



# Tooth surface treatment strategies for adhesive cementation

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**PURPOSE.** The aim of this study was to evaluate the effect of tooth surface pre-treatment steps on shear bond strength, which is essential for understanding the adhesive cementation process. **MATERIALS AND METHODS.** Shear bond strengths of different cements with various tooth surface treatments (none, etching, priming, or etching and priming) on enamel and dentin of human teeth were measured using the Swiss shear test design. Three adhesives (Permaflo DC, Panavia F 2.0, and Panavia V5) and one self-adhesive cement (Panavia SA plus) were included in this study. The interface of the cement and the tooth surface with the different pre-treatments was analyzed using SEM. pH values of the cements and primers were measured. **RESULTS.** The highest bond strength values for all cements were achieved with etching and primer on enamel ( $25.6 \pm 5.3$  -  $32.3 \pm 10.4$  MPa). On dentin, etching and priming produced the highest bond strength values for all cements ( $8.6 \pm 2.9$  -  $11.7 \pm 3.5$  MPa) except for Panavia V5, which achieved significantly higher bond strengths when pre-treated with primer only ( $15.3 \pm 4.1$  MPa). Shear bond strength values were correlated with the micro-retentive surface topography of enamel and the tag length on dentin except for Panavia V5, which revealed the highest bond strength with primer application only without etching, resulting in short but sturdy tags. **CONCLUSION.** The highest bond strength can be achieved for Panavia F 2.0, Permaflo DC, and Panavia SA plus when the tooth substrate is previously etched and the respective primer is applied. The new cement Panavia V5 displayed low technique-sensitivity and attained significantly higher adhesion of all tested cements to dentin when only primer was applied. [*J Adv Prosthodont 2017;9:85-92*]

**KEYWORDS:** Dual-cure dental bonding; Shear strength; Dentin; Enamel; Dental etching

## INTRODUCTION

The retention and consequently the longevity of indirect restorations strongly rely on the bond strength of the cement.<sup>1</sup> The adhesion between dentin and cement is generally more susceptible to failure than enamel-cement or ceramic-cement interfaces.<sup>1,2</sup>

The micromechanical bond of adhesive cement to den-

tin is based on the infiltration and polymerization of a synthetic resin into the collagen fibril network, referred to as hybrid layer.<sup>3,4</sup> The hybrid layer can seal dentin and prevent post-operative sensitivity and secondary caries and may as well act as an elastic buffer that compensates the tension generated by polymerization shrinkage of composite resin cement.<sup>4</sup>

To achieve a hybrid layer with the etch-and-rinse approach, dentin is pre-treated with an acidic agent, followed by priming and the application of a low viscous resin. Current adhesive cement systems use an etching step plus one bottle priming system, where primer and adhesive resin are combined into one application. In order to simplify the cementation process, self-etch adhesive cements containing non-rinsing acidic monomers for simultaneous etching and priming of the dental tissues were developed.<sup>4</sup> Due to their reduced ability to completely dissolve the smear layer, self-adhesive cements only superficially interact with dentin.<sup>5</sup> The absence of a dentin conditioning step creates partially demineralized dentin substrates that make it more difficult

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for the resin monomers to diffuse into the tubules.<sup>5,6</sup> The relatively high viscosity of the self-adhesive cement possibly contributes to this low infiltration.<sup>7,8</sup>

Each self-etch system or adhesive system contains its specific functional monomer that determines its actual adhesive performance. In addition to the micromechanical retention, specific carboxyl or phosphate groups of functional monomers chemically interact with residual hydroxyapatite on the partially demineralized dentin surface.<sup>9</sup> Adhesives containing 10-MDP have revealed a significantly stronger bonding potential to hydroxyapatite than 4-META or phenyl-P.<sup>9</sup>

To investigate cements and their adhesives, shear and microtensile bond strength tests are most commonly used.<sup>10</sup> The tests measure the adhesion between tooth and cement material in order to evaluate the ability to later resist stress imposed by oral function. In one study, the bond strengths of a conventional adhesive cement (Panavia F 2.0, Kuraray-Noritake, Kurashiki, Japan) and a self-etch cement (Clearfil SA, recently renamed Panavia SA, Kuraray-Noritake) to dentin were  $14.1 \pm 2.4$  MPa and  $13.8 \pm 1.9$  MPa, respectively, with etching and priming,  $12.5 \pm 2.3$  MPa and  $9.8 \pm 1.6$  MPa when only primer was applied, and  $12.8 \pm 2.6$  MPa and  $9.3 \pm 1.4$  MPa without pre-treatment.<sup>11</sup> Shear bond strength values cannot be considered as a material property because they depend on the substrate material and surface morphology and vary from test design to test design.<sup>12</sup>

