

수 종의 복합레진 접착 시스템에서의 미세 누출의 비교

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국문초록

최근 자가 부식형 접착 시스템이 개발되어 법랑질 및 상아질에 모두 적용할 수 있고, 임상적 술식을 단순화 시켰다. 이 접착 시스템은 적용하기 용이하고 임상적으로도 법랑질 및 상아질에 높은 결합강도를 보인다고 보고되고 있다.

이 연구는 in vitro 상에서 복합레진 5급 와동의 법랑질 및 상아질 경계에서 전부식 처리를 동반한 one-bottle 접착 시스템과 자가 부식형 접착 시스템을 사용하였을 때 미세 누출을 비교하기 위해 시행하였다.

교정적 목적으로 발치된 30개의 소구치를 무작위적으로 3개 군으로 나누었다: 1 군 (Single Bond® + Filtek Z250®), 2 군 (Clearfil SE Bond® + Filtek Z250®), 3 군 (Adper Prompt L-Pop® + Filtek Z250®).

표준화된 5급 와동을 각 소구치 협면, 설면에서 백악-법랑경계에 평행하게 형성하였으며, 와동의 치은측 1/2은 백악-법랑경계보다 1mm 치근단으로 연장하였다. 접착 시스템을 제조자의 지시대로 적용한 다음 복합레진을 충전하고 제조자의 지시대로 광중합하였다.

시편을 37℃ 증류수에 5일 간 보관 후 5℃±2℃ 와 55℃±2℃에서 1000회 열순환한 다음, 2% methylene blue 용액에 12시간 침잠시켰다. Isomet™ (Buehler Co., Lake Bluff, IL, USA)을 사용하여 치아를 충전물 중앙에서 종절 단한 후 입체 현미경하에서 25배의 배율로 색소 침투도를 평가하였다. 결과는 t-test 와 one-way ANOVA를 이용하여 통계처리 하였다. 결과는 다음과 같았다.

- 실험에 사용 된 접착 시스템 중에 미세 누출을 완벽하게 방지하는 접착 시스템은 없었다.
- 상아질 경계에서의 미세 누출이 법랑질 경계에서보다 통계학적으로 유의하게 더 많았다(p<0.0001).
- 법랑질 경계에서 자가 부식형 접착 시스템은 전부식 처리를 동반한 one-bottle 접착 시스템과 통계학적으로 유의한 차이를 보이지 않았다.
- 상아질 경계에서, 자가 부식형 접착 시스템은 전부식 처리를 동반한 one-bottle 접착 시스템과 통계학적으로 유의한 차이를 보이지 않았다.

주요어 : 미세 누출, 자가부식, 복합레진 수복

I. Introduction

In recent years, resin composites have become widely used as a restorative material in the posterior area¹⁾. This is due to the increase in patients' demand for better esthetics and clinicians' interest in esthetic and conservative restoration. Besides the ability to bond to enamel and dentin, they feature the advantage of esthetics and are less costly than

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ceramic inlays and cast gold inlays²⁾. And resin composites have some other advantages. These contain no mercury, are thermally nonconductive³⁾, and bond to tooth structure with the use of adhesive systems^{4,5)}, so preserve tooth structure.

However, resin composites also have several undesirable characteristics. Polymerization shrinkage may be thought a one of the most considerable disadvantage of the current resin composites. The polymerization process results in shrinkage or contraction of the composite, causing stresses that may exceed the strength of the bond with the surrounding tooth structure, with possible failure at the adhesive joint⁶⁾. Polymerization shrinkage leads to gap formation between the composite restoration and the walls of the preparation at the weakest bond. Microleakage is the diffusion of substances within the cracks that contain fluids or within gaps between restorative material and dentinal substrates⁷⁾. Microleakage may result in postoperative sensitivity, and recurrent dental caries⁸⁻¹⁰⁾.

