

Effects of Application Methods of
Dentin Bonding Agents
& Eugenol Containing
Temporary Cement
on Micro-Tensile Bond Strength

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감사의 글

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기도로 빛이 되어주시고 사랑으로 길이 되어주신 부모님, 며느리 역할도 잘 못하는 저를 딸이 되게 해주시고 언제나 든든한 힘이 되어주신 시부모님께도 진심으로 감사드립니다.

마지막으로 하나됨의 울타리 안에서 저의 반 쪽이 되어준 남편에게 이 글이 나눔을 함께 축하드립니다.

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조 유 나 드림

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Abstract

Effects of Application Methods of Dentin Bonding Agents & Eugenol Containing Temporary Cement on Micro Tensile Bond Strength

Due to the recent elevated of interest in esthetic restorations, the clinical use of metal free restorations such as all-ceramic is increased. This trend has led to a frequent use of composite resin cements, which are esthetic, and have good mechanical properties.

When resin cements are used with indirect restoration, some problems may lie especially in eugenol containing temporary cements.

The purpose of this study was to determine the effect of application methods of dentin bonding agents(DBAs) on bond strength between composite resin and dentin. Micro-tensile bond strength(μ -TBS) was measured.

One-Step (Bisco. Inc. Schaumburg, IL, USA) for DBAs and Temp-Bond® (Kerr corporation. Orange CA, USA) for temporary cement were used. Surface treatment and application of DBAs were fresh dentin as it is(Control), DBAs following Temp-Bond(Group 1), pre-treatment of DBA and then Temp-Bond(Group 2), dual application without temporary cement(Group 3), dual application of DBA with intermediate use of temporary cement(Group 4), dual

application of DBA and amphiphilic copolymer(Group 5). Temporary cements were applied and then specimens were stored for 7 days in distilled water. The dentin surfaces of all groups were cleaned with pumice (non fluoride) and chlorhexidine.

Control and Group 3 had higher μ -TBS than other groups, and Group 2 had lowest values. By the results, method of Group 2 not recommended. Within the limits of this experiments, dual application of dentin bonding agent could not improve the bonding strength between composite resin and dentin. Function of amphiphilic copolymer cannot be ascertained because of insufficient storage time. Eugenol containing temporary cements lowered the μ -TBS of composite resin and dentin. In this experiment, accurate cause can not be distinguished.

Key words : composite resin, dentin, one-bottle dentin bonding agents, eugenol containing temporary cement, micro tensile bond strength, dual application, amphiphilic copolymer.

Effects of Application Methods of
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I. INTRODUCTION

Due to the recent escalated interest in esthetic restorations, the clinical use of metal free restorations has increased. Adhesive luting

procedures are usually required with indirect metal free restoration, which has led to a frequent use of composite resin cements along with dentin bonding agents. Dentin bonding agents is also used for the treatment or prevention of hypersensitivity. Sealing or impregnating the dentinal tubules with dentin bonding materials has been suggested, especially in the light of the hydrodynamic theory (Brannstrom and Nordental, 1977).

The dentin bonding materials are with good mechanical and adhesive strength, however preparing the dentin surface still has many problems. Residual monomers of dentin bonding agents can be cytotoxic to the pulp (Murray et al., 2001), causing post-operative hypersensitivity (Demarco et al., 2001). As multiple applications of dentin bonding agents increase the film thickness, final marginal integrity may be interfered and the bonding strength is decreased (Swift et al., 1997).

Whereas dentin bonding agents are applied to fresh dentin in direct composite resin filling, indirect restorations usually require a provisional restoration that is cemented to the abutment teeth with temporary cements. Eugenol containing temporary cement are usually used in cementation of provisional restoration because eugenol containing temporary cement are cheap, easily removed, have good sealing properties, and have sedative effects on hypersensitive teeth. However, eugenol containing materials have adverse effects on resin bonding restorative materials. Eugenol changes the wettability and reactivity of dentin (Baier, 1992) and

remnants of eugenol on the surface may interfere with the setting of resin and resulted in impair polymerization (Terata, 1993). This leads to surface roughness, reduced microhardness, and reduced color stability of composite resin (Lingard et al., 1981). Stephan et al(2001) said that use of eugenol containing temporary cement are not recommended prior to the resin cement.

