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Effect of Surface Preparation on Bond Strength of Resin Luting Cements to Dentin

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Clinical Relevance

For Panavia F2.0, higher bond strengths are achieved on dentin surfaces prepared with tungsten carbide bur. Proper bur selection is essential to optimize dentin adhesion of self-etch resin luting cements.

SUMMARY

This study examined the effects of using two different burs for dentin surface preparation on the microtensile bond strength (µTBS) of three resin luting cements. Flat, deep dentin surfaces from 45 extracted human third molars were divided into three groups (n=15) according to bur type: (i) diamond bur and (ii) tungsten carbide bur. The controls were abraded with #600-grit SiC paper. Both burs operated in a high-speed handpiece under water-cooling. Composite blocks were luted onto the dentin using one of three cements: RelyX ARC (ARC, 3M ESPE), Panavia F2.0 (PF, Kuraray) and RelyX Unicem (UN, 3M ESPE) following the manufacturers' instructions. For ARC, the dentin surface was treated with 32% phosphoric acid. The bonded specimens were stored at 37°C for 24 hours and sectioned into 0.9 x 0.9 mm beams for µTBS testing. The data were analyzed using the two-way ANOVA and Student-Newman-Keuls tests. Representative fractured beams from each group were prepared for fractographic analysis under SEM. Two-way ANOVA revealed that the effects of "dentin surface preparation" and "luting cement" were statistically significant (*p*<0.001); however, the interaction of these two factors was not significant (*p*>0.05). ARC showed no significant difference in µTBS among the three differently prepared dentin surfaces. The µTBS of PF and UN was significantly lower when bonding to dentin prepared with a

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diamond bur (p<0.05), compared to the control. For Panavia F2.0, higher bond strengths were achieved on the dentin surface prepared with a tungsten carbide bur. Proper bur selection is essential to optimizing the dentin adhesion of self-etch resin luting cements.

INTRODUCTION

The adhesion of restorative materials to enamel has become a routine and reliable aspect of modern restorative dentistry, but dentin adhesion has proved to be more difficult and less predictable, due to its complex histological structure and variable composition. Dentin bonding is further complicated by the formation of a smear layer, which is created when dentin is cut or ground. The smear layer is a mixture of enamel, dentin or cementum that is usually contaminated with blood, saliva or micro-organisms.[1] Although the smear layer acts as a "diffusion barrier," which decreases the permeability of dentin by 86% in dogs,[2] it can also be considered an impediment for proper resin infiltration into underlying dentin. Since the smear layer interacts poorly with the prepared dentin surface, it must be removed, modified or impregnated for optimal bonding between the restorative material and dentin.[3-4]

Two main approaches have been used to produce an effective bond between direct restorations and dentin.[5] The etch-and-rinse adhesive system employs phosphoric acid to remove the smear layer, followed by primer adhesive applications, while the self-etch adhesive system utilizes acidic monomers to modify the smear layer. Several studies have shown that differences in the smear layer thickness, as generated by burs and abrasive papers, affected the bond strength of self-etch adhesives to dentin,[6-11] while other investigators have reported the contrary.[12-15]

Resin cements used to bond indirect restorations to tooth surfaces are classified into two categories: those that employ etch-and-rinse and self-etch adhesives based upon the need for pretreatment of the bonding substrate.[16-17] More recently, self-adhesive resin cements that eliminate the pretreatment of dentin have been developed to simplify the bonding procedure. These self-etch and self-adhesive cements claim to be less technique sensitive, as they reduce luting procedures and are less likely to cause postoperative sensitivity, because the smear layer and smear plugs are not removed. However, as a result of their weak acidity, it is not clear whether the bond strength of these self-etch and self-adhesive resin cements to dentin may be affected by the quantity and/or quality of the smear layer.

With the growing popularity of the use of indirect restorations, it is of clinical importance to determine the effect of different burs on the bond strength of resin-luting cements to dentin. To date, a limited number of studies have evaluated the bond strength of resin-luting cements to dentin prepared by different dental burs. Therefore, the current study evaluated the effect of cutting dentin with diamond and tungsten carbide burs at high speed on the microtensile bond strength (µTBS) of three resin-luting cements to dentin. The null hypothesis tested was that different dentin surface preparations had no effect on the microtensile bond strength of three resin-luting cements to dentin.

METHODS AND MATERIALS

Tooth Preparation

Forty-five caries-free human third molars stored in 0.5% Chloramine T solution at 4°C were used within one month following extraction. A flat dentin surface was created perpendicular to the long axis of the tooth, using a slow-speed saw with a diamond-impregnated disk (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under copious water lubrication.

Experimental Design

The teeth were randomly assigned to three groups, with 15 teeth in each group, according to the dentin surface preparation.

Group A: The dentin surface was prepared using flat-end tapered medium diamond burs (Hi-Di, #556, Dentsply Ash Instruments, Surrey, UK).

Group B: The dentin surface was prepared using plain-cut tapered-fissure tungsten carbide burs (Ash Size 2, Dentsply Ash Instruments).

