Effect of additional coating of bonding resin on the microtensile bond strength of self-etching adhesives to dentin

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ABSTRACT

This study investigated the hypothesis that the dentin bond strength of self-etching adhesive (SEA) might be improved by applying additional layer of bonding resin that might alleviate the pH difference between the SEA and the restorative composite resin. Two SEAs were used in this study; Experimental SEA (Exp, pH: 1.96) and Adper Prompt (AP, 3M ESPE, USA, pH: 1.0). In the control groups, they were applied with two sequential coats. In the experimental groups, after applying the first coat of assigned SEAs, the D/E bonding resin of All-Bond 2 (Bisco Inc., USA, pH: 6.9) was applied as the intermediate adhesive. Z-250 (3M ESPE, USA) composite resin was built-up in order to prepare hourglass-shaped specimens. The microtensile bond strength (MTBS) was measured and the effect of the intermediate layer on the bond strength was analyzed for each SEA using t-test. The fracture mode of each specimen was inspected using stereomicroscope and Field Emission Scanning Electron Microscope (FE-SEM). When D/E bonding resin was applied as the second coat, MTBS was significantly higher than that of the control groups. The incidence of the failure between the adhesive and the composite or between the adhesive and dentin decreased and that of the failure within the adhesive layer increased. According to the results, applying the bonding resin of neutral pH can increase the bond strength of SEAs by alleviating the difference in acidity between the SEA and restorative composite resin. 〔J Kor Acad Cons Dent 31(2):103-112, 2006〕

Key words : Self-etching adhesive, Microtensile bond strength test, pH, Intermediate layer, Fracture mode

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Ⅰ. INTRODUCTION

Most dentin adhesive systems in which the bonding procedure was performed with three steps, obtained reliable resin-dentin bond strengths. In order to simplify the clinical procedures, manufacturers introduced self-etching primer system or self-etching adhesive system

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(also termed 'all-in-one' adhesive system). With these systems, etching and priming dentin occurred simultaneously by infiltrating the smear-layer-covered dentin with acidic resin monomer. As a result, critical procedures like rinsing the etchant and priming the hydrated collagen fibers were eliminated. However, it is still unclear if these materials can produce strong, durable bonds when applied to dentin.

Several studies showed that most all-in-one adhesives exhibited relatively low bond strength when compared with two-step self-etching primer systems[1-3]. Recently, self-etching adhesives are also recommended to be applied at least twice to ensure that the etched dentin is adequately covered[4,5]. Although thicker adhesive layers were reported to contribute to absorb shrinkage stress of the polymerizing composite resin, protect the bond integrity, and reduce microleakage[6], there are still controversies about the relationship between the adhesive layer thickness and the bond strength[7,8].

Although thin adhesive layer was suggested as the cause of the relatively low bond strength of self-etching adhesives, two coat of self-etching adhesives still show relatively low bond strength than that of the adhesives of earlier generation[5]. Tay *et al*[9] reported "water tree" which passed through the adhesive layer from the hybrid layer to the composite resin in the transmission electron micrographs (TEM) of self-etching adhesives. From the scanning electron micrographic (SEM) images of the fractured surfaces, they also reported lots of voids being suspected as water blister from the fracture surfaces[10]. The pH of most self-etching adhesives ranges from 2.0 to 1.0[11]. The low pH might also interfere with the copolymerization of self-etching adhesive and restorative composite.

The hypothesis tested in this study was that the dentin bond strength of self-etching adhesives might be improved by applying additional layer of bonding resin that could alleviate the pH difference between the self-etching adhesive and the restorative composite resin. To investigate the hypothesis, after treating the dentin surface with self-etching adhesives, the same self-etching adhesive or bonding resin of a three-step dentin boning system was coated as the second coat and the microtensile bond strength (MTBS) was compared.

Ⅱ. MATERIALS & METHODS

Preparing the experimental adhesives

Two self-etching adhesives were used in this study, an experimental one-bottle and a commercial two-bottle self-etching adhesives. The adhesives used in this study were shown in Table 1.

