# Bond Strength of Two Resin Cements on Dentin Using Different Cementation Strategies

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## ABSTRACT

*Purpose:* This study evaluated the microtensile bond strength of two resin cements to dentin either with their corresponding self-etching adhesives or employing the three-step "etch-and-rinse" technique. The null hypothesis was that the "etch-and-rinse" adhesive system would generate higher bond strengths than the self-etching adhesives.

*Materials and Methods:* Thirty-two human molars were randomly divided into four groups ($N = 32$, $n = 8$/per group): G1) ED Primer self-etching adhesive + Panavia F; G2) All-Bond 2 "etch-and-rinse" adhesive + Panavia F; G3) Multilink primer A/B self-etching adhesive + Multilink resin cement; G4) All-Bond 2 + Multilink. After cementation of composite resin blocks ($5 \times 5 \times 4$ mm), the specimens were stored in water (37°C, 24 hours), and sectioned to obtain beams ($\pm 1$ mm$^2$ of adhesive area) to be submitted to microtensile test. The data were analyzed using 2-way analysis of variance and Tukey's test ($\alpha = 0.05$).

*Results:* Although the cement type did not significantly affect the results ($p = 0.35$), a significant effect of the adhesive system ($p = 0.0001$) was found on the bond strength results. Interaction terms were not significant ($p = 0.88751$). The "etch-and-rinse" adhesive provided significantly higher bond strength values (MPa) with both resin cements (G2: $34.4 \pm 10.6$; G4: $33.0 \pm 8.9$) compared to the self-etching adhesive systems (G1: $19.8 \pm 6.6$; G3: $17.8 \pm 7.2$) ($p < 0.0001$). Pretest failures were more frequent in the groups where self-etching systems were used.

*Conclusion:* Although the cement type did not affect the results, there was a significant effect of changing the bonding strategy. The use of the three-step "etch-and-rinse" adhesive resulted in significantly higher bond strength for both resin cements on dentin.

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CLINICAL SIGNIFICANCE

Dual polymerized resin cements tested could deliver higher bond strength to dentin in combination with "etch-and-rinse" adhesive systems as opposed to their use in combination with self-etching adhesives.

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## INTRODUCTION

Current trends in dental research tend to simplify the clinical procedures, especially for the applications of adhesives not only in restorative dentistry for direct applications of resin-based materials but also in prosthetic dentistry in adhesive cementation of fixed partial dentures (FPDs). Advances in these procedures primarily include the development of simplified bonding systems, when compared to the classical three-step systems.[1] On the other hand, traditional "etch-and-rinse" adhesive systems rely on the application of adhesive monomers to acid-etched dentin or the use of simplified self-etching, self-priming agents that contain hydrophilic and acidic monomers, acidic molecules, diluent monomers, photoinitiators, and solvents (usually at low pH). Such adhesives simultaneously etch the dentin and infiltrate the adhesive monomers into the dentin.[1–3]

Different bonding strategies may affect the coupling of resin cements to dentin. The application of simplified adhesive systems in combination with resin cements has not been widely studied.[4] In fact, long-term survival of FPDs may be very much dependent on the function of the resin cement that adheres both to the restorative materials and the tooth substance.[5] The reliable adhesion of the resin cements to both the dentin and the ceramic becomes particularly important in the application of glassy matrix all-ceramics or surface-retained resin-bonded FPDs where mechanical retention does not dominate. Also, when enamel margins do not exist after tooth preparation as a consequence of caries removal or tissue loss due to trauma, adhesion of the cement is dictated by dentin conditioning through etching and application of the bonding agents. Previous studies have shown that self-etch adhesives may result in lower bond strength to dentin.[6,7] In addition, due to the acidic nature of the self-etch adhesives and their permeability, their adhesion to resin-based cements are compromised.[8] Since etching dentin separately would contribute to dentin bonding[9] and due to their lesser hydrophilicity,[8] it can be anticipated that "etch-and-rinse" adhesive systems compensate for other factors and lead to higher bond strength of the resin cements to dentin. Therefore, this study aimed to evaluate the microtensile bond strength (μTBS) of two dual-polymerized resin cements to dentin either using their corresponding self-etching primers or with an "etch-and-rinse" system. Thus, the null hypothesis tested was that there is no effect of bonding protocol in the bond strength results.