Group C: The dentin surface was abraded with #600-grit SiC paper and served as the control group.

For groups A and B, the burs were mounted in an air turbine handpiece (Synea HS, W & H Dentalwerk Burmoos GmbH, Bürmoos, Austria) and run at 200,000 rpm under water-cooling. The RPM in the air turbine handpiece was constantly monitored using a handheld digital tachometer (HR-6800 Digital Tachometer, Ono Sokki, Yokohama, Japan).

The same operator conducted all the dentin surface preparations by gently passing the burs 20 times across the dentin under copious water spray. For control Group C, the surfaces were abraded with #600-grit SiC paper for 15 seconds under running water.

Cylindrical Composite Block Preparation

A heat- and light-activated hybrid resin composite (Estenia C&B, Kuraray Medical Inc, Tokyo, Japan) was used for the experiment. The cylindrical composite blocks were prepared following the protocol of Hiraishi and others.[18] Layers of composite were dispensed into flat Teflon molds (5 mm thick and 10 mm in diameter). The uncured composites were initially light-cured using a quartz-tungsten-halogen light-curing unit

(Optilux 500, Demetron Research Corporation, Danbury, CT, USA) operated at 600 mW/cm^2. To improve polymerization, the cylindrical composite blocks were then placed inside a composite inlay-processing chamber (Dentacolor XS, Heraeus Kulzer GmbH & Co KG, Wehrheim, Germany) and heat-cured at 100-110°C for five minutes. The bonding surface of each composite was ground with #180-grit SiC paper to create a roughened surface, followed by etching with 32% phosphoric acid gel (Uni-Etch, BISCO, Inc, Schaumburg, IL, USA) and rinsed for 10 seconds. A mixture of Clearfil SE Primer and Porcelain Bond Activator (Kuraray Medical Inc) was applied for five seconds on the bonding surface of the composite and dried.

Cementation of Indirect Composite Blocks

Each dentin surface preparation group was further divided into three subgroups (n=5) based on the luting cement used. Three resin cement systems were used in this study: RelyX ARC (ARC, 3M ESPE, St Paul, MN, USA), Panavia F2.0 (PF, Kuraray Medical Inc) and RelyX Unicem (UN, 3M ESPE). All the materials were used according to the manufacturers' instructions (Table 1). The use of three dentin surface preparations and three luting cements resulted in nine subgroups with five teeth in each.

After the resin cement was applied on the dentin surface, the composite block was placed under a constant seating pressure of 3.0 kg, which was maintained for three minutes. The seating pressure was applied to the composite block by means of a plunger that was loaded by a box filled with lead pellets according to Chieff and others.[19] The weight of the lead pellet-filled box was adjusted based on the bonding surface area of the specimen to obtain the standard seating pressure of 3kg. The selection of 3.0 kg was based on the findings of previous studies,[20-21] in which a greater seating force enhanced interfacial adaptation and subsequently improved the bonding strength of resin cements. For Panavia F2.0, Oxyguard II was liberally applied around the resin cement to ensure complete anaerobic polymerization. Light-curing was then performed from four parallel directions for 20 seconds each along the cement interface using an Optilux 500 light-curing unit at 600 mW/cm^2. The output of the light-curing unit was assessed using a commercial hand held halogen-based radiometer (Demetron 100, SDS/Kerr, Orange, CA, USA).

Microtensile Bond Strength Test

After storage in distilled water at 37°C for 24 hours, the luted teeth were sectioned occluso-gingivally into 0.9 x 0.9 mm composite-dentin beams, according to the "non-trimming" technique of the microtensile test.[22] Eight beams were retrieved from the two widest slabs of each tooth. Five teeth from each group yielded 40 beams for bond strength evaluation. Each beam was attached to the test apparatus with a cyanoacrylate adhesive and stressed to failure under tension in a Bencor Multi-T device (Danville Engineering, San Ramon, CA, USA) using a universal testing machine, Model 4440 (Instron, Inc, Canton, MA, USA) at a crosshead speed of 1 mm per minute. Any beams that failed during specimen preparation were recorded as null bond strength, and those values were included in the statistical analy-

Table 1: *Composition and Application Techniques of the Tested Luting Cements*

Luting Cement	Dentin Treatment	Composition
Rely X ARC (ARC)	1. Etch with phosphoric acid	Adper Single Bond 2 (Lot: 6JF): Ethyl alcohol, Bis-GMA, silane-treated silica, HEMA, glycerol 1,3-dimethacrylate, copolymer of acrylic and itaconic acids, diurethane dimethacrylate, water
Two-step etch-and-rinse luting cement	2. Apply Adper Single Bond 2 and light cure	Dual-cured-filled resin cement (Lot:EYGH): Bis-GMA, TEDGMA, zirconia filler, silica
Panavia F 2.0 (PF)	1. Apply ED primer for 30 seconds and gently air-dry	ED primer 2.0 A (Lot: 00226A): HEMA, 10-MDP, 5-NMSA, water, accelerator
One-step self-etch luting cement		ED primer 2.0 B (Lot: 00105A): 5-NMSA, accelerator, water, sodium benzene sulfinate
		Paste A (Lot: 00239A): 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, benzoyl peroxide
		Paste B (Lot: 00128A): hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass
Rely X Unicem Aplicap (UN)	No pretreatment	Powder (Lot: 262075): glass powder, silica, calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator
Self-adhesive luting cement		Liquid: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizor, initiator