Despite manufacturers' efforts in the development and marketing of user-friendly self-etching cement products, the question still remains whether clinicians should consider using the self-etching products over the conventional adhesive pre-treatment with etching and priming. According to the manufacturer's recommendations for a new adhesive cement (Panavia V5, Kuraray-Noritake), etching is no longer indicated for dentin and for enamel. The aim of this study is to evaluate the influence of the various surface pre-treatment steps on the bond strength of adhesive cements to enamel and dentin, including one conventional adhesive cement without MDP, one cement containing MDP, one self-etching cement, and the new conventional cement Panavia V5. The hypotheses are that (1) the bond strength is the highest when the substrate surface is etched and primed, and (2) the lowest when no pre-treatment is used.

## MATERIALS AND METHODS

Shear bond strengths of different cements with various tooth surface treatments (none, etching, priming, or etching and priming) on enamel and dentin of human teeth were measured using the Swiss shear test design.<sup>13-15</sup> Three adhesives and one self-adhesive cement system were included in this study (Table 1). The interface of the cement and the tooth surface with the different pre-treatments was analyzed using a replica technique with SEM. pH values of the

**Table 1.** Cements and primers used

Name	Code	Lot No.	Composition	Manufacturer
Panavia F 2.0	PF2	630066/7A0013	MDP, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, Silanated silica filler, Silanated colloidal silica, dl Campherquinone, Catalysts, Initiators, Silanated barium glass filler, Surface treated sodium fluoride, Accelerators, Pigments	
ED Primer II	EDP2	00309B/00183B	HEMA, MDP, Water, 5-NMSA, Accelerators, Catalysts	
Panavia V5	PV5	1T0001	Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, Initiators, Accelerators, Silanated barium glass filler, Silanated fluoroaluminosilicate glass filler, Colloidal silica, Silanated aluminium oxide filler, dl Camphorquinone, Pigments	Kuraray Noritake Dental, Kurashiki, Japan
Panavia V5 Tooth Primer	PVTP	3H0001	MDP, HEMA, Hydrophilic aliphatic dimethacrylate, Accelerators, Water	
Panavia SA Cement Plus	PSA	8X0015	MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, HEMA, Silanated barium glass filler, Silanated colloidal silica, dl, Camphorquinone, Peroxide, Catalysts, Pigments, Hydrophobic aliphatic dimethacrylate, Surface treated sodium fluoride, Accelerators	
Clearfil Universal Bond	CUBO	3E 0007	MDP, Bis-GMA, HEMA, Hydrophilic aliphatic dimethacrylate, Colloidal silica, Silane coupling agent, dl Camphorquinone, Ethanol, Water	
Permaflo DC	PDC	BB3y3	TEGDMA, Bis-GMA	Ultradent Products, South Jordan, UT, USA
Peak Universal Bond	PUBO	B996D	Ethyl Alcohol, HEMA, Methacrylic Acid, Chlorhexidine diacetate	

cements and primers were measured.

For this study, 144 human third molar teeth were used. The use of human teeth for laboratory studies was approved by the Ethics Committee of northwest/central Switzerland (EKNZ UBE-15/111). The teeth were cleaned from any remnant tissue or contamination and stored at room temperature in an aqueous solution of 0.9% NaCl and 0.02% thymol. All teeth were tested in less than 6 months after extraction. The teeth were sectioned in mesio-distal direction using a wire saw under permanent water cooling (well Walter Ebner, type 3241, Le Locle, Switzerland) and then embedded in a square form in cold-curing resin (Demotec 20, Nidderau, Germany).

The surface of the teeth was ground and then polished with silicon carbide polishing paper (Struers, Ballerup, Denmark P180, 400, 800, 1200) using a polishing machine (Knuth Rotor, Struers) from the buccal or oral side to expose an enamel surface of at least 5 mm in diameter.

After performing the shear bond strength test on enamel, the specimens were ground down until a dentin surface of at least 5 mm in diameter was exposed. The tooth surface was polished again with polishing paper and used for testing shear bond strength on dentin. The specimens were divided into 32 different groups (Table 2). 320 ( $n = 10$ ) specimens were prepared in total for shear bond strength test and 64 for SEM analysis ( $n = 2$ ).

