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Effect of dentin surface removal on the NaOCl-treated dentin

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차아염소산나트륨 용액으로 처리된
상아질 표면의 삭제 효과

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이 논문을 치의학 석사학위신청 논문으로 제출함.

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CONTENTS

Table legends ····· ii

Figure legends ····· iii

Abstract ····· iv

Ⅰ. Introduction ····· 1

Ⅱ. Materials and methods ····· 2

Ⅲ. Results ····· 6

Ⅳ. Discussion ····· 8

Ⅴ. Conclusion ····· 12

References ····· 13

TABLE LEGENDS

Table 1. Materials used in the study ····· 2

Table 2. Mean microtensile bond strength (MPa) to dentin after different surface treatment····· 6

FIGURE LEGENDS

Fig. 1. (a) Microtensile tester (b) Beam attached on microtensile device 4

Fig. 2. Schematic representation of the specimen preparation. 5

Fig. 3. Mean microtensile bond strength (MPa) to dentin after different surface treatment. 7

국 문 초 록

차아염소산나트륨 용액으로 처리된 상아질 표면의 삭제 효과

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본 연구에서는 차아염소산나트륨으로 처리된 상아질 결합강도를 높이기 위해서 항산화제 (10% 아스코르브산)를 사용한 것과 표면 상아질을 삭제한 경우의 효과를 비교하였다. 인간 제 3대구치 20개를 사용하였으며 미세인장강도 시험을 위해 편평한 상아질 면이 얻어지도록 법랑질을 제거하고 무작위로 4가지 군으로 분류하였다. 대조군은 차아염소산나트륨의 영향을 받지 않은 군으로 0.9% 생리식염수로 10분간 처리하였다. 1군은 5.25% 차아염소산나트륨으로 10분간 처리하였다. 2군은 1군과 같은 방법으로 처리한 후 10% 아스코르브산으로 10분간 처리하였다. 3군은 1군과 같은 방법으로 처리한 후 거친 다이아몬드 버를 이용하여 0.1 mm 가량 삭제하였다. 모든 치아의 표면을 건조시킨 후 Clearfil SE bond를 사용하여 접착제를 도포한 후 즉시 Filtek Z250 복합레진을 사용하여 수복하였다. 24시간 보관 후 미세인장 결합강도를 측정하였다. 실험 결과, 차아염소산나트륨만 처리한 1군의 결합강도 (19.44 ± 4.49 MPa) 가 다른군들에 비해 유의적으로 낮게 나타났다 ($p<0.01$). 10% 아스코르브산를 처리한 2군 (31.96 ± 8.48 MPa) 이 다른 군들보다 유의적으로 높은 수준의 결합 강도를 보였다 ($p<0.05$). 반면에 버로 상아질 표층을 삭제한 3군 (26.08 ± 5.25 MPa) 은 대조군과 통계적으로 유의한 차이가 나타나지 않았다. 결론적으로, 5.25%의 차아염소산나트륨 세척은 two-step self-etch adhesive의 상아질 결합 강도를 유의적으로 감소시켰으며 10% 아스코르브산의 사용 또는 표층 상아질 삭제는 감소된 결합강도를 회복시키는 효과가 있었다.

I. Introduction

Effective cleaning and shaping of the root canal, as well as creation of an apical seal is an essential goal for successful endodontic treatment. Recently, more attention has been focused on procedures performed to achieve an effective coronal sealing soon after the completion of root canal therapy. The immediate sealing of endodontically treated teeth using restorative materials is powerful tool in preventing early coronal leakage.[1-3] The importance of the coronal restoration in successful endodontic outcomes is widely accepted and has been supported by Ray and Trope.[4] Among definite restorative materials, dentin adhesives have been advocated for use within the pulp chamber in an attempt to work as a durable barrier against microleakage hampering apical and coronal microleakage.

Sodium hypochlorite (NaOCl) is the most widely recommended irrigant in endodontics because of its ability to dissolve necrotic tissue remnants.[5] According to many articles, NaOCl reduces the bond strength between resin composites and dentin.[6-8] This is thought to be due to remnants and by-products of NaOCl exhibiting negative effect on the polymerization of dental adhesive systems.

To restore the bond strength to normal range, 10% sodium ascorbate or 10% ascorbic acid were introduced to apply on the NaOCl-treated dentin.[6,9] Weston CH et al.[10] recommended treatment with ascorbic acid or sodium ascorbate for 10 min. But it is an impractical recommendation for clinical situation.