Abbreviations: Bis-GMA: bisphenol A diglycidyl ether dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; 10-MDP: 10- methacryoloyloxydecyl dihydrogen phosphate; 5-NMSA: N-methacryloxyl-5-aminosalicylic acid.
Note: The brand name of Adper Single Bond 2 is used in Latin America and Oceania, while Adper Scotchbond 1 XT is used in Europe, Adper Single Bond Plus in the USA and Adper Single Bond 1XT in South Africa.

sis. After testing, the cross-sectional area of each beam was measured using a pair of digital calipers (Model CD-6BS, Mitutoyo, Tokyo, Japan) and the microtensile bond strength was calculated.

The fractured surfaces were examined using a scanning electron microscope (SEM) to determine the failure mode. The failures were classified according to Hiraishi and others[18] as: 1) adhesive failure along the cement-dentin interface, 2) adhesive failure along the cement-composite interface, 3) cohesive failure within resin cement, 4) mixed failure of 1 and 3 and 5) mixed failure of 2 and 3. Representative fractured beams from each subgroup with µTBS close to the mean bond strength of that group were selected for fractographic analysis by SEM. The fractured sides of the specimens were air-dried, sputter-coated with gold/palladium and examined using SEM (FEI Quanta 200 3D, FEI Company, Hillsboro, OR, USA) operating at 10-20kV.

Scanning Electron Microscopy Evaluation of Treated Dentin Surfaces

Approximately 1-mm thick dentin disks were prepared from the mid-coronal dentin of 20 extracted human third molars by slow-speed Isomet saw under water cooling. For SEM evaluation of the treated surface, the dentin side of 10 disks was prepared with a diamond bur, and the remaining disks were prepared with a tungsten carbide bur. For each group, the dentin surface was further treated with either ED primer or Unicem without light curing. ED primer 2.0 was applied onto the smear layer-covered surface for 30 seconds. A thin layer of RelyX Unicem (ca 1.0 mm) was placed on the smear-layer-covered dentin with light pressure (40 g/mm^2) and remained for one minute. The disks were immediately soaked in 100% acetone for five minutes to remove the applied adhesive or cement. Removal of RelyX Unicem was enhanced using a shaker during immersion in acetone. All of the disks were dehydrated in ascending concentrations of ethanol, followed by immersion in HMDS (hexamethyldisilazane) for 10 minutes. The disks were then placed on filter paper inside a covered glass vial and air-dried at room temperature.[23] They were then mounted on aluminum stubs and sputter-coated with gold/palladium prior to examination with SEM (Cambridge Stereoscan 440, Leica, Cambridge, UK) operating at 10-20 kV.

Statistical Analysis

The bond strength data were analyzed using a statistical software package (SigmaStat Version 2.03, SPSS, Chicago, IL, USA). As the normality (Kolmogorow-Smirnoff test) and homoscedasticity assumptions (Levene test) of the data appeared to be valid, two-way ANOVA was used to examine the effects of "dentin surface preparation" and "luting cement," along with the interaction of these two variables on microtensile bond strength. The total number of tested beams in each group was used in the statistical analysis, with each beam taken as an independent specimen. Multiple comparisons were carried out using the Bonferroni and Student-Newman-Keuls tests, with the statistical significance set at α=0.05. A Pearson Chi Square test was used to examine the association among the three types of cement with the three types of dentin preparation under the five different failure modes, with the statistical significance set at $p<0.05$.

RESULTS

Microtensile Bond Strength

The mean µTBS and standard deviations of resin-luting cements to dentin after different surface preparations are given in Table 2. Two-way ANOVA revealed that significant differences were observed for the factors "dentin surface preparation" ($p<0.001$) and "luting cement" ($p<0.001$). However, the interaction of these two factors was not significant ($p>0.05$), indicating that the bond strength results of the luting cements were not dependent on the dentin surface preparation (Table 3). For the ARC groups, no significant difference ($p>0.05$) was found among the dentin surfaces prepared with #600-grit SiC paper (20.7 ± 3.7 MPa), diamond (18.8 ± 3.2 MPa) or tungsten carbide (20.9 ± 5.5 MPa) burs. The µTBS of PF to dentin cut with a diamond bur (14.8 ± 3.4 MPa) was significantly lower ($p<0.001$) than to dentin prepared with a tungsten carbide bur (19.0 ± 4.9 MPa) and #600-grit SiC paper (20.6 ± 4.3 MPa). Similarly, for UN, the bond strength to dentin prepared with a diamond bur (15.6 ± 4.1 MPa) was significantly lower ($p<0.05$) than that prepared with #600-grit SiC paper (18.9 ± 5.0 MPa). The µTBS of ARC (18.8±3.2 MPa) to dentin prepared with a diamond bur was significantly higher than that of PF (14.8 ± 3.4 MPa) and UN (15.6 ± 4.1 MPa) ($p<0.05$).