Holderegger et al. (1997), Hansen and Asmussen (1987) and Al-Wazzan et al. (1997) evaluated the effect of eugenol-containing temporary cements on composite resin and dentin. Their results indicated that pretreatment of the dentin with eugenol-based temporary cement adversely affected the bonding strength of composite resin and dentin, and increased marginal contraction gap.

However, the results of a study by Leirskar (2000) showed that eugenol had no negative effects on bonding strength. According to Schwartz (1992), Ganss and Jung (1998), shear bonding strength was not affected by temporary cement, if the dentin had been cleaned with pumice and etched with 37% phosphoric acid.

Nevertheless, the oily and greasy additives in provisional cements are not effectively removed by simply cleaning the dentin surface with pumice (Bertschinger et al., 1996). This characteristic causes remained remnant of temporary cements.

Many clinicians tried to resolve this problem by using different application methods of dentin bonding agents. Bertschinger (1996) and Holderegger (1997) argued that dual application of DBAs seems to have a beneficial effect on bond strength to dentin surface

in the case of use of eugenol containing temporary cement by necessity. Previous application of DBAs before cemented with temporary cement can prevent penetration of eugenol containing temporary cement. And many investigators tried to utilize amphiphilic copolymers to improve bonding strength of composite resin. Amphiphilic copolymer with dual application of DBAs may have beneficial effect on bond strength through that amphiphilic copolymer activates pretreated DBAs and supports bonding of two DBAs layer. However no preceding study that comparative effects of dual application of DBAs, amphiphilic copolymer and previous treatment of DBAs in the case of one-bottle DBAs used exists

The purpose of this study was to compare the micro-tensile bond strength of composite resin to dentin which it follows in application methods of one-bottle DBAs. The hypothesis to be tested was that compared to conventional application method, tensile bond strength values of dual application of one-bottle DBAs would be lower for adhesive luting purpose on dentin, and amphiphilic copolymers with dual application of DBAs would have higher bonding strength values than conventional methods.

II. MATERIALS & METHODS

One-Step® for DBAs was selected for the experiment. Composite activator® was amphiphilic copolymers. Bisfil core resin® was used for composite resin based on its favorable manipulation and its same ingredient, bisphenol diglycidyl methacrylate with resin cement, and Temp-Bond® for temporary cements.

Table I. Used materials for dentin surface treatment

Materials	Manufacturers	Essential Ingredients (batch No.)
Uni-Etch	Bisco. Inc. Schaumburg, IL, USA	32% Phosphoric Acid, Benzalkonium Chloride (0300001210)
ONE-STEP	Bisco. Inc. Schaumburg, IL, USA	BPDM, HEMA, Aceton (0300001011)
Composite activator	Bisco. Inc. Schaumburg, IL, USA	Methyl Methacrylate, Isobutyl Methacrylate (0200005228)
Temp-Bond®	Kerr corporation Orange CA, USA	4-Allyl-2-Methoxy Phenol (24617C)

Fabrication of Test Specimens

Six extracted, caries free, and unrestored human molars were selected for this study.

The teeth were mechanically cleaned with hand scaling instruments and stored in distilled water at room temperature before specimen preparations and during the entire experimental period. A flat dentin surface perpendicular to the long axis of tooth was prepared using a slow-speed diamond grinding wheel (Wehmer corporation, Addison IL 60101, USA) with water coolant. To create a smear layer, the dentin surface was abraded with wet silicon carbide paper (600 grit, 60 sec). The root surface was flattened with a grinding wheel and was attached to a mounting block with Kerr Sticky Wax (Kerr corporation, Orange CA, USA)

Specimen Preparation and Material Application

A random simple blinded allocation of the trimmed teeth into five groups were made. Each specimen was treated as described in Fig. 1 in sequence from left to right. In control group, composite resin was directly built up to fresh dentin applied DBAs without temporary cement and storage. This was to reproduce a clinical situation in which composite resin was directly applied without any provisional restoration. Group 1 was to ordinary and conventional clinical steps in which DBAs and composite resin applied after removal of temporary cement. In Group 2, DBAs was once only before temporary cement. In this instance, DBAs used for

prevention of penetration of temporary cements and of hypersensitivity. Dual application of dentin bonding agents was used in Group 3, 4 and 5. In Group 3, temporary cements were not applied and stored for 7 days following to first DBAs. Composite activator was amphiphilic copolymers and it was utilized for activate DBAs in Group 5.