The procedure of the Swiss shear test used in this study is described in detail elsewhere<sup>14-16</sup> and will only be briefly summarized. The tooth surfaces of the different groups were pre-treated as listed in Table 2, either without any treatment, by etching with phosphoric acid (Panavia, Etching Agent V, Kuraray Noritake Dental, Kurashiki, Japan), by application of the corresponding primer, or by both etching and primer application. Subsequently, the specimens were fixed in a customized holding device. An acrylic cylinder (D+R Tec, Birmensdorf, Switzerland) with an inner diameter of 2.9 mm was fastened vertically on the exposed tooth

surface. Cement was applied through the opening of the acrylic cylinder onto the tooth surface. The cement was compressed with a headless steel screw with a force of 1 N and light-cured (Elipar S10, 3M ESPE, Seefeld, Germany) for 20 seconds from 3 different directions. Subsequently, the specimens were removed from the holding device and stored in water at 37°C for 24 hours. Shear bond strength test was performed at a crosshead speed of 1 mm/min using a universal testing machine (Z020, Zwick/Roell, Ulm, Germany). The force at fracture was recorded (software testXpert II 3.61, Zwick/Roell).

For SEM replica analysis, two specimens of each group were prepared according to the process described above. The 64 specimens were then stored in 37% hydrochloric acid (Sigma-Aldrich, Steinheim, Germany) for 3 days until all dental tissue was dissolved. The specimens then were prepared for SEM analysis (Philips XL30 FEG ESEM, Philips Electron Optics, Eindhoven, Netherlands) to observe the bonding area. SEM imaging was performed at 15 kV using magnifications of  $\times 25$ ,  $\times 250$ ,  $\times 1000$ , and  $\times 5000$ .

pH of all cements and primers were measured three times each using pH-indicator strips within a range of pH 0-14 and 0.5-5.0 (Merck, Darmstadt, Germany). After applying cement to the indicators, the strips were dipped in distilled water for 1s. No water was added when primer was tested. The color matching was performed in a wet condition.

Two-way ANOVA was performed on the shear bond strength values to test for differences among different cements, pre-treatments, and tooth surfaces (Minitab 16, Minitab, Coventry, UK).  $P$  values  $< .05$  were considered statistically significant.

