

# Bond Strengths of Resin Cements to Astringent-contaminated Dentin

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## Clinical Relevance

The contamination of the dentin surface with an astringent-containing aluminum chloride does not reduce the bond strength of either the resin cement used in conjunction with an etch-and-rinse or the resin cement with a self-etching adhesive. However, the contamination probably interferes with the etching ability of the self-etching primer and the adaptation of the resin cement to the dentin surface.

## SUMMARY

**The current study evaluated the micro-shear bond strength of two resin cements to astringent-contaminated dentin. Twelve occlusal dentin discs were prepared from extracted caries-free human molars and divided into two groups subjected to two types of resin cements, Panavia F**

**(PF) and Variolink II (VL). Each disc was ground with 600 grit SiC paper and sectioned into two semi-disks, one for the normal dentin surface and the other for the contaminated dentin surface. For contaminated dentin, an astringent containing aluminum chloride was applied for two minutes and rinsed before the bonding procedures. A micro tygon tube was placed on the dentin surface following the bonding application and then filled with a resin cement. After the resin was polymerized, the specimen was kept in water for 24 hours before the micro-shear bond strengths evaluation. The micro morphology of the treated surfaces and resin-dentin interfaces were observed under a scanning electron microscope (SEM). Aluminum content under different dentin conditions was also examined. No significant differences were found between the dentin bond strengths to normal dentin and contaminated dentin surfaces in both the PF and VL groups ( $p>0.05$ ). PF showed similar bond strengths to VL on normal and contaminated dentin ( $p>0.05$ ). SEM observations of the VL groups revealed no**

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**differences in the treated dentin surfaces and the resin-dentin interfaces between normal and contaminated dentin. However, for the PF group, an inconsistent etching pattern of the self-etching primer and gap formation at the interface of resin cement to contaminated dentin were observed.**

## INTRODUCTION

The use of adhesive resin cements for bonding indirect tooth-colored and casting restorations, such as inlays, onlays and crowns, is increasing. These cements bond to both the fitting surface of the restoration and the tooth structure. At the tooth surface, an adhesive system is used to bond the resin cement to both enamel and dentin surfaces. Currently, adhesive resin cements can be categorized according to the adhesive system used as either etch-and-rinse or self-etching system.<sup>1</sup>

During the cementation procedures of indirect restorations at the cervical area, gingival fluid and blood sometimes appear as a result of trauma from tooth preparation. In this clinical situation, hemostatic agents are frequently used to control bleeding and gingival fluid. The pH of these hemostatic agents has been reported to vary from 0.7-3.0.<sup>2,3</sup> The tooth structure, especially dentin, which is a major part of the preparation, may be contaminated with these highly acidic astringents. Of these hemostatic agents, aluminum chloride, with a concentration between 20%-25%, is frequently used.<sup>3</sup> Land and others<sup>4</sup> reported that dentin surfaces treated with 21.3% aluminum chloride for five minutes exhibited complete smear layer removal with some degree of demineralization.<sup>3,4</sup> Since some effects of the smear layer on the adhesion of self-etching adhesives have been reported,<sup>5,6</sup> smear layer removal by hemostatic agents could affect the bonding mechanism of the self-etching adhesive used with a resin luting cement.

In a previous study by the current authors, a light-cured, two-step self-etching adhesive used in conjunction with a direct resin composite exhibited lower bond strength to dentin contaminated with 25% aluminum chloride solution compared to normal dentin, but an etch-and-rinse adhesive exhibited similar bond strength to both contaminated and normal dentin.<sup>7</sup> However, there is no report regarding the bonding efficiency of adhesives used with resin cements to dentin contaminated with a hemostatic agent. Therefore, the purpose of the current study was to evaluate the micro-shear bond strengths of two resin cements to astringent-contaminated dentin, one used with an etch-and-rinse adhesive and the other with a self-etching adhesive. The null hypothesis is that the bond strengths to astringent-contaminated human dentin of resin cements used with these adhesives are not different from the bond strengths to normal dentin. To observe

the micro morphological differences among the test groups, non-stress, resin-dentin interfaces and dentin surfaces with and without contamination were observed under SEM. The aluminum content under different dentin conditions was also determined.

