Influence of Acid-base Conditioning on the Bond Strength of Five Luting Agents Employing Self-etching Primer to Enamel and Dentin

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The purpose of this study was to evaluate the effect of multi-step conditioning (PA-AD conditioning) with phosphoric acid and sodium hypochlorite on the bond strength of five luting materials to enamel and dentin. Three commercial self-etching/priming systems (Panavia, Linkmax, and Multibond) and two experimental systems (ED/Super-Bond and EDFe/Super-Bond) were used. The surfaces of bovine enamel or dentin were bonded to a stainless steel rod. Tensile bond strength was determined after 24-hour immersion in water. PA-AD conditioning significantly improved the bond strength between enamel and three of the systems (Panavia, ED/Super-Bond, and EDFe/Super-Bond), but did not have any effect on Linkmax and Multibond. Likewise, PA-AD conditioning did not significantly improve the bond strength of Panavia, Linkmax, Multibond, and ED/Super-Bond to dentin. Highest bond strength to dentin (19.7 MPa) was obtained when self-etching primer containing ferric chloride (EDFe/Super-Bond) was used, but additional PA-AD conditioning significantly weakened the bonding (12.6 MPa).

Key words : Adhesion, Tooth, Ferric chloride

INTRODUCTION

Adhesive bonding between luting material and tooth substance is essential for restoring teeth with minimal intervention at the preparation stage. Recently, a self-etching primer system that combines the functions of both etching and priming steps has been widely used in the bonding of indirect restorations, orthodontic brackets, and resin composite materials^{1–5}. However, such a self-etching primer system does not always provide a sufficiently strong bond with enamel or dentin, as could be achieved with conventional acid etching systems^{6–8}.

Dissolution of the exposed dentin collagen following acid etching is another interesting approach to achieving adhesion. The organic fraction of the smear layer is not always removed by acid etching^{9,10}. In a surface modification technique (PA-AD conditioning), the smear layer is removed with 40wt% phosphoric acid solution and the exposed dentin collagen is dissolved with 10wt% sodium hypochlorite (NaClO)^{11–13}.

Although phosphoric acid etching prior to the application of a self-etching primer was found to improve enamel bonding, additional etching occasionally reduced the durability of the bond between resin and dentin¹⁴. Moreover, some adhesive resins were adversely affected when dentin was irrigated with NaClO¹⁵. To improve adhesive bonding to enamel and dentin, a manufacturer recommended that PA-AD conditioning be used followed by a self-etching primer system (Panavia F 2.0, Kuraray Medical Co.

Ltd., Osaka, Japan). However, limited information is available as to whether PA-AD conditioning would improve the bond strength of a luting agent which employs a self-etching primer^{16,17}.

A commercially available bonding system (Super-Bond C&B, Sun Medical Co. Ltd., Moriyama, Japan) employs a dentin conditioner containing citric acid with ferric chloride. In a previous study, when self-etching primer was modified using iron (II) perchloride, it successfully increased the bond strength between tri-*n*-butylborane (TBB)-initiated resin and dentin¹⁸. It was also reported that self-etching primers containing ferric chloride promote adhesion to enamel as well as phosphoric acid etching^{19,20}.

The purpose of this study was to investigate the effect of PA-AD conditioning on the bond strength of five different luting materials employing self-etching primer to enamel and dentin.

MATERIALS AND METHODS

The materials used in the present study are listed in Table 1. A total of 120 mandibular incisors extracted from cattle were randomly divided into 20 groups of six each, 10 groups for enamel and the remaining 10 groups for dentin (Table 2). The labial portion of 60 teeth was ground using a water-cooled model trimmer (Y-230, Yoshida Dental Distribution, Tokyo, Japan) and abraded on wet #400 and #600 silicon carbide papers to achieve a flat enamel surface. The remaining 60 teeth were also ground and