Tooth preparation

After removing soft tissue debris, caries-free human molars were stored in 0.5% chloramines T solution for 24h, and then moved into distilled water. These teeth were used for the study within 6 months after extraction. These teeth were embedded in a cubic-shaped stainless steel mold with self-curing acrylic resin. The occlusal enamel was removed using a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under copious water irrigation. The exposed occlusal dentin surface was polished with 500 grit silicon carbide paper under running water.

Bonding procedure

The teeth were divided randomly into four groups. Adper Prompt (3M ESPE, St. Paul. MN, USA), and the experimental all-in-one adhesive were used as self-etching adhesives. Each adhesive was coated twice in the control groups. In the experimental groups, each adhesive was coated first, and as an alternative to the second coat of the assigned self-etching adhesives, D/E bonding resin of All-Bond 2 (Bisco, Itasca, IL, USA) was coated. Z-250 (3M ESPE, St. Paul. MN, USA) shade #A2 was used as restorative composite resin.

Table 1. Composition of the materials used in this study

	Composition of the materials	Manufacturer
Adper Prompt	Liquid 1:	3M ESPE,
	Methacrylated phosphoric esters	St. Paul. MN, USA
	Bis-GMA	
	Initiators based on camphoroquinone	
	Stabilizers	
	Liquid 2:	
	Water	
	2-Hydroxyethyl methacrylate (HEMA)	
	Polyalkenoic acid	
	Stabilizers	
Experimental self-etching adhesive	Ethylene glycol methacrylate Phosphate (EGMP)	
	MONO-2-(Methacryloyloxy) Ethyl phthalate (MEP)	
	Urethane dimethacrylate	
	2-Hydroxyethylmethacrylate Ethanol	
D/E resin of All-Bond2	Bisphenol A diglycidylmethacrylate	Bisco, Itasca, IL, USA
	Urethane dimetyhacrylate	
	Hydroxyethyl methacrylate	
Z-250	Matrix:	3M ESPE,
	UDMA, Bis-EMA and Bis-GMA	St. Paul. MN, USA
	Filler:	
	Zirconium glass and Coloidal silica	

Group 1: Experimental SEA, two coats

The polished dentin surface was air-dried shortly to be a little shiny and to have no excess water. Experimental adhesive was applied to the entire dentin surface and agitated for 5 seconds. After waiting for 20 seconds, the adhesive was gently air-dried and spread into a homogenous, slightly shiny film. Then, the second coat was applied in the same manner with the first coat. The adhesive-coated surface was light-cured for 20s using dental light curing unit (Hilux Ultra Plus, Benlioglu Dental Inc., Ankara, Turkey; Light intensity: 600 ㎽/㎠). Z-250 composite resin was built-up to approximately 3.5 ㎜ thick incrementally; the first layer in 0.5 ㎜ thick and the other layers in 1.0 ㎜ thick. Each layer was polymerized for 20s according to the manufacturer's instruction.

Group 2: Experimental SEA + D/E bonding resin

Experimental adhesive was applied to the entire dentin surface and agitated for 5 seconds. After waiting for 20s, the adhesive was gently air-dried and D/E bonding resin of All-Bond 2 was applied as the second coat. The adhesive-coated surfaces were light-cured for 20 seconds using the same curing unit. Composite resin was built-up with same manner described above.

Group 3: Adper Prompt, two coats

The same procedure with group 1 was done, except that Adper Prompt was used instead of the experimental adhesive.

Group 4: Adper Prompt + D/E bonding resin

The same procedure with group 2 was done, except that Adper Prompt was used instead of the experimental adhesive.

After bonding, the teeth were stored in distilled water at 4℃ until the preparation of the hourglass-shaped specimens for testing MTBS.