In routine endodontic therapy, most clinicians remove a little amount of pulpal wall to obtain clean dentinal surface after canal obturation procedure. However, there was no study whether removing dentin surface after NaOCl-treatment affects on microtensile bond strength or not.

The purpose of this study was to compare the effect of removing superficial dentin surface with that of antioxidant (ascorbic acid) on microtensile bond strength of sodium hypochlorite-treated dentin.

II. Materials and Methods

1. Specimen preparation

Twenty non-carious human third molars were used in this study. The teeth were cleaned of periodontal tissue residue using a periodontal scaler and were stored in distilled water at room temperature. Storage water was changed daily. The occlusal enamel was removed by sectioning the crown perpendicular to the long axis of the tooth using a model trimmer (Se-ki, Seoul, Korea.) under copious water lavage to achieve a flat superficial dentin surface. And dentin surface was wet-polished by abrasive paper (600 grit; Silicon Carbide Water Proof Abrasive Paper Electro Coated, Daesung, Korea) with running tap water for 1 min to obtain smooth dentin surface and rinsed for 1 min.

The teeth were randomly divided into four groups and each group had 5 specimens. The materials used in the study are listed in Table 1.

Table 1. Materials used in the study.

Brand	Material	Composition	Manufacturer
Clearfil™ SE Bond	two-step self-etch adhesive system	**Primer**; MDP, HEMA, hydrophilic dimethacrylate **Bond**: MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, DL-camphorquinone, N,N-diethanol-p-toluidine and silanated colloidal silica	Kuraray, Osaka, Japan
Filtek™ Z250	composite resin	Bis-GMA, BisEMA, UDMA, TEGDMA, Silane treated ceramic	3M ESPE, St. Paul, MN, USA

The dentin surface treatment of each groups are as follows;

Control : dentin surface was irrigated with 0.9% NaCl for 10 min, then rinsed with 10 mL of distilled water.

Group 1 : dentin surface was irrigated with 5.25% NaOCl (Yuhanclorox, Seoul, Korea) for 15 min per tooth, then rinsed with 10 mL of distilled water.

Group 2 : after treatment as in group 2, freshly made 10% ascorbic acid (JUNSEI, Tokyo, Japan) was applied to the dentin surfaces for 10 min, followed by rinsing with 10 mL of distilled water.

Group 3 : after treatment as in group 2, the dentin surface was removed about 0.1 mm with coarse diamond bur (TR 14, MANI, INC., Utsunomiya Tochigi, Japan), then rinsed with 10 mL of distilled water.

After pre-treatment, the specimens were dried with air syringe, and Clearfil SE Bond (Kuraray, Osaka, Japan) as dentin adhesive applied to dentin surfaces following the manufacturer's instructions. Spectrum 800 (Dentsply Caulk, Milford, DE. USA) with output intensity of 400 mW/cm^2 was used for light curing. Resin composites (Filtek Z250; 3M ESPE, St. Paul, MN, USA) build-ups were constructed in three 1.5 mm increments. Each increment was light-cured for 40 s. Then the specimens were stored in distilled water at 25℃ for 24 h

2. Microtensile bond strength test

After being stored in distilled water at 25℃ for 24 h, the specimens were embedded in acrylic blocks using sticky wax (Kerr corporation, Orange, CA, USA). Then they were placed on a low-speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) to produce 1 mm X 1 mm adhesive surface area beams under water cooling. Each beam consisted of composite resin and dentin. The dimension of each beam was measured using digital caliper and the bonded area was calculated for subsequent conversion of microtensile bond strength values

into units of stress (MPa).

The beams were attached with cyanoacrylate adhesive (Zapit; DVA, Corona, CA, USA) to a testing apparatus and tensile load was applied with a microtensile tester (Micro Tensile Tester; Bisco, Schaumburg, IL, USA) at a cross-head speed of 0.5 mm/min (Figure 1).

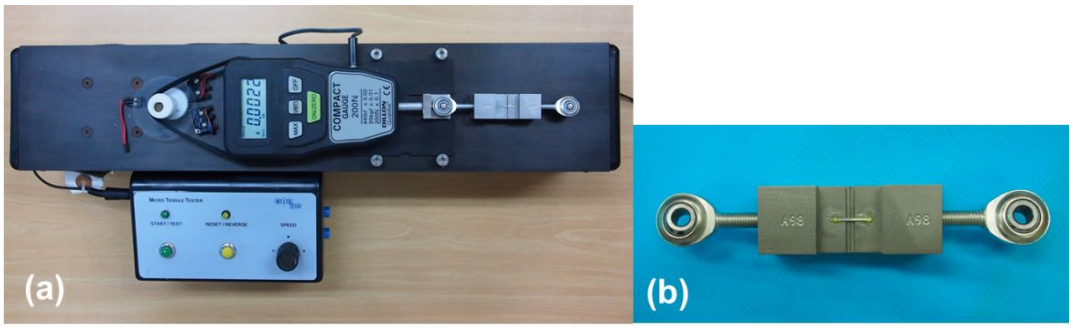

Figure 1. (a) Microtensile tester (b) Beam attached on microtensile device.