## METHODS AND MATERIALS

### Preparation of Dentin Surface (Figure 1)

Twelve 2-mm thick dentin discs were prepared by perpendicular section to the long axis of the extracted caries-free human molars using a slow-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under water lubrication. The surfaces of the dentin discs were hand ground with 600 grit silicon carbide papers (Struers, Ballerup, Denmark) under running water to standardize the resulting smear layers. The ground dentin discs were then hemi-sectioned into 12 pairs of dentin semi-discs. All semi-discs were equally divided into two groups, the control and contaminated groups. In the control group, the dentin surface of each semi-disc was rinsed and dried. In the contaminated group, the dentin surface of each semi-disc was treated with 25% aluminum chloride (Racestypine, Septodont, Cedex, France) for two minutes, rinsed with water spray for 30 seconds and dried with oil-free air. Semi-

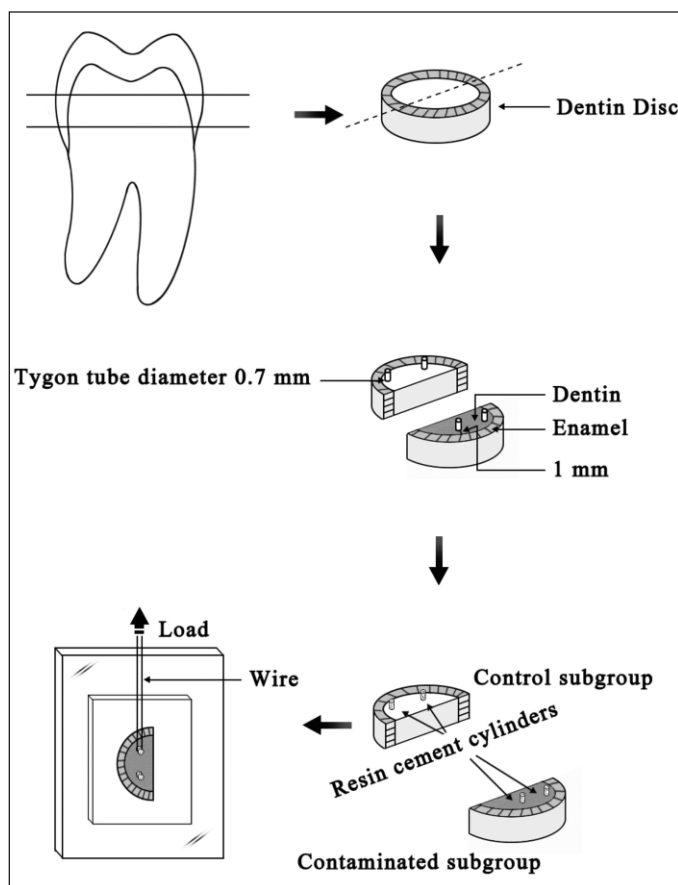


Figure 1. Schematic illustration of the experimental design.

Table 1: <i>Resin Cements and Their Adhesives Used</i>			
Resin Cements	Composition	Manufacturer	Batch #
Panavia F ED II Primer	Primer A: HEMA, MDP, 5-NMSA, water, accelerator Primer B: 5-NMSA, accelerator, Water, sodium benzene sulfinate	Kuraray Medical Inc Okayama, Japan	00225A
Luting Resin	Base paste: hydrophobic aromatic (and aliphatic) dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate (TPBSS), N,N-diethanol-p-toluidine, functionalized sodium fluoride, silanized barium glass Catalyst paste: MDP, hydrophobic aromatic (and aliphatic) dimethacrylate, hydrophilic dimethacrylate, silanized silica, photoinitiator, dibenzoyl peroxide		00104A  00197A  00108A
Variolink Excite DSC	Etchant Total Etch: 37% phosphoric acid Primer-adhesive: HEMA, DMA phosphoric acid acrylate, silica (0.5 wt%), ethanol, initiators	Ivoclar Vivadent, Schaan, Liechtenstein	F40503
Luting Resin	Base paste: Bis-GMA, UDMA, TGDMA, fillers (72.3 wt%), pigments and stabilizers Catalyst paste/low viscosity: Bis-GMA, UDMA, TGDMA, fillers (71.2 wt%), pigments, stabilizers and catalysts		G26397  H17779

discs of both the control and contaminated groups were further divided into two subgroups of six pairs of semi-discs each according to the resin cement systems used (Table 1).