However, used in combination with the appropriate adhesive systems, composite restorations form a reliable and durable bond to tooth structure¹¹⁾. To minimize polymerization shrinkage, certain procedures, such as keeping dentin wet^{12,13)}, applying the adhesive according to manufacturers' recommendation and placing resin composite incrementally⁹⁾, could be applied.

In enamel, many adhesive systems use acid-etching technique to improve adhesion to enamel. Adhesion to enamel is achieved through acid-etching of this highly mineralized substrate, which substantially enlarges its surface area, and makes it irregular surface with high surface-free energy^{14,15)}. Kubo et al¹⁶⁾, reported current dentin adhesive systems use two different means to achieve the goal for micromechanical retention between resin and dentin. The first method removes the smear layer completely and demineralizes the subsurface intact dentin via acid-etching with chelating agent or mineral acids. Following rinsing, a multiple-step application of a primer and an adhesive, or a simplified self-priming adhesive is applied to the conditioned substrate to complete the bonding procedure. The second method uses the smear layer as a bonding substrate. Known as self-etching primers, they are applied to the

smear layer-covered dentin for a designated period of time. Without further rinsing, a layer of adhesive resin is then applied to the treated dentin. In these systems, the goal is to incorporate the smear layer into the hybrid layer.

Recently, many investigations using self-etching primer adhesive systems were performed. And the clinical effects of self-etching primer systems are controversial. Many studies reported that both adhesive systems show high bond strengths to enamel and dentin¹⁷⁻²⁰⁾. The use of self-etching primer is attractive because they are used on dry dentin and, after mixing, require one primer application which is subsequently air-dried rather than rinsed. So these adhesive systems are less technique-sensitive, more convenient to be used in dental clinic and less time-consuming than previous adhesive systems. Furthermore, self-etching primer system is divided two subgroups²¹⁾. One is bonding resin is separate with self-etching primer, the other is bonding resin is combined with self-etching primer.

Although, many reports show self-etching primer systems' good experimental results, it is true that there are many different opinions about the clinical effect of self-etching primer systems^{22,23)}. The purpose of this study is to evaluate in vitro the microleakage on the enamel and cementum/dentin walls in composite resin restoration of Class V cavities, regarding the use of different adhesive systems.

II. Materials and Method

1. Selection of teeth

30 premolars, extracted for orthodontic purpose within a 3-month period and stored in a physical saline solution, were selected for this study. Teeth with large carious lesions, extensive wear, fractured cusps, and cracked enamel were discarded. Each tooth was scaled to remove calculus and remaining periodontal tissue.

2. Preparation of the Class V cavities

A standardized Class V preparation was prepared on the buccal and lingual surface of each premolar tooth. The mesio-distal, occlusogingival widths of

each preparation were 2mm; the axial depth was 1.5mm, measured by periodontal probe. The preparation were made parallel to the cementsoenamel junctions, with the gingival half of the preparation extending 1mm apical to the cementsoenamel junction and the carvosurface walls finished to a butt joint. All preparation were performed using a carbide pear shaped bur (No. 330, Komet, Germany) in a high-speed hand piece with a water spray. Each bur was replaced after 6 preparations.

3. Composite

The resin composite chosen for this study is universal hybrid (Filtek Z250®, 3M ESPE Dental Products, St. Paul, MN, U.S.A.). Shade A4 was chosen for the test material.

4. Adhesive systems

The adhesive systems chosen for this study are 1 one-bottle adhesive system used in combination with total-etching (Single Bond®, 3M ESPE Dental Products, St. Paul, MN, U.S.A.) and 2 self-etching primer systems. One is Clearfil SE Bond® (Kuraray Co., Osaka, Japan) and the other is Adper Prompt L-Pop® (3M ESPE Dental Products, St. Paul, MN, U.S.A.).

5. Placement of the restorations

Before the restorative procedure, the teeth were randomly divided into three gorups.