## MATERIALS AND METHODS

After approval of the university institutional ethical review board (São Paulo State University, São Jose dos Campos Dental School, Brazil), the teeth were selected from a pool of extracted human third molars ($N = 32$). The teeth were extracted at the surgery department 3 months prior to the study. They were cleaned and stored in distilled water with 0.1% thymol solution at room temperature up to 3 months. Then the root portions of the teeth were embedded in the polyethylene rings with autopolymerized acrylic resin (Condular AG, Wager, Switzerland). The occlusal enamel of the teeth was removed using wet 320-grit silicon carbide abrasive papers

(Buehler, Lake Bluff, IL, USA) in order to expose the mid-coronal dentin surface. Smear layer was created on the dentin surface by using wet 600-grit silicon carbide abrasive papers (Buehler) for 60 seconds. The teeth were then randomly divided into four testing groups ($n = 8$/per group):

1. G1: Dentin was treated with the self-etching ED primer and resin cement Panavia F was applied,
2. G2: Dentin was treated with "etch-and-rinse" system (All Bond-2) and resin cement Panavia F was applied,
3. G3: Dentin was treated with self-etching Multilink primer A/B and resin cement Multilink was applied,
4. G4: Dentin was treated with "etch-and-rinse" system (All Bond-2) and resin cement Multilink was applied.

Commercial names, manufacturers, compositions of the materials, and manufacturer's recommended protocol of the materials investigated are presented in Table 1.

Resin composite blocks (Filtek Z250, 3M ESPE, St. Paul, MN, USA) with dimensions of $5 \times 5 \times 6$ mm were obtained by incremental buildup and light polymerized (XL3000, 3M ESPE, light output: 550 mW/cm$^2$) in a silicone matrix (Zetaplus, Zhermack, Badia Polesine, Italy) according to the manufacturer's instructions. The surface of composite blocks to be bonded to the dentin was sandblasted (Micro-Etcher, Danville Inc., San Ramon, CA, USA) with 50 µm aluminum oxide particles for 5 seconds and air-dried at 2.9 bar pressure.

The resin cements were mixed according to each manufacturer's recommendations and placed on the composite blocks. The resin blocks were then luted on the respective bonded dentin surfaces under 750 g load for 1 minute. The excess cement was removed from the margins with hand instruments and light polymerized for 40 seconds from each side. Oxygen blocking gel (Oxyguard, Kuraray) was applied and left for 5 minutes on all bonded interfaces. The composite-cement-dentin assemblies were washed with air-water spray and stored in distilled water for 24 hours at 37°C.

**Production of Non-trimmed Beam Specimens**

The specimens were bonded with cyanoacrylate glue (Super Bonder Gel, Loctite Ltd, São Paulo, Brazil) to a metal base that was attached to a cutting machine. The dentin surfaces were positioned perpendicular to the diamond disk. The specimens were sectioned in *x* and *y* direction using a slow-speed diamond disk (# 34570) (Microdont, São Paulo, Brazil) under water cooling. The peripheral slices (0.5 mm) were eliminated to avoid possible specimens with excess, insufficient amount of resin cement, or irregularities at the interface. Thus, nontrimmed beams with adhesive area of $1 \pm 0.1$ mm$^2$ and length of $10 \pm 0.1$ mm were produced from the test specimens.

**µTBS Test**

The ends of each beam specimen were fixed with cyanoacrylate adhesive (Super Bonder, Henkel Loctite Adhesives Ltd, Itapevi, SP, Brazil) to the aligned jig of the Universal Testing Machine (EMIC DL-1000, EMIC, São José dos Pinhais, PR, Brazil). µTBS tests were then performed (cross-head speed: 1 mm/min). The bond strength $\sigma$ (MPa) was calculated according to the formula $\sigma = L/A$, where $L$ is the load for rupture of specimen (N) and $A$ is the interfacial area (mm$^2$) measured with a digital caliper prior to testing.

Statistical analysis was performed using SAS System for Windows, release 8.02/2001 (Cary, NC, USA). The µTBS data were analyzed using two-way analysis of variance and Tukey's test with the adhesives and the resin cement types as independent and bond strength values as dependent variables ($\alpha = 0.05$).