### Bonding Procedures

The composition and batch number of the adhesive resin cements used are presented in Table 1.

**Subgroup 1 (PF)** ED primer A and B were mixed and applied to the dentin surfaces of both the control and contaminated groups, left for 30 seconds and gently air dried. Irises that had been cut from micro bore tygon tubing (TYG-030, Small Parts Inc, Miami Lakes, FL, USA), with an internal diameter and a height of approximately 0.75 and 0.50 mm, respectively, were placed at two positions on the primed dentin surface 1 mm from the dentino-enamel junction. Freshly mixed dual-cured resin cement of Panavia F (Kuraray Medical Inc, Okayama, Japan) was used to fill the tubing and then light cured for 20 seconds using a halogen light-curing unit (Curing Light XL 3000, 3M ESPE, St Paul, MN, USA) with an output of 700 mW/cm<sup>2</sup>. The bonding interface was covered entirely with liquid glycerin gel (Oxyguard II, Kuraray Medical Inc) for three minutes to enable optimal anaerobic polymerization and the gel was then rinsed out. The bonded specimens were left at room temperature (25°C) for one hour before removal of the tygon tubing by longitudinally

cutting with a razor blade. This resulted in 12 resin cement cylinders bonded to the dentin surface of the control and contaminated groups.

**Subgroup 2 (VL)** The dentin surfaces of the semi-discs of both the control and contaminated groups were etched with 37% phosphoric acid for 15 seconds, then thoroughly rinsed with water spray. Excess water on the dentin surface was blot-dried with lint-free absorbent tissue prior to the application of primer-adhesive (Excite DSC) and agitated gently for 30 seconds. The treated dentin surface was gently air dried for three seconds and light cured for 20 seconds before placing the tygon tubing in the same manner as in Subgroup 1. Variolink II low viscosity base and catalyst paste (1:1 ratio) were mixed, filled the tygon tubing and were light cured for 20 seconds. The specimens were left at room temperature for one hour before removing the tygon tubing as in Subgroup 1. All the specimens were stored in distilled water at 37°C for 24 hours before the micro-shear bond test.

### Micro-shear Bond Test

After 24 hours, the resin cement cylinders were checked under an optical microscope (30x) for bonding defects. The cylinders, which showed interfacial gap formation and/or bubble inclusion, were excluded and replaced. Twelve specimens were tested for each test group. The micro-shear bond test was performed with



the bond test apparatus (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) attached to a universal testing machine (EZ-test 500N, Shimadzu Co, Kyoto, Japan).<sup>8</sup> The dentin semi-disc with the resin cement cylinder was fixed to the apparatus with a cyanoacrylate adhesive (Zapit, DVA, Corona, CA, USA). A thin wire (0.2 mm in diameter) was looped around the resin cement cylinder, making contact through the lower half of its circumference and gently held flat against the resin/dentin interface. The resin cement cylinder and the center of the load cell were aligned as straight as possible to ensure the desired orientation of the shear test force. A shear force was applied to each specimen at a crosshead speed of 1 mm/minute until fracturing occurred.

After debonding, bond strengths were recorded and the fracture modes of all the specimens were observed under a SEM (JSM-5310V, JEOL Ltd, Tokyo, Japan). The fracture mode was classified as follows: adhesive failure at the resin-dentin interface, cohesive failure in dentin or cohesive failure in resin cement. The percentage of each type of failure in the specimens was recorded.

The data were statistically analyzed by two-way ANOVA (type of dentin, adhesive resin cement system) followed by *post hoc* multiple comparisons with the Student's *t*-test. For the fracture modes, the Kruskal-Wallis test was used to compare differences among each experimental group. All analyses were performed using the SPSS program. Statistical significance was considered to be  $p < 0.05$ .