Group 1 : The preparations were etched with 32% phosphoric acid gel (UNI-ETCH®, Bisco, Inc., Schaumburg, IL, U.S.A.) for 15 seconds. The acid was rinsed throughly for 10 seconds. Absorbent paper was used to blot excess water, but the dentin was kept moist. Two consecutive applications of adhesive Single Bond® were made, softly dried for 5 seconds and cured for 10 seconds. The resin composite, Filtek Z250® (3M ESPE Dental Products, St. Paul, MN, U.S.A.) was placed in bulk into the cavity and light cured according to manufacturer's instructions for each composite.

Group 2 : The primer of Clearfil SE Bond®(Kuraray Co., Osaka, Japan) was applied to preparations for

20 seconds. After 20 seconds the bonding resin was applied for 10 seconds and cured for 10 seconds. The resin composite, Filtek Z250® (3M ESPE Dental Products, St. Paul, MN, U.S.A.) was placed in bulk into the cavity and light cured according to manufacturer's instructions for each composite.

Group 3 : Prior to placement, a blister pack of Adper Prompt L-Pop® (3M ESPE Dental Products, St. Paul, MN, U.S.A.) was activated by squeezing and emptying the liquid out of the red cushion into the yellow cushion. The activated liquid mixture was then emptied into the green section of the blister pack and then applied to the specimens using the disposable applicator. A new blister pack was used for each specimen. The all-in-one adhesive was applied to the entire surface and agitated for 15 seconds. The liquid then gently air-dried and spread into a homogenous, shiny film and cured for 10 seconds. The resin composite, Filtek Z250® (3M ESPE Dental Products, St. Paul, MN, U.S.A.) was placed in bulk into the cavity and light cured according to manufacturer's instructions for each composite.

The same light unit was used through out the study, maintaining the tip not more than 1mm from the surface of the specimen. All restorations were finished flush to the margins with disks (SuperSnap, Shofu Inc, Kyoto, Japan) within 5 minutes after light polymerization. The restored teeth were stored in distilled water at 37°C for 5 days.

6. Thermocycling Procedure

All teeth were thermocycled for 1000 cycles at 5 ± 2 °C and 55 ± 2 °C with a dwell time of 30 seconds in distilled water and a five-second transfer time.

7. Dye Leakage Test

The apices were sealed with wax and flowable resin. Then the entire teeth were coated with two applications of fingernail varnish except for 1mm around the restoration body. When the fingernail varnish was dry, the teeth were then immersed in a 2% methylene blue solution for 12 hours then washed in water for 3 hours. Then the fingernail varnish was removed.

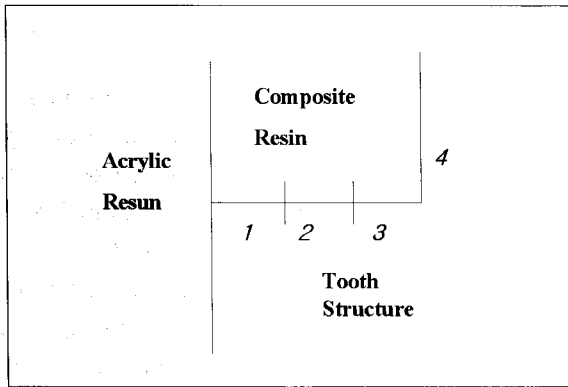


Fig. 1. Dye leakage test score. 0: no leakage, 1: leakage within 1/3 of the preparation depth, 2: leakage greater than 1/3, within 2/3 of the preparation depth, 3: leakage greater than 2/3, within the preparation depth, 4: leakage pass the axial wall

Table 1. Materials invested in this study

	Adhesive system	Composite resin
Group I	Single Bond®	Filtek Z250®
Group II	Clearfil SE Bond®	Filtek Z250®
Group II	Adper Prompt L-Pop®	Filtek Z250®

