Regional bond strength of dentin bonding systems to pulp chamber dentin

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국문초록

치수강 상아질에 대한 상아질 접착제의 결합 강도에 관한 연구

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본 연구는 치수강 상아질 부위 및 상아질 접착제 종류에 따른 결합 강도를 측정하고 이들 사이의 상관관계를 구명하고자 시행되었다. 45개의 대구치를 포매 후, 대조군에서는 교합면 법랑질 제거 후 #600 SiC paper까지 순차연마하여 상아질을 노출시켰고, 실험군에서는 치수강 개방 후 1시간동안 NaOCl에 보관 후 axial wall과 pulpal floor를 노출시켰다. 노출된 상아질 면에 상아질 접착제를 적용한 후 Z 100을 충전한 다음 40초간 광중합하였다. 사용된 상아질 접착제는 Scotchbond Multi Purpose와 Single Bond, Clearfil SE Bond였다. 37℃ 증류수에 24시간 보관 후, 저속 diamond saw를 이용하여 0.7mm 두께로 수직절단하고 고속 diamond point(#104)로 단면적 $1mm^2$가 되도록 시편을 제작하고, Universal testing machine에서 미세인장 결합강도를 측정하였다.

1. 모든 상아질 접착제의 미세인장강도는 대조군, axial wall군, pulpal floor군 순으로 감소하는 경향을 보였다.
2. 대조군에서 SM과 SB는 SE에 비해 유의성있게 높은 결합강도를 나타내었다($p<0.05$).
3. SM과 SB는 대조군에 비해 axial wall군과 pulpal floor군에서 유의성있게 낮은 결합강도를 보였으나, SE에서는 pulpal floor군만이 유의성있게 낮은 결합강도를 보였다($p<0.05$).
4. Axial wall군과 pulpal floor군에서는 상아질 접착제의 종류에 따른 유의차를 보이지 않았다.
5. 전자현미경 소견에서는 대조군에 비해 axial wall군과 pulpal floor군이 더 부드러운 접착 계면을 나타내었다. 혼성층의 두께는 결합강도의 감소와는 관련이 없었다. [J Kor Acad Cons Dent 29(1):13-22, 2004]

주요어 : 치수강 상아질, 상아질 접착제, 결합강도, 혼성층, Axial wall군, Pulpal floor군

Ⅰ. INTRODUCTION

The development of new dentin bonding systems has brought about great improvements in adhesive dentistry and the use of resin composite has become widely accepted for treatment of both anterior and posterior teeth. Also resin composites used as core materials after endodontic treatment are becoming more and more popular in the clinical practice of dentistry because of their esthetics, ability to bond to tooth structure, strength, and the fact that they allow immediate continuation of a crown preparation. Good adhesion between the restorative material and the cavity wall results in good marginal sealing, less microleakage, and longevity of the restoration[1].

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Endodontic treatment has become a routine procedure for treating and retaining nonvital teeth. Endodontic treatment consists of removing all contents of the root canal system before and during cleaning and shaping. Successful cleaning entails the use of instruments to mechanically remove dentin, irrigant to flush loosened debris away, and chemicals to dissolve contaminants from inaccessible regions[2].

Sodium hypochlorite and hydrogen peroxide are common endodontic irrigants that are used for the debridement and deproteinization of mechanically prepared, smear layer covered radicular dentin[3]. Sodium hypochlorite is also frequently used for chemomechanical caries removal and the arrest of hemorrhage in pulpal exposures before bonding to coronal dentin occurs[4,5]. The use of these irrigants provides gross debridement, lubrication, destruction of microbes, and dissolution of tissues[6]. Recent studies showed that bond strength of some adhesives was compromised by the use of these reagents on root and crown dentin[7-9], as well as enamel[10]. Contamination of dentin with blood or other body fluids can also be detrimental to bond strength with dental adhesives[11,12].