Microtensile bond strength test

Each specimen was trimmed to a rectangular shape using an Isomet low-speed diamond saw. In order to make hourglass-shaped slab specimens, two grooves were cut at both sides of the bonded interface under water irrigation with a diamond bur mounted in a low-speed drill press (Pressdrill, Samchully machinery Co. Ltd, Shiheung-City, Korea). And then, the tooth was serially sectioned with a thin diamond wheel (Buehler® Diamond Wafering Blade, Buehler Ltd.) mounted on the same low-speed saw, so that the dimension of the bonded surface area of the hourglass-shaped slab specimens were 1.05 ± 0.06 ㎜ wide and 0.65 ± 0.07 ㎜ thick (Figure 1). Copious water irrigation was always used through the procedures for sectioning specimens.

For measuring the MTBS, both the composite and tooth ends of each specimen were glued with gel-type cyanoacrylate cement (Super Glue Gel, 3M, St. Paul, MN, USA) on a specially designed measuring device, which had two parallel pins to guide the tensile load at a right angle to the bonded interface. Specimens were loaded to failure under tension using a universal testing machine (model 4466, Instron Inc., Canton, MA, USA) at a crosshead speed of 1.0 ㎜/min. The data were analyzed using t-test at a 5% level of significance. The statistical analysis was done using SPSS ver 10.0.

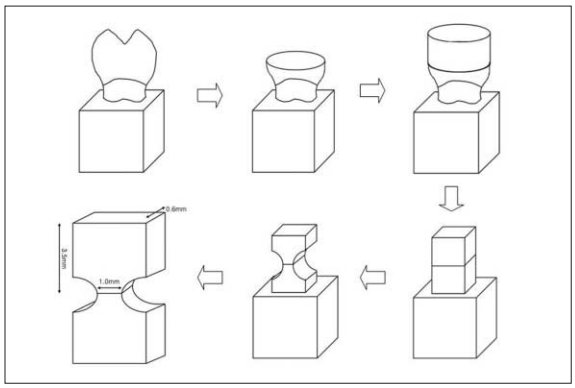

Figure 1. Schematic diagram of the specimen preparation.

Fracture mode analysis

After verifying and classifying fracture patterns with Field Emission Scanning Electron Microscope (FE-SEM), all the fractured surfaces of the specimens were evaluated under stereomicroscope. From the observations, the fractures were classified into six modes; cohesive failure in the composite resin, failure at the interface between the composite resin and the adhesive layer, failure within the adhesive layer, failure at the interface between the dentin and the adhesive layer, cohesive failure in the dentin, and failure of mixed modes including that within the adhesive layer.

Ⅲ. RESULT

The mean MTBS are presented in Figure 2. The MTBS of the group bonded with the experimental SEA plus D/E bonding resin (30.8 ± 10.8 ㎫, n = 22, t-test, $p < 0.05$) was significantly higher than that of the group bonded with two coats of the

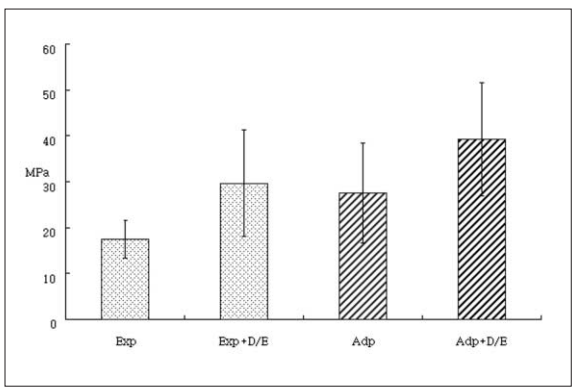

Figure 2. Microtensile bond strength (MTBS) to dentin. When D/E bonding resin was used as the second coat over the first coat of SEA, the MTBS of each SEA increased significantly.
Abbreviations. Exp: Experimental self-etching adhesive; D/E: D/E bonding resin of All-Bond 2; Adp: Adper Prompt.

experimental SEA (17.0 ± 4.2 ㎫; n = 21). The MTBS of the groups bonded with two coats of Adper Prompt (26.3 ± 10.6 ㎫, n = 22) and Adper Prompt plus D/E bonding resin (39.0 ± 11.5 ㎫, n = 21) also showed significant difference ($p < 0.05$).