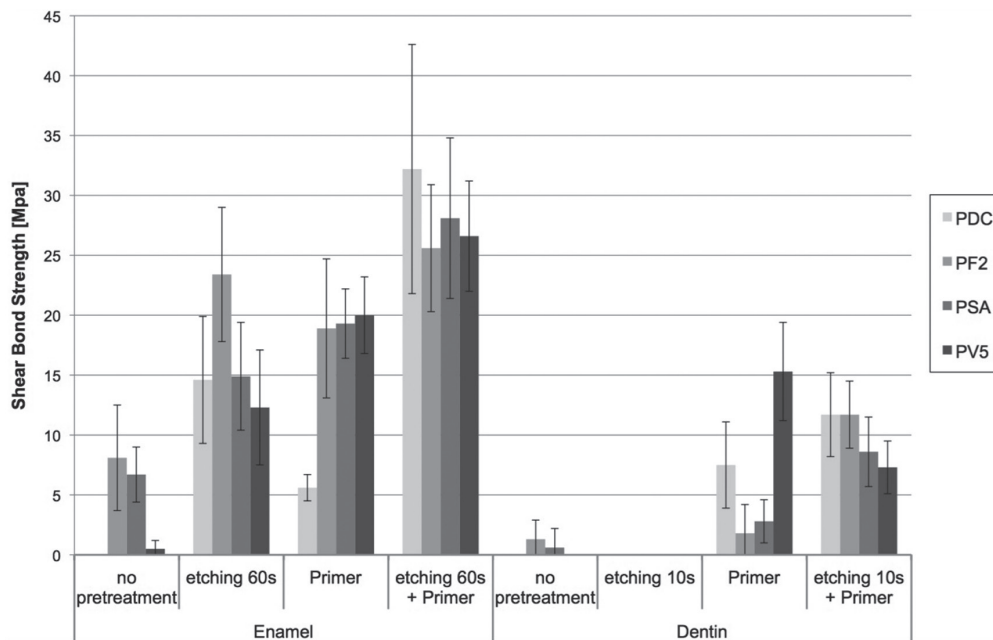
## RESULTS

The shear bond strength values for the cements on enamel and dentin are presented in Fig. 1 and Table 3. The highest values for all cements were achieved with etching and primer on enamel. On dentin, etching and priming produced the highest bond strength values for all cements except for PV5, which achieved a significantly higher bond strength when pre-treated with primer only ( $P < .001$ ). Spontaneously de-bonded specimens were recorded as 0 MPa. No bond was attained for PDC on enamel and dentin without pre-treatment, for PV5 on dentin without pre-treatment, and for all cements in the etching group on dentin.

In the enamel group without pre-treatment, PF2 and PSA attained significantly higher bond strengths than PDC and PV5 ( $P < .001$ ). In the enamel etching group, the values of PF2 differed significantly from those of the other cements ( $P < .001$ ). PDC achieved significantly lower bond strengths than the other cements in the enamel primer group ( $P < .001$ ). No statistical difference was found among the cements in the enamel etched and primed group ( $P = .190$ ). Without pre-treatment, PF2 revealed significantly higher values on dentin than PDC and PV5 ( $P < .001$ ) but

**Table 2.** Specimen surface treatments for shear bond strength (320 specimens,  $n = 10$  per group) and SEM analysis (64 specimens,  $n = 2$ )

	Surface treatment
Enamel	none
	etching 60s (phosphoric acid 37%)
	Primer 20s, blow-dry 5s
	etching 60s and Primer 20s, blow-dry 5s
Dentin	none
	etching 10s (phosphoric acid 37%)
	Primer 20s, blow-dry 5s
	etching 10s and Primer 20s, blow-dry 5s



**Fig. 1.** Shear bond strength means for the cements with different pre-treatments on enamel and dentin. Debonded specimens during water storage were counted as 0 MPa.

**Table 3.** Shear bond strength (MPa) means and standard deviations (n = 10). Debonded specimens during water storage were counted as 0 MPa

		PDC	PF2	PSA	PV5
Enamel	no pretreatment	0.0 (0.0)	8.1 (4.4)	6.7 (2.3)	0.5 (0.7)
	etching 60s	14.6 (5.3)	23.4 (5.6)	14.9 (4.5)	12.3 (4.8)
	Primer	5.6 (1.1)	18.9 (5.8)	19.3 (2.9)	20.0 (3.2)
	etching 60s + Primer	32.3 (10.4)	25.6 (5.3)	28.1 (6.7)	26.6 (4.6)
Dentin	no pretreatment	0.0 (0.0)	1.3 (1.6)	0.6 (1.6)	0.0 (0.0)
	etching 10s	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)	0.0 (0.0)
	Primer	7.5 (3.6)	1.8 (2.4)	2.8 (1.8)	15.3 (4.1)
	etching 10s + Primer	11.7 (3.5)	11.7 (2.8)	8.6 (2.9)	7.3 (2.2)