## RESULTS

The number of beams stressed to failure in the groups formed using

TABLE 1. BRANDS, MANUFACTURERS, COMPOSITIONS OF THE MATERIALS, AND MANUFACTURERS' RECOMMENDED PROTOCOL FOR THE TWO RESIN CEMENTS AND THE THREE ADHESIVE SYSTEMS INVESTIGATED.

| Adhesive system/manufacturer | Composition | Manufacturers' recommended protocol |
| --- | --- | --- |
| ED Primer A (Kuraray Medical, Inc. Okayama, Japan) | MDP, HEMA, water, MASA, DEPT | Mix equal amounts of Primer A and B. Apply the liquid and leave undisturbed for 30 seconds before thinning by a stream of air. |
| ED Primer B (Kuraray) | DEPT, Na-benzene sulfinate, MASA, water | |
| Panavia F, cement catalyst paste (Kuraray) | Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, MDP, filler, BPO | Apply mixed cement paste, cover with glycerine gel. |
| Panavia F, cement universal paste (Kuraray) [Batch#:00022E] | Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophobic dimethacrylate, filler, DEPT, sodium aromatic sulfinate | |
| Multilink Primer A (Ivoclar Vivadent, Schaan, Liechtenstein) | DEPT, Na-benzene sulfinate, water | Mix equal amounts of Primer A and B. Apply the liquid and agitate the primer on the surface for 15 seconds before thinning by a stream of air. |
| Multilink Primer B (Ivoclar Vivadent) | HEMA, EAEPA, MAEPA | |
| Multilink Cement Catalyst (Ivoclar Vivadent) | HEMA, dimethacrylates, filler, BPO | Apply mixed cement paste, cover with glycerine gel. |
| Multilink Cement Base (Ivoclar Vivadent) | HEMA, dimethacrylates, filler, t-amine | |
| Phosphoric acid All Bond 2 (Bisco, IL, USA) [Batch#:03000877] | 32% phosphoric acid | Etch with 32% phosphoric acid for 15 seconds, rinse with water, dry with absorbent paper. |
| Primer A All-Bond 2 (Bisco) | Acetone, ethanol, NTG-GMA | Mix equal amounts of Primer A and B and leave undisturbed for 10 seconds before thinning by a stream of air. Apply "Pre-Bond resin." |
| Primer B All-Bond 2 (Bisco) | Acetone, Ethanol, BDPM | |
| Pre-Bond resin All-Bond 2 (Bisco) [Batch#:03000014662] | Bis-GMA, TEGDMA, BPO | |

BDPM = biphenyl dimethacrylate; Bis-GMA = Bisphenol A diglycidylmethacrylate; BPO = dibenzoyl peroxide; DEPT = N,N-di(2-hydroxyethyl) p-toluidine; EAEPA = ethyl 2-[4-(dihydroxyphosphoryl)-2-oxabutyl]acrylate; HEMA = 2-hydroxyethyl methacrylate; MAEPA = 2, 4, 6 trimethylphenyl 2-[4-(dihydroxyphosphoryl)-2oxabutyl]acrylate; MASA = N-methacryloyl 5-aminosalicylic acid; MDP = 10-methacryloyloxydecyl hydrogen phosphate; NTG-GMA = Na-N-tolylglycine glycidylmethacrylate; TEGDMA = triethyleneglycol dimethacrylate.

the All-Bond 2 system was higher than that of the groups using the ED primer, which reflects premature failures that occurred in the latter groups. Initially, a uniform number of beams per tooth and group were not expected, not only because the bonding agents had different natures but also because one bonding agent might not uniformly interact with dentin in the same tooth. In total, eight beams were obtained per tooth from both G2 ($n = 64$) and G4 ($n = 64$) etch-and-rinse adhesive-containing systems without any pretest failures. From the obtained beams randomly chosen, 32 beams were subjected to microtensile testing. On the other hand, approximately three beams were obtained per

**TABLE 2. MEANS AND STANDARD DEVIATIONS OF THE µTBS DATA (MPA) FOR THE EXPERIMENTAL GROUPS.**

| Adhesive system | Resin cements* | |
|---|---|---|
| | Panavia F | Multilink |
| Self-etching adhesives | G1: 19.8 ± 6.6[b] ($n = 23$) | G3: 17.8 ± 7.2[b] ($n = 22$) |
| Total etching adhesive | G2: 34.4 ± 10.6[a] ($n = 32$) | G4: 33.0 ± 8.9[a] ($n = 32$) |

G1: ED-Primer self-etching adhesive + Panavia F; G2: All-Bond 2 "etch-and-rinse" adhesive + Panavia F; G3: Multilink primer A/B self-etching adhesive + Multilink resin cement; G4: All-Bond 2 + Multilink.

*Same letters indicate no significant differences (Tukey's test, $\alpha = 0.05$).

tooth in G1 ($n = 23$) and G3 ($n = 22$) self-etch adhesive-containing groups, with more than 50% pretest failures. Finally, the number of beams tested to failure ($n$) in each group was as follows: G1, $n = 23$; G2, $n = 32$; G3, $n = 22$; and G4, $n = 32$. No failures were experienced in the glue used for the attachment of the beam specimens to the jig of the testing device.