Temp-Bond of test groups was sealed with Soft Putty (Aquasil, Densply) to prevent the temporary cement from washing out. After teeth of test groups were stored, temporary cements were mechanically removed with hand instruments and the dentin surfaces of all groups were cleaned by scrubbing with pumice (non fluoride) and chlorhexidine.

Acid etching with 32% phosphoric acid etchant (UNI-ETCH) and treatment with ONE-STEP was done according to the manufacturer's recommendation. The core resin (BISFIL CORE) was built up and cured with VIP (Bisco. Inc. Schaumburg, IL, USA) as manufacturer's direction, to approximate 4mm thickness. Teeth were soaked with distilled water for 24 hours at room temperature to optimize curing and bonding. Teeth were sectioned into 0.9mm thick slabs from the occlusal to gingival direction using water irrigated diamond saws (R&B Low speed precision diamond saw, Topmet, Daejeon, Korea) at 200 rpm with 75 g of force. The slab sections were mounted onto another plastic block with Kerr Sticky Wax(Kerr corporation, Orange, CA) and were sectioned into

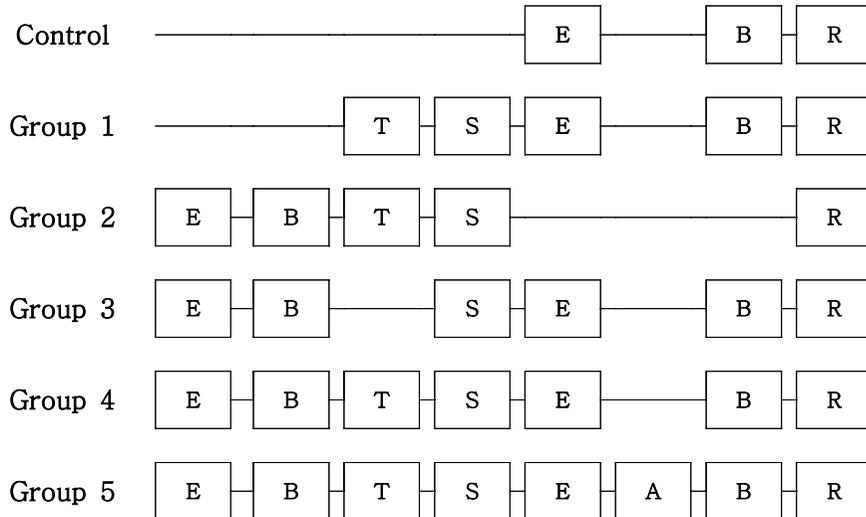


Fig. 1. Flow chart of materials application methods on dentin surface

E(acid etching with 32% phosphoric acid etchant). B(dentin bonding agent(One-Step)). T(Temp-Bond®). S(storage in distilled water for seven days at room temperature). A(Composite activator). R(composite resin build up).

0.9mm*0.9mm bars. Dimension of sectioned specimens were measured using Digimatic Caliper (Mitutoyo. Corp. Japan)

Method of Testing Micro Tensile Bond Strength

Specimens were then fixed to jaws with Zapit (DVA Inc, USA) and the micro tensile bond strength(μ -TBS) was evaluated with Micro Tensile Tester (Bisco. Inc. Schaumburg, IL, USA) at a cross-head speed of 0.5mm/min.

Examination of fractured surface

The nature of failure was noted by visual examination under a stereomicroscope. Representative samples which had average μ -TBS value selected per each group, and were observed under an SEM (Scanning electron micrographs).

Statistic Analysis

Values of μ -TBS for the control group and four test groups were statistically analyzed using One-way ANOVA with LDS method at 95% confidence level for comparing each groups.