In the specimens bonded with two coats of the experimental SEA, most failures were occurred at the interface between the dentin and the adhesive layer (16 out of 21 specimens, Table 2). Most of the specimens bonded with two coats of Adper Prompt failed at the interface between the composite resin and the adhesive layer (16 out of 22 specimens). On the other hand, the groups coated with D/E bonding resin as the second coat showed increasing numbers of the failure within the adhesive layer and mixed failures including that within the adhesive layer (Experimental SEA + D/E bonding resin; 12 out of 22, Adper Prompt + D/E resin; 10 out of 22, Table 2).

From the fractured surfaces on the dentin side of the specimens bonded with two coats of the experimental SEA, in some area numerous open dentinal tubules were observed without any covering and in other area the tubules were observed with resinous covering that was expected to be hybrid layer (Figure 3). It revealed that the fracture occurred at the bottom or at the top of the

Table 2. Fracture modes of tested specimens

Fracture modes	Exp 2coat	Exp + D/E	Adp 2coat	Adp + D/E
Composite resin cohesive	4	0	3	6
Composite resin - Adhesive layer	0	3	16	0
Within adhesive layer	0	7	1	7
Adhesive layer - Dentin	16	6	2	5
Dentin cohesive	0	1	0	0
Mixed	1	5	0	3
Total	21	22	22	21

Abreviations. Exp: Experimental adhesive; D/E: D/E bonding resin of All-Bond2; Adp: Adper Prompt.

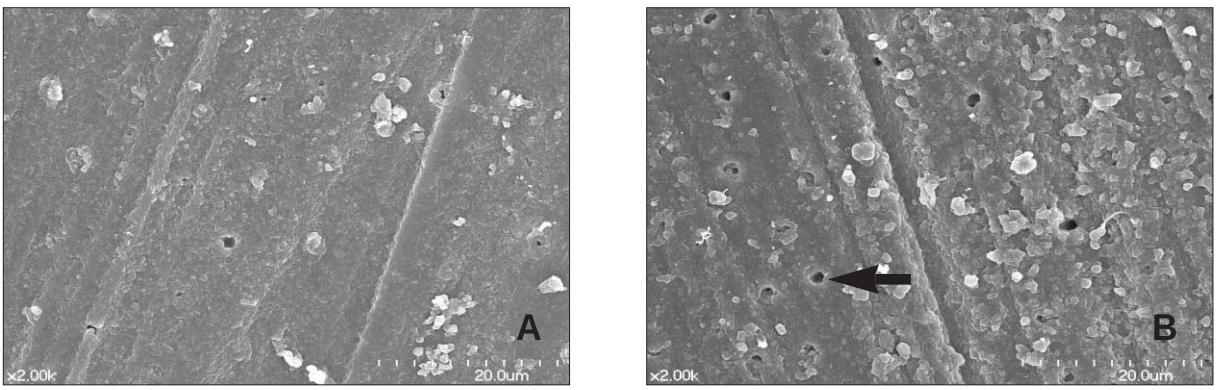

Figure 3. FE-SEM micrograph of two coats of the Experimental SEA. (A) fractured surface of resin side. (B) fractured surface of dentin side. Dentinal tubules were seen (arrow). Failures at the bottom or at the top of the hybrid layer locating between the adhesive layer and the dentin was observed.

hybrid layer locating between the adhesive layer and the dentin. The dentin side of the specimen bonded with two coats of Adper Prompt showed a lot of irregular voids that were similar to so called 'honeycomb appearance (Figure 5). The specimens of the groups bonded with D/E bonding resin as the second coat showed failure within the adhesive layer and failure of mixed modes including that within the adhesive layer (Figures 4 and 6).