### EDS Microanalysis of the Dentin Surfaces

The surfaces of normal dentin after grinding as a control, the surfaces of astringent contaminated dentin and both dentin conditions after etching with phosphoric acid and self-etching primer (ED primer) were all measured for aluminum (Al) content on the surface using an energy dispersive spectrometer (Oxford ISIS Pentafet link model 6647, Highway Combe, England) operated at 20 KV. The relative amounts of Al to Ca were measured at 500x magnification.

### SEM Observation of the Treated Dentin Surfaces and the Bonding Interface

The surfaces of ground dentin and astringent-contaminated dentin and both dentin conditions after etching with phosphoric acid and self-etching primer were observed. For the PF group, ED primer A and B were mixed and applied to the dentin surface for 30 seconds before being thoroughly rinsed with acetone and water to remove residual

resin. They were then air-dried. The morphological changes induced on the dentin surfaces were observed using SEM.

The interfacial morphology between both normal and contaminated dentin and the resin cements were also observed after the acid-base challenge. Another 12 pairs of semi-discs were divided into four groups of three semi-discs each and received the same treatment as for the micro-shear bond test. Twenty-four hours after bonding, the specimens were sectioned perpendicular to the bonded surface using the slow-speed diamond saw (Isomet, Buehler Ltd) under water spray. The cut specimens were fixed in 10% buffered formalin before being embedded in a self-cured epoxy resin (Epon 812, Nisshin M Co, Ltd, Tokyo, Japan), then ground and polished using wet silicon carbide papers and diamond pastes of decreasing abrasiveness down to 0.25  $\mu\text{m}$ . The surfaces of the polished specimens were subjected to 10% phosphoric acid for five seconds and 5.25 % NaOCl for five minutes. After rinsing thoroughly, the specimens were dried overnight, sputter coated with gold and observed under high vacuum in SEM.

### pH Measurement

The pH determination of the astringent was preformed using a pH meter (Twin pH, Horiba, Tokyo, Japan). The pH reference solutions at pH 7 and pH 4 were used to standardize the electrode. The measurements were done in triplicate.

## RESULTS

The pH of the 25% aluminum chloride Racestypine was 0.8. The micro-shear bond strength of both resin cements to normal and contaminated dentin and the mode of fracture are presented in Tables 2 and 3, respectively.

The bond strengths of both resin cements to contaminated dentin were not significantly different from those of normal dentin. The bond strengths of PF and VL to normal and contaminated dentin were also not signifi-

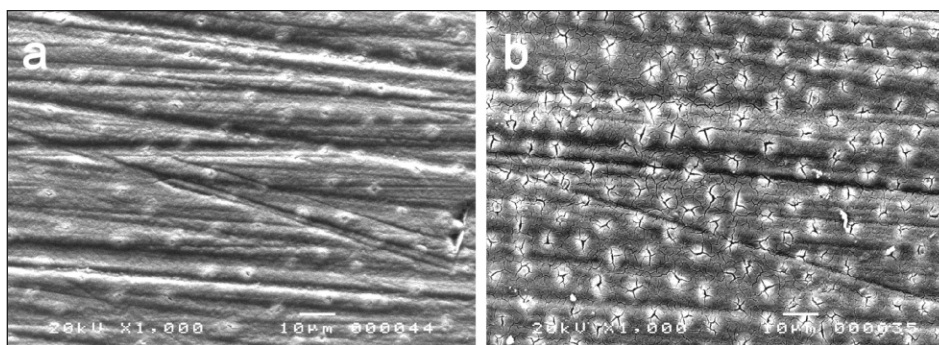


Figure 2. (a) SEM photomicrograph of a normal dentin surface after grinding with 600 grit SiC paper. A thick smear layer covered the entire surface. (b) Dentin surface after two-minute contamination with 25% aluminum chloride and washed out. Part of the smear layer was removed. Opening of the dentinal tubules were occluded with smear plug (original magnification 1000x).