Fig. 2. Micro tensile tester used in experiment.

II. RESULTS

The micro tensile bond strength(μ -TBS) of all groups tested are presented in Table II. and Fig. 3. Fig. 3 shows the bar graphs over all groups and significant differences between groups could be observed.

The control group and Group 3 obtained higher μ -TBS values than Group 1, 2, 4, and 5($p < 0.05$). Group 2 had lowest μ -TBS values. μ -TBS of Group 1 and Group 2 were significantly different($p = 0.023$) and μ -TBS of Group 2 and Group 5 were also significantly different($p = 0.033$) each other. μ -TBS of the other groups had not statistical differences one another. The control group obtained the highest μ -TBS values in this study, whereas Group 2 had the lowest.

When visually inspected, four samples fractured cohesively in dentin (one of Control, one of Group 1, two of Group 4), one samples in resin (Control), and the remaining samples at adhesive layer. Cohesive fractures in resin were owing to defects within the built up material, and fractures in dentin were due to an insufficient dentin thickness with large pulp chamber.

The remnants of residual temporary cement were seen in SEM of Group 1, 2, 4 and 5.

Table II. Number of specimens in each group, mean tensile bond strength value(MPa), and standard deviation for the different experimental groups

	n	Mean(MPa)	Std. Deviation
Control	18	30.1	7.2
Group 1	17	23.2	9.0
Group 2	21	16.9	7.3
Group 3	19	29.4	11.9
Group 4	17	20.3	12.0
Group 5	25	22.2	6.3

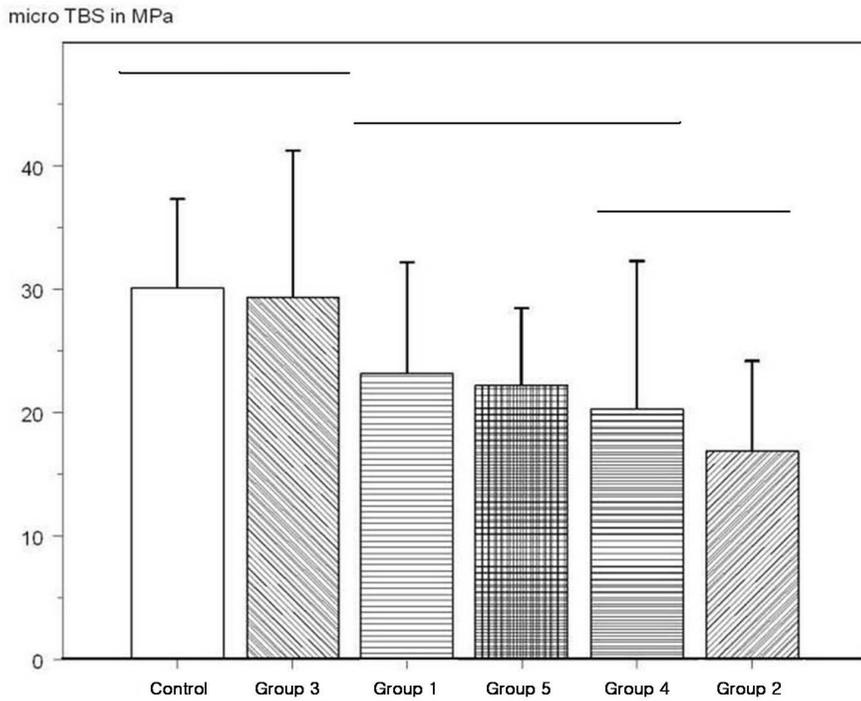


Fig. 3 Bar graphs of μ -TBS. Lines present that statistical difference does not exist (results of LSD method at significance level 0.05)