The incomplete removal of the partially denatured or destabilized collagen matrix has been proposed as a possible reason for compromised bond strength in sodium hypochlorite treated, acid etched dentin[13]. Sodium hypochlorite, apart form being an effective deproteinizing agent, is similar to hydrogen peroxide in that it is also a potent biological oxidant[14]. Sodium hypochlorite breaks down to sodium chloride and oxygen. This oxygen causes strong inhibition of the interfacial polymerization of resin bonding materials[15]. After the chemical irrigation of the root canal, the residual chemical irrigants and their products are likely to diffuse into the dentin along the dentinal tubules, which must result in decrease of bond strength.

The tooth structure that remains after endodontic treatment may be undermined and weakened by caries, fracture, tooth preparation, and restoration. Endodontic manipulation further removes important intracoronal and intraradicular dentin. Finally, the endodontic treatment changes the actual composition of the remaining tooth structure[16].

The major changes in the endodontically treated tooth include loss of tooth structure, altered physical and esthetic characteristics of the residual tooth. The decreased strength evaluated in endodontically treated teeth is primarily due to the loss of coronal tooth structure and is not a direct result of the endodontic treatment. Endodontic access into the pulp chamber destroys the structural integrity provided by the coronal dentin of the pulpal roof and allows greater flexing of the tooth under function[17]. However, the tooth structure remaining after endodontic treatment also exhibits irreversibly altered physical characteristics. Changes in collagen cross linking and dehydration of the dentin result in a 14% reduction in strength of endodontically treated molars[17]. The internal moisture loss has been shown to average approximately 9%[18].

Dentin is not a uniform tissue but differs from each region. With age, dentin formation continues slowly, and the regular secondary dentin is laid down at the pulpal end of the primary dentin. In response to noxious stimuli, the odontoblasts may evacuate the tubules, giving rise to the so called dead tract, and/or seal off the tubules at their pulpal ends with irregular secondary dentin, or form sclerotic dentin. Irregular secondary dentin has many fewer tubules with irregular orientation, than primary dentin[19].

Associated with physiological aging, especially in root dentin, the dentinal tubules become completely occluded by mineral in a process similar to that of peritubular dentin formation[19]. Fogel et al[20] showed that the permeability of root dentin is much lower than that of coronal dentin. The number of dentinal tubules per unit area is less for radicular dentin, meaning that the area of intertubular dentin available for bonding is greater in the root than the crown[21]. The structure of pulp chamber floor is complicated, including primary dentin, and regular and irregular secondary dentin.

Restorations for endodontically treated teeth should be designed to replace the missing tooth structure and to protect the remaining tooth structure from fracture. Not every endodontically treated tooth needs a crown or a dowel. Recently, the opportunity for the restoration of nonvital teeth with resin composite has increased based on the development of better dentin bonding sys tems. A resin composite enables a nonvital tooth to be restored by only replacement of the missing tooth structure, because adhesive restoration can reinforce remaining tooth structure[22 23].

Adhesive restoration for endodontically treated teeth offers many advantages over the use of tradi tional, nonadhesive materials. For instance bonded resins permit transmission of functional stresses across the bonded interface to the tooth, with the potential to reinforce weakened tooth structure[24]. Using adhesive materials properly can reduce microleakage of interface between these materials and tooth structure. Application of adhesive to acid etched dentin creates an acid resistant, resin infiltrated collagen layer, the so called hybrid layer that not only retains composites to dentin, but also can seal dentin from oral fluids[25].

Recently, Sano et al[26] developed micro tensile bond strength test, permitting the measurement of small bounded areas as small as 1 mm^2. As this new method permits measurement on small areas, it can be used to compare regional bond strengths with different surface of the pulp chamber.

The purpose of this study is to evaluate the regional bond strengths of three dentin bonding systems to pulp chamber dentin of endodontically treated teeth and to evaluate the effect of NaOCl pretreatment on resin dentin bonding interface.

Ⅱ. MATERIALS AND METHODS

Forty five caries free human molars extracted for the periodontal reasons were used in this study. The teeth were stored frozen after extrac tion until use. Initially, the teeth were embedded in epoxy resin using acrylic ring. Then, teeth were divided into three groups of fifteen for each control and experimental groups. Three dentin bonding systems and one resin composite were used in this study and their components, manu facturers were listed in table 1.