Table 2: *Microtensile Bond Strength of Resin Luting Cements to Dentin After Different Surface Preparations*

	Microtensile Bond Strength (MPa)*		
Cement	#600 Grit SiC Paper	Diamond	Tungsten Carbide
ARC	20.7 ± 3.7 (40)^{A}	18.8 ± 3.2 (40)^{A}	20.9 ± 5.5 (40)^{A}
PF	20.6 ± 4.3 (40)^{A}	14.8 ± 3.4 (40)^{B}	19.0 ± 4.9 (40)^{A}
UN	18.9 ± 5.0 (40)^{A}	15.6 ± 4.1 (40)^{B}	17.8 ± 4.0 (40)^{A,B}

Values are means ± standard deviations. The number of specimens tested is included in parentheses. Groups identified with different superscripts indicate statistically significant differences (p<0.05).

Table 3: *Summary of the Results of Two-way ANOVA*

Source of Variation	Sum of Variation	Df	Mean Square	F-ratio	p
Corrected Model	1528.539	8	191.067	10.407	0.0000
Cements	480.295	2	240.147	13.081	0.0000
Dentin Surface Preparations	878.575	2	439.287	23.927	0.0000
Cements x Dentin Surface Preparations	169.669	4	42.417	2.310	0.0575
Error	6444.074	351	18.359		

Scanning Electron Microscopy Examination of Treated Dentin Surfaces

SEM micrographs of the prepared dentin surfaces treated with ED primer are shown in Figures 1a and 1b, while those treated with Unicem are shown in Figures 1c and 1d.

The thick smear layer produced by a diamond bur was unevenly removed with ED primer. The majority of the tubules remained occluded with residual smear plugs (Figure 1a). In contrast, when applied to the dentin surface prepared by tungsten carbide bur, the smear layer and smear plugs were completely dissolved. Mild etching of the peritubular dentin was observed (Figure 1b). Similarly, the thick smear layer produced by diamond bur was incompletely removed by RelyX Unicem. Furthermore, varying thicknesses of the smear layer could be observed on the treated dentin surfaces (Figure 1c). When RelyX Unicem was applied to the dentin surface prepared with a tungsten carbide bur, smear layer removal was more complete, but orifices of the tubules remained occluded with smear plugs (Figure 1d).

Examination of Fractured Interfaces

The failure modes are summarized in Table 4. Morphological differences were observed among the