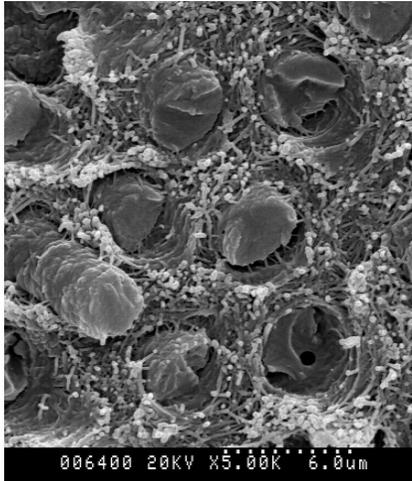


Fig.4. Residual temporary cements present in dentin surface of Group 2

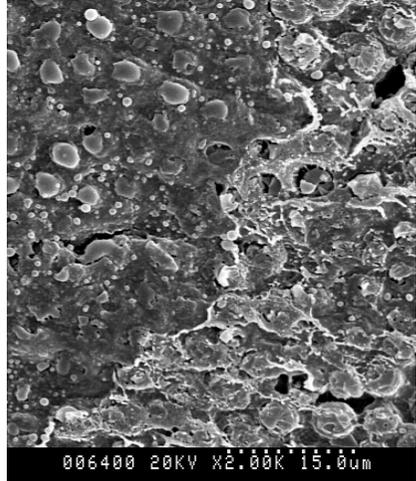


Fig.5. Residual temporary cements present and architecture of resin tag was incomplete in dentin surface of Group 4

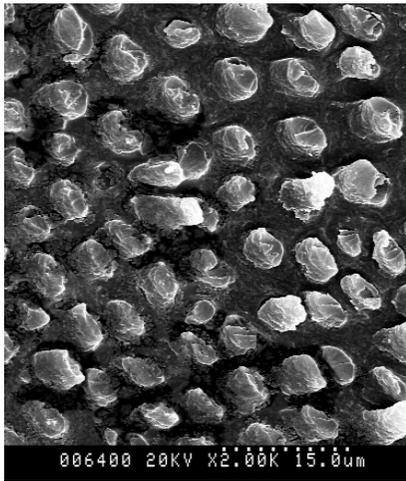


Fig.6. Residual temporary cements attached to resin tag of resin surface were seen in Group 5

IV. DISCUSSION

The result of present study shows that conventional application method and dual application method did not have statistical difference. According to Bertschinger (1996) and Holderegger (1997), dual application of dentin bonding agent can reduce the effect of eugenol. Bertschinger (1996) argued that dual application of two or three bottle DBAs with intermediate use of Temp-Bond seems to have a beneficial effect on their bond strength to dentin surface. Holderegger (1997) compared one-bottle DBAs with two or three-bottle DBAs and concluded that dual application of one-bottle DBAs had no beneficial effects on shear bonding strength of composite resin and dentin, while two or three bottle DBAs did.

The result of this study was same some result as that of Holderegger. On the contrary, dual application method resulted in lower μ -TBS than conventional method. It can be said that dual application of one-bottle DBAs has no beneficial effect on the bonding strength of composite resin. Holderegger explained that one-bottle bonding resin may result in a more pronounced oxygen inhibition, and this lead to lower bond strength. However many investigators like Li (1997) asserts unreacted double bonds of the oxygen inhibition layer function as a bonding medium between two increments of dental composites and improve bonding strength of resin. Oxygen inhibition layer formed by first applied DBAs did not

interrupt bond of resin. Dual application method resulted in lower bonding strength because either the failure of adhesion between resin bonding agents or the interruption of polymerization of composite resin due to remained temporary cements.

Resin tags formed by first applied DBAs employed as interrupt penetration of temporary cements and prevention of post operative hypersensitivity were not removed by scrubbing with pumice and acid etching. Instead, these resin tags did not combine with DBAs applied afterward and deteriorated bonding strength. The previous application of DBAs may adversely affect the bonding strength of resin in this experiment. However, dual application of DBAs without intermediate provisional cement obtained similar high bonding strength with Control group and had statistically higher bond strength than conventional method with eugenol containing temporary cement. By this results, it can be said that dual application method of one-bottle DBAs do not lower the dentin bonding strength and intermediate use of provisional cement lowers the bonding strength.

Dentin bonding agents can be used to prevent post-operative hypersensitivity. In the methods of Group 2, 4 and 5, first applied DBAs can function in such a way. Even though bond strength was relatively low in dual application methods than conventional method, the results of this study shows that conventional application method and dual application methods had no statistical difference. Therefore in the case that need prevention post-operative hypersensitivity,

dual application methods can be used without great disturbance bonding strength of composite resin cement.