1. Specimen preparation

(1) Control group Intact dentin group

The teeth were sectioned to remove occlusal

Table 1. Materials used in this study

Material (code)	Component	Composition	Manufacturer
Scotchbond Multi-Purpose (SM)	Etchant	35% Phosphoric acid	3M Dental Products, St. Paul, MN, USA
	Primer	HEMA, water, Polyalkenoic acid compolymer	
	Adhesive	HEMA, Bis-GMA	
Single Bond (SB)	Etchant	35% Phosphoric acid	3M Dental Products, St. Paul, MN, USA
	Adhesive	HEMA, Bis-GMA, Polyalkenoic acid compolymer, ethanol, water	
Clearfil SE Bond (SE)	Primer	MDP, HEMA, water	Kuraray Co., Osaka, Japan
	Adhesive	MDP, HEMA, dimethacrylate, microfiller	
Z-100 Restorative	Resin composite	Zirconia/silica filler Bis-GMA, UDMA	3M Dental Products, St. Paul, MN, USA

Bis-GMA–Bisphenol-A glycidyl methacrylate
HEMA–Hydroxyethylmethacrylate
MDP–Methacryloyloxydecyl dihydrogen phosphate

enamel using a Low speed diamond saw (ISOMET; Buhler, USA) and exposed dentin surface was ground with #600 grit SiC paper serially under a stream of running water. Three dentin bonding systems, Scotchbond Multi Purpose(3M, USA), Single Bond(3M, USA), Clearfil SE Bond(Kuraray, Japan), were applied according to manufacturer's instruction (Table 2). Then, teflon mold(diameter : 6mm, height : 2mm) was placed on bonding area and filled with resin composite(Z 100; 3M, USA) and light cured (Spectrum 800; Dentsply, USA) for 40 sec.

(2) Experimental group

Initially, the access cavity preparation was performed using high speed diamond point under copious water spray. Following this, pulp tissue in pulp chamber was carefully removed using endodontic file. The teeth were then stored in 5% NaOCl for 1 hour.

Table 2. Instruction for dentin boding systems

Product	Instruction
Scotchbond Multi-Purpose	Etching 15 sec, priming, air dry 5 sec Adhesive, light-cure 10 sec
Single Bond	Etching 15 sec Adhesive (two coat), air dry 5 sec Light-cure 10 sec
Clearfil SE Bond	Primer 20 sec, air dry Adhesive, light-cure 10 sec

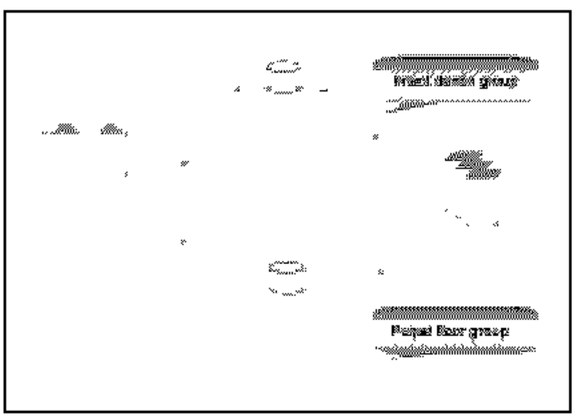

Fig. 1. Specimen preparation for micro-tensile bond strength test (Intact dentin group, Pulpal floor group)

1) Axial wall group

The teeth were sectioned mesio distally parallel to the long axis of teeth using Low speed diamond saw. Then, three dentin bonding systems were applied to axial wall area in pulp chamber according to manufacturer's instruction and Z 100 was filled in pulp chamber and light cured for 40 sec.

2) Pulpal floor group

The teeth were sectioned at pulpal floor level perpendicular to the long axis of teeth and ground with #600 grit SiC paper serially. Three dentin bonding systems were applied according to manufacturer's instruction. Then, teflon mold was placed on the cured bonding resin and filled with Z 100 and light cured for 40 sec.