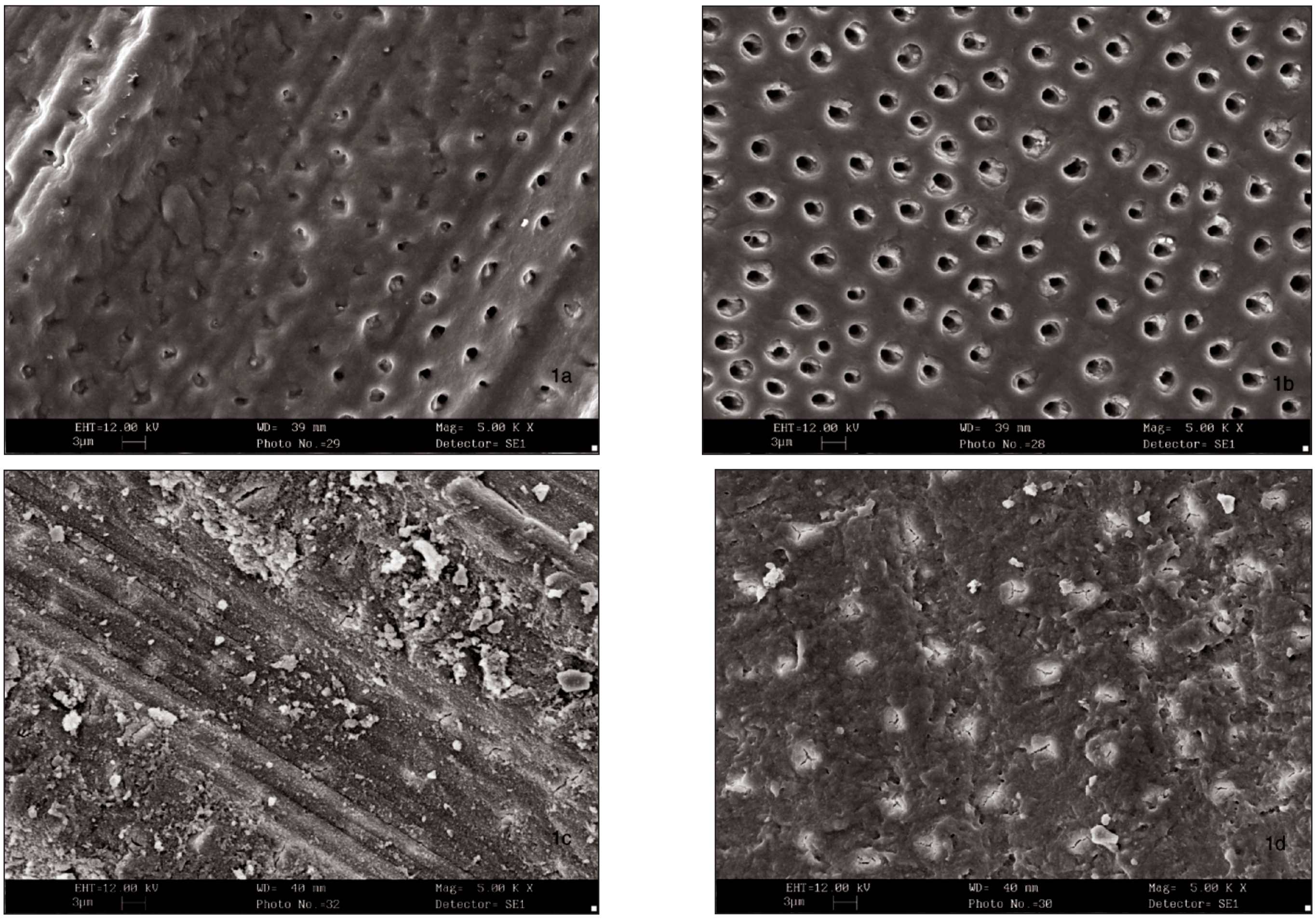

Figure 1. *SEM micrograph of the dentin surface (Figure 1a) prepared with a diamond bur and conditioned with ED primer; (Figure 1b) prepared with carbide bur and conditioned with ED primer; (Figure 1c) prepared with diamond bur and conditioned with RelyX Unicem and (Figure 1d) prepared with carbide bur and conditioned with RelyX Unicem. (Original magnification = 5000x)*

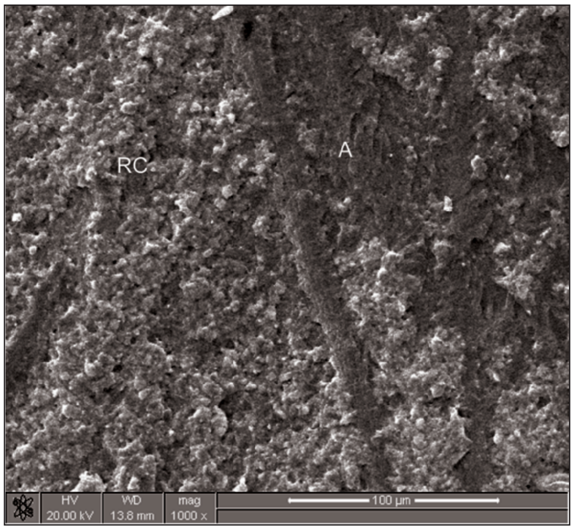

Figure 2. *Representative SEM micrographs of the dentin side of a fractured beam from the ARC-tungsten carbide group. Mixed failure involved adhesive failure at the composite-cement interface and cohesive failure between the adhesive and cement. The resin cement was found laying on top of the fractured adhesives. RC: fractured resin cement; A: fractured adhesive. (Original magnification = 1000x)*

fractured interfaces of the specimens in all three cement groups. SEM micrographs of the dentin sides of representative fractured beams from the ARC, PF and UN groups are shown in Figures 2-4. In the ARC groups, the predominant mode of failure was adhesive between composite and cement and cohesive between cement and adhesive. No adhesive failure along the dentin interface was observed. Since the fracture patterns were similar in all ARC groups, irrespective of the type of dentin surface preparation, only the representative SEM micrograph ARC-tungsten carbide group is shown. A high magnification of the dentin surface revealed fractured resin cement lying on top of the fractured adhesives (Figure 2).

For the PF-SiC paper, PF-diamond and PF-tungsten carbide groups, the predominant mode of failure was adhesive along the dentin surface. High magnification views from the PF-SiC paper and PF-tungsten carbide groups were similar and revealed hybridized dentin with dentinal tubules occluded by resin tags. Circular, rosette-like fracture patterns, originating from a patent dentinal tubule, were identified on the dentin surface (Figure 3a). The dentin surface from the PF-diamond group was irregular and traversed by deep, wide grooves. The hybridized smear layer was observed at the top of the grooves formed by the diamond bur. Areas of incompletely infiltrated dentinal tubules were found at the bottom of the grooves (Figure 3b).

For the UN-SiC paper, UN-tungsten carbide and UN-diamond groups, the mode of failure was predominantly adhesive failure along the dentin surface. A high magnification view of the UN-SiC paper group revealed failure at the top of demineralized dentin with partial infiltration of dentinal tubules by resin cement (Figure 4a). Remnants of resin cement were observed on the smooth and regular dentin surface in the UN-tungsten carbide group (Figure 4b). A porous layer of resin cement and residual smear debris was observed on the rough and irregular dentin surface (Figure 4c). Significant association ($p<0.05$) was only found among cements, surface preparation and mixed failure of 1 and 3.

