

수종의 상아질 접착시스템이 즉시 및 지연 상아질 봉쇄의 미세인장결합강도에 미치는 영향

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ABSTRACT

THE EFFECT OF VARIOUS BONDING SYSTEMS ON THE MICROTENSILE BOND STRENGTH OF IMMEDIATE AND DELAYED DENTIN SEALING

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The purpose of this study was to compare the effect of various dentin bonding systems on microtensile bond strength of immediate dentin sealing (IDS) and delayed dentin sealing (DDS). Eighteen extracted permanent molars were used in this study. The teeth for DDS group were restored with a provisional restorations, and immersed in saline solution for 1 week, and divided into 3 subgroups according to various dentin bonding adhesives: SB subgroup (3 step total-etch adhesive), SE subgroup (2 step self-etch adhesive), XE subgroup (1 step self-etch adhesive). In IDS group, the teeth were divided into 3 subgroups, and applied with bonding adhesives as in DDS group. The teeth were restored with provisional restorations, and immersed in saline solution for 1 week. Indirect composite disc was cemented with resin cement, and all specimens were subjected to microtensile bond strength. The data were statistically analyzed with one-way ANOVA and Student t-test.

The results were as follows:

1. The IDS group showed significantly higher μ TBS than DDS group in 3 step total-etch and 2 step self-etch adhesive ($p < 0.05$).
2. In IDS and DDS group, 3 step total-etch adhesive showed the highest μ TBS value, followed by 2 step self-etch, and 1 step self-etch adhesive. In IDS group, the μ TBS value for 1 step self-etch adhesive was significantly different from those of the other subgroups ($p < 0.05$), and in DDS group, there were statistical differences in all subgroup ($p < 0.05$).
3. Failure modes of tested dentin bonding adhesives were mostly mixed failure and only 1 step self-etch adhesive showed adhesive failure. [J Kor Acad Cons Dent 33(6):526-536, 2008]

Key words: Immediate dentin sealing, Delayed dentin sealing, Indirect resin composite restoration, Dentin bonding adhesives, Microtensile bond strength

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I . Introduction

Direct resin composite restoration is considered as most conventional restoration in anterior and posterior restoration. However, there are several problems of posterior direct resin composite restoration such as

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wear, polymerization stress, and inadequate proximal contact. Especially due to the residual polymerization stress, direct resin composite restorations are often contraindicated in large cavities¹⁾. Indirect resin composite and ceramic inlay / onlay restorations are advocated as alternatives in that situation²⁾. Indirect resin composite restorations have improved clinical conditions with respect to proximal contact, occlusal anatomy and marginal adaptation³⁾.

Successful dentin bonding is particularly important in the case of indirect resin composite restorations such as inlays, onlays, and veneers, because the final strength of the tooth-restoration complex is highly dependant on adhesive procedures⁴⁾. Paul et al.⁵⁾ and Bertschinger et al.⁶⁾ described freshly cut dentin is the ideal substrate for dentin bonding, while dentin contamination due to provisional cements could reduce the potential for dentin bonding. "Immediate dentin sealing (IDS)" method was introduced. This method defined immediate application and polymerization of the dentin bonding adhesive to the freshly cut dentin, prior to impression taking⁴⁾. This procedure appears to achieve increased bond strength, improved restoration adaptation, fewer gap formations, decreased bacterial leakage, and reduced dentin sensitivity during provisional period. This approach to adhesion also has a positive influence on the structure preservation, patient comfort, and long-term survival of indirect bonded restoration. On the contrary, the conventional technique for indirect restorations, traditionally, consists in impression taking the cavity immediately after preparation. Then, after the fabrication of the indirect restoration, provisional material and remnants of luting cement were removed and the adhesive or luting procedures are performed. This procedure was called "Delayed dentin sealing (DDS)" method¹⁾.

The success of "IDS" method might be attributed to the dentin bond strength. Although there is a tendency to simplify dentin bonding procedures, some authors^{7,8)} described 3 step total-etch adhesive is indicated for IDS because of its ability to form a consistent and uniform layer. But, recently self-etch systems have been produced as an alternative to total-etch system⁹⁾. Recent studies^{10,11)} of self-etch systems have reported high bond strengths to dentin.