2. Micro-tensile bond strength test

All restored specimens were stored in distilled water at 37℃ for 24 hours. The teeth were serially sectioned into slice of mean thickness 0.7 mm perpendicular to the bonded surface using a Low speed diamond saw under copious water supply. These specimens were then trimmed into an hour glass shape to give a bonded surface of 1 mm^2 using a high speed diamond point (#104; Shofu, Japan). The trimmed specimens were attached to testing zig with cyanoacrylate adhesive(Zapit; MDS Products Co., USA), then subjected to tensile forces in a universal testing machine(EZ Test, Shimadzu, Japan) at a cross head speed 1 mm/min.

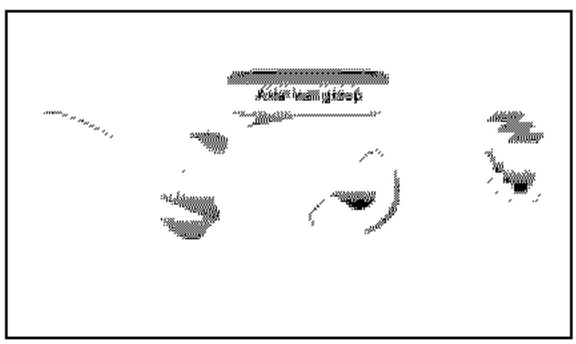

Fig. 2. Specimen preparation for micro-tensile bond strength test (Axial wall group)

3. SEM evaluation

For the evaluation of the morphology at the resin dentin interface, specimens used for hybrid layer observation were bonded with each system in the same manner as for the micro tensile bond test. One day later, the teeth were sectioned per pendicular to bonding surface and then embedded in epoxy resin. Then the sectioned surfaces were serially ground to #2000 grit SiC papers, and highly polished with a diamond paste. The speci mens were subjected to 10% phosphoric acid treatment for 3~5 sec[27]. Then specimens were rinsed with water for 15 sec and treated with 5% sodium hypochlorite for 5 min[28]. After being extensively rinsed with water, the treated speci mens were air dried, gold sputter coated and examined in SEM(S 2300; Hitachi Co., Japan).

4. Statistical analysis

The maximum tensile force was divided by the area of the specimen and the measured micro tensile bond strength values were analysed using ANOVA/ Newman Keuls multiple comparisons test at a significance level of 0.05.

Table 3. Micro-tensile bond strength of each bonding system to dentin(MPa+S.D, n=10)

Group	Control	Axial	Pulpal
SM	36.71±5.36	24.64±9.73	21.73±8.12
SB	34.69±6.31	23.85±6.86	20.54±6.80
SE	28.04±6.23	22.64±5.71	19.43±5.39

Ⅲ. RESULTS

1. Micro-tensile bond strength

The micro tensile bond strengths of Scotchbond Multi Purpose(SM), Single Bond(SB) and Clearfil SE Bond(SE) with intact dentin, axial wall and pulpal floor are shown in Table 3.

As shown in Table 3, the micro tensile bond strength of all dentin bonding systems were decreased in order of control group, axial wall group, pulpal floor group. In control group, SM and SB showed significantly higher bond strength than SE($p<0.05$). However, in axial wall and pul pal floor groups, there were no significant differ ence between dentin bonding systems.

The micro tensile bond strengths of SM and SB were much higher in control group than that of axial wall and pulpal floor group ($p<0.05$).

For SE, control group showed significantly high er bond strength than axial wall and pulpal floor group, also pulpal floor group showed significantly lower bond strength than control and axial wall group($p<0.05$).

The micro tensile bond strengths according to locations of dentin and to dentin bonding systems are showed in Fig. 3 and 4.

