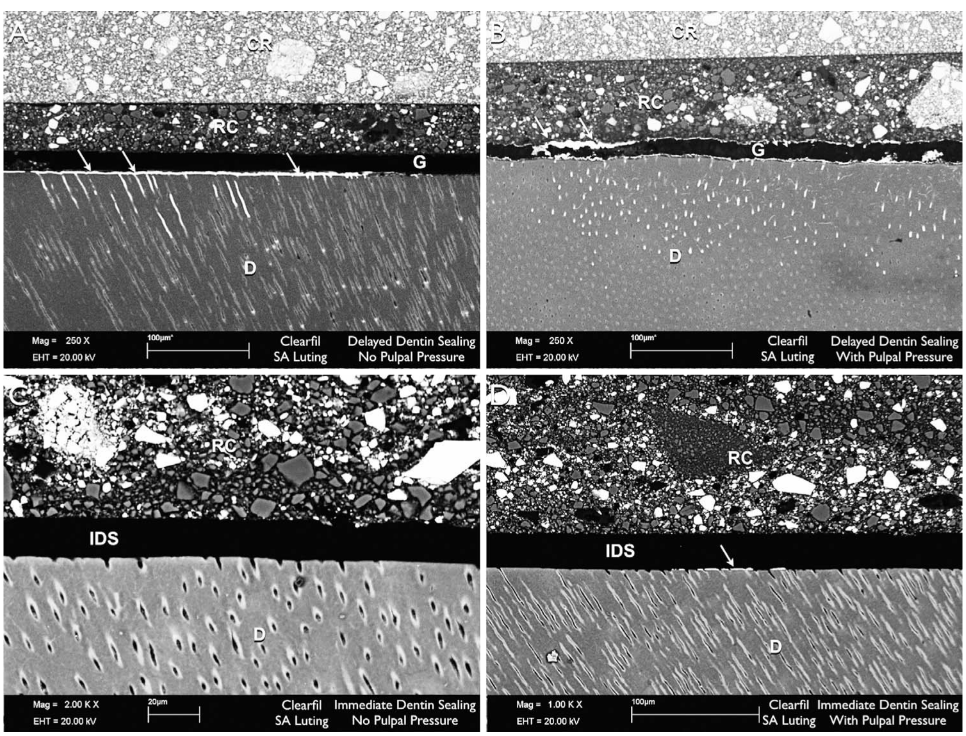


Figure 4. *Representative SEMs of specimens luted with the self-adhesive cement Clearfil SA luting. (A and B): DDS. (A): NPP. (B): Silver deposition was observed when specimens were subjected to SPP. (C and D): IDS. (C): NPP. (D): SPP. The arrows indicate silver deposition. G, gap.*

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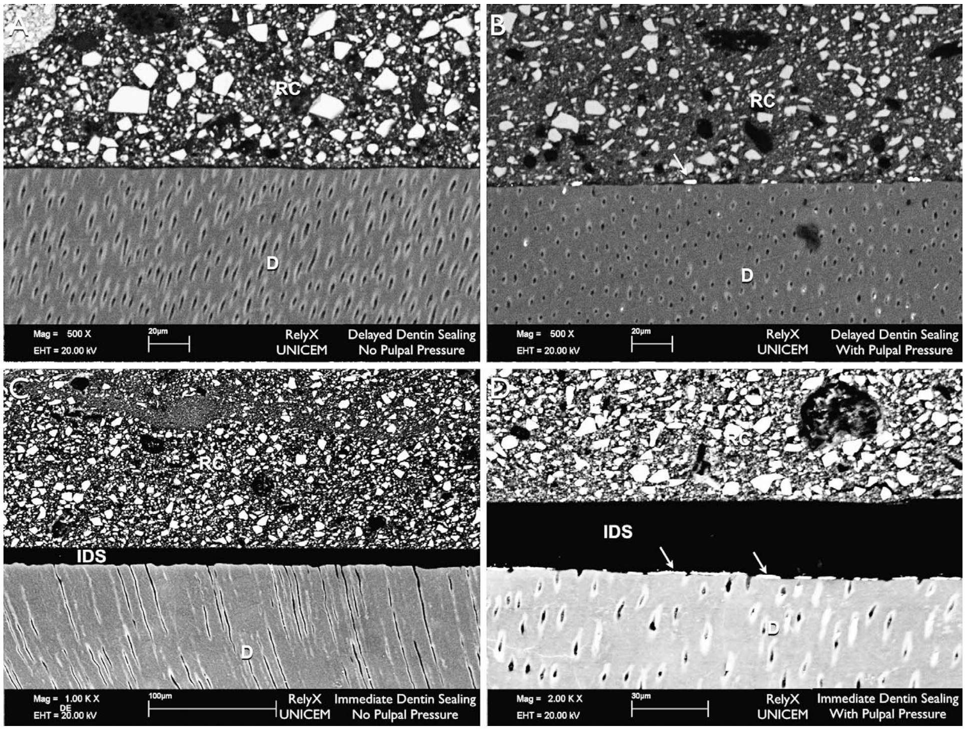


Figure 5. *Representative SEMs of the self-adhesive resin cement RelyX Unicem. (A and B): DDS. (A): NPP. (B): SPP. Almost no silver deposition was observed. (C and D): IDS. (C): NPP. (D): SPP.*

observed for all groups in which IDS was performed (Figure 5C,D).