Webster et al. (1983) have announced a new method of living polymerization termed "group transfer polymerization" and group transfer polymerization by amphiphilic copolymer is available to improve bonding strength of composite resin. Composite activator was an acrylic based amphiphilic copolymer which hydrolyzed polymers and initiated polymerization. The amphiphilic copolymers are used as detergent, compatibilizer, and surface modifier(Kwon, S. K. et al. 1992). μ -TBS values of the group which used amphiphilic copolymer did not have statistical difference with those not used, although the group of amphiphilic copolymer had higher mean μ -TBS value. This can be explained by insufficient inactivation of first applied DBAs. Group 3 with storage time and Control group without storage time had similar high bond strength and had no statistical difference. It can be said that storage time of 7 days are not sufficient for aging of DBAs and for lowering bond strength. The function of amphiphilic copolymer was the activation aged resin. However, first applied DBAs of Group 5 did not inactivated after storage of seven days because of insufficient aging time. Increased bonding strength of composite resin by function of amphiphilic copolymer cannot be ascertained.

From the results, Group 2 showed the lowest mean μ -TBS, 16.9 MPa which is lower than polymerization shrinkage force of composite luting resins (17 MPa)(Davidson et al., 1984). The result

shows relatively low bonding strength in comparison with other groups. The first applied DBAs did not function as adhesives of composite resin. It can be speculated that after removal of temporary cement, acid-etching is not accomplished. This is resulted in incomplete removal of eugenol containing cement and in more remaining eugenol remnant than other test groups. This is supported by microscopically detection of remnant of eugenol containing cement (Fig. 4). Multiple application of DBAs can interfere marginal integration and formation of uniform DBAs layer can be difficult because of complicated morphology of prepared teeth and bulky film thickness can be form especially in internal finish line of prepared teeth. If final impression can be taken after application of DBAs, interference of marginal integration owing to DBAs can be prevent. However, the result shows that Group 2 had lower dentin bond strength than conventional method. This application method of one-bottle DBAs is not favorable to obtaining sufficient dentin bonding strength.

Asmussen (Hansen and Asmussen. 1987) said that eugenol containing temporary cements have adverse effect on dentin bond strength. 12 years after releasing (1987), Asmussen (Peutzfeldt and Asmussen. 1999) reversed himself and stated that eugenol did not influence the efficacy of dentin bonding agents in micro-leakage and shear bond strength thanks to the development of DBAs. Ganss et al. (1998) and Schwartz et al. (1992) also came to the same conclusion and stated that phosphoric acid etching of enamel has

been found to remove any residual cement and increase wettability.

However, Hume (1984) stated that eugenol reached concentrations of 10^{-2} M or more in the dentin just beneath ZOE, 10^{-4} M or less adjacent to the pulp space, and dentin thickness penetrated by eugenol is in the range 1-9mm. Thus, complete removal of eugenol penetrated into the dentinal tubules by cleaning or using etching systems was difficult. Woody and Davis (1992) saw dentin surface with cement removed with hand instrument and pumice under SEM. They concluded that microscopically detectable cement remained following removal with pumice-water slurry. In this experiment, non-fluoride pumice and 32% phosphoric acid were used for removing eugenol containing temporary cements. However eugenol containing temporary cements cannot be removed completely, and it is conformed by that remnants of residual temporary cement are seen in SEM of Group 1, 2, 4 and 5. (Fig. 4, 5 and 6)

In this study, the μ -TBS values of the control group was higher than those of the test groups in which eugenol containing temporary cement was applied. Difference on the μ -TBS values between the control group and the four test groups may have resulted from the existence of storage period or adverse effect of eugenol containing temporary cement.