De Munck et al.¹²⁾ reported that the 2 step self-etch adhesive resulted in bond strength close to that obtained with the 3 step total-etch adhesive. Recently, 1 step self-etch adhesives have been introduced that combined the etchant, primer, and adhesive into one bottle¹³⁾. 1 step self-etch adhesives are more hydrophilic due to a higher concentration of acidic monomers to properly etch the dentin surface¹⁴⁾. These adhesives are clinically available and now widely used throughout the world.

However, the difference in bond strength between IDS and DDS for cementation of indirect restorations with various dentin adhesives has not yet to be evaluated. The purpose of this study was to compare the effect of various dentin bonding adhesives on microtensile bonding strength (μ TBS) of IDS and DDS for indirect resin composite restoration.

II . Materials and methods

1. Tooth preparation

Eighteen extracted permanent molars without caries were stored in distilled water. The teeth were ground flat to expose occlusal dentin surface. The flat dentin surface was polished with wet 600-grit silicon carbide paper. The exposed dentin surface was evaluated for the presence of remaining enamel under operating microscope (OPMI pico; Carl zeiss, Obercohen, Germany), which was removed by additional trimming. The teeth were then randomly assigned to two groups of 9 teeth each, according to application of the dentin adhesive such as IDS and DDS (Figure 1).

2. Experimental group

Delayed dentin sealing (DDS)

The teeth for DDS group were restored with a provisional restoration material (Clip; VOCO, Cuxhaven, Germany), and immersed in saline solution for 1 week. Following that delay, the provisional restoration was removed and dentin was cleaned by an intraoral air abrasion device (Danville Engineering Inc., Danville, CA, USA) filled with 50 μ m aluminum oxide particles. And this group was