Table 2: Micro-shear Bond Strengths (MPa) of Panavia F and Variolink II to Normal Dentin and Contaminated Dentin (n=12)		
Groups	Normal Dentin	Contaminated Dentin
Panavia F (PF)	22.23 (9.94) <sup>a</sup>	24.72 (5.72) <sup>a</sup>
Variolink II (VL)	22.29 (5.86) <sup>a</sup>	23.89 (3.19) <sup>a</sup>
Groups with the same superscript are not statistically different (p<0.05).		

Table 3: Mean Percentage of Failure Mode After Micro-shear Bond Test			
	Adhesive Failure <sup>a</sup>	Cohesive Failure <sup>b</sup>	
		In Dentin	In Composite
Variolink			
Normal Dentin	96.67	3.33	0
Contaminated Dentin	97.72	0	2.08
Panavia F			
Normal Dentin	83.33	9.58	7.09
Contaminated Dentin	72.08	0	27.92
There is no significant difference among each group (p<0.05).			
<sup>a</sup> Adhesive failure = failure between resin and dentin.			
<sup>b</sup> Cohesive failure = failure that occurred within the dentin or in the resin composite.			

mode of failure did not show significant differences between that of normal and contaminated dentin. Most failures were adhesive. Variation in failure mode was found in the PF groups, which demonstrated a slight increase in cohesive failure in resin, especially in the contaminated group.

Surface morphological study showed that, in the control group, a thick smear layer was left on the dentin surfaces (Figure 2a). In the contaminated group, noticeable etching effects were observed. The smear layer was partially removed and the dentinal

tubules can be located with smear plugs still occluding the tubule orifices (Figure 2b). The surfaces of normal and contaminated dentin after phosphoric acid etching were similar, with the absence of the smear layer and peritubular dentin, as well as clearly visible patent dentinal tubules (Figures 3a and 4a). The normal dentin after treatment with ED primer revealed consistent etching patterns of a clear surface without the smear layer and with opened dentinal tubules with the remaining peritubular dentin (Figure 5a). However, the contaminated surfaces treated with ED primer showed inconsistencies in etching patterns. The surfaces were clear without the smear layer and also with open dentinal tubules with and without peritubular dentin. In a few tubules, the remaining smear plugs still occluded the tubule openings (Figure 6a).

No difference was found between the SEM's appearance of the bonded interfaces of VL II to normal dentin and contaminated dentin (Figures 3b and 4b). Approximately 2 µm thick hybrid layers with funnel-shaped resin tags were observed. Many resin tags showed lateral extension of the micro-tags branching off from the main tags in the dentinal

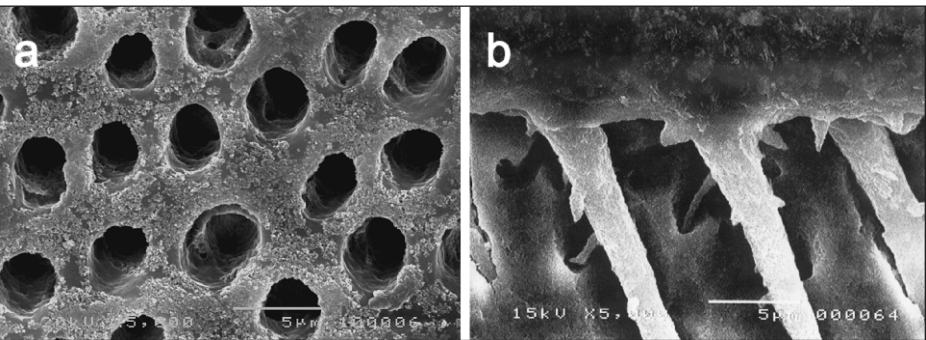


Figure 3. (a) SEM photomicrograph of the normal dentin surface after etching with 37% phosphoric acid. The smear layer was completely removed. Widening of the dentinal tubules was observed. (b) The interface between Variolink resin cement and normal dentin with a 2 µm thick hybrid layer and funnel-shape resin tags with lateral branches (original magnification 5000x).