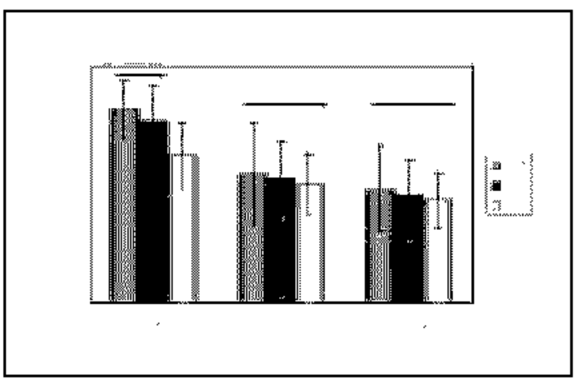

Fig. 3. Micro-tensile bond strengths according to location of dentin

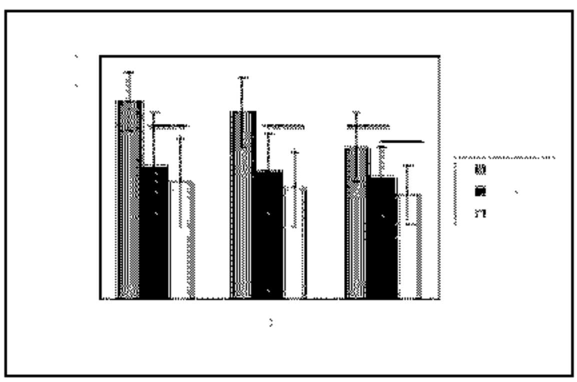

Fig. 4. Micro-tensile bond strengths according to location of dentin

2. SEM Evaluation

In SEM observation, there were several notable regional differences in dentin structure. The tubule density was much lower on the floor of the pulp chamber compared with the intact dentin. For axial wall and pulpal floor group, which were treated with 5% NaOCl, predentin matrix was removed, leaving a smoother, mineralized matrix for bonding. This lead to smoother interfaces. Most of the bond failures occurred at the top of the hybrid layer. For SM, the hybrid layer thick ness ranged from between 4 5 ㎛. Resin tags were

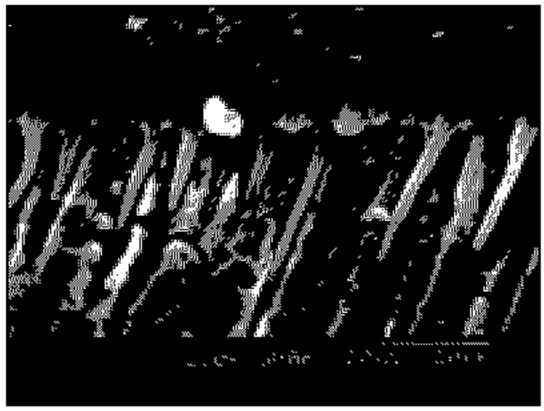

Fig. 5. SEM photograph of the adhesive interface of SM-control group(×2000)

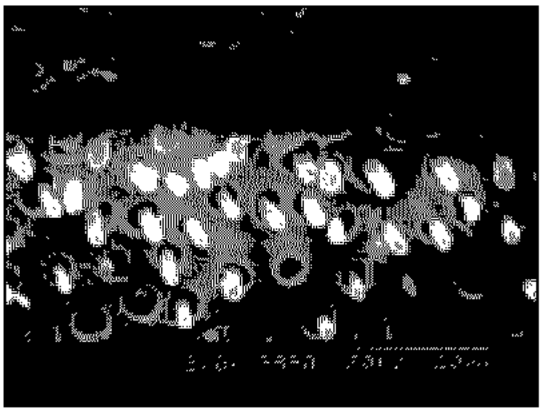

Fig. 6. SEM photograph of the adhesive interface of SM-axial wall group(×2000)

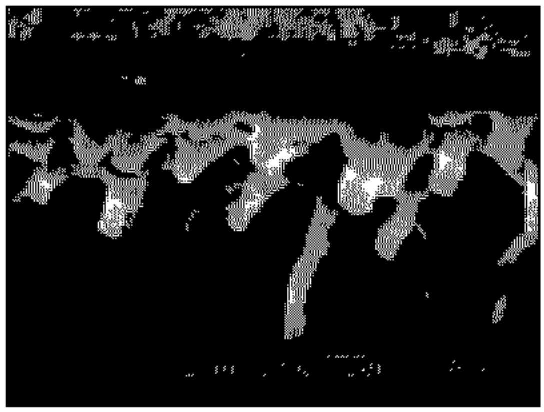

Fig. 7. SEM photograph of the adhesive interface of SM-pulpal floor group(×2000)