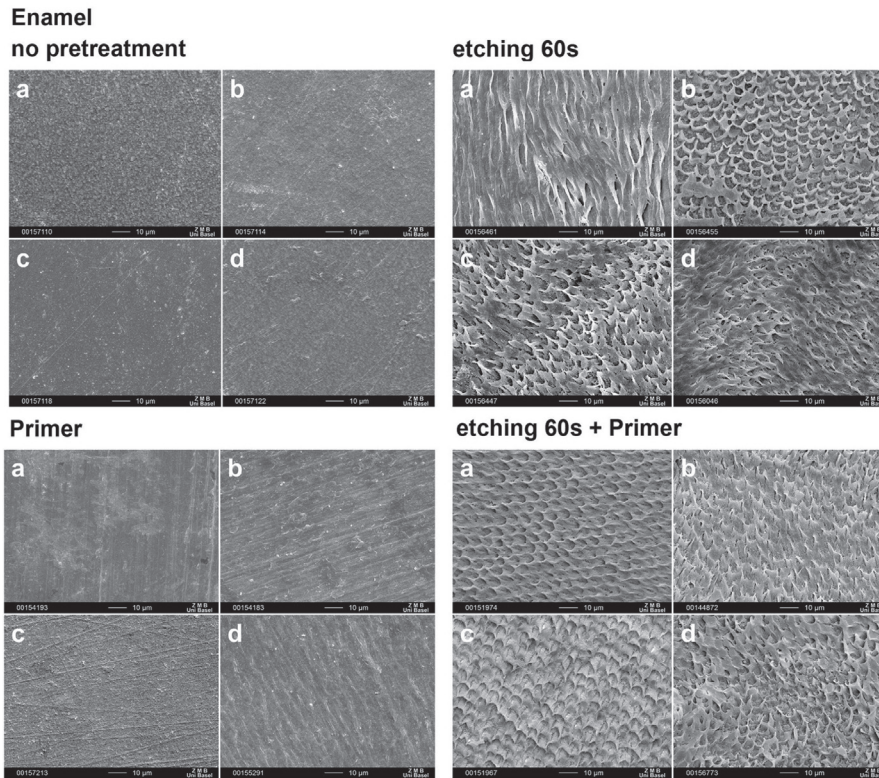
not than PSA ( $P = .348$ ). No statistical difference was found among the etched dentin specimens ( $P < .001$ ). When only primer was applied on dentin, PV5 achieved significantly higher values than all the other cements ( $P < .001$ ). In the same group, PDC attained significantly higher values than PF2 and PSA ( $P < .001$ ). PV5 revealed significantly lower values than PDC ( $P = .003$ ) and PF2 ( $P = .001$ ) in the etched and primed dentin group. Among the different pre-treatments on enamel and dentin, a significant difference was noted ( $P < .001$ ). Cements differed significantly from each other regarding all pre-treatments ( $P < .001$ ).

Pure adhesive failures of enamel specimens occurred for

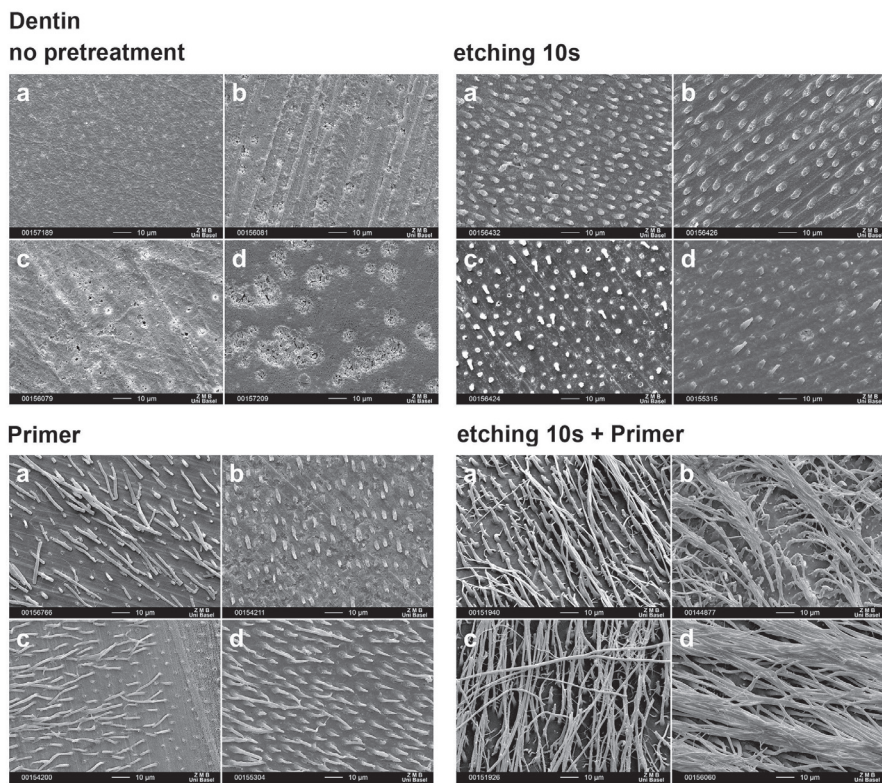
bond strengths below 20 MPa. A tendency to cohesive failure modes was observed for bond strengths higher than 20 MPa. On dentin specimens, only adhesive failures were recorded.

SEM images of the replicas at a magnification of  $\times 1000$  are presented in Fig. 2 and Fig. 3. Across the specimen with a diameter of 2.9 mm, different patterns were observed depending on the variation in tooth structure. The most representing pattern of each specimen was chosen for the analysis. The specimens bonded to tooth surfaces without pre-treatment revealed a smooth surface without any imprints of tooth structures.