3. Statistical analysis

The microtensile bond strength data was analyzed for statistically significant differences by one-way analysis of variance (ANOVA) and Tukey's test using SPSS™ Ver 12.0 (SPSS Inc, Chicago, IL, USA). Statistical significance was defined as $p<0.05$.

The specimen preparation procedure is schematically illustrated in Figure 2.

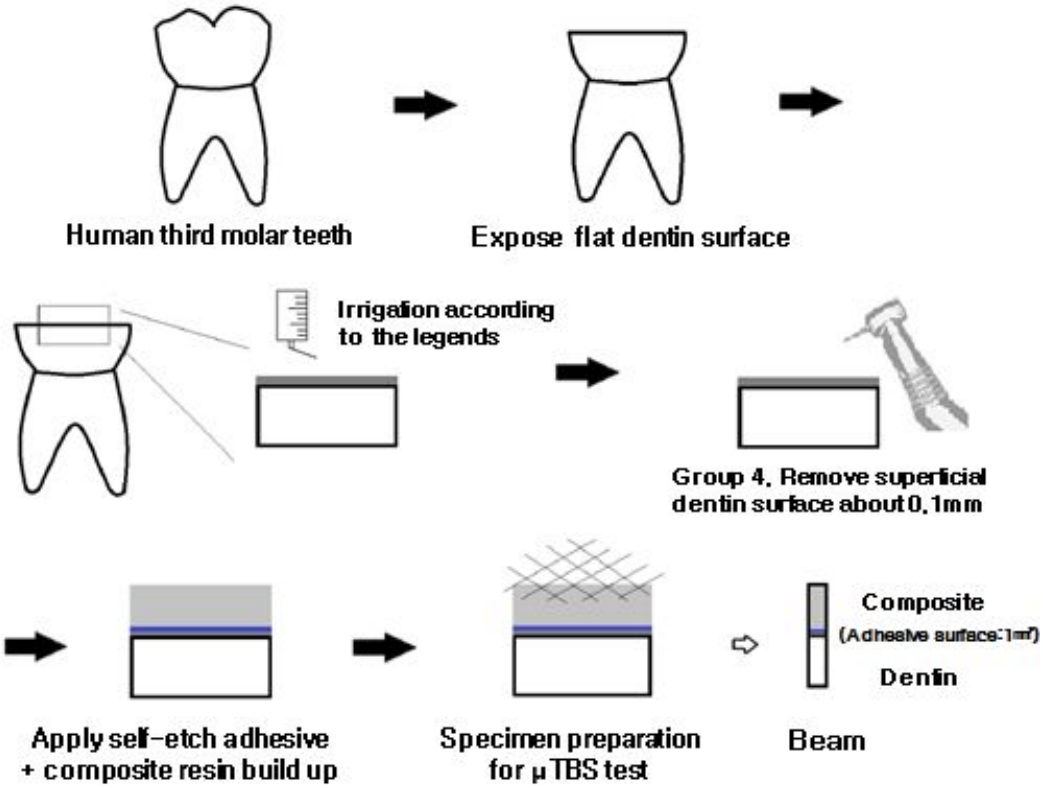

Figure 2. Schematic representation of the specimen preparation.

III. Results

1. Microtensile bond strength

Mean microtensile bond strength values and standard deviation of each group are shown in Table 2 and Figure 3. One-way ANOVA revealed that there were significant differences between surface treatment methods ($p<0.001$). The microtensile bond strength of the NaOCl-treated dentin (group 1) demonstrated significantly lower bond strength than the control group ($p<0.01$). Treatment with 10% ascorbic acid (group 2) demonstrated significantly higher bond strength than other groups ($p<0.05$). There was no significant differences in bond strength between removal of superficial dentin surface (group 3) and control group ($p>0.05$).

Table 2. Mean microtensile bond strength (MPa) to dentin after different surface treatment

Group	Number	Mean	S.D.
Control(NaCl)	30	27.26 [a]	5.74
1(NaOCl)	30	19.44 [b]	4.49
2(Ascorbic acid)	30	31.96 [c]	8.48
3(Dentin remove)	30	26.09 [a]	5.25

Groups identified by different superscript letters are significantly different ($p<0.05$).