DISCUSSION

In the current study, the microtensile bond strength of three resin cements was compared using different dentin surface preparation methods. The results of the study showed that the factors, such as "dentin surface preparation" and "luting cement," were statistically significant ($p<0.001$), but their interaction was not significant. For RelyX ARC, bond strength was unaffected by preparation of the dentin surface. For Panavia F2.0 and RelyX Unicem, the bond strength to the diamond bur-prepared dentin surface was significantly lower than that of the #600-grit SiC paper-prepared surface. Hence, the null hypothesis

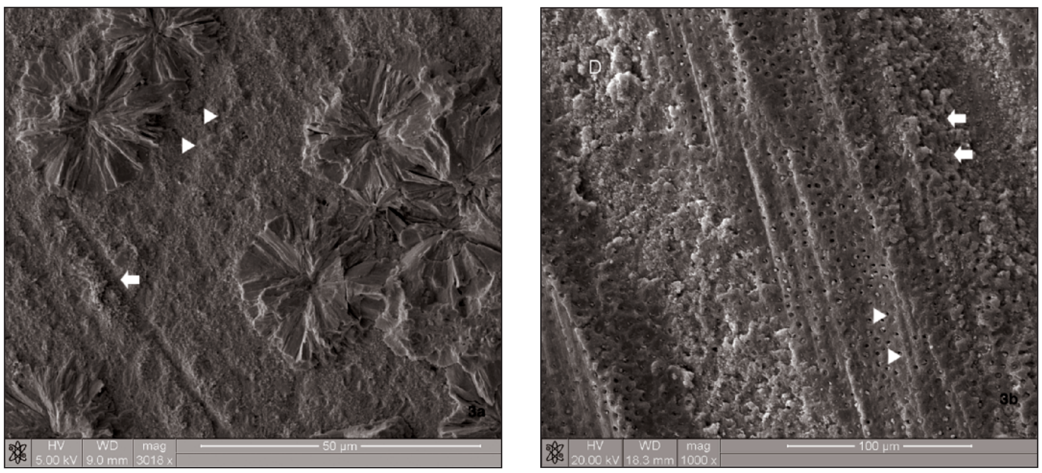

Figure 3. *Representative SEM micrographs of the dentin side of a fractured beam from PF-tungsten carbide (Figure 3a) and the PF-diamond groups (Figure 3b). (Figure 3a): A high magnification view from the PF-tungsten carbide group shows hybridized smear plugs (arrows) at the top of the hybrid layer. Circular, rosette-like fracture patterns (arrowheads), originating from patent dentinal tubules, are identified on the dentin surface. (Original magnification = 3018x). (Figure 3b): A high magnification view from the PF-diamond groups show the hybridized smear layer (arrows) at the top of the grooves produced by diamond burs and areas of incompletely infiltrated dentinal tubules (arrowheads) at the bottom of the grooves. D: dentin. (Original magnification = 1000x)*

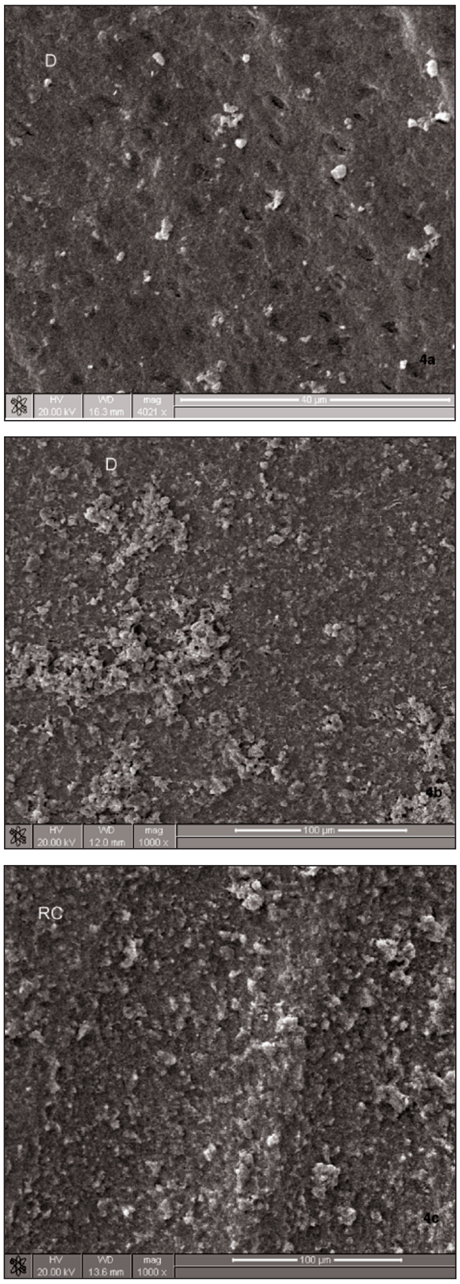

Figure 4. *SEM micrographs of the dentin side of the representative fractured beam from UN-SiC paper (Figure 4a) UN-tungsten carbide (Figure 4b) and UN-diamond (Figure 4c) groups. (Figure 4a): A high magnification view from the UN-SiC paper group showed a partially removed smear layer and slight infiltration of the resin cement into dentinal tubules. (Original magnification = 4021x). (Figure 4b): High magnification from the UN-tungsten carbide group showing islands of resin cement on the smooth dentin surface. (Original magnification = 1000x). (Figure 4c): A high magnification view from the UN-diamond group shows thick remnants of porous cement and the smear layer on rough, irregular dentin. (Original magnification = 1000x). D: dentin; RC: fractured resin cement.*