**Fig. 2.** SEM specimen replicas of enamel for all cements with different pre-treatments ( $\times 1000$ ). a) PDC, b) PF2, c) PSA, d) PV5.



**Fig. 3.** SEM specimen replicas of dentin for all cements with different pre-treatments ( $\times 1000$ ). a) PDC, b) PF2, c) PSA, d) PV5.

**Table 4.** pH values for cements and primers

Name	pH
PF2	3.0
EDP2	2.0
PV5	6.0
PVTP	2.0
PSA	4.0
CUBO	2.5
PDC	5.0
PUBO	3.5

On the etched enamel specimen replicas, an imprint of the etched surface was developed. The etching patterns differed due to the orientation of the enamel prisms. On the dentin specimens, all cements developed small resin tags. Similar surfaces as for the non-treated enamel specimens were observed for the specimens where primer was previously applied. Slight imprints of enamel prisms were detected on the PV5 specimens. On the dentin replica of PF2 with primer, formation of small thick resin tags was observed. On the PDC and PSA specimens, longer, fragile resin tags were formed. A more regular pattern of thicker and longer resin tags was noted for PV5.

Clear-cut replicas were found for the etched and primed enamel specimens for all cements. Differences in patterns were due to the prism orientation of the enamel. Long resin tags with lateral branches were observed for all the etched and primed dentin specimens.

pH values of cements and primers are listed in Table 4. The lowest values were recorded for primers EDP2 and PVTP (pH 2.0) and the highest for PV5 cement (pH 6.0).

## DISCUSSION

The present study was designed to demonstrate the impact of different tooth surface treatments on shear bond strength. The first hypothesis that the bond strength of the different cements would be the highest when the substrate surface was previously etched and primed was confirmed for all cements except PV5 on dentin. This cement revealed significantly higher shear bond strength values than the other cements on dentin in absence of etching when only primer was applied. The second hypothesis that the lowest values would be generated when no pre-treatment was used was confirmed for the enamel substrate. When bonding to dentin, an etched surface even led to lower values for PF2 and PSA values than a polished surface.

Surface treatment with phosphoric acid enhances the topography of enamel, changing it from a low-reactive surface to a surface that is more susceptible to adhesion due to

its higher surface free energy.<sup>16</sup> Difference of orientation of the enamel prisms caused irregular etching patterns in SEM replicas. By capillary attraction, resin cement is drawn into the microporosities of the demineralized enamel. The viscosity of the resin cement, which seems to be related to the amount of fillers,<sup>17</sup> affects the penetration depth and subsequently the bond strength. For all cements, significantly higher values were measured when the enamel surface was etched compared to no pre-treatment. Bond strengths were achieved without pre-treatment of the enamel for the two cements containing MDP (PF2 and PSA). These cements also revealed the lowest pH-values (PF2 pH 3, PSA pH 4). pH levels of 3 and 4 were not acidic enough to etch the enamel surface according to the SEM imaging, where specimens without pre-treatment revealed a smooth surface. Chemical bonding of MDP to hydroxyapatite seems to have added to the bond obtained by microretention for the etched MDP containing specimens as well, especially for PF2. In a clinical study, selective enamel etching significantly increased the survival rate of partial crowns over 6.5 years, confirming the results found in this study.<sup>18,19</sup>

Etching of dentin removes the smear layer and exposes the dentin tubules. Adhesion to dentin is generally achieved through microretention and chemical bond. For the etched dentin specimens, SEM analysis revealed that the cement was able to minimally (1 - 2  $\mu\text{m}$ ) penetrate into the tubules. Due to the high viscosity of the cements, a deeper infiltration of the tubules for a sufficient microretention could not be achieved. A slight chemical bond of MDP to hydroxyapatite (PF2, PSA) of the non-etched dentin specimens was attained. With the removal of the smear layer by etching and therefore the removal of hydroxyapatite-crystals, no chemical bond could be accomplished for the etched specimens. Remaining collagen fibers might have formed an isolating layer that prevented the cement from bonding to the dentin.