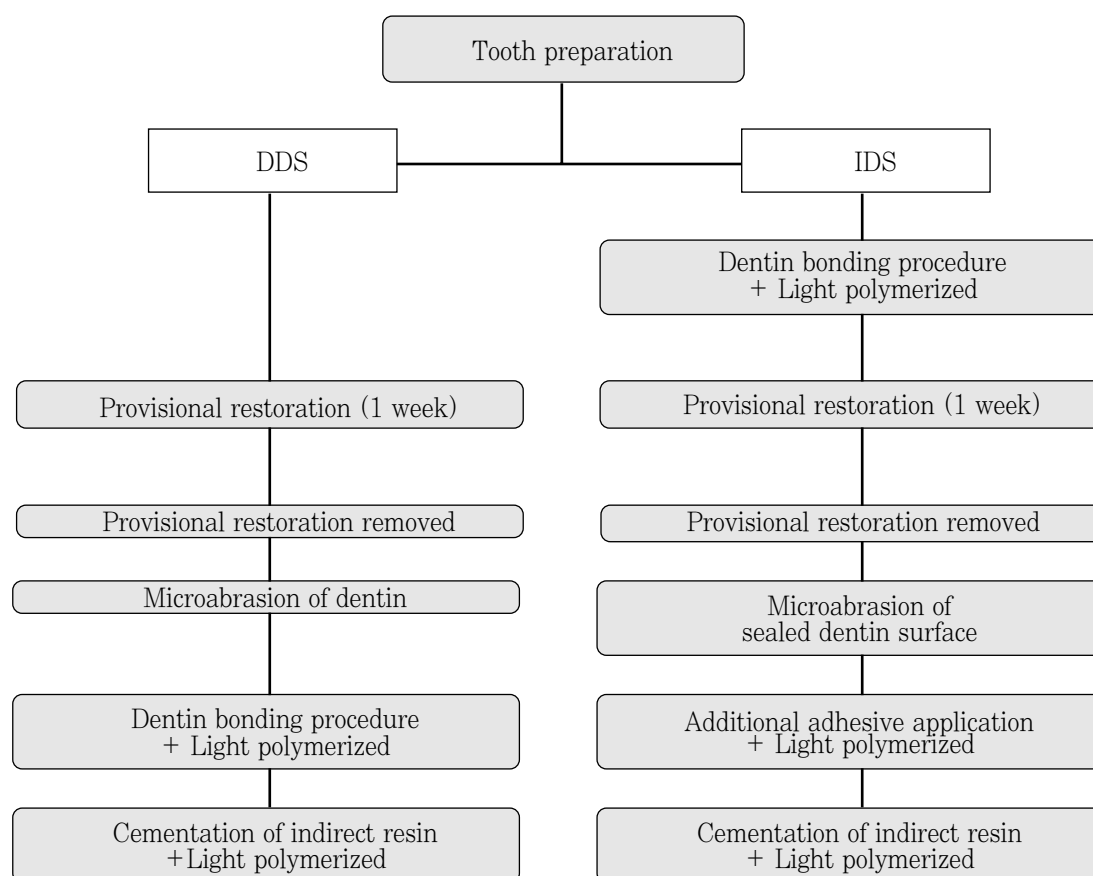


Figure 1. Experimental groups and sequences

divided into 3 subgroups according to dentin bonding adhesive. All dentin bonding adhesives (Table 1) were manipulated and applied to the dentin surfaces according to the manufacturers' instructions (Table 2). The specimen was light polymerized with LED curing unit (Bluephase; Ivoclar Vivadent, Shann, Liechtenstein) at 600 mW/cm².

Immediate dentin sealing (IDS)

The teeth for IDS group were also divided into 3 subgroups. Like DDS group, all dentin bonding adhesives were manipulated and applied to the fresh dentin surfaces according to the manufacturers' instructions. The specimen was light polymerized with LED curing unit at 600 mW/cm².

Polymerization of the adhesive was followed by the application of an air-blocking barrier (glycerin jelly) and 10 seconds of additional light exposure with the same light unit to polymerize the oxygen-inhibition

layer as proposed by Magne⁴. The bonded surfaces were then isolated with petroleum gel not to attach between bonding surface and resin-based provisional restoration. Teeth were restored with a provisional restoration material, and immersed in saline solution for 1 week. Following that delay, the provisional restoration was removed and the sealed dentin was cleaned by an intraoral air abrasion device filled with 50 μ m aluminum oxide particles. An adhesive resin was applied once and polymerized.

3. Cementation

Eighteen indirect composite discs of 7mm in height and 10mm in diameter (Sinfony; 3M ESPE, St. Paul, MN, USA) were prepared to simulate overlying laboratory-processed resin composite restorations. The surface of each composite disc was sandblasted with 50 μ m aluminum oxide particles for 10 seconds

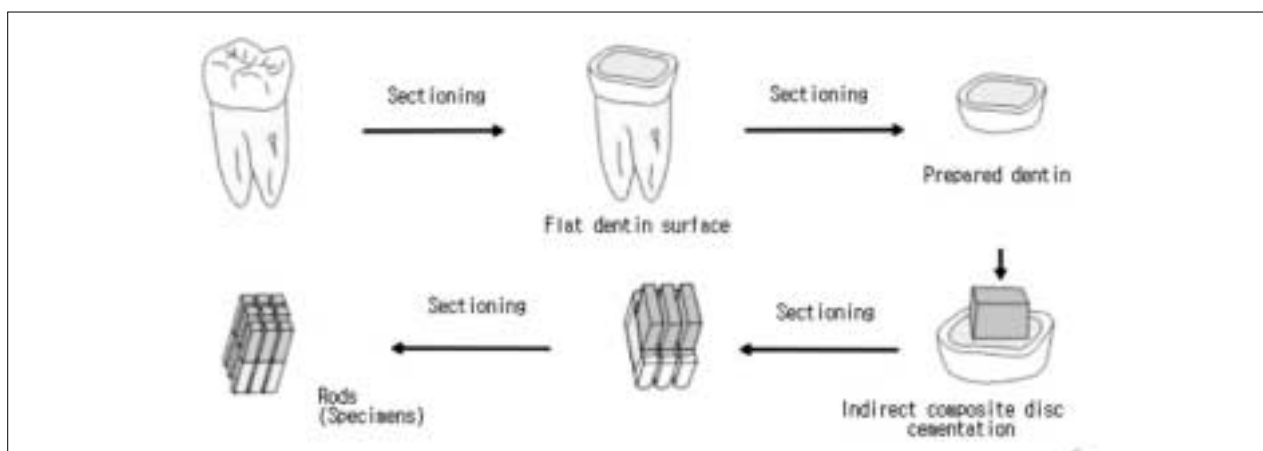
Table 1. Adhesives Investigated in This Study

