

EFFECT OF AN INTERMEDIATE BONDING RESIN AND FLOWABLE RESIN ON THE COMPATIBILITY OF TWO-STEP TOTAL ETCHING ADHESIVES WITH A SELF-CURING COMPOSITE RESIN

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ABSTRACT

This study compared the effect of an activator, intermediate bonding resin and low-viscosity flowable resin on the microtensile bond strength of a self-curing composite resin used with two-step total etching adhesives. Twenty extracted permanent molars were used. The teeth were assigned randomly to nine groups (n=10) according to the adhesive system and application of additional methods (activator, intermediate adhesive, flowable resin). The bonding agents and additional applications of each group were applied to the dentin surfaces. Self-curing composite resin buildups were made for each tooth to form a core, 5mm in height. The restored teeth were then stored in distilled water at room temperature for 24h before sectioning. The microtensile bond strength of all specimens was examined. The data was analyzed statistically by one-way ANOVA and a Scheffé's test. The application of an intermediate bonding resin (Optibond FL adhesive) and low-viscosity flowable resin (Tetric N-flow) produced higher bond strength than that with the activator in all groups. Regardless of the method selected, Optibond solo plus produced the lowest μ TBS to dentin. The failure modes of the tested dentin bonding agents were mostly adhesive failure but there were some cases showed cohesive failure in the resin. [J Kor Acad Cons Dent 34(5):397-405, 2009]

Key words : activator, intermediate bonding resin, low-viscosity flowable resin, self cure composite resin, microtensile bond strength

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I . Introduction

The remarkable evolution of contemporary dentin adhesive systems has focused on simplifying the clinical steps. Factors, such as time and simplification of the clinical steps have led to the increased use of simplified-step adhesives in association with resin materials.

Conventional (three-step) and simplified-step adhesive systems are currently available for etch and rinse systems. The bonding protocol of three-step systems requires separate acid-etching of the enamel

and dentin surfaces, the application of a primer containing hydrophilic resin and solvents to etched dentin, and the subsequent application of an adhesive containing resin monomers to both enamel and dentin¹⁾. However, in order to simplify the bonding protocol, manufacturers have attempted to produce several one-bottle dentin bonding systems that combine the primer and adhesive resin in a single solution.

Simplified-step adhesive systems have replaced their predecessors in most clinical applications because of their reported versatility, ease of use and time-saving protocols. Most of these products are composed of hydrophilic and hydrophobic resins dissolved in a high vapor pressure organic solvent, such as ethanol or acetone, which chase the water and bring the monomers into intimate contact with the exposed collagen fibers²⁾. These adhesive systems

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(one-bottle) have low and varied pH³⁾.

Some of the two-step total etch adhesives are incompatible with a self-curing composite resin³⁾. The decrease in dentin bond strength of self-curing composite resin is inversely proportional to the acidity of the uncured adhesive remaining on the dentin surface^{4,5)}. Sanares et al.³⁾ reported that there is an inverse correlation between the acidity of these single bottle adhesives and the microtensile bond strength (μ TBS) obtained from the use of a self-curing composite resin. This phenomenon has been attributed to an acid-base reaction between the basic tertiary amine in the self-curing composite resin and uncured acidic monomer of the adhesive, which prevents the tertiary amine from participating in the redox reaction and impedes free radical generation and complete curing of the resin composite⁶⁾. Therefore, the degree of polymerization of this superficial layer can be reduced, resulting low bond strengths^{3,7)}.

In order to overcome this problem, some manufacturers have developed so-called activators, which give the practitioner the option to use either dual-curing or light-curing bonding systems. Most of these activators contain salts of aromatic sulfinic acids, which can react with acidic monomers to produce free radicals and initiate polymerization of the self-curing composite⁸⁾. For adhesive systems, these components must be strong reducing agents and oxygen scavengers, which improve polymerization, reduce the thickness of the oxygen inhibition layer and amount of uncured acidic monomer on the bonded surface⁹⁾.

Some studies suggested that this incompatibility problem can be solved using either an additional adhesive resin layer without acidic monomers⁷⁾ or an intermediate low-viscosity composite liner over bonded dentin¹⁰⁻¹²⁾, thereby improving the bond strength between the adhesive and resin composite core material. The use of bonding systems associated with a low-viscosity resin has been suggested to improve the bond strength, marginal sealing and interfacial adaptation of restorative composites to dentin¹²⁻¹⁵⁾. In addition, some studies¹⁶⁻¹⁸⁾ suggested that the application of a low-viscosity resin can preserve and protect the hybrid layer during the shrinkage of restorative composites or resin cements, minimizing the postoperative sensitivity and increasing longevity of

the restoration.