Most adhesive systems that use the total-etch technique contain low-viscosity hydrophilic monomers diluted in organic solvents with a high potential of volatilization, such as acetone or water. Bipolar molecules with a hydrophilic and a hydrophobic end (e.g. HEMA, BPDM, 4-META, MDP) are able to interact under moist conditions with the tooth and with the other end to the resin matrix.<sup>20</sup> For the non-etched enamel specimens that were treated with primer, chemical bond strength was probably achieved from HEMA for all cements. PF2, PSA, and PV5 contain MDP in the primer, which might have attributed to the chemical bond that resulted in significantly higher bond strength values. The low pH value of 2.0 of EDP2 and PVTP resulted in a slight etching of the enamel surface.

For the dentin specimens pre-treated with primer only, the highest values were measured for PV5/PVTP. Due to its low pH of 2, PVTP seemed to be able to simultaneously etch and sufficiently penetrate the tubules. The resin tags on the PV5 replica were not as long (5 - 20  $\mu\text{m}$ ) as those observed for the other cements (up to 100  $\mu\text{m}$ ) in the etched and primer group, although higher bond strengths



were achieved. PV5/PVTP seemed to be able to attain a strong chemical bond in addition to reduced micromechanical retention. EDP2 (PF2), of which a pH of 2 was recorded, was not able to sufficiently penetrate the tubules to achieve a bond to hydroxyapatite and therefore revealed the lowest values. The primer containing chlorhexidine PUBO (PDC) achieved higher bond strength than EDP2 (PF2) and CUBO (PSA). Longer and more fragile resin tags were observed for PUBO (PDC) and CUBO (PSA) (Fig. 3), which also exhibited higher pH values.

When phosphoric acid and primer were applied on enamel surface, best bonding quality was achieved for all cements. Etching provided an enlargement of the surface, in which the viscous primer could penetrate through capillary attraction for sufficient micromechanical retention. Chemical bond might have added to these high bond strength values, but the effect could not be evaluated since the bond strengths were so high that cohesive failures occurred.

Etching and primer application seemed to be necessary in order to achieve a bond to dentin for PDC, PF2, and PSA. When the surfaces of the PV5 specimens were previously etched, lower bond strengths were achieved than for the non-etched specimens.

The values found in this study cannot be directly compared to the values from other studies due to variations in test set-ups. The results in a study with a comparable test design,<sup>11</sup> where similar cements on different pre-treatments of dentin were tested for shear bond strength, do not correlate to the findings in this study. For PF2, similar bond strengths were found<sup>11</sup> when the teeth were pre-treated either with etching and primer, primer only, or not at all. In the present study, significantly higher values were measured for the etched and primer treated specimens than for only primed or no pre-treated ones. In a clinical study where partial crowns were cemented in a split-mouth design with self-adhesive or adhesive cement, significantly higher survival rates over 2 years were recorded for the adhesive procedure.<sup>21</sup> Debondings were recorded for the self-adhesive cement, confirming the results found in this study that enamel etching and primer application was essential for a successful bonding strategy.

Thermocycling should be added to the test design in future studies to investigate the impact of aging on shear bond strength.

The highest bond strength can be achieved for PF2, PDC, and PSA when the tooth substrate is previously etched and the respective primer is applied. For PSA, which is commonly used as self-adhesive cement, it is strongly recommended to use an additional primer such as CUBO to achieve a significant improvement in bond strength. The new cement PV5 revealed the highest adhesion of all tested cements to dentin. No phosphoric acid should be applied when restorations such as crowns or bridges are bonded with PV5 to both dentin and enamel since previous etching reduces the bonding quality to dentin. Cohesive fractures that occurred for the enamel specimens achieving bond

strengths of 20 MPa indicate a sufficient bond to enamel without etching, although thermocycling and micro-leakage should be investigated in further studies. When cementing enamel based restorations such as veneers or adhesive bridges, etching of enamel should be performed to achieve the highest bond strengths for PV5.

The approach, chosen in the development of PV5 to simplify a conventional adhesive cement system in combining easy handling by using a single primer bottle without previous etching as well as an auto-mixed cement, revealed promising results. The incorporation of a self-etch component and a multi-step adhesive into a single cement system reduces technique-sensitivity and facilitates daily clinic routines.

A merging of self-adhesive and conventional adhesive cement systems may become the future trend in development strategies.

## CONCLUSION

Etching and primer application should be performed to achieve sufficient bond strengths for conventional adhesive and self-adhesive cements. A primer (Panavia V5 Tooth Primer) can supersede tooth etching in the adhesive cementation process and increase bond strength values to dentin of reliable cements presently on the market. Based on the present results, Panavia V5 has the potential to realign future concepts of adhesive cementation.

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