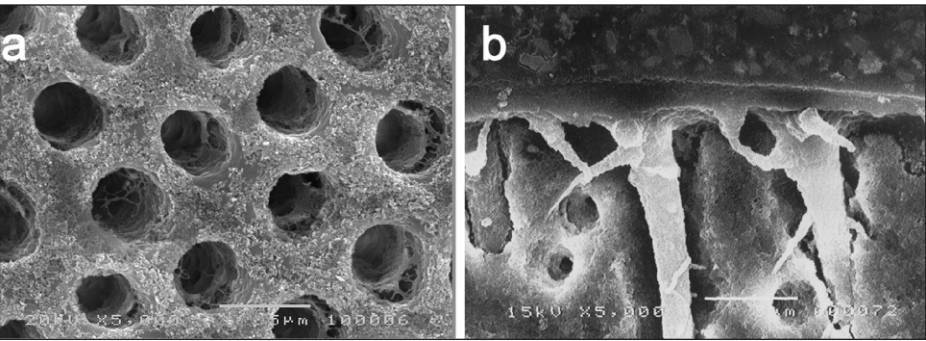


Figure 4. (a) SEM photomicrograph of the contaminated dentin surface after etching with 37% phosphoric acid and (b) the interface between Variolink resin cement and contaminated dentin, which was not different from that of normal dentin (original magnification 5000x).

cantly different from each other. Most failures were adhesive (Table 3). For the VL groups, analysis of the



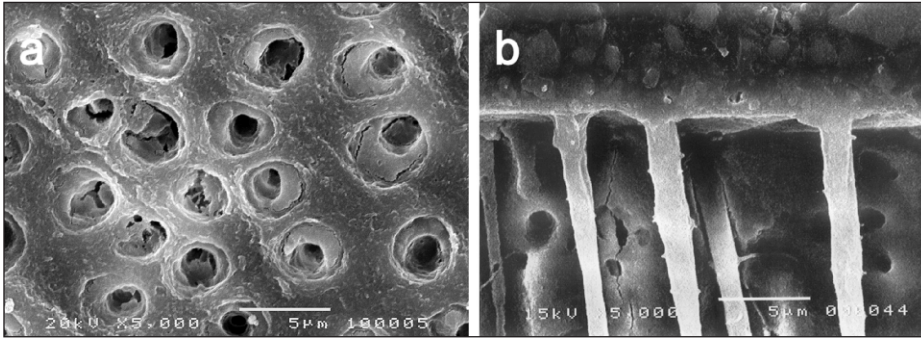


Figure 5. (a) SEM photomicrograph of a normal dentin surface after application of the ED primer. The smear layer was removed and most dentinal tubules were opened with the remaining peritubular dentin. (b) The interface between Panavia resin cement and normal dentin with approximately a 0.5 µm thick hybrid layer and long cylindrical resin tags (original magnification 5000x).

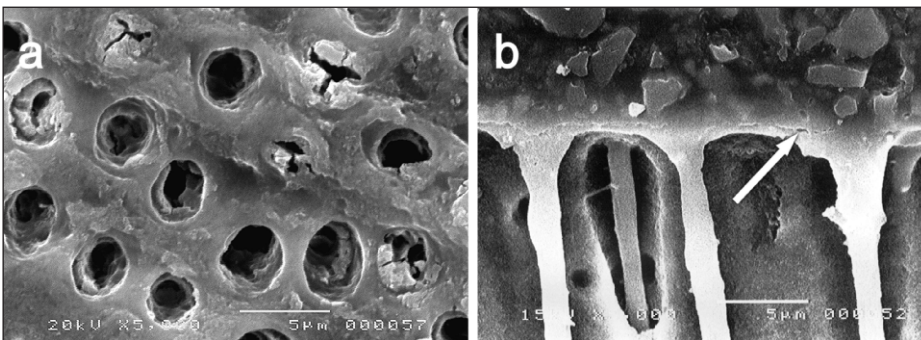


Figure 6. (a) SEM photomicrograph of the contaminated dentin surface after the application of ED primer. The smear layer was removed. Dentinal tubules are clearly visible with different degrees of tubular opening. (b) The interface between Panavia resin cement and contaminated dentin. A thin hybrid layer with small gaps between resin cement and the top of the hybrid layer (arrow). Funnel-shape and cylindrical resin tags are observed in the same area (original magnification 5000x).