Therefore, this study compared the effect of an activator, intermediate adhesive and low-viscosity flowable resin on μ TBS of self-curing composite resin and compared the μ TBS according to the dentin bonding agent in the same application method.

II. Materials and Methods

1. Tooth preparation

Twenty extracted permanent molars without caries were stored in distilled water. The crown portions were sectioned horizontally at the mid-coronal level using a slow-speed diamond-saw sectioning (Accutom-50; Struers, Rødovre, Denmark) under continuous water cooling. The flat dentin surface was polished with 600 grit silicon carbide paper under running water.

2. Dentin bonding and resin composite buildups

The teeth were assigned randomly to nine groups, according to the adhesive system and application of an intermediate adhesive, flowable resin between the adhesive and self-curing composite resin. Table 1 and Figure 1 show the materials and groups used in this study.

The dentin surface was etched for 15 sec with a 35% phosphoric acid etchant, and rinsed and air-dried gently for 5 sec, to leave a glossy surface. The bonding agents and additional applications in each group were applied to the dentin surfaces according to the manufacturer's instructions (Table 2). Optibond FL adhesive (Kerr, Orange, CA, USA) and Tetric N-flow (Ivoclar Vivadent, Schaan, Liechtenstein) were used as the intermediate adhesive and flowable resin, respectively. After the bonding procedure, the self-cure composite resin (BisFil 2B; BISCO, Inc., Schaumburg, IL, USA.) buildups were made to each tooth to form a core. The restored teeth were then stored in distilled water at room temperature for 24h.

Table 1. Materials used in this study

Materials	Compositions	Manufacturer
Etchant	Phosphoric acid(37wt.% in water)	Ivoclar Vivadent
Optibond FL adhesive	Bis-GMA, HEMA, GDMA, Ba-Al-silicate glass, silicate glass, Na ₂ SiF ₆ , CQ	Kerr
Tetric N-flow	Dimethacrylates (including TEGDMA), fillers(barium glass, ytterbium trifluoride, highly dispersed silica and mixed oxide), catalyst, stabilizers, pigments	Ivoclar Vivadent
BisFil 2B	Bisphenol A diglycidyl methacrylate, Ethoxylated bisphenol A dimethacrylate (base only), Glass frit, Triethyleneglycol dimethacrylate, Silica, Amorphous silica	Bisco
XP bond	Bonding-carboxylic acid modified dimethacrylate phosphoric acid modified acrylate resin (PENTA), UDMA, TEGDMA, HEMA, Butylated benzendiol (stabilizer) Ethyl-4-dimethylaminobenzoate, camphorquinone Functionalized amorphous silica Self-cure activator- Urethane dimethacrylate(UDMA); 2-hydroxyethyl methacrylate(HEMA), catalysts, photoinitiators, stabilizers, acetone, water	Dentsply Caulk, Milford, DE, USA
Prime & Bond NT	Bonding- Di-/tri-methacrylate resins, functionalized amorphous silica, PENTA, photoinitiators, stabilizers, CH, acetone Self-cure activator- Mono-/di-methacrylate resins, catalysts, photoinitiators, stabilizers, acetone, water	Dentsply De Trey
Optibond solo plus	Bonding- Bis-GMA, HEMA, GPDM, ethanol, CQ, anorganic fillers Activator- Benzene sulfinic acid sodium salt, ethanol, Bis-GMA, HEMA	Kerr

* Abbreviations: BIS-GMA=bisphenol-A-glycidyl ether dimethacrylate; UDMA=urethane dimethacrylate;

BIS-EMA=ethoxylated bisphenol-A-dimethacrylate; HEMA=2-hydroxyethyl methacrylate;

MDP=10-methacryloyloxydecyl dihydrogen phosphate; PENTA=dipentaerythritol penta acrylate monophosphate,

TEGDMA=triethylene glycol dimethacrylate; BHT=butylhydroxytoluene.

3. μ TBS testing

The restored teeth were cut longitudinally to produce specimens, approximately 1×1 mm thick and 10 mm long. Each group contained 10 specimens. The specimens were glued to the jig of the microtensile testing machine (BISCO Inc, Schaumburg, IL, USA) using cyanoacrylate cement (Zapit; Dental Ventures of America, Corona, CA, USA). A tensile load was applied at a cross-head speed of 1 mm/min until the specimen failed.