Table 2. Composition of adhesive systems investigated in this study

	Conditioner	Adhesive
Single Bond®	Phosphoric acid	HEMA, Ethanol, Water, Bis-GMA, Dimethacrylates, Amines, Methacrylate-functional copolymer of polyacrylic and polyitaconic acids
Clearfil SE Bond®		Primer : MDP, HEMA, Hydrophilic dimethacrylate, dl-Camphorquinine, N,N-diethanol-p-toluidine, Water Bond : MDP, Bis-GMA, HEMA, Hydrophobic dimethacrylate, dl-Camphorquinine, N,N-diethanol-p-toluidine, Silanated colloidal silica
Adper Prompt L-Pop®		Liquid 1 : Methacrylated phosphoric esters, Bis-GMA, Initiators based on camphorquinone, Stabilizers Liquid 2 : Water, HEMA, Polyalkenoic acid, Stabilizers

MDP 10-methacryloyloxydecyl dihydrogen-phosphate
 HEMA 2-hydroxyethyl-methacrylate
 BIS-GMA bis-phenol-A-diglycidylmethacrylate

8. Evaluation of Microleakage

Finally, each tooth was embedded in auto-polymerizing acrylic resin (Orthodontic Resin, Dentsply de Tray Germany). They were sectioned longitudinally, in a buccolingual direction coincident with the center of the restoration using a slow rotating diamond saw (Isomet™, Buehler Co., Lake Bluff, IL, U.S.A.) The sectioned parts were both analyzed for microleakage. Microleakage at the occlusal and cervical margin was evaluated with an optical stereomicroscope (SZ-PT 40, Olympus Optical Co., Ltd., Tokyo, Japan) at × 25

magnification and scored the following criteria (Fig. 1):

- 0 - No dye penetration
- 1 - Dye penetration that extended up to 1/3 of preparation depth.
- 2 - Dye penetration greater than 1/3, up to 2/3 of preparation depth.
- 3 - Dye penetration that extending to the axial wall.
- 4 - Dye penetration past the axial wall.

III. Results

There was no adhesive system which prevents microleakage perfectly.

Result of dye penetration scores in each group is presented in Table 3 and Fig. 2.

The difference among adhesive systems in enamel and dentin is analyzed by one-way ANOVA and the difference between enamel and dentin in each adhesive system was analyzed by t-test using SAS Ver 8.01.

The results were presented in Table 4.

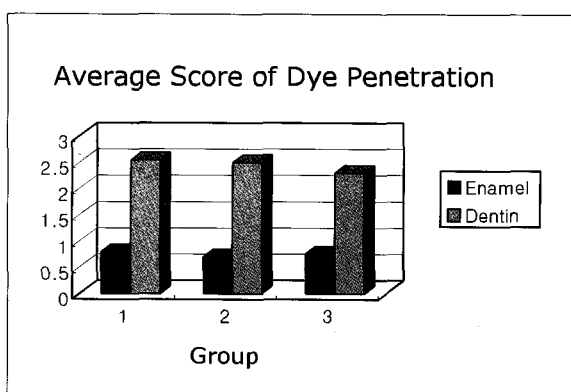


Fig. 2. Average Score of Dye Penetration

IV. Discussion

A goal of restorative dentistry is to develop adhesive restorative materials that are durable and provide an effective seal at the restoration/tooth interface. To achieve this purpose, many enamel-dentin bonding systems were introduced and improved. These new adhesive materials not only attempt to improve the quality of the bond, but also simplify the clinical procedures. In order to simplify the clinical procedures and improve the quality of the bonding, a self-etching primer was introduced. The self-etching primer system has several improvements in clinical application^{24,25}.

In this study, there was microleakage in all groups, regardless of the location of restoration. However, the microleakage of enamel margin is less than that of dentin margin in all groups ($p < 0.0001$). It represents that successful bonding to dentin is more difficult than to enamel. The result seems to be caused by the differences of composition and characteristic between enamel and dentin.