Table 1 Surface treatment agents, self-etching primers, and luting agents used

Material name (Abbreviation)	Components	Manufacturer	Lot number
<i>Surface treatment agent</i>			
K-etchant (PA)	40wt% phosphoric acid, Water	Kuraray Medical Co. Ltd., Osaka, Japan	254
AD Gel (AD)	10wt% sodium hypochlorite, Water		00474
<i>Self-etching primer</i>			
ED Primer II (ED)	Liquid A: HEMA, MDP, 5-NMSA, Accelerator, Water	Kuraray Medical Co. Ltd.	00181A
	Liquid B: 5-NMSA, Accelerator, Water		00062A
Linkmax Self-etching Primer	EP-A: 4-MET, Methacrylate, Ethanol, Water	GC Corp., Tokyo, Japan	0301241
	EP-B: Ethanol, Initiator		0301241
Multibond Primer	Primer A: Phosphoric monomer, Acetone	Tokuyama Dental Corp., Tokyo, Japan	110
	Primer B: Borate initiator, Acetone, Water		209
Experimental (EDFe)	33.3wt% ED Primer II Liquid A	Kuraray Medical Co. Ltd.	00181A
	33.3wt% ED Primer II Liquid B		00062A
	0.1wt% Ferric chloride hexahydrate	Katayama Chemical Industries Co. Ltd., Osaka, Japan	3747
	Water		
<i>Luting agent</i>			
Panavia F 2.0	Paste A: MDP, Methacrylate monomer, Filler, Photoinitiator, Initiator	Kuraray Medical Co. Ltd.	00023A
	Brown Paste B: Methacrylate monomer, Filler, NaF, Photoinitiator, Initiator		00012A
	Oxyguard II: Polyethyleneglycol, Accelerator		00464A
Linkmax	Paste A: Aluminosilicate glass powder, Methacrylate	GC Corp.	0301071
	Paste B: Aluminosilicate glass powder, Methacrylate		0301071
Multibond	Liquid: MAC-10, MMA, Amine, Multifunctional methacrylate	Tokuyama Dental Corp.	325
	Ivory powder: PMMA, BPO		403
Super-Bond C&B	Initiator: Tri- <i>n</i> -butylborane derivative	Sun Medical Co. Ltd., Moriyama, Japan	GE31
	Monomer liquid: 5wt% 4-META, MMA		GG5
	Opaque ivory powder		FV1

HEMA: 2-hydroxyethyl methacrylate;
 MDP: 10-methacryloyloxydecyl dihydrogen phosphate;
 5-NMSA: 5-(*N*-methacryloylamino)salicylic acid;
 4-MET: 4-methacryloyloxyethyl trimellitate;
 4-META: 4-methacryloyloxyethyl trimellitate anhydride;
 MMA: methyl methacrylate;
 PMMA: poly(methyl methacrylate);
 BPO: benzoyl peroxide;
 MAC-10: 11-methacryloyloxyundecan-1,1-dicarboxylic acid.

Table 2 Bonding systems evaluated

Group name	K-etchant	AD gel	Self-etching primer	Luting agent	Light cure
Panavia	None	None	ED Primer II (A/B), 30 sec	Panavia F 2.0, Oxyguard II	20 sec
Linkmax	None	None	EP-A/EP-B, 30 sec	Linkmax	3 sec
Multibond	None	None	Primer A/Primer B, 30 sec	Multibond	None
ED/Super-Bond	None	None	ED Primer II (A/B), 30 sec	Super-Bond C&B	None
EDFe/Super-Bond	None	None	EDFe Primer, 30 sec	Super-Bond C&B	None
PA-AD/Panavia	15 sec (15 sec)	60 sec (15 sec)	ED Primer II (A/B), 30 sec	Panavia F 2.0, Oxyguard II	20 sec
PA-AD/Linkmax	15 sec (15 sec)	60 sec (15 sec)	EP-A/EP-B, 30 sec	Linkmax	3 sec
PA-AD/Multibond	15 sec (15 sec)	60 sec (15 sec)	Primer A/Primer B, 30 sec	Multibond	None
PA-AD/ED/Super-Bond	15 sec (15 sec)	60 sec (15 sec)	ED Primer II (A/B), 30 sec	Super-Bond C&B	None
PA-AD/EDFe/Super-Bond	15 sec (15 sec)	60 sec (15 sec)	EDFe Primer, 30 sec	Super-Bond C&B	None

(): rinse time in seconds

abraded to expose a sufficient area on the dentin surface for bonding.

The surfaces were modified with 40wt% phosphoric acid etchant for 15 seconds, followed by 10wt% sodium hypochlorite agent for 60 seconds. A 50- μ m thick adhesive masking tape, with a circular hole of 5-mm diameter, was positioned on the surface of each specimen to demarcate the bonding area. Self-etching primer was applied to the bonding area, and then using finger pressure a stainless steel rod (8 mm in diameter and 30 mm in length) was bonded to the specimen with the luting material. Specimens without PA-AD conditioning were also prepared as control. For Super-Bond C&B system, no original dentin conditioner was used as it was not a self-etching primer system. All bonded specimens were left in an atmospheric environment for 30 minutes, and then stored in water at 37°C for 24 hours. Tensile bond strength was determined using a universal testing device (AGS-10kNG, Shimadzu, Kyoto, Japan) at a cross-head speed of 2.0 mm/min.

Mean and standard deviation of six specimens were calculated for each group. All data were analyzed using analysis of variance (ANOVA) and a *post-hoc* Tukey compromise test ($p=0.05$). Fractured tooth surfaces were observed using an optical microscope (SMZ-10, Nikon, Tokyo, Japan) at $\times 80$ magnification. Failure modes were categorized into eight groups as follows: within luting material (C), resin-tooth interface (A), fracture within enamel (E), fracture within dentin (D), and four combinations of these modes (AC, CE, ACE, and ACD).