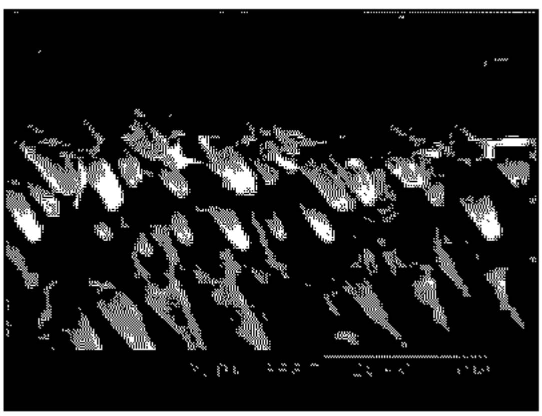

Fig. 8. SEM photograph of the adhesive interface of SB-control group(×2000)

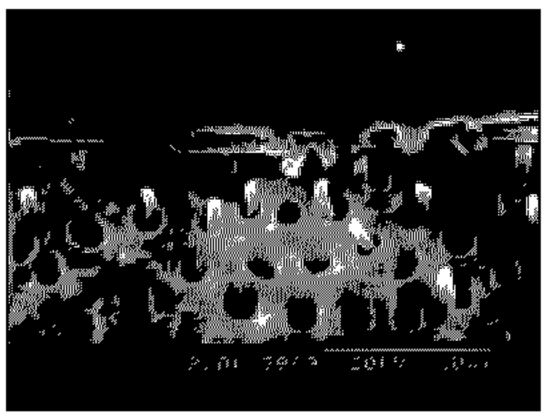

Fig. 9. SEM photograph of the adhesive interface of SB-axial wall group(×2000)

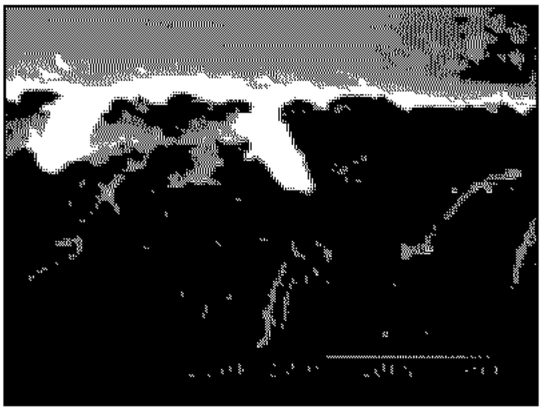

Fig. 10. SEM photograph of the adhesive interface of SB-pulpal floor group(×2000)

Fig. 11. SEM photograph of the adhesive interface of SE-control group(×2000)

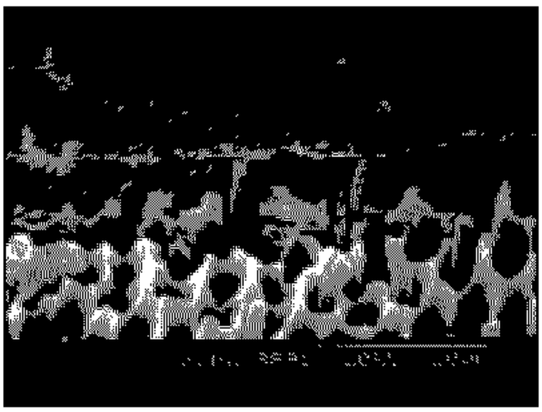

Fig. 12. SEM photograph of the adhesive interface of SE-axial wall group(×2000)