that different dentin surface preparations had no effect on the microtensile bond strength of three resin luting cements to dentin was partially rejected.

When the tooth surface is prepared with rotary or hand instruments, the debris is smeared over the enamel and dentin surfaces to produce a smear layer,[24-25] the quantity and quality of which varies with the technique. Dentin surfaces prepared with high-speed medium-grit diamond burs exhibit an irregular, rough surface with a thick smear layer, traversed by deep grooves. In contrast, when prepared with a tungsten carbide bur, the smear layer was thinner, with uniform, smooth scratches on the dentin surface.[26-27]

In the current study, three different types of resin cements were used: etch-and-rinse adhesive cement (RelyX ARC), self-etch adhesive cement (Panavia F2.0) and self-adhesive cements (RelyX Unicem). RelyX ARC employs a two-step etch-and-rinse adhesive in which the priming and bonding steps are combined. RelyX ARC showed the highest bond strength values across the three surface preparation methods. However, there was no significant difference between the surface preparation and bond strength values (Table 1), indicating that the surface preparation method did not influence bond strength. This finding is in agreement with previous studies that show that etch-and-rinse adhesive systems are not sensitive to the method used to create the smear layer.[28] Hosoya and others[9] demonstrated that, due to the stronger demineralization effect of the phosphoric acid etchant, the smear layers and smear plugs were completely removed, regardless of how the surface had been prepared.

It appeared that, in the current study, phosphoric acid-etching was able to fully remove the smear layer created by the diamond and tungsten carbide burs, allowing acidic resin monomers from Adper Single Bond 2 to penetrate into the demineralized collagen fibrils and achieve optimal bonding to dentin. From the SEM fractographic analysis, the predominant mode of failure in RelyX ARC was between the composite and resin cement, indicating that the bond between the adhesive and dentin was relatively strong. This was in agreement with the findings of Mak and others,[29] who also reported a high percentage of adhesive failures along the composite-cement interface with Rely X ARC.

Self-etching primers combine the etching and priming steps into one procedure. The acidic component demineralizes through the smear layer into the underlying dentin, resulting in the creation of a thin hybrid layer.[30-31] Removal of the smear layer by self-etch adhesives is dependent on the pH of the acidic primer used.[5] The mode of failure within Panavia F2.0 was predominantly adhesive between the dentin and ED primer. This failure mode had also been reported in several other studies.[16-17,29,32-33] This may be explained by an increased permeability associated with one-step self-etch ED primer,[16,33] which behaved as a semi-permeable membrane after polymerization and compromised the bond strength of the resin cement. "Mushroom-like blisters," which were continuous with the lumen of patent dentinal tubules, were observed in the SEM of the PF-control and PF-tungsten carbide groups (Figure 2a). These blisters are assumed to be incompletely polymerized regions within the primer layer that resulted in the entrapment of water or were filled with water that permeated from the dentinal tubules.[16]

It is interesting to note that a constant seating force of 3.0 kg had been applied to the bonded specimen for five minutes before curing the cement in order to prevent water diffusion from dentin as a result of increased permeability of the ED primer. The appearance of "mushroom-like blisters" at the bonded interface, despite the application of sustained seating pressure,

Table 4: *Summary of Fracture Modes*

[1] adhesive failure along the cement-dentin interface

Cement	#600 Grit SiC Paper	Diamond	Tungsten Carbide
ARC	2	0	0
PF	24	32	17
UN	30	32	36

[1]p=0.0852

[2] adhesive failure along the cement-composite interface

Cement	#600 Grit SiC Paper	Diamond	Tungsten Carbide
ARC	30	26	22
PF	5	0	6
UN	1	0	0

[1]p=0.1122

[3] cohesive failure within resin cement

Cement	#600 Grit SiC Paper	Diamond	Tungsten Carbide
ARC	0	0	2
PF	3	3	8
UN	5	3	4

[1]p=04267

[4] mixed failure of 1 and 3

Cement	#600 Grit SiC Paper	Diamond	Tungsten Carbide
ARC	8	4	16
PF	7	5	7
UN	4	5	0

[1]p=0292

[5] mixed failure of 2 and 3

Cement	#600 Grit SiC Paper	Diamond	Tungsten Carbide
ARC	0	0	0
PF	1	0	2
UN	0	0	0

[1]Chi-Square

suggests that seating pressure alone may be insufficient to stop water movement across the bonded interface, and the additional use of a hydrophobic light-cured bonding layer is necessary to improve the final bond strength of Panavia F2.0.[20]