Four additional tooth specimens, two enamel and two dentin, were prepared for micro-photographic evaluation. The specimens were ground flat and abraded on wet #400 and #600 silicon carbide pa-

pers. Two specimens, enamel and dentin, were then treated with 40wt% phosphoric acid agent and 10wt% sodium hypochlorite gel as described above. The surfaces were sputter-coated with gold (Ion Coater IB-3, Eiko Engineering, Mito, Japan) and then observed using a scanning electron microscope (S-3500N, Hitachi, Tokyo, Japan) at $\times 3000$ magnification.

RESULTS

Three-way ANOVA results revealed that bond strength was influenced by the following factors: adherend (F-value: 175.3, P-value: 0.0001), self-etching system (F-value: 34.0, P-value: 0.0001), and PA-AD conditioning (F-value: 15.2, P-value: 0.0002). With the exception of self-etching system/PA-AD conditioning, the interactions were significant in all combinations. Then, all bond strength data were analyzed with one-way ANOVA (F-value: 23.0, P-value: 0.0001) followed by post-hoc test.

Table 3 shows the mean and standard deviation of tensile bond strength for each group together with its mode of bonding failure. The mean bond strength obtained in enamel varied from 8.2 to 25.3 MPa. In the absence of PA-AD conditioning, the bond strength to enamel with Panavia, ED/Super-Bond, and EDFe/Super-Bond was higher than that with Linkmax. Between Multibond and Linkmax, there were no significant differences in bond strength to enamel. When PA-AD conditioning was applied, the bond strengths of Panavia, ED/Super-Bond, and EDFe/Super-Bond to enamel were significantly greater. The strongest bonds were achieved by PA-AD/ED/Super-Bond (25.1 MPa) and PA-AD/EDFe/Super-Bond (25.3 MPa).

Table 3 Tensile bond strength results

Group Name	Enamel		Dentin	
	Mean (SD)* (MPa)	Bonding failure** (number of specimens)	Mean (SD)* (MPa)	Bonding failure** (number of specimens)
Panavia	14.7 (1.8) ^{cde}	AC(6)	6.1 (2.7) ^a	A(4) AC(2)
Linkmax	8.2 (3.0) ^{ab}	C(3) AC(3)	5.7 (0.6) ^a	A(2) AC(3) ACD(1)
Multibond	13.1 (4.0) ^{bcd}	AC(6)	5.6 (1.2) ^a	A(1) AC(3) ACD(2)
ED/Super-Bond	18.2 (4.5) ^{def}	AC(4) ACE(2)	8.7 (3.8) ^{abc}	AC(6)
EDFe/Super-Bond	17.7 (3.5) ^{def}	AC(6)	19.7 (4.5) ^{efg}	AC(4) ACD(1) D(1)
PA-AD/Panavia	21.6 (2.9) ^{fg}	AC(6)	9.9 (1.9) ^{abc}	A(1) AC(3) ACD(2)
PA-AD/Linkmax	11.0 (3.4) ^{abc}	C(4) AC(2)	9.5 (1.5) ^{abc}	AC(2) ACD(4)
PA-AD/Multibond	14.7 (4.0) ^{cde}	C(3) AC(3)	7.3 (3.4) ^{ab}	C(1) A(1) AC(3) ACD(1)
PA-AD/ED/Super-Bond	25.3 (3.8) ^g	C(1) CE(2) E(3)	4.5 (4.0) ^a	A(4) AC(2)
PA-AD/EDFe/Super-Bond	25.1 (3.9) ^g	C(2) AC(2) CE(1) E(1)	12.6 (3.8) ^{bcd}	AC(5) ACD(1)

*Identical letter indicates that values are not statistically different ($p \geq 0.05$)

**C: Cohesive failure within resin;

A: Adhesive failure at resin-enamel or resin-dentin interface;

E: Fracture at enamel;

D: Fracture at dentin;

AC, CE, ACE, ACD: Combined failure involving these failure modes.

Fig. 1 Scanning electron micrograph of an enamel surface: specimen ground with #600 silicon carbide paper.

Fig. 2 Scanning electron micrograph of an enamel surface: specimen treated with 40wt% phosphoric acid agent for 15 seconds followed by 10wt% sodium hypochlorite gel for 60 seconds. Prismatic and interprismatic substances were removed, thereby increasing microscopic roughness.

Fig. 3 Scanning electron micrograph of a dentin surface: specimen ground with #600 silicon carbide paper.

Fig. 4 Scanning electron micrograph of a dentin surface: specimen treated with 40wt% phosphoric acid agent for 15 seconds followed by 10wt% sodium hypochlorite gel for 60 seconds. Dental tubules were opened, accompanied by irregularity on the intertubular dentin surface.