## DISCUSSION

Intrapulpal pressure has been described as one of the factors able to negatively interfere with dentin adhesion, and lower bond strengths have been consistently produced in the presence of positive intrapulpal pressure.[5,8,9,29] The intrapulpal pressure setup used in the present investigation was designed to simulate the *in vivo* bonding condition using water as dentinal fluid. However, besides water, dentinal fluid contains electrolytes, proteins, and enzymes that can provide a greater challenge to the resin-dentin interfaces produced *in vivo*.[29] The sensitivity of some materials to regional variations in dentin morphology can be responsible for the high standard deviation values observed for some of the tested groups.

Conventional resin cements such as RX that use the phosphoric acid etch-and-rinse technique for dentin pretreatment promote complete smear layer removal. This process increases dentin permeability, allowing a greater flow of dentin fluid within dentinal tubules, due to the presence of positive pulpal pressure.[19,22] In this study, RX exhibited reduced bond strength and increased silver deposition when subjected to SPP and DDS. This result may be related to water diffusion in the hybrid layer, reducing mechanical properties of the polymer matrix.[5,14,30,31]

According to Hiraishi and others,[8] water sorption through the adhesive layer may result in an unstable porous region, making it prone to degradation, rendering this interface a weak point in the presence of pulpal pressure.[32,33] For RX, with SPP, porous regions were observed, suggesting the presence of water channels. Despite the presence of silver deposits, the bond strength values observed for groups restored with the etch-and-rinse system RX were the highest under all studied conditions. Although nanoleakage analysis provides important information on the performance and behavior of resin-dentin interfaces, no correlation between bond strength and nanoleakage has been reported, which may explain these results.[34-37]

Even in the absence of SPP, RX and PF presented a certain amount of nanoleakage. In RX, the nanoleakage pattern produced was probably due to the difficulty in completely eliminating water during the adhesive procedure. In addition to water presence in its composition, a two-step etch-and-rinse adhesive system produces a semipermeable membrane due to its high hydrophilic monomers and solvent concentrations.[22,38]

The self-etching resin cement PF subjected to SPP revealed silver deposition by SEM, suggesting permeability within this system. Although no signif-

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icant difference was detected for PF μTBS values when SPP was applied with DDS, a considerable reduction in bond strength was observed compared with the DDS group applied with no SPP. This performance is probably related to a higher hydrophilic monomer concentration in the ED Primer, resulting in a highly permeable layer after curing.[2,38] Previous studies have reported a negative influence of pulpal pressure to Panavia F.[8,32] However, when IDS was performed, bond strength values increased significantly compared with the groups without the IDS, despite the presence of SPP. The Panavia F failure pattern was predominantly adhesive between resin cement and dentin.

The role of water is crucial in self-adhesive luting agents, because it promotes the release of hydrogen ions necessary for smear layer demineralization.[39] It is believed that the change from hydrophilic to hydrophobic after curing, which occurs in UC, enhances the stability of the interface. When SPP is applied, water coming from the dentinal tubules can increase aggressiveness of acidic monomers by improving smear layer dissolution and dentin demineralization.[8] This fact probably contributed to the increase in bond strength observed for UC when applied to dentin submitted to SPP.

Except for RX, IDS resulted in higher bond strength values for all tested materials either with or without SPP. Furthermore, nanoleakage reduction was observed when IDS was performed for most materials, including RX, both in the presence and absence of SPP. Due to its higher demineralization ability, the hybrid layer formed by Clearfil SE Bond (≈0.5 μm) is thicker than that produced by self-adhesive cements.[5,7,13,30] In addition, the presence of a hydrophobic adhesive layer may contribute to greater interface stability.[5,10,13]

The IDS technique has been proposed in an attempt to improve the quality of bonded interfaces in indirect restorations, in which dentin is hybridized with a two-step self-etch or a three-step etch-and-rinse adhesive system after preparation. This procedure contributes to the occurrence of increased bond strength and reduces the dentin sensitivity during the provisional phase.[23,24,26]

Effective adhesion between an immediate dentin sealing layer and resin cement probably occurs due to the presence of unreacted methacrylate groups still present in the adhesive layer. Thus, a copolymerization between fresh resin cement and adhesive previously applied during the sealing may occur. Failure pattern analysis demonstrated that fractures in IDS groups usually occurred between the luting agent and the sealing, suggesting that even in the case of bond disruption, the dentin still remains sealed. Magne and others[26] reported that resin cement/sealed dentin bonding might occur due to the presence of residual free radicals,[40,41] van der Waals interactions (intermolecular forces), and micromechanical retention.

Therefore, results of the present investigation suggest that the use of the IDS technique is effective in promoting greater bond strength values and reduced nanoleakage patterns in indirect restorative procedures, especially in the presence of SPP. Our null hypotheses were rejected, because IDS, pulpal pressure, and material selection significantly affected bonded interfaces.

## CONCLUSION

SPP decreased the quality of resin-dentin interfaces produced by the multistep resin cements and did not affect the tested self-adhesive materials. However, the IDS technique improved the quality of interfaces of all tested materials, counteracting the negative effects of SPP for the multistep etch-and rinse system RelyX ARC and increasing bond strength values for the self-etching cement Panavia F and the self-adhesive cements RelyX Unicem and Clearfil SA Luting.

### Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of: Guarulhos University Ethics Committee. The approval code for this study is: (SISNEP/384).

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### Conflict of Interest

The authors 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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