Panavia F2.0 showed significantly higher bond strength values for the control and tungsten carbide bur groups, when compared to the diamond bur group (p<0.05). It is believed that the bond strength of the self-etching primer bonding systems to dentin could be affected by differences in the quantity and quality of the smear layer due to the weak acidity of self-etching primers.[8] The ED primer that was used had a pH of 3.0, which is a mild self-etch adhesive, when compared to 35% phosphoric acid with a pH of 0.6.[9] As a result of its weak acidity, ED primer can easily be neutralized by buffering components in the thick smear layer,[34] thereby limiting its penetration in underlying dentin. Furthermore, the deep grooves produced by a diamond bur may cause uneven distribution of smear debris, with thicker accumulations at the bottom of the grooves.[26] This regional variation in smear layer thickness could have caused the uneven infiltration and irregular interaction between the adhesive and dentin (Figure 2b). This could explain the low bond strength observed in the PF-diamond group. Failure in this group occurred predominantly at the top of the hybridized smear layer, with areas of incompletely infiltrated dentinal tubules at the bottom of the grooves created by the diamond burs.

Similarly, Ogata and others[34] also observed areas of smear layer remnants on the dentin surface cut with a diamond bur and treated with a self-etching primer. In general, thick smear layers interfere with adhesion capabilities of the self-etching primer, suggesting that the self-etching primer should be used with a surface preparation that creates a thin smear layer, such as a tungsten carbide bur.[27-28] Higher bond strengths were observed in the PF-tungsten carbide and PF-SiC paper groups. Bond strength was improved by 29% (tungsten carbide bur) and 39% (#600-grit SiC paper), when compared to diamond burs. This showed that ED primer successfully etched through the thin smear layer produced by the tungsten carbide bur and SiC paper to demineralize the underlying dentin to create a thin hybrid layer. This is supported by SEM findings that the failure of these two groups occurred mainly at the top of the hybridized dentin, with cohesively fractured resin tags occluding the dentinal tubules.

A multi-step application technique is time-consuming and technique-sensitive, which, as a consequence, may compromise bonding effectiveness.[29] Recently, RelyX Unicem, a self-adhesive universal cement not requiring surface pretreatment, has been introduced. It attempts to reduce the number of steps involved in the luting procedure. The organic matrix consists of multifunctional phosphoric acid methacrylates. In the presence of water, phosphoric acidic methacrylates demineralize the smear layer and infiltrate the underlying dentin, resulting in micro-mechanical retention. The acidic resin monomers also interact with the hydroxyapatite from tooth structure to form secondary chemical adhesion.

However, it differs from self-etch resin cement, in that no distinct demineralization and hybridization was observed during TEM morphological interface examination.[36-38] A limited ability to etch through the clinically relevant smear layers into the underlying intact dentin, together with relatively high viscosity and porosity, were described for the self-adhesive material RelyX Unicem.[39] When compared with RelyX ARC, this may explain the low bond strength observed in this group.

The pH of mixed Rely X Unicem is less than two during the first minute of setting (3M ESPE Product Profile). When compared to the control, the bond strength of RelyX Unicem was significantly lower when bonded to dentin prepared with a diamond bur. RelyX Unicem is heavily filled (72 wt% reactive glass fillers) (3M ESPE Product Profile). The high viscosity of RelyX Unicem was unable to etch through the thick smear layers produced by the diamond burs to decalcify the underlying dentin within the limited interaction time. The thick smear layer probably rapidly buffers the acidity of the acidic resin monomers, thereby limiting its etching ability.[37] Furthermore, a pH neutralization effect may also occur during the acid-base reaction between the phosphoric acidic methacrylates and the basic ion-leachable fillers. Hence, SEM findings showed a porous layer of cement and smear debris on the rough dentin surface, which compromised the interfacial strength of RelyX Unicem. Consequently, failure in the group occurred predominantly between the cement and the weakly infiltrated smear layer (Figure 3c). The superficial interaction of RelyX Unicem with dentin and the absence of the hybrid layer/resin tags formation was similar to the morphological findings reported by De Munck and others.[36] By contrast, the thin smear layer produced by #600-grit SiC paper and tungsten carbide burs had a higher permeability and allowed the acidic resin monomers to partially remove the smear layer and interact superficially with the underlying dentin. Hence, the mode of failure in this group occurred mainly along the surface of the dentin (Figure 3a and 3b). The bond strength of RelyX Unicem to dentin prepared with a tungsten carbide bur was higher than that prepared with a diamond bur, but this was not statistically significant.

CONCLUSIONS

Within the limitations of this study, it may be concluded that:

1. Panavia F2.0 showed higher bond strength when bonded to a dentin surface prepared with a tungsten carbide bur than with a diamond bur.
2. The selection of bur had no effect on the adhesion of RelyX Arc and RelyX Unicem luting cements to dentin.

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