With regard to dentin bonding, the mean bond strength ranged from 4.5 to 19.7 MPa. The bond strengths of Panavia, Linkmax, Multibond, ED/Super-Bond, PA-AD/Panavia, PA-AD/Linkmax, PA-AD/Multibond, and PA-AD/ED/Super-Bond were statistically identical. The mean bond strength of ED/Fe/Super-Bond, at 19.7 MPa, was significantly higher than those of the other groups.

Combined adhesive and cohesive failure (AC) was found in the largest number of specimens, followed by complete cohesive failure and complete adhesive failure. For enamel specimens, none showed complete adhesive failure. For dentin specimens with mean bond strength ≥ 8.7 MPa, complete adhesive failure

did not occur too. As for the interface between resin and stainless steel rod, no failures occurred.

Enamel surfaces modified by PA-AD conditioning showed a pattern wherein the smear layer and prism peripheries were removed, preferentially leaving the prism cores intact (Figs. 1 and 2). On the other hand, PA-AD conditioned dentin surface showed opened dental tubules, microscopically roughened intertubular dentin, and an absence of smear layer (Figs. 3 and 4).

DISCUSSION

Though no significant effects were detected in enamel, the addition of 0.1wt% ferric chloride to ED Primer II significantly improved the bonding between TBB-initiated resin and dentin. The concentration of ferric chloride used in this study was 1/20th of the optimal concentration of iron (II) perchloride¹⁸⁾ and 1/50th of the ferric chloride concentration reported¹⁹⁾.

The reason why TBB-initiated resin bonds well to dentin treated with ferric chloride has been accounted for in terms of two essential mechanisms. The first mechanism is about preventing the collapse of demineralized dentin matrix following the drying step^{21,22)}. However, there was no need for drying step in the present self-etching primer system — which meant that the risk of demineralized dentin matrix collapsing was eliminated. The comparison between PA-AD/ED/Super-Bond and PA-AD/EDFe/Super-Bond further indicated the positive effect of ferric chloride, in the light when exposed dentin collagen was depleted by PA-AD conditioning (Fig. 4).

The other mechanism focuses on the polymerization of resin after monomer diffuses into the demineralized dentin surface. Once polymerization is initiated at the resin-dentin interface, contraction stress would be reduced^{23,24)}. Free radicals are created when TBB is oxidized by oxygen, and water may be a co-catalyst with TBB in the polymerization of MMA²⁵⁾. It was also speculated that by the other redox reaction between TBB and ferric ions, more free radicals were produced as a result²⁶⁾. In the present study, the findings suggested that accelerated polymerization was the main factor that increased the bond strength of EDFe/Super-Bond by the addition of ferric chloride — which promoted polymerization.

After PA-AD conditioning, a significant reduction in bond strength was especially observed for PA-AD/EDFe/Super-Bond. This could be due to neutralization of ferric chloride by NaClO¹⁵⁾. It was suggested that NaClO remained on the adherend surface after PA-AD conditioning, even in cases when the surface was rinsed with water²⁷⁾. Neutralization could locally decrease the ferric ion, thereby suppressing radical polymerization.

PA-AD conditioning did not significantly affect the bond strength between Panavia and dentin. However, bonding durability might be improved as long as the smear layer and the underlying organic fraction were removed from the surface¹⁶⁾. The use of PA-AD conditioning with ED Primer II was effective with respect to enamel bonding (PA-AD/Panavia, PA-AD/ED/Super-Bond, and PA-AD/EDFe/Super-Bond). Phosphoric acid etching of PA-AD conditioning removed the inorganic fraction of the smear layer and increased the roughness of underlying enamel, thereby contributing to high bond

strength in the case of enamel bonding. Self-etching primer could demineralize tooth substrate and infiltrate the tubules simultaneously. It was therefore thought that the self-etching primer further changed the surface texture as shown in Figs. 2 and 4.

As shown in Table 3, failure modes shifted from adhesive to cohesive as bond strength increased. However, differences in failure mode among the different self-etching primer systems stemmed from differences in their chemical composition and mechanical properties. For example, pure cohesive failure observed in bonding systems using Linkmax or Multibond suggested that their cohesive strengths were lower than those of Panavia, ED/Super-Bond, and EDFe/Super-Bond.

In conclusion, PA-AD conditioning improved the bonding between Panavia and enamel, and between ED/Super-Bond and enamel, but not between these materials and dentin. Tensile bond strength to enamel was also significantly increased following PA-AD conditioning when tri-*n*-butylborane-initiated luting agent was used with a ferric chloride-modified primer (EDFe/Super-Bond). Although bonding between EDFe/Super-Bond and dentin was weakened by PA-AD conditioning, its bond strength was significantly higher than those of the other dentin bonding systems evaluated. As for Linkmax and Multibond systems, PA-AD conditioning did not significantly improve their bonding to enamel or dentin.

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