Product Name	Compositions	Manufacturer
Scotchbond Multipurpose (SB)	Etchant - 35% phosphoric acid Primer - HEMA, polyalkenoic acid, copolymer Adhesive-BIS-GMA, HEMA, photoinitiator	3M ESPE, St. Paul, MN, USA
Clearfil SE bond (SE)	Self-etching primer-MDP, HEMA, photoinitiator, water, hydrophilic dimethacrylate Bonding resin-MDP, Bis-GMA, HEMA, microfiller, hydrophilic dimethacrylate	Kuraray Co., Osaka, Japan
Xeno III (XE)	Liquid A-HEMA, water, ethanol, silicon dioxide Liquid B-phosphoric acid modified methacrylate resin, UDMA, BHT, camphorquinone, ethyl-4-dimethylaminobenzoate	Dentsply Caulk Milford, DE, USA

* Abbreviations: HEMA = 2-hydroxyethyl methacrylate; BIS-GMA = bisphenol-A-glycidyl ether dimethacrylate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; UDMA = urethane dimethacrylate; BHT = butylhydroxy-toluene.

Table 2. Bonding Procedures

Bonding adhesive	Procedures
SM (3 step total-etch adhesive)	Etchant-Applied for 15 seconds, then rinsed thoroughly and gently dried for 2 seconds. Primer-Applied, then dried gently for 5 seconds. Adhesive- Applied, then light cured for 10 seconds.
SE (2 step self-etch adhesive)	Self-etching primer -Primer is applied for 20 seconds, air blown gently. Bonding resin-Applied, then light cured for 10 seconds.
XE (1 step self-etch adhesive)	Equal amounts of liquid A and B mixed for 5 seconds. Applied for 20 seconds, then light cured for 10 seconds.

**Figure 2.** Schematic diagram of the specimen preparation for the microtensile bond test

from a distance of approximately 5mm. The composite disc surface to be cemented was silanized with a Monobond-S (Ivoclar vivadent AG, Schaan, Liechtenstein) for one minute, and then air dried. Rely X ARC resin cement (3M ESPE, St. Paul, MN, USA) was mixed according to the manufacturers' instructions, and applied to the tooth specimen. A5 kg mass was applied to the composite disc during cementation. The excess resin cement was removed with a probe. The resin cement was photopolymerized for 40 seconds at 600 mW/cm². The restored specimens were subsequently stored in distilled water at room temperature for 24 hours before testing.

4. Microtensile bond strength (μ TBS) testing

The teeth were then cut longitudinally into six or seven sections perpendicular to the tooth / adhesive interface, with each slab being 1mm thick and 10mm long using the hard tissue cutter (Accutom-50; Struers, Rødovre, Denmark) under water cooling. The sections were left attached to the remainder of the tooth for further sectioning to obtain sticks approximately 1×1mm thick and 10mm long (Figure 2). Each group was consisted of 36 rods. The specimens were glued to the jig of microtensile testing machine (BISCO Inc, Schaumburg, IL, USA) using cyanoacrylate cement (Zapit; Dental Ventures of America, Corona, CA, USA). Tensile load was applied until specimen was failed. Failure load was recorded for each specimen and then the μ TBS was calculated.

5. Fracture mode investigations

1) Optical microscopy observation

After testing, the failure mode of each beam was determined under operating microscope. Fractured test specimens were examined to record the type of bond failure (adhesive, cohesive, or mixed). Bond failure was characterized according to the area of resin remaining on the dentin surface. Adhesive failures were characterized as having less than 25% resin remaining at the interfacial bond area. Cohesive failures had greater than or equal to 75% resin remaining at the interfacial bond area, and

mixed failures had 25% to 75% resin remaining at the interfacial bond area.

2) Scanning electron microscopy (SEM) observation

The dentin sides of 6 fractured beams (mixed failure or adhesive failure) from each group were air dried, sputter coated with gold / palladium (E1010 Ion Sputter, Hitachi Co., Mito City, Japan), and examined using SEM (S-3500N SEM, Hitachi Co., Mito City, Japan).

6. Statistical analysis

In each dentin bonding adhesive, the difference of μ TBS between IDS and DDS were analyzed statistically by Student t-test. In IDS and DDS group, one-way ANOVA and Tukey's test were used to determine statistical difference of μ TBS between the dentin bonding adhesives using SPSS 12.0 software (SPSS, Chicago, IL, USA). The level of significance was set at $p < 0.05$.