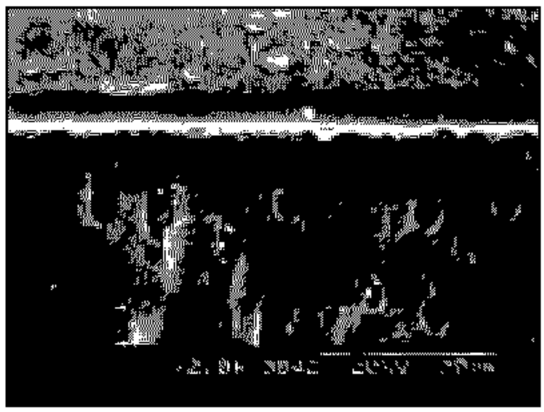

Fig. 13. SEM photograph of the adhesive interface of SE-pulpal floor group(×2000)

clearly observed with the typical funnel shape at the top of the tubules and more than 10 ㎛ in length. SB exhibited similar pattern to SM but the thickness of the hybrid layer was thin (2~3 ㎛) and the length of resin tag was relatively short (5~7㎛). For SE, the thickness of the hybrid layer was measured between 4 5 ㎛. Resin tags were not observed in the pulpal floor dentin. In other regions where resin tags were present, they were thin and pooly formed.

Ⅳ. DISCUSSION

Previous studies reported that endodontically treated teeth are not reinforced by a full coverage crown combined with post and core systems[29 30]. Those studies stated that preservation of the tooth structure is an important factor to prolong the longevity of the tooth. In addition, minimizing the amount of tooth structure loss is reported to be essential for a favorable prognosis[16]. With development of adhesive dentistry, the weakened tooth structure can be reinforced by the use of resin bonding system after endodontic treatment.

However, both the composition and morphology of pulp chamber dentin are different from those of intact dentin. At the surface of the dentin, or the dentino enamel junction, dentinal tubules range between 15,000 and 20,000/mm^2. At the pulpal surface, the number of dentinal tubules increases three fold to 45,000 to 60,000/mm^2 and the tubule diameter increases. Dentin permeability is greatest on thin axial surfaces, particularly mesial surfaces[16]. Also, with aging or in response to noxious stimuli, secondary dentin is laid down at the pulpal end of the primary dentin. This secondary dentin has fewer dentinal tubules, irregular orientation, and lower permeability than primary dentin. Especially, most of pulpal floor composed with calcified secondary dentin.

In addition, pulp chamber dentin might be affected by root canal irrigants and disinfectants during endodontic procedure. NaOCl is one of the most common root canal irrigants used for debridement, lubrication, destruction of microbes and dissolution of organic tissues[2]. Nikaido et al[7] reported that NaOCl treatment adversely affected the bond strengths to dentin. In contrast, others reported that NaOCl treatment improved the adhesion of the bonding system to dentin when using phosphoric acid[31].

In this study, the micro tensile testing method was used for regional bond strength of pulp chamber dentin. Since, each region of pulp chamber dentin is too small to permit conventional bond test and micro tensile testing method has been shown that the bonded interface of small specimens distributes stress better, which can result in more consistent adhesive failures and higher apparent bond strengths. It also allows for more specimens to be generated from the same tooth. One of the disadvantage of the test is that small bond strengths are difficult to measure because specimens can be broken easily during preparation[32].

The dentin bonding systems used in this study represent the conventional bonding system(SM), self priming system(SB) and self etching system(SE). In conventional and self priming system, the etchant removes the smear layer and demineralizes dentin, and the adhesive resin penetrate the etched dentin. However, the acid component of the self etching primer mildly demineralizes dentin so that it does not remove smear plugs completely. At the same time, the primer component modifies the demineralized dentin and the bonding resin infiltrates the primed dentin.