The inorganic content of enamel is 95% to 98% by weight and 86% by volume²⁶. The surface of enamel is smooth, and enamel is almost homogeneous in structure and composition. Unlike enamel, dentin contains a higher percentage of water and organic

Table 3. Frequency of Dye Penetration Score

		0	1	2	3	4	계
Single Bond®	Occlusal	8	8	4			20
	Cervical	1	4	3	7	5	20
Clearfil SE Bond®	Occlusal	7	12	1			20
	Cervical		4	6	6	4	20
Adper Prompt L-Pop®	Occlusal	5	15				20
	Cervical		4	8	6	2	20

Table 4. The results of statistic analysis

Bond	Single Bond	Clearfil SE Bond	Adper Prompt L Pop	p-value ¹
Enamel	0.80±0.77	0.70±0.57	0.75±0.44	0.8742
Dentin	2.55±1.23	2.50±1.05	2.30±0.92	0.7408
p-value ²	< 0.0001	< 0.0001	< 0.0001	

Values are mean±S.D.

material²⁶⁾. In addition, these constituents are unevenly distributed, so heterogeneous. Due to its heterogeneity, dentin can be described as a dynamic substrate and therefore represents a challenging substrate for bond. Another major reason why successful bonding to dentin was difficult to achieve is that dentin is an intrinsically wet substrate. The bonding area is connected with the pulp by dentin tubules which are filled with dentinal fluid, a serum-like tissue fluid. The dentinal permeability and the internal dentinal wetness depend on several factors²⁷⁾. The variability in dentinal permeability makes it a more difficult substrate for bonding than enamel. Dentinal bonding is further complicated by the formation of smear layer, while the dentin is cut of ground.

In this study, there are not significant differences among 3 adhesive systems at enamel margin ($p=0.8742$). Acid-etching technique, proposed by Buonocore²⁸⁾, is used to obtain micro-mechanical bonding of restorative materials in enamel surface. To obtain sufficient adhesion, two materials being joined must be in close and sufficient wetting is important. Sufficient wetting of the adhesive will only occur if its surface tension is less than the surface-free energy of the adherend^{14,15)}. Acid-etching technique enlarges surface area and transforms the smooth surface into an irregular surface with a high surface-free energy. The pH of the acidic primers may play a role in their effectiveness in penetrating the enamel. In the past the performance of self-etching adhesives has been a controversial issue, and there was concern that the manufacturers sacrificed enamel bond strength. The pH value of 32% UNI-ETCH[®], Clearfil SE Bond[®] primer, and Adper Prompt L-Pop[®] is 0.1, 1.6, and 1.0 respectively. According to Barkmeier et al.²⁹⁾ a primer agent with a pH 1.4 presumably allows mineralized tissue to be conditioned and primed in one treatment step. Scanning electron microscopy investigations of current self-etching adhesive also have shown enamel etching patterns morphologically similar to that of phosphoric acid-etching enamel³⁰⁾. Perdigão et al³¹⁾, Torii et al³²⁾, reported that no difference in shear bond strength and tensile bond strength to enamel among traditional acid etching and self-etching priming adhesive systems.

This study showed there is no significant difference among 3 adhesive systems ($p=0.7408$). In the ma-

jority of current dentin bonding system, smear layer is dissolved. Unlike total-etching systems, self-etching primer systems don't completely resolve or remove the smear layer, but rather partly integrate into hybrid layer³³⁾. One advantage of self-etching primer system is that it simultaneously demineralizes dentin and infiltrates it with monomers to the same depth³³⁾. In total etching systems, some questions have been raised about the possibility that the primer can't reach the demineralized zone completely. The pH of self-etching primers is low enough to promote dissolution of the smear layer and conditioning of the mineralized tissue. Tay et al³⁴⁾, Hayakawa et al³⁵⁾, reported that self-etching primers etch beyond the smear layer to form hybrid layers within intact dentin.