III. Results

1. Microtensile bond strength (μ TBS)

1) Comparison between IDS and DDS in each dentin bonding adhesive

Table 3 lists the μ TBS values of three dentin bonding adhesives to dentin in IDS and DDS group. The mean μ TBS values from 12 to 25 MPa.

In 3 step total-etch SB and 2 step self-etch SE subgroup, IDS group showed higher bond strength than DDS group ($p < 0.05$). However, in 1 step self-etch XE subgroup, there was no significant difference between IDS and DDS group ($p > 0.05$).

2) Comparison with each dentin bonding adhesive in IDS and DDS group

The μ TBS values of three dentin bonding adhesives in IDS and DDS group were showed in Figure 3.

In IDS group, 3 step total-etch SB subgroup showed the highest μ TBS, followed by 2 step self-etch SE, and 1 step self-etch XE subgroup. But, there

was no significant difference between 3 step total-etch SB and 2 step self-etch SE subgroup ($p > 0.05$). 1 step self-etch XE subgroup showed the lowest μ TBS value and was significantly different from the other subgroups ($p < 0.05$).

In DDS group, 3 step total-etch SB subgroup also exhibited the highest bond strength, followed by 2 step self-etch SE, and 1 step self-etch XE subgroup. And, there was significant difference among all groups ($p < 0.05$).

2. Fracture mode

1) Optical microscopy observation

The fracture mode was different among the dentin bonding adhesives. These results were summarized in Table 4. In IDS and DDS group, the fractured beams for 3 step total-etch SB and 2 step self-etch SE subgroup demonstrated cohesive or mixed failure, and there was no specimen with adhesive failure.

However, 1 step self-etch XE subgroup showed adhesive failure both IDS and DDS group (25% and 22% respectively).

2) SEM observation

The fractured beam of 3 step total-etch SB and 2 step self-etch SE subgroup in both IDS and DDS groups showed a case of mixed failure. But, fractured beam of 1 step self-etch XE subgroup in IDS and DDS group showed a case of adhesive failure (Figure 4 and 5).

IV. Discussion

This study compared the μ TBS of three dentin bonding adhesives with IDS and DDS. The results of this study demonstrated that the IDS method used when cementing indirect resin composite restoration may affect the μ TBS of indirect restoration, depending on the dentin bonding adhesive used.

For 3 step total-etch and 2 step self-etch subgroup,

Table 3. Microtensile bond strength of IDS and DDS according to dentin bonding adhesive (Mean \pm SD, MPa)

Dentin bonding adhesive	IDS	DDS	Student t-test p - value
SB (n=36)	24.0 \pm 4.4	21.4 \pm 4.1	$p < 0.05$
SE (n=36)	22.1 \pm 5.6	17.2 \pm 3.9	$p < 0.05$
XE (n=36)	12.8 \pm 3.2	13.3 \pm 3.0	$p > 0.05$

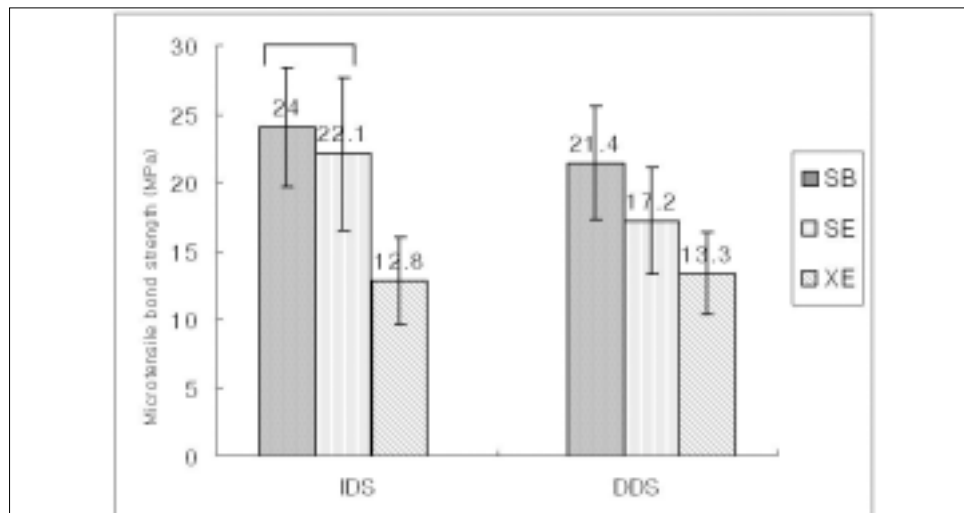


Figure 3. Microtensile bond strength of three dentin bonding adhesives according to IDS and DDS. Subgroups under the horizontal line were not significantly different ($p > 0.05$).