4. Failure mode investigation

The failure mode was examined using an operating microscope (OPMI pico; Carl zeiss, Obercohen, Germany) at 25× magnification, and the results were classified as follows: adhesive, if the composite resin cone had fractured at the adhesive-tooth interface; cohesive in resin, if the composite resin cone had fractured inside the composite resin; cohesive in dentin, if the composite resin cone had fractured with a large portion of the dentin attached; or mixed, a combination of adhesive and cohesive in the dentin or cohesive in the resin.

5. Statistical analysis

Statistical analysis was carried out using one-way ANOVA with SPSS 12.0 software (SPSS, Chicago,

IL, USA). A Scheffé's test was used for post-hoc multiple comparison. The level of significance was set to $p < 0.05$.

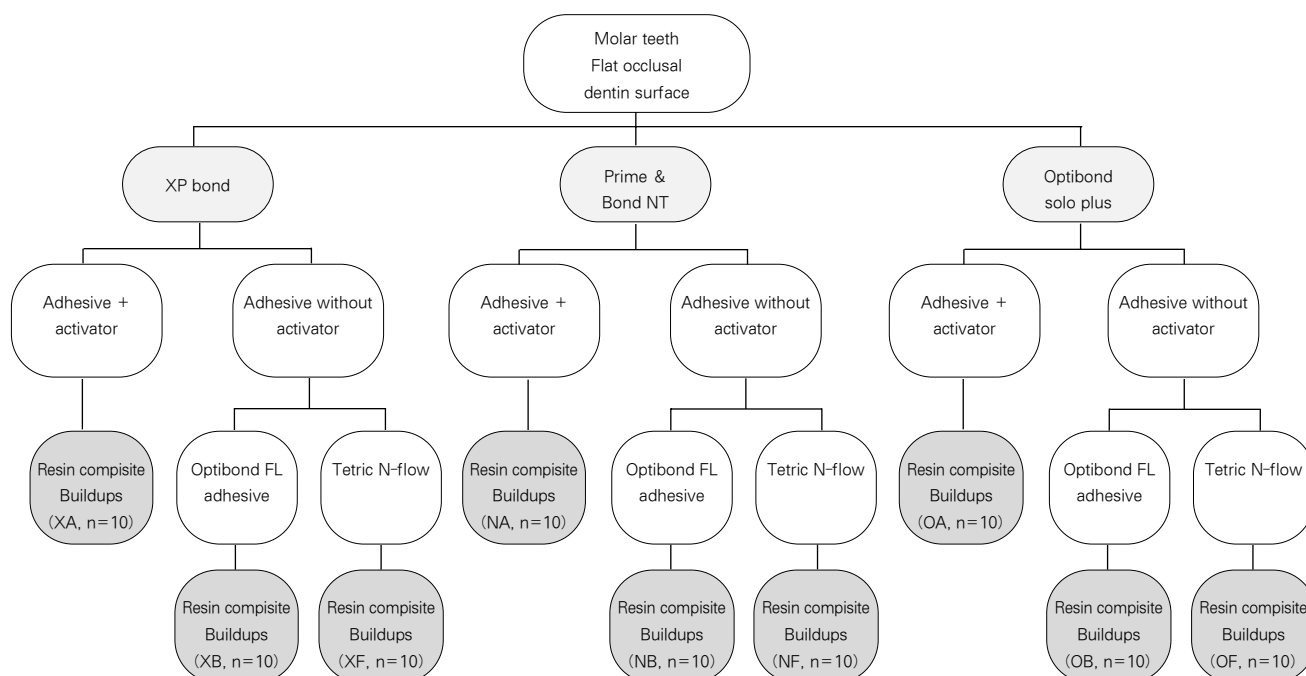


Figure 1. Restorative procedure of experimental groups

Table 2. Bonding procedures according to groups

Groups	Procedures
XA	Bond: Mix with Self-Cure Activator in equal amounts, apply the mixture and leave undisturbed (20s), thoroughly air dry (5s), light cure (10s)
XB	Bond: Apply and leave undisturbed (20s), thoroughly air dry (5s), light cure (10s) Intermediate bonding resin: Apply to a thin layer, light cure (20s)
XF	Bond: Apply and leave undisturbed (20s), thoroughly air dry (5 s), light cure (10 s) low viscosity flowable resin: Apply as a thin coat, light cure (20s)
NA	Bond: Mix with Self-Cure Activator in equal amounts, apply the mixture and leave undisturbed (20s), gently air dry (5s), light cure (10s)
NB	Bond: Apply and leave undisturbed (20s), thoroughly air dry (5 s), light cure (10 s) Intermediate bonding resin: Apply to a thin layer, light cure (20s)
NF	Bond: Apply and leave undisturbed (20s), thoroughly air dry (5 s), light cure (10s) low viscosity flowable resin: Apply as a thin coat, light cure (20s)
OA	Bond: Mix with Optibond Solo Plus Activator in equal amounts, apply the mixture with light scrubbing motions (15s), light cure (20s)
OB	Bond: Apply with light scrubbing motions (15s), light cure (20s) Intermediate bonding resin: Apply to a thin layer, light cure (20s)
OF	Bond: Apply with light scrubbing motions (15s), light cure (20s) low viscosity flowable resin: Apply as a thin coat, light cure (20s)

III. Results

1. Comparison between μ TBS according to the intervening method of each dentin bonding agent

Table 3 shows a comparison of μ TBS according to the intervening method in the same dentin bonding agent.