Ⅳ. DISCUSSION

In this study, the MTBS test was used to measure the bond strength of two SEAs and to evaluate the effect of D/E bonding resin as the second coat of the SEAs on their bond strength. The greatest advantage of the test is that the test mechanics can reduce the number of cohesive failures being observed in other tests and result in most failures within the adhesive layer[12,13]. This may be come from the facts that the gluing procedure was done under a microscope so that the testing load could be applied in a relatively well-controlled direction and there must have been a self-aligning stage during loading, which was observed from the load-deflection curve. Using the MTBS test, multiple specimens can be obtained from a single tooth and one can calculate regional bond strengths of small, irregular surfaces under clinical conditions. The disadvantage of the method is that it is technically difficult and labor intensive. And with the materials that produce low bond strengths (below 5 ㎫), the specimens frequently fail during preparation.

SEAs provided simple clinical procedure and removed the need of rinsing and drying, but their bond strengths were lower than those of the adhesives of the earlier generations[1-3,5]. Water transduction was claimed to be the cause of the weak bonding of SEA to dentin. Dentinal fluid droplets were observed as interconnecting, dendritic silver deposits along the surface of the hybrid layer in TEM micrographs[9,14,15]. Those were called "water tree". The permeability associated with these adhesives was not caused by a loss of integrity between the adhesive and dentin, but by the presence of dentinal tubules, as water channels that probably expedite such water movement via capillary fluid flow[9]. "Water tree" reported to interfere with the coupling of SEA and chemically-cured composite resin[16]. This phenomenon was aggravated by an increased concentration of hydrophilic resin component in contemporary SEAs, since the hydrophilicity and hydrolytic stability of resin monomers were generally antagonistic[17,18]. The "honeycomb appearance" found in an overwet phenomenon or when SEAs were used was also observed in this study (Figure 5B). Such defects at the hydrophilic domains might act as cracks in the crack mechanism of brittle fracture of bonded complex[7].

To get higher bond strength, multiple-coat was recommended for SEAs[4,5]. The method could get the adhesive layer thick, but the adhesive layer thickness failed to correlate with the bond strength[7,8]. Rather than it, bond strength may be affected more by physical or mechanical properties such as degree of conversion. The pH of the experimental SEA, Adper Prompt, and D/E bonding resin of All-Bond 2 were 1.96, 1.0, and 6.9, respectively. The high acidity of SEAs with acidic monomer hindered the polymerization reaction, resulting in considerably low degree of conversion. The effect of acidic monomer on tertiary amine was already mentioned in the study using total-etch adhesive system[19-21]. Incompatibility between the adhesives containing acidic resin monomers and chemically-cured composites was first reported in the study that the decrease in the MTBS of chemically-cured composites to dentin was directly proportional to the acidity of these adhesives[22]. High concentration of acidic monomer in oxygen-inhibited layer interfered with tertiary amine of composite resin, so that tertiary amine could not accelerate the polymerization reaction[10]. That is, high acidity of SEA interfered not only with the polymerization of itself, but also copolymerization of the adhesive and the composite resin. As a result, the adhesive layer might not stand against shrinkage stress during polymerization of compos-