In dentin adhesion, the formation of hybrid layer³⁶⁾, is very critical. Current dentin bonding theories suggest that there are two fundamental processes involved in bonding an adhesive to dentin. First, the mineral phase must be extracted from the dentin substrate without damaging the collagen matrix, and second, the voids left by the mineral must be filled with an adhesive resin that penetrates the exposed collagen fibril network. If the exposed collagen collapses during the bonding procedure, the porosity of the dentin substrate is reduced, and many of the sites available for resin penetration are eliminated^{37,38)}. It has been reported that as long as the dentin is kept fully hydrated, the surface morphology of the demineralized layer does not change³⁹⁾. Results from previous studies with a "wet" bonding technique support these findings. It is speculated that moist dentin provides a more porous collagen network and thus greater infiltration of adhesive monomers^{36,37)}. However, in conventional total etching system, it is very technique-sensitive to keep an adequate dentinal wetness to form adequate hybrid layer. The advantage of self-etching primer systems is that problems associated with moist application technique are avoided. These agents contain water as a component, so, these agents can be used at dry dentin.

In this study, Clearfil SE Bond[®] and Adper Prompt L-Pop[®] were used as self-etching primer system. There are some considerations about bonding procedure and composition. Both agents contain water as a important component, so, these agents can be used

at dry dentin. However, being excessive water, the acidity is diluted and the bonding strength may be weakened. The pH value of these adhesive systems are 1.6 and 1.0 irrespectively. These value are low enough to infiltrate the smear layer and subsurface. And it must be considered that the pH value is defined as the negative decadic logarithm of the hydrogen ion concentration. Thus, small differences in pH values can mean that the substance is 100times more acidic. Regarding this fact, Clearfil SE Bond® is less aggressive to tooth than Adper Prompt L-Pop®. With a filler content of about 10%, Clearfil SE Bond® is considered as a filled adhesive. Filled low-viscosity resins are thought to have a strain capacity sufficient to relieve stresses between the shrinking composite restoration and the rigid dentin substrate. However, in this study there is no difference between both agents regarding the filling of low-viscosity resins.

In present study, all self-etching adhesives were able to achieve good in vitro results, and it can be assumed that the adhesives used are capable to producing sufficient etch patterns, showing bond strengths. The fact that self-etching primer system showed the similar results validates that smear layer modifying adhesives must not perform more poorly than total etching systems.

V. Conclusion

In conclusion, although any adhesive system can not prevent microleakage, there is no significant difference between total etching system and self-etching primer system. And, the self-etching primer systems efficiently bonded to tooth structure. For the evaluation of the clinical performance of self-etching primer systems on enamel and dentin bonding, further studies are needed by various products, various test method and various clinical condition.

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Abstract

COMPARISON OF MICROLEAKAGE WITH THREE DIFFERENT ADHESIVE SYSTEMS

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Recently, self-etching adhesive system have been developed and bonding procedures simplified into one or two steps, which are simultaneously applied to both enamel and dentin. These systems are easy to use and have the potential for good clinical success.

The purpose of this study is to evaluate in vitro the microleakage on the cementum/dentin and enamel walls in composite resin restoration of Class V cavities, regarding the use of different adhesive systems.

30 human premolars were divided into 3 groups. A standardized Class V preparation was prepared on the buccal and lingual surface of each premolar. The preparation were made parallel to the cemento-enamel junctions, with the gingival half of the preparation extending 1mm apical to the cemento-enamel junction. After adhesive system was applied to teeth as manufacture's recommendation, hybrid resin composite was filled in bulk into the preparation and light polymerized according to manufacturer's recommendations

Specimen were stored in distilled water at 37°C for 5 days and thermocycled 1000 times (5°C±2°C and 55°C±2°C), then immersed in a 2% methylene blue solution for 12 hours. After sectioning mesio distally through the restorations, the degree of dye penetration was scored under a stereomicroscope at × 25 magnification. The data were analyzed statistically using t-test and one-way ANOVA.

The results were as follows:

- There is no adhesive system which can prevent microleakage perfectly.
- There is significant difference in microleakage between enamel margin and dentin margin ($p < 0.0001$).
- In enamel margin, self-etching primer systems did not show any significant difference comparing total-etching system.
- In denin margin, self-etching primer systems did not show any significant difference comparing one-bottle adhesive system used in combination with total-etching.

Key words : Microleakage, Self-etching, Composite resin restoration