The micro tensile bond strength of pulp chamber dentin(axial wall, pulpal floor) was lower than that of intact dentin(control) in all dentin bonding systems. This result suggests that endodontic treatment which use chemical irrigant such as NaOCl interfere the adhesion of bonding system to dentin. It is thought that residual NaOCl may interfere with polymerization of the bonding resin due to oxygen generation. For SM and SB, micro tensile bond strength was decreased so much in pulp chamber dentin, but SE showed relatively gradual decreation though there were no significant difference between dentin bonding systems in pulp chamber dentin. Several factors could explain this difference between wet bonding system(SM, SB) and self etching system(SE). The self etching primer of SE might not be effective for removing degenerated dentin and residual NaOCl, while the etchant of SM, SB might be strong enough to remove both. The remained smear layer after self etching primer application might prevent oxygen from penetrating through dentinal tubules from the pulp chamber dentin, while the removal of the smear layer by the etchant treatment of SM, SB might allow oxygen penetration. In highly calcified pulpal floor dentin, self etching primer might not etch mineral enough to infiltrate by monomers to form a hybrid layer. Self etching primer could potentially eliminate the risk of overetching dentin. Overetching leaves a layer of demineralized dentin below the hybrid layer, leading to long term weakening of the dentin bond and subsequent leakage[33]. Nikaido et al[7] have shown that bond strength of self priming system was significantly decreased, while self etching system did not affected by NaOCl treatment. Ishizuka et al[34], on the other hand, found that the bond strength of self etching system decreased following NaOCl treatment whereas that of wet bonding system did not change. In this study, although bond strengths of all dentin bonding systems were affected by NaOCl, self etching system was less affected by NaOCl treatment. It is thought that as the self etching primer acts on the dentin surface by modifying the smear layer, and partially dissolved smear plugs may remain within the tubules, low ering the dentinal permeability. Therefore residual chemical irrigants and their products in dentinal tubules are main reason that affect the penetration of resin into the dentin structure and/or the polymerization of the monomer in the demineralized dentin.

In SEM observation, the thickness of hybrid layer or the length of resin tag were not affect the bond strength, supporting previous study that there is no correlation between bond strength and hybrid layer thickness[35]. But smoother bonding interfaces due to use of NaOCl as well as lower dentinal tubule density were thought another reason of lower bond strength in pulp chamber dentin.

Although the bond strength of SE was slightly lower than that of SM, SB in pulp chamber dentin, the intent of use of adhesive resins inside the pulp chamber is to seal the root canal to prevent microleakage of oral microorganisms and their products, then high bond strength are not required, because the correlation between microleakage and bond strength is not high[36]. Rather the use of adhesive system that is simple, easily retreatable, and technique insensitive would be a good choice.

In addition, we tried to reduce the C factor(cavity configuration factor) in order to exclude the effect of C factor that may affect the bond strength[37]. For instance, in control and pulpal floor group we made flat dentin surface, also dentin bonding systems were applied only to the axial wall of pulp chamber in axial wall group. However, when the pulp chamber is restored with resin composite, special care must be taken to reduce C factor which may cause microleakage.

In this study, endodontic procedure prove to have an adverse effect on bond strengths of composite. Wet bonding system was more influenced by chemical irrigant than self etching system even though bond strengths of wet bonding system were slightly higher. Then, for the recovery of bond strength in endodontically treated teeth, the use of anti oxidant such as sodium ascorbate before resin bonding may be considerable.

Ⅴ. CONCLUSION

In restoring endodontically treated teeth, treatment goals must be based upon a multitude of factors that include occlusion, patient's function, tooth position, periodontal status, prosthetic needs, amount of remaining tooth structure, and root morphology. Recent development of adhesive dentistry enables a non vital tooth to be restored with minimal intervention.

This study was designed to evaluate the effect of endodontic treatment on bond strength to pulp chamber dentin. From the results of this study, we can conclude as follows:

1. The micro tensile bond strengths of all dentin bonding systems were decreased in order of control (intact dentin) group, axial wall group, and pulpal floor group.
2. In control group, SM and SB showed significantly higher bond strengths than SE ($p<0.05$).
3. SM and SB showed significantly lower bond strengths in axial wall and pulpal floor group when compared with control group($p<0.05$). But SE showed significantly lower bond strength only in pulpal floor group than control group($p<0.05$).
4. In axial wall and pulpal floor group, there were no significant differences between dentin bonding systems.
5. In SEM observation, the tubule density was much lower on the floor of the pulp chamber compared with the intact dentin. For axial wall and pulpal floor group, which were treated with 5% NaOCl, the smoother bonding interface was shown. There was no correlation between the bond strength and the thickness of hybrid layer.

This study suggests that the procedure of endodontic treatment using chemical irrigants can adversely affect the adhesion to dentin, but self etching system is less affected than wet boding system which needs total etching procedure. Therefore, proper selection of dentin bonding system is important in restoring endodontically treated teeth with resin composite.

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