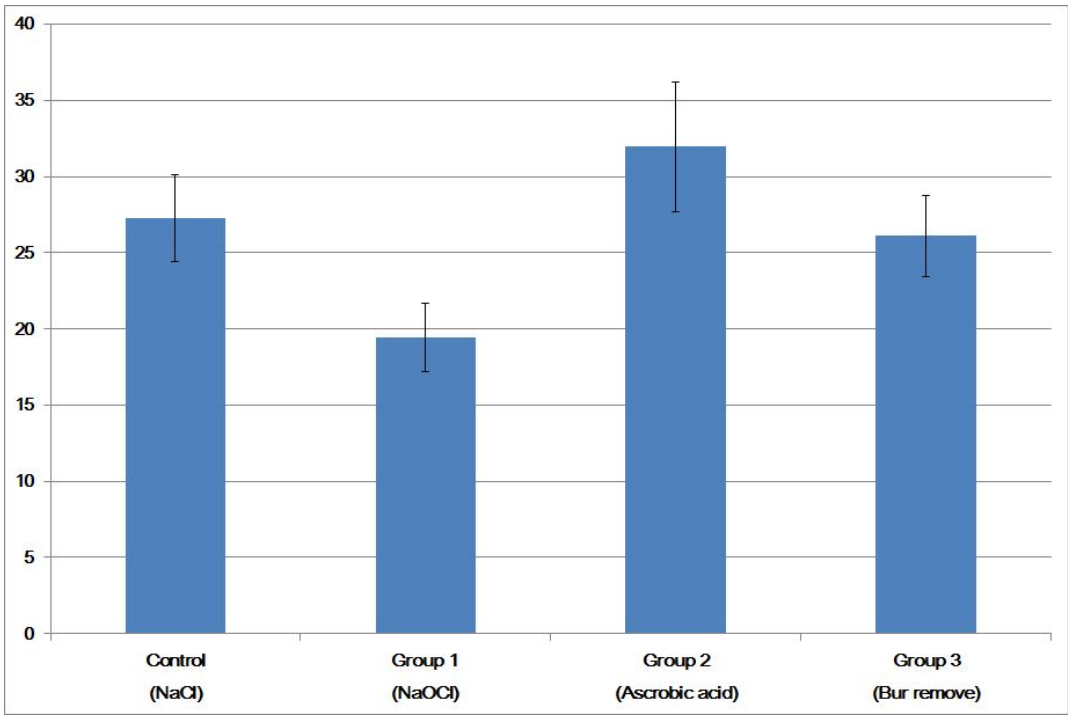

Figure 3. Mean microtensile bond strength (MPa) to dentin after different surface treatment.

IV. Discussion

The aim of this study was to investigate the effect of removal of superficial dentin surface to restore the decreased bond strength caused by NaOCl.

Sodium hypochlorite (NaOCl) has been widely used in root canal treatment to provide gross debridement, disinfection, lubrication, and dissolution of tissues.[11] NaOCl causes alterations in cellular metabolism and phospholipid destruction. It has oxidative actions that cause deactivation of bacterial enzymes and causes lipid and fatty acid degradation

Several studies have reported that dentin that has been exposed to NaOCl exhibits significantly lower bond strengths than untreated dentin.[6-9,12] One study reported bond strengths as low as 8.5 MPa.[7] Increased microleakage was also reported.[13] The results of this study showed that NaOCl irrigation generally decrease the bond strength of self-etch adhesive to dentin. This result was in agreement with previous studies.[7,14,15] NaOCl removes any exposed organic matrix, mainly the collagen or soft tissue from the dentin and leaves a mineralized surface less receptive to bonding with resin composites.[10] It is likely that NaOCl acts to oxidize a component in the dentinal matrix that interferes with free radical propagation at the resin - dentin interface, leading to lower bond strength. In addition, when NaOCl breaks down, oxygen will be made and it causes strong inhibition of the interfacial polymerization of adhesive materials.[16] According to Lai et al., in NaOCl-treated dentin, there might be some reactive residual free-radicals, which might compete with the propagating vinyl free radicals generated during light activation of the adhesive system, resulting in premature chain termination and incomplete polymerization.[9] Furthermore, reductions in calcium and phosphorus levels[17] and in mechanical properties of dentin, such as elastic modulus, flexural strength, and microhardness[18], were reported after irrigation of root canals with 5% sodium hypochlorite, which can also contribute to a decrease in the micromechanical interaction between adhesive resins and NaOCl-treated dentin.