Table 4. Number of beams showing fracture modes

	IDS			DDS		
	SB	SE	XE	SB	SE	XE
Adhesive	0	0	9	0	0	8
Cohesive	1	5	7	19	3	10
Mixed	35	31	20	17	33	18

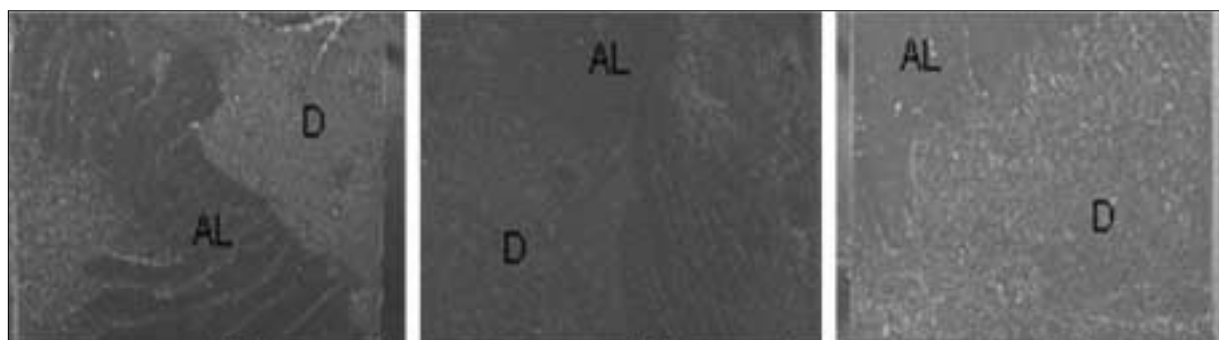


Figure 4. Fractured beam in IDS exhibiting failure pattern (X 100)

- (Left) Mixed failure in a beam of 3 step total-etch SB subgroup was shown
- (Middle) Mixed failure in a beam of 2 step self-etch SE subgroup was shown.
- (Right) Adhesive failure in a beam of 1 step self-etch XE subgroup was shown.
- (D: dentin,AL : adhesive layer)

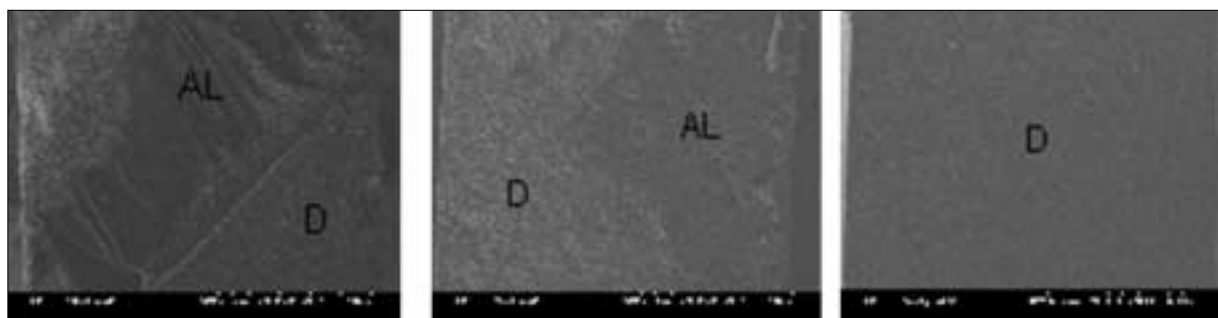


Figure 5. Fractured beam in DDS exhibiting failure pattern (X 100)

- (Left) Mixed failure in a beam of 3 step total-etch SB subgroup was shown .
- (Middle) Mixed failure in a beam of 2 step self-etch SE subgroup was shown.
- (Right) Adhesive failure in a beam of 1 step self-etch XE subgroup was shown.
- (D : dentin,AL : adhesive layer)