Group XB and XF showed higher bond strength than group XA ($p < 0.05$) but there was no significant difference between groups XB and XF ($p > 0.05$). Groups NB and NF had a higher bond strength than group NA ($p < 0.05$) but there was no significant difference between groups NB and NF ($p > 0.05$). In Optibond solo plus, group OB had the highest μ TBS, followed by group OF and OA. There were significant differences between all groups ($p < 0.05$).

2. Comparison between μ TBS according to the dentin bonding agent of each intervening method

Figure 2 shows the μ TBS of the three dentin bonding agents in the activator, bonding resin, and low viscosity flowable resin.

In the activator group, Prime & Bond NT showed

the highest μ TBS, followed by a XP bond, Optibond solo plus. However, there was no significant difference between the XP bond and Prime & Bond NT ($p > 0.05$). Optibond solo plus showed the lowest μ TBS of all groups ($p < 0.05$).

In the bonding resin group, Prime & bond NT also had the highest bond strength, followed by the XP bond, Optibond solo plus. However, there was no significant difference between the XP bond and Optibond solo plus ($p > 0.05$). Prime & Bond NT had the highest μ TBS of all groups ($p < 0.05$).

In the low viscosity flowable resin group, Prime & Bond NT had the highest μ TBS, followed by the XP bond, Optibond solo plus. However, there was no significant difference between the XP bond and Prime & Bond NT ($p > 0.05$). Optibond solo plus had lowest μ TBS value of the groups ($p < 0.05$).

3. Failure mode

Table 4 shows the failure modes. Adhesive failure was observed predominantly in all groups. Adhesive failure was observed more often in Optibond solo plus than the other groups. All specimens in group OA showed adhesive failure.

Table 3. Mean μ TBS (Mean \pm SD, MPa)

each group n=10	Activator	Intermediate bonding resin	Low viscosity flowable resin
XP bond	XA (36.54 \pm 3.66) ^a	XB (43.85 \pm 3.63) ^b	XF (46.59 \pm 1.32) ^b
Prime & Bond NT	NA (36.84 \pm 2.18) ^a	NB (48.15 \pm 2.10) ^b	NF (47.85 \pm 4.95) ^b
Optibond solo plus	OA (13.60 \pm 2.20) ^a	OB (41.55 \pm 2.32) ^b	OF (36.49 \pm 1.70) ^c

μ TBS with same superscript in the same horizontal row were not significantly different ($p < 0.05$).

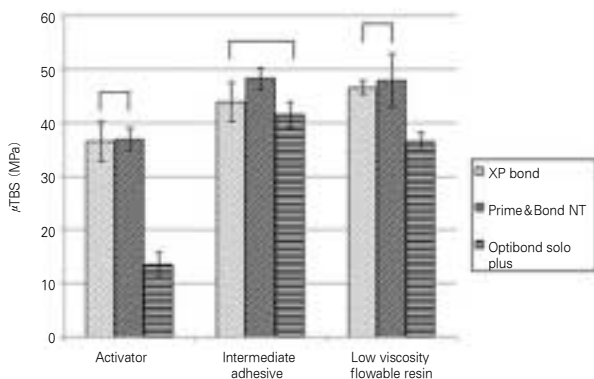


Figure 2. μ TBS of three dentin bonding agents in same application method. The subgroups under the horizontal line were not significantly different ($p > 0.05$).