tubules. The interface between PF to normal and contaminated dentin was similar, with a 0.5 µm thick hybrid layer and cylindrical resin tags with lateral protrusion of micro tags from the side of the main tags (Figures 5b and 6b). However, at the interface of the PF and contaminated dentin, small gaps were found at the junction between the top of the hybrid layer and the bottom of the resin cement layer, which were not found in normal dentin. In addition, regular cylindrical tags extending into the dentin were found in the normal dentin. In the contaminated dentin, irregular shapes of resin tags were observed, especially at the upper part of the tags. Cylindrical resin tags were frequently revealed, but funnel-shaped resin tags were also observed in some areas due to more aggressive etching patterns that removed the peritubular dentin. Fewer resin tags exhibited a constriction at the upper end next to the hybrid layer.

EDS analysis showed slightly higher aluminum content in the groups of contaminated dentin treated with ED primer (2.46%Al) compared with normal dentin treated with ED primer (0.75%Al) and contaminated dentin treated with phosphoric acid (0.46%Al).

## DISCUSSION

Many studies have reported that the results of the bond strength test will vary due to different dentin substrate conditions, such as the age of the tooth and storage conditions.<sup>9-10</sup> However, these factors could be omitted in the current study, since all specimens in the control and contaminated groups were prepared from the same dentin disks. In addition, the location of the bonding area was controlled by placing the resin cement cylinders at the same distance from the dentino-enamel junction.

In the current study, the bonding ability of two resin cements, one utilizing an etch-and-rinse, single bottle adhesive, and the other utilizing a self-cured, all-in-one self-etching adhesive to astringent-contaminated dentin were comparable to those in normal dentin. The results of the current study thus lead to acceptance of the null hypothesis that the bond strengths to a hemostatic agent-contaminated human dentin of both resin cements are similar to that of normal dentin.

The self-etching ED primer of PF with pH 2.4 has less etching effect on the dentin surface than phosphoric acid. On the normal dentin surface, this weak acid removed the smear layer and the smear plug as well as slightly demineralizing the intertubular dentin, but it was not strong enough to demineralize the peritubular dentin. This resulted in a 0.5 µm thick hybrid layer and uniform cylindrical resin tags of PF compared to a 2 µm thick hybrid layer and funnel-shaped resin tags of the etch-and-rinse adhesive in the VL group.

The hemostatic agent containing 25% AlCl<sub>3</sub> Racetyptine and the two-minute application time used in the current study were in accordance with the methods used in the previous study conducted by the current authors.<sup>7</sup> The results showed that the two-minute application of this agent removed the smear layer on the surface and, to a small extent, it removed the smear plug from most dentinal tubules. The demineralization effect of AlCl<sub>3</sub> also seems to have enhanced the etching effect of the self-etching ED primer on the contaminated dentin in some areas, since few open dentinal tubules without peritubular dentin were observed (Figure 6a). Even though the results of the EDS analy-

sis showed that, after application of the ED primer, a higher Al content (2.46%) remained on the contaminated dentin surface than on the normal non-contaminated dentin surface (0.75%), the remaining Al seemed to have no obvious effect on the etching ability of the ED primer. This result is in contrast to the previous study in which a self-etching primer of a light-cured, two-step self-etching adhesive, Clearfil SE Bond, was applied for 20 seconds on the contaminated dentin and showed a subsequently less etching effect than to normal dentin. The authors of the current study ascribe this to the displacement of Ca in the hydroxyl apatite by Al, which resulted in the formation of the insoluble  $\text{Al}(\text{OH})_2\text{H}_2\text{PO}_4$  compound. The compound might have increased resistance to acid of the dentin surface.<sup>11</sup> However, this explanation could only be partially applied to the contaminated dentin of the PF group, since the results of SEM observation revealed inconsistencies in etching patterns on the contaminated dentin. More or less localized etching effects were found on the same surfaces of contaminated dentin after the ED primer was rinsed off. This varied etching effect corresponded to the SEM pictures of the resin-contaminated dentin interface of PF, which showed greater variation in resin tag formation.