IDS group had a statistically higher mean value of bond strength than DDS group. Dietschi et al.¹⁵⁾ suggested that using IDS and indirect bonded restorations, because of the delayed placement of the restoration and postponed occlusal loading, the dentin bond can increase over time and residual stress can dissipate, resulting in significantly improved restoration adaptation. And several studies^{5,6)} incorporating various dentin bonding adhesives

and application methods have shown that the IDS method increased bond strength values compared to the DDS method. This was related to formation of longer resin tags and a thicker hybrid zone. Moreover, IDS method can protect the tooth from the consequences of microleakage by sealing the dentin tubules that are vulnerable to bacteria invasion, immediately after completion of the preparation¹⁶⁾. Sealing of the dentin tubules also reduces sensitivity

by preventing hydraulic fluid flow within the dentin tubules, which is associated with postoperative sensitivity¹⁷⁾.

In this study, 3 step total-etch subgroup in IDS group exhibited the highest bond strength. According to recent study¹⁸⁾, total-etch systems have shown high bond strength to dentin. Bouillaguet et al.¹⁹⁾ demonstrated 3 step total-etch adhesive exhibited significantly higher bond strength values than some self-etch systems. In total-etch system, an acid etching of dentin is necessary to efficiently dissolve the smear layer and the smear plugs and to promote a strong and impervious bond between dentin and adhesive mediated by a hybrid layer²⁰⁾. This system could be considered as more reliable in spite of their more time-consuming procedures and technique sensitivity.

The 2 step self-etch adhesive in this study presented good bond strength to dentin. The advantage of this adhesive is that it combines conditioning and priming into one step, avoiding a gap between inorganic component demineralization and primer infiltration. Tanumiharja et al.²¹⁾ reported that 2 step self-etch adhesive provides the simplest bonding technique and exhibited the highest bond strength to dentin. Laboratory studies^{22,23)} have demonstrated the capability of self-etch systems to bond equally as well as phosphoric acid-etch based systems which demineralize the tooth surface and require a wash and dry step prior to dentin application. In this study, SE Bond was used as 2 step self-etch adhesive, and then showed as high as μ TBS of 3 step total-etch adhesive in IDS group. This is probably due to SE Bond's mild acidic monomer content and high filler particle content⁹⁾. Another reason to show high μ TBS of 2 step self-etch adhesive in IDS group described twice application of adhesive layer. First adhesive layer to the freshly cut dentin was done before impression taking. Second adhesive layer was applied before cementation. Several studies^{24,25)} have reported that bond strength to dentin may be improved by application of a second adhesive layer. And due to the second adhesive layer, thicker adhesive layer contribute to greater reduction in polymerization shrinkage stress and the extent of microleakage in cavities²⁴⁾. IDS method is somewhat similar to a twice applica-

tions of adhesive on dentin bonding. Another advantage of 2 step self-etch adhesive has been associated with less postoperative sensitivity than 3 step total-etch adhesive²⁶⁾. It seems that 2 step self-etch adhesive in IDS showed as an alternative 3 step total-etch adhesive in IDS. But, the results of this study show that the bond strength of 2 step self-etch subgroup resulted in lower bond strength values than 3 step total-etch subgroup in DDS group.

In this study, XE subgroup bond strength is the lowest value in IDS and DDS group. 1 step self-etch adhesives have been shown to contain a higher concentration of acid derivatives, methacrylated phosphoric acid esters, water, and organic solvents than conventional bonding agents to simultaneously etch and infiltrate the dentin surface in 1 step. The low pH (1.5-2.5) of these 1 step self-etch adhesives makes them hydrolytically unstable as a result of the methacrylate-based components²⁷⁾. Meerbeek et al.⁸⁾ described that μ TBS of 1 step self-etch adhesive was significantly least favorable. Low bond strengths recorded with 1 step self-etch adhesive may indicate the single step material cannot yet fulfill all requirements for the production of effective adhesive layers. As reported in several studies^{28,29)}, most simplified 1 step self-etch adhesives are the least durable, while 2 step self-etch adhesives continue to show the best performance in terms of bond strength, aging, and stability of the bonded interface for IDS. Inoue et al.³⁰⁾ found that 1 step self-etch adhesive tended to have lower bond strengths than 2 step self-etch adhesive. In this study, in both IDS and DDS group, μ TBS value of 1 step self-etch subgroup was lower than those of 2 step self-etch subgroup.