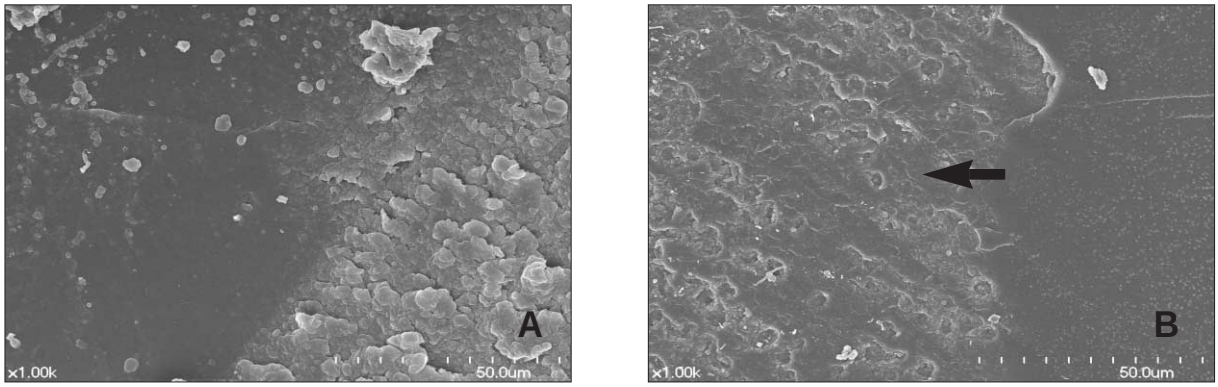

Figure 4. FE-SEM micrograph of the Experimental SEA and D/E bonding resin. (A) fractured surface of resin side. (B) corresponding surface of dentin side. Fracture within the adhesive layer was observed as smooth surface. The fracture plane deviated into the composite resin (arrow).

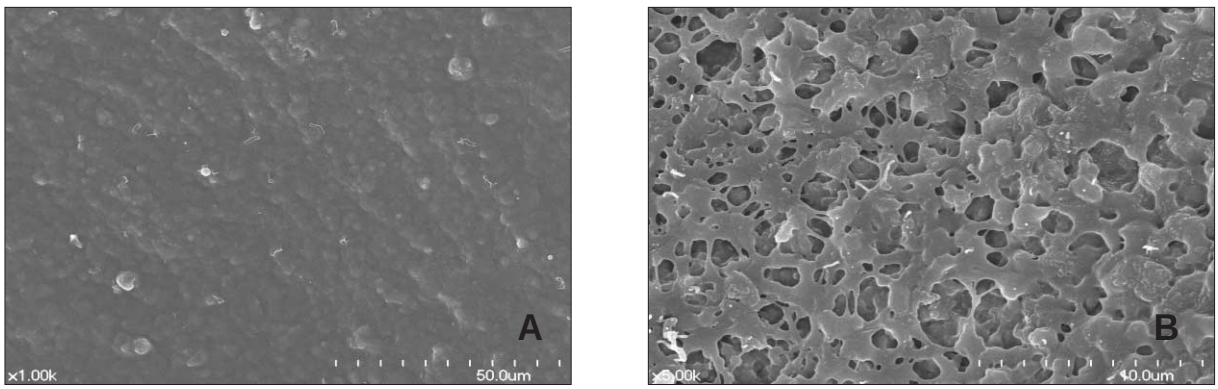

Figure 5. FE-SEM micrograph of two coats of Adper Prompt. (A) fractured surface of resin side. (B) fractured surface of dentin side. Honeycomb appearance with much irregular voids was seen in dentin side (B).

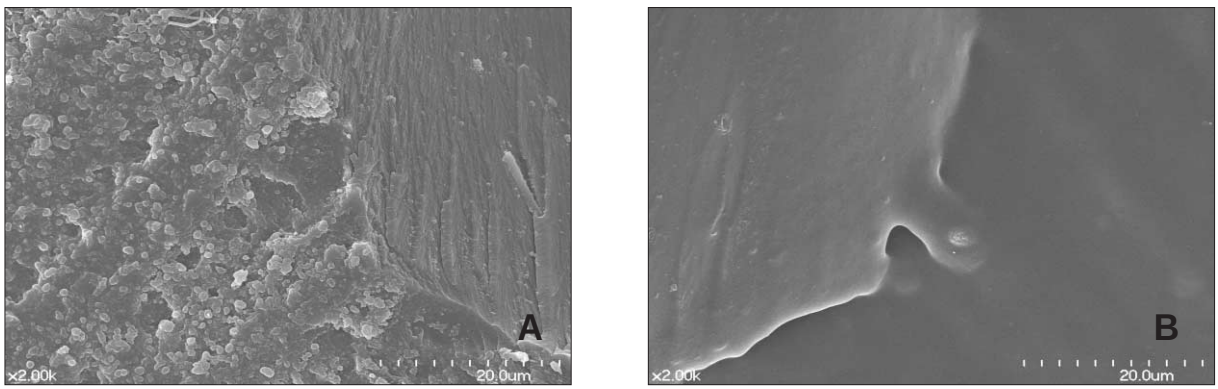

Figure 6. FE-SEM micrograph of Adper Prompt and D/E bonding resin. (A) fractured surface of resin side. (B) corresponding surface of dentin side. The failure was classified as a mixed one.

ite resin, and crack could be made within the adhesives[23]. This might reduce the bond strength of SEA to dentin.