Although the cement type did not significantly affect the µTBS results ($p = 0.35$), a significant effect of the adhesive system ($p = 0.00001$) was found on the bond strengths. Interaction terms were not significant ($p = 0.88751$). Table 2 displays the mean µTBS and standard deviations of the testing groups.

Both cements when used in combination with the "etch-and-rinse" system showed significantly higher bond strength values (MPa) (G2: 34.4 ± 10.6; G4: 33 ± 8.9) compared to their combination with self-etching adhesive systems (G1: 19.8 ± 6.6; G3: 17.8 ± 7.2) (Tukey's test) ($p < 0.0001$).

## DISCUSSION

The simplicity of the application procedure of self-etching, self-priming adhesive systems is appealing for the clinicians. Therefore, several manufacturers have developed their own systems and indicated their product for use with any resin cements. The present study showed that the "etch-and-rinse" adhesive system resulted in higher bond strengths to dentin than those of the self-etching adhesive when used in combination with two dual-polymerized cements.

Some studies have previously indicated a possible chemical incompatibility between the adhesive systems with low pH and resinous materials.[6–8,10,11] This incompatibility occurs when chemically activated or dual-activated resins are placed in contact with the acidic monomer components of simplified adhesive systems ("etch-and-rinse" or self-etch) or indirectly by drawing water from dentin, resulting in delayed polymerization and/or hydrolysis of the interface, and ultimately compromising the bond between the adhesive and the resin cement.[7,8,12–15] Carvalho and colleagues[4] applied an additional coat of experimental non-acidic hydrophobic resin, after a self-etching primer prior to application of Panavia, and noted that the bond strength was improved significantly by 35%. The application of this adhesive impaired the acidic and hydrophilic nature of the self-etching primer, preventing the contact of dual-polymerized resin cement with the adhesive layer and decreasing its permeability to water from the dentin tubules.[10–15]

In this study, the greatest possible advantage of the investigated "etch-and-rinse" adhesive system in comparison to the self-etching system was the application of the phosphoric acid. Demineralization of the dentin substrate and penetration of the resin monomers create micromechanical retention that surely contributes to the overall adhesion to a great extent.[1–3] Self-etching primers act on the dentin surface only by modifying the smear layer and dissolving the smear plugs within the dentin tubules.[3,16,17] However, the "etch-and-rinse" adhesives require a moist substrate for optimal bonding,[4] making it highly sensitive, meaning that the collapse of over-dried, exposed collagen acts as a difficult substrate for the monomer infiltration. However, this also indicates that, in the case of increased dentin wetness in deeper dentin, an overly wet condition may be created.[18] This makes three-step "etch-and-rinse" adhesives more technique sensitive compared to self-etch adhesives. That, however, did not affect our findings, as the operator was careful in maintaining ideal bonding conditions for the adhesive. Thus, the results of our study suggest that, when moist technique is meticulously followed, mid-coronal dentin is a favorable substrate for the "etch-and-rinse" system used. The results may vary in deep dentin substrate. Although employing "etch-and-rinse" adhesives combined with conventional luting resin composites seems to be a better protocol for adhesive cementation, morphological characteristics and variations in dentin should also be taken into consideration when assessing dentin bond strengths.[19,20]

Our findings also suggest that the particular combination of All-Bond 2 with Panavia and Multilink might be acceptable for use. This does not imply that all combinations of three-step "etch-and-rinse" adhesives with resin cements would result in improved bond strengths. There might be chemical peculiarities of individual adhesives and cements that make them incompatible, and thus, this must be taken into account when interpreting our findings. Based on the outcomes of this study, including the high incidence of pretest failures, the simplification of adhesive cementation should be questioned. The data obtained in this study resulted in failure to reject the null hypothesis.

After debonding, it was not possible to identify the adhesive failures between the adhesive and the cement layer under the optical microscope. We are aware that the mode of failure analysis provides important information leading to predictions of clinical performance limits[21] and that the lack of proper failure analysis is a limitation of the present study.

Although clinically ideal bond strength is not known to date, the difference between the two adhesive cementation approaches was significant within the limitations of this study. This, together with the high pretest failure rate with self-etching adhesives, puts the use of simplified techniques in question when using adhesive cements for luting purposes. Further research is needed on the stability of such bonded interfaces.

## CONCLUSIONS

Both dual-polymerized resin cements tested in combination with self-etching adhesive systems resulted in lower bond strengths to dentin when compared to their use with a three-step "etch-and-rinse" adhesive system.

## DISCLOSURE

The authors do not have any financial interest in the companies whose materials are included in this article.

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