In this study, failure modes of tested dentin bonding adhesive were mostly mixed or cohesive failure. Cohesive failures represent integrity in the adhesive layer, protecting the dentin. In contrast, adhesive failures denote a rupture at the dentin / resin interface, characterized by open dentinal tubule and intertubular dentin. In this study, 1 step self-etch subgroup showed adhesive failure. This result is somewhat supported by the SEM microscopes showing poorly infiltrated smear layer. The low μ TBS and the relatively high number of adhesive failures of the 1 step self-etch adhesive strongly suggest that the

incorporation of the smear layer might have decreased the adhesive properties or that they failed to optimally hybridize the smear layer covered dentin³¹⁾.

For many years, some authors^{7,8)} claimed the IDS method needed 3 step total-etch adhesive. But, the problem of 3 step total-etch adhesive showed technique sensitivity and postoperative sensitivity. To overcome this problem, 2 step self-etch adhesives were produced. In this study, the μ TBS of 2 step self-etch subgroup was as high as that of 3 step total-etch subgroup and failure mode of 2 step self-etch subgroup are similar to that of 3 step total-etch subgroup. As the result of this study, when clinician use the IDS method, 2 step self-etch adhesive may be advocated as alternatives of 3 step total-etch adhesive. But, 1 step self-etch adhesive is not recommended.

V. Conclusions

With the limitations of the study, the following conclusions were drawn,

1. The IDS group showed significantly higher μ TBS than DDS group in 3 step total-etch and 2 step self-etch adhesive ($p < 0.05$).
2. In IDS and DDS group, 3 step total-etch adhesive showed the highest μ TBS value, followed by 2 step self-etch, and 1 step self-etch adhesive. In IDS group, the μ TBS value for 1 step self-etch adhesive was significantly different from those of the other subgroups ($p < 0.05$), and in DDS group, there were statistical differences in all subgroups ($p < 0.05$).
3. Failure modes of tested dentin bonding adhesives were mostly mixed failure and only 1 step self-etch adhesive showed adhesive failure.

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국문초록

수종의 상아질 접착시스템이 즉시 및 지연 상아질 봉쇄의 미세인장결합강도에 미치는 영향

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이 연구의 목적은 수종의 상아질 접착시스템이 즉시 및 지연 상아질 봉쇄에서 미세인장결합강도에 미치는 영향을 평가하는 것이었다. 18개의 발거된 대구치를 사용하여 지연 상아질 봉쇄그룹은 노출된 상아질면을 임시수복하였으며, 1주간 보관 후, 접착제에 따라 3개의 소그룹으로 나누어 도포하였다: SB 그룹 (3 단계 산 부식 접착제), SE 그룹 (2단계 자가 부식 접착제), XE 그룹 (1단계 자가 부식 접착제). 즉시 상아질 봉쇄그룹은 3개의 소그룹으로 나누어 접착제를 도포하고 임시수복 후 1주간 보관하였다. 모든 시편은 간접 복합레진과 레진 시멘트로 합착하고 미세인장결합강도를 측정하여, 다음과 같은 결과를 얻었다.

1. 즉시 상아질 봉쇄그룹은 지연 상아질 봉쇄그룹에 비해 3단계 산 부식과 2단계 자가 부식 접착제에서 높은 미세인장결합강도를 보였다 ($p < 0.05$).
2. 즉시 및 지연 상아질 봉쇄그룹 모두 미세인장결합강도는 3단계 산 부식, 2단계 자가 부식, 1단계 자가 부식 접착제 순으로 감소하였고, 즉시 상아질 봉쇄그룹에서는 1단계 자가 부식 접착제와 다른 소그룹간 유의한 차이가 있었으며 ($p < 0.05$), 지연 상아질 봉쇄 그룹에서는 모든 소그룹간 유의한 차이가 있었다 ($p < 0.05$).
3. 파절 양상은 대부분 혼합성 파절을 보였으며, 1단계 자가 부식 접착제에서만 접착성 파절을 보였다.

주요단어 : 즉시 상아질 봉쇄, 지연 상아질 봉쇄, 간접 복합레진 수복물, 상아질 접착제, 미세인장결합강도