In order to overcome these problems, it may be suggested to cover these SEAs with a hydrophobic adhesive (e.g., D/E bonding resin of this study). According to the inspection of the fractured surface, the interference of copolymerization confirmed indirectly (Table 2, Figures 3-6). Two-coat applications of experimental SEA and Adper Prompt resulted in most failures at the interfaces between the adhesive layer and the dentin and between the adhesive layer and the composite resin, respectively. However, applying D/E bonding resin as the second coat increased the incidences of the failures within the adhesive layer and the mixed failures including that within the adhesive layer. The changes in the fracture modes resulting from the application of D/E bonding resin as the second coat coincided with the increase of the bond strengths of each SEA.

In this study, the hypothesis that applying D/E bonding resin as a second coat might improve the bond strength of SEAs by alleviating pH difference between the adhesive and the composite resin was accepted. In order to increase the mechanical properties of the adhesive layer and improve co-polymerization between the acidic SEA and the composite resin, applying neutral bonding resin as a second coat, e.g., D/E bonding resin of All-Bond 2 or bonding agent of Scotchbond Multi-Purpose, may be an alternative to multiple coats of the SEA itself. Applying a second coat of bonding resin having neutral pH may improve the degree of conversion, mechanical properties, and the thickness of the adhesive layer, and accordingly improve the stress distribution or resist against the polymerization stress of composite resin. In addition to the advantage of removing the water-rinsing step, the adhesion strategy adopted in the procedure of self-etching primer systems which has additional step of applying adhesive might demonstrate high efficiency in dentin bonding.

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국문초록

접착레진의 추가도포가 자가부식형 접착제의 상아질에 대한 미세인장접착강도에 미치는 영향

정문경[1] · 조병훈[1,2,3*] · 손호현[1,2] · 엄정문[1,2] · 한영철[1] · 정세준[1]

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본 실험에서는 자가부식형 접착제와 콤포짓트 레진 사이의 산도의 차이를 완화시킬 수 있는 접착레진을 자가부식형 접착제 위에 추가적으로 도포할 경우, 상아질에 대한 접착력을 개선할 수 있는지를 연구하였다. 자가부식형 접착제로는 실험실에서 직접 제작한 실험용 자가부식형 접착제 (pH: 1.96)와 Adper Prompt (3M ESPE, USA, pH: 1.0)를 사용하였으며, 중성의 접착레진으로 All-Bond 2의 D/E bonding resin (Bisco Inc., USA, pH: 6.9)을 사용하였다. 두 대조군에서는 두 가지 자가부식형 접착제를 각각 두번씩 도포하였으며, 두 실험군에서는 각 자가부식형 접착제를 한번 도포한 후 그 위에 D/E bonding resin을 추가 도포하였다. Z-250 하이브리드 복합레진을 쌓아올려 모래시계 형태의 시편을 제작하여 미세인장강도를 측정하고 t-test를 이용하여 비교하였다. 파절 양상은 입체현미경과 주사전자현미경을 이용하여 관찰하였다. D/E bonding resin을 추가 도포한 미세인장접착강도는 유의하게 증가되었고, 접착층과 복합레진 또는 접착층과 상아질 사이의 파절을 보인 시편의 수는 감소하고, 접착층 내의 파절을 보인 시편의 수는 증가 되었다. 따라서 자가부식형 접착제와 복합레진의 산도의 차이를 완화할 수 있는 중성의 접착레진을 추가 도포할 경우, 미세인장접착강도를 증가시킬 수 있음을 확인하였다.

주요어: 자가부식형 접착제, 미세인장접착강도, 산도, 중간층, 파절양상