Table 4. Failure mode

Group	Adhesive failure	Cohesive failure in resin	Cohesive failure in dentin	Mixed
XA	8	2	0	0
XB	6	4	0	0
XF	9	0	0	1
NA	8	1	0	1
NB	6	3	0	1
NF	9	1	0	0
OA	10	0	0	0
OB	9	1	0	0
OF	9	0	1	0

IV. Discussion

Recently, several one-bottle dentin bonding systems that combine the primer and adhesive resin into a single solution were introduced. These materials have attracted considerable attention from the dental profession because some two-step total etch adhesives are incompatible with self-curing composite resins. In some studies, an adverse interaction between the tertiary amine catalytic component of the restorative composites placed over an uncured acidic resin adhesive layer was previously reported^{3,7,18}.

Compromised bond strength was observed when a self-curing composite resin was coupled with unactivated adhesives, which can compromise their longevity. Swift¹⁹ reported two reasons for this incompatibility. The first is that the oxygen-inhibition layer of polymerized adhesives contains uncured acidic resin monomer, which can react with the amines from the self-curing composite resin in the adhesion-promoting monomers to form charge-transfer complexes, resulting in incomplete polymerization and compromised adhesion. The second reason is that water movement occurs through the polymerized adhesive layer, which contains acidic and hydrophilic resin monomers. The slow rate of polymerization of the self-curing composite resin enables water to diffuse from the hydrated dentin, which can trap water, resulting in osmotic blistering or water trees²⁰.

Some manufacturers have developed activators for self-curing composite resin to overcome the dilemma of incompatibility. Activators have been used with dentin adhesives containing acidic resin monomers to improve their bonding to self-cured composites³. The activator components must be strong reducing agents and oxygen scavengers, which improve polymerization, in order to reduce the thickness of the oxygen inhibition layer and amount of uncured acidic monomer on the bonded surface⁹. For adhesive systems, some alternative initiators, such as sulphinic acid salts, organoboron compounds or barbituric acid/cupric chloride have been included in adhesive compositions as initiator systems. In addition, sulfonamides can be effective coactivators for photoinitiation²¹⁻²³. Munksgaard et al.²⁴ suggested that the

inclusion of reducing agents, such as aromatic sulphinic acid salts or propanal in dentin adhesives, could improve the bond strength and marginal integrity by reducing the thickness of the oxygen inhibition layer.

In another solution, Tay et al.⁷ suggested the application of an adhesive resin intermediate layer to avoid contact between the uncured acidic adhesive and unpolymerized resin composite, and prevent adverse interactions. In this study, intermediate adhesive (Optibond FL adhesive) and a low-viscosity flowable resin (Tetric N-flow) were used as an intermediate layer over bonded dentin. The application of an intermediate adhesive and low-viscosity flowable resin eliminates the incompatibility between resin-based materials, thereby avoiding the formation of a weakened bonded interface⁹. Jayasooriya et al.¹¹ suggested that the application of an intermediate bonding resin and a low-viscosity flowable resin layer can improve the degree of conversion of the underlying adhesive resin, and increase the bond strength.

In this study, the application of an intermediate adhesive and a low-viscosity flowable resin produces higher bond strength than the applying activator. Therefore, applying an intermediate adhesive and low-viscosity flowable resin should be more useful in preventing adverse interactions between the acidic monomers on the adhesive surface and the tertiary amine catalytic component of the self-curing composite resin.

The mean bond strengths of XP bond and Prime & Bond NT were significantly higher than Optibond Solo Plus. According to the manufacturer's composition, XP bond, Prime & Bond NT and Optibond Solo Plus contain butanol, acetone and ethanol as solvents, respectively. In many studies²⁵⁻²⁷, the effect of the solvents on the efficacy of dentin bonding has been demonstrated. These chemical agents, known as "water-chasers", increase the dentin wettability and help replace the water on the acid-etched and rinsed dentin surface with hydrophilic resin monomers. Acetone is used frequently as a solvent because it can efficiently remove water from surfaces^{25,26,28}. By adding 10% acetone to water, the vapor-pressure increases by more than 300%, leading to volatilization of some of the surface water²⁹.

Ethanol is another organic solvent that is used as a vehicle in adhesives. However, but it has a higher boiling temperature and lower vapor pressure than acetone. Alcohol triggers more hydrogen bonding than acetone. Therefore, alcohol does not chase water as effectively as acetone, which can compromise the durability of dentin bonding^{25,26,30}. Therefore, it is likely that Optibond solo plus containing ethanol as a solvent has low bond strength.