Dentin bonding is based on the formation of a resin-infiltrated layer in the conditioned intertubular and peritubular dentin. After polymerization, resin monomers may form a micro-mechanical bond with the primed dentin, so-called hybrid layer.[19] Thus, to achieve satisfactory dentin adhesion, open tubules and exposed collagen-rich meshwork should be infiltrated by resin monomers entirely and homogeneously. However, NaOCl damaged the organic matrix, mainly the collagen. Therefore, weak hybrid layer was formed and microtensile bond strength was significantly decreased.

Morris et al.[6] showed that application of 10% ascorbic acid or 10% sodium ascorbate, both of which are antioxidant/reducing agents, reversed the effects of sodium hypochlorite and restored bond strengths to normal levels. Lai et al. and Yiu et al. also reported similar results.[9,13] 10% ascorbic acid (pH=4) was as effective as 10% sodium ascorbate (pH=7) to reverse the lowered bond strength of NaOCl treated dentin.[6] In this study, application of NaOCl-treated dentin with 10% ascorbic acid for 10 min (group 2) demonstrated significantly higher bond strength than other groups ($p<0.05$). By treating dentin with 10% ascorbic acid, the micro-environment of the dentin is converted from an oxidized substrate to a reduced substrate. It seems that this redox potential recovery can restore the bond strength to normal range.

The influence of endodontic irrigants on adhesion was tested with two step self-etch adhesive system. Restoration of endodontically treated teeth with self-etch adhesives and composites may offer some advantages over the use of conventional total-etch dental adhesives. Self-etch adhesives have weak acids in their primer composition, resulting less change in the dentinal wall structure than the strong acids of total-etch systems. In addition, primer application is performed without air-drying, collapse of collagen fibrils is avoided, reducing technique-sensitivity.[20] Self-etching systems (Clearfil SE Bond, Prompt L-pop) were more successful than one bottle (Prime&Bond NT) and multistep (Scotchbond MultiPurpose Plus) in bonding to pulp chamber dentinal walls.[14] So, self-etch adhesive (Clearfil SE Bond) was used as a bonding agent in this study.

Numerous studies have described the impact of many antioxidant to reverse the bond strength of the NaOCl treated dentin.[6,10,21] But in clinical situation, application of 10% ascorbic acid to pulp chamber for 10 min is time-consuming. Therefore, this study was designed to find a simple way to restore the decreased bond strength caused by NaOCl. A study about effect of sodium hypochlorite on human root dentin, reported that exposure to 5% NaOCl rendered the superficial 80-100 of the intertubular dentin permeable to basic fuchsin.[22] The loss of tooth structure decreases tooth stiffness by only 5%.[23] In fact, the largest reduction in tooth stiffness results from additional preparation, especially the loss of marginal ridges. Therefore, additional removal of superficial dentin layer about 0.1 mm without destruction of marginal ridge before bonding procedure will exposure the fresh dentin and restore the bond strength and reduce the coronal leakage.

There are some limitations in this study. The microtensile bond test allows bond testing of small areas promoting a better stress distribution throughout the specimen and induces failures of materials that are closer to their true ultimate strengths and are mostly adhesive failures.[24] However, the correlation between bond strength and microleakage is not well established, and bond strength data alone are not sufficient to evaluate the sealing ability of resins. According to this, the rationale involving microtensile bond testing of endodontic surfaces is that a better adhesion of restorative materials to dentin increases the opportunity for good marginal sealing, longer life of the restoration, and withstanding of mechanical stresses.

Further research should focus on the optimal amount of dentin removal after exposure to NaOCl. Microleakage studies should also evaluate the sealing properties of resin over time. The structure of pulp chamber wall dentin differs from that of the other dentinal regions of teeth. Additional experiment should be done with pulp chamber wall dentin because flat occlusal dentin was used in this study.

Because NaOCl is likely to remain the primary irrigant used in endodontics for the near future, and because adhesive resin materials are used routinely in

restoring endodontically treated teeth, this issue will have to be addressed. Future adhesive resin products for endodontic applications may contain a reducing agent to reverse the effects of the sodium hypochlorite. A non-oxidizing irrigant would also solve this problem.

V. Conclusions

Irrigation with NaOCl significantly reduced the microtensile bond strength. Antioxidant application and superficial dentin removal significantly restored the decreased bond strength of NaOCl-treated dentin.

Within the limitations of this study, it is concluded that removal of superficial dentin surface could be used as a simple way of restoring the bond strength to NaOCl-treated dentin.

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