The finding that the contamination of dentin with the  $\text{AlCl}_3$  solution did not adversely affect the bond strength of this self-cure, self-etch primer used with Panavia was different from the results of previous studies.<sup>7,12</sup> In previous studies, the light-cured, self-etch adhesives used with direct resin composite restoration showed lower bond strengths to astringent-contaminated dentin. The main reason is probably the greater etching ability of the self-cured ED primer compared to that of the light-cured self-etching SE primer. The greater etching effects of the ED primer may not be explained by the acidity of the primer, since the pH of the ED primer (pH 2.4) is higher than that of the SE primer (pH 2).<sup>13-14</sup> This is probably due to the longer etching time (30 seconds) of the ED primer compared to the 20 second-etching time of the SE primer as per the manufacturer's instructions. This may support the previous study, in which the etching effect of self-etching primer was greater when priming time was extended from 20 seconds to 40 seconds.<sup>7</sup> The other reasons may be the different composition of the self-etching primer/adhesives utilized and the different mode of curing that may have enabled a good infiltration of the ED primer into the dentin. These reasons may have resulted in substantially similar bond strengths to that of normal dentin.

Although the bond strength of Panavia to contaminated dentin was not different from that of normal dentin, this result should be cautiously interpreted, since the ultramorphology of the bonding interfaces of Panavia to contaminated dentin revealed small gaps

between the top of the hybrid layer and the resin cement (Figure 6b). Previous studies have reported that no correlation was found between the bond strength values and degree of microleakage.<sup>15-16</sup> These gaps may be the result of either incompatibility between the dual-cured resin cements and the acidic monomers in the ED primer or the semi-permeable property of the cured one-step self-etch primer/adhesive as reported in a previous study.<sup>17</sup> This may result in the susceptibility of this adhesive system to microleakage or lower durability of the bonding. However, in the current study, this adverse effect was noticeable only with bonding to astringent-contaminated dentin, since no such gaps were found at the interface between this cement and normal dentin. The remaining aluminum content on the surface of hemostatic-contaminated dentin may increase the adverse effect of ED primer on the adaptation and strength of resin cement. This may be the reason why a higher cohesive failure of resin was found in the contaminated subgroup of PF when compared to that of the normal dentin subgroup.

In many studies regarding the bond of adhesives to dentin contaminated by other agents, such as zinc oxide eugenol, hydrogen peroxide or astringents, the etch-and-rinse adhesive generally provided similar bond strengths to contaminated dentin compared with normal dentin.<sup>7,18-21</sup> It was assumed that the phosphoric acid used in etch-and-rinse adhesives would remove most of the contaminants from the dentin surface before the adhesive resin application.<sup>20</sup> This may explain the similar bond strengths to normal and astringent-contaminated dentin of the etch-and-rinse system of VL in the current study. The aggressive etching effect of phosphoric acid with pH 0.5 might have demineralized and removed all contaminant-induced effects on the dentin surface. This was supported by the result of the EDS analysis, which showed similar remaining aluminum content on the surfaces of normal and contaminated dentin after etching with phosphoric acid. The acid etching patterns of the dentin surfaces of the normal and contaminated groups of VL were also similar.

The astringent used in the current study contained 25% aluminum chloride. Its acidity had a demineralizing effect on dentin surfaces. However, this did not affect the dentin bond strengths of both resin cements. From the SEM pictures, the ED primer has less of an etching effect when compared with the phosphoric acid used in the adhesive of Variolink II. It seems that, in terms of bond strength, this self-etching effect is sufficient to remove any contaminants from the astringents and provides similar bond strength. However, further study regarding the sealing ability and long-term durability is needed.

## CONCLUSIONS

Dentin contaminated with astringent containing 25% aluminum chloride, Racestypine, had comparable bond strengths to normal dentin of both resin luting cements. These results are limited to the materials used in the current study. Other materials might exhibit differently from the current report.

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