Sanares et al.^{3,31} reported that there was a linear relationship between the pH of the adhesives and their mean μ TBS when using a self-cured composite. According to their study, Prime & Bond NT, which is more acidic than Optibond solo plus, exhibited the largest decrease in bond strength. However, in this study, Prime & Bond NT, which is the most acidic of the three adhesives, exhibited higher bond strength than Optibond Solo Plus. These findings are inconsistent with those of Sanares's study. Therefore, it is unlikely that a decrease in the bond strength of simplified-step adhesives using self-cured composites is due to the pH of adhesives. Factors other than pH, such as the monomer composition, appear to be responsible for the bond strength.

Adhesive failure was the main failure mode observed in all groups. In Optibond solo plus, adhesive failure was observed more often than the other groups. Some cohesive failure in resin was observed. This might due to the incorporation of air-voids within the self-cured composite during hand-mixing of the two-paste systems. The ultimate mechanical properties of the polymerized material are compromised by these voids, due to their inherent potential to increase the stress level within the self-curing composite resin³².

Composite build-up materials have different curing modes that vary from self-cured, light-cured or dual-cured. Light-cured resin composites have displaced the use of self-curing composites in esthetic dental applications. However, self-curing composite resins have a variety of clinical applications as direct core buildup materials. The success of the treatment depends on the appropriate selection of adhesive system in combination with a resin core composite. However, it was reported that there are incompatibilities between two-step total etch adhesives and self-

curing composite resins. Based on these findings, it is possible that applying an intermediate adhesive and a low-viscosity flowable resin would more effective on dentin bonding of a self-curing composite resin using two-step total etch adhesives with low pH. More study should be carried out using other two-step total etch adhesives to determine if intermediate adhesive and flowable resin are as effective as the one examined in this study.

V. Conclusions

This study compared the effect of an activator, intermediate bonding resin and low-viscosity flowable resin on the microtensile bond strength of a self-curing composite resin. The results showed that applying an intermediate adhesive (Optibond FL adhesive) and low-viscosity flowable resin (Tetric N-flow) produces a higher bond strength than applying an activator. Regardless of the method selected, Optibond solo plus produced the lowest μ TBS to dentin. The failure modes of the tested dentin bonding agent were mainly adhesive failure with some cases of cohesive failure in resin.

These results suggest that the application of an intermediate adhesive and low-viscosity flowable resin may be made more effective on dentin bonding to a self-curing composite resin using two-step total etch adhesives with a low pH.

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국문초록

자가 중합 복합 레진과 두 단계 산 부식 접착제의 친화성에 대한 중간 접착제와 흐름성 레진의 효과

최속경 · 염지완 · 김현철 · 허 복 · 박정길*

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이 연구의 목적은 자가 중합 복합 레진과 두 단계 산 부식 접착제의 친화성에 대한 중간 접착제와 흐름성 레진의 효과를 미세 인장 결합 강도를 통해 비교하는 것이었다. 20개의 발거된 대구치를 사용해 접착제와 부가적인 적용(활성제, 중간 접착제, 흐름성 레진)에 따라 XP bond 그룹 (XA, XB, XF 그룹), Prime & Bond NT 그룹 (NA, NB, NF 그룹), Optibond solo plus 그룹 (OA, OB, OX 그룹) 으로 나누었다. 접착제 적용 후 활성제, 중간 접착제, 흐름성 레진을 각각 적용하고 상부에 자가 중합 레진을 축적하였다. 24시간 동안 실온의 증류수에 보관 후 각 그룹당 10개의 시편을 준비하였다. 모든 시편의 미세인장 결합 강도를 측정하여 다음과 같은 결과를 얻었다.

1. 모든 그룹에서 중간 접착제와 흐름성 레진의 적용은 활성제 적용보다 더 높은 결합 강도를 보였다.
2. 선택된 방법에 관계없이, Optibond solo plus 그룹은 가장 낮은 미세인장 결합강도를 보였다.
3. 파절 양상은 대부분 접착성 파절을 보였고 일부 레진 내에서 응집성 파절을 보였다.

이번 연구의 결과로부터, 중간 접착제와 흐름성 레진의 적용이 낮은 pH를 가지는 두 단계 산 부식 접착제와 자가 중합 복합 레진의 상아질 접착에 더 효과적이라 예상된다.

주요단어 : 활성제, 중간 접착제, 흐름성 레진, 자가 중합 레진, 미세인장 결합 강도