Teeth of test groups were stored for 7 days, whereas tooth of the control group had built up immediately on fresh dentin with surface treatment without delay. Asmussen (1987) removed the temporary cement 3 hours after application to reduce the interference of

storage, and Holderegger after 24 hours (1997). However, temporary cement is usually used provisionally for more than one week. According to Hume (1984), penetration of eugenol continues for more than one week. It is clinically relevant to design an experiment with a storage period of more than one week. Group 3 also had storage time. Control and Group 3 had no statistical difference in μ -TBS, and Group 5 was also statistically different with other four test groups. Therefore lower bond strengths observed in Group 1, 2, 4 and 5 are not resulted from storage time.

Lower bond strength of eugenol containing temporary cement may be explained by well-known adverse effect of eugenol on composite resin. However, Woody and Davis (1984) concluded in their study that negative effects of eugenol containing temporary cements on resin cement were caused not by eugenol but by the presence of residual cement. In this experiment, lower bond strength of groups which used eugenol containing temporary cement is because either adverse effect of eugenol or remnants of temporary cement. Accurate cause can not be distinguished in this study. It is necessary to continue the experiments with non-eugenol containing temporary cement to investigate the cause of this result. `

V. CONCLUSION

Esthetic metal free restorations usually require adhesive luting procedures with dentin bonding agents(DBAs) and mainly composite resin cements. Unlike direct filling treatment, indirect restoration requires provisional restoration with temporary cement for at least a few days. Previous studies reported conflicting results about the influence of temporization on the bonding strength of adhesive luting cements. To ensure predictable bonding strength, various application methods have been suggested.

Dentin adhesive materials and technology have been advanced significantly last decade. Although "one-bottle" dentin bonding system have been popular, few reports compared the effect of various application methods with "one-bottle" DBAs for adhesive luting procedures.

The compared application methods of DBAs were as follows: conventional application method, application of DBAs once before temporization, dual application of DBAs before and after temporization, dual application coupled with amphiphilic copolymer only after temporization.

Micro-tensile bond strengths of composite resin to dentin were measured after one week temporization period for all experimental groups except control group.

Within the limitations of this study, following conclusion are drawn:

1. Utilization of eugenol containing temporary cements lowered

-TBS of dentin and composite resin.

2. Although dual application method of one-bottle DBAs had no beneficial effect on μ -TBS of dentin and composite resin, dual application method can be used because μ -TBS of this method was not lower than that of conventional application method.
4. Function of amphiphilic copolymer cannot be ascertained.

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국문 초록

상아질 접착제의 적용방법과 유지놀 함유 임시 합착제에 따른 미세인장강도

전부도재관이나 고식적 전장관등 고정성 보철치료 시 유지력의 증가를 위해서, 또는 최근의 심미 보철에 대한 관심의 증가로 전부 도재관등 심미 보철물의 접착을 위해 사용되는 composite resin cement의 사용이 늘어나고 있는 추세이다.

유지놀이 함유된 치과용 임시 접착제는 물성이 좋고 치수진정작용을 가지는 등 여러 장점으로 임상에서 가장 많이 사용되는 임시 접착제이나, Composite resin cement과 같이 사용할 시에는 composite의 중합을 방해하는 문제점을 가지고 있다.

유지놀 함유 임시 접착제를 불소를 함유하지 않은 퍼미스로 제거하고 산부식 처리 후에는 유지놀에 의한 효과를 줄일 수 있다는 연구들이 있으나 임시 접착제가 완전히 제거되는 것은 힘이 들기 때문에, 유지놀의 composite resin 중합 방해 작용을 방지하게 위해 유지놀이 함유되지 않은 임시접착제를 사용하거나, 상아질 접착제의 도포법을 다양화하는 방법이 그 동안의 많은 연구를 통하여 소개되어졌다.

이 연구는 상아질과 composite resin cement의 결합시 상아질 결합제의 적용법을 달리 하였을 때, 결합력에 미치는 영향을 비교하고자 한다. 이를 위해 micro tensile tester 를 이용하여 치질과 composite resin 사이의 인장강도를 측정하였다.

상아질 접착제로는 one-bottle dentin bonding agents 인 One-Step

(Bisco. Inc. Schaumburg, IL, USA)이 사용되었다. 상아질 접착제의 도포 방법은 대조군에는 임시접착제를 사용하지 않고 상아질의 처리 직후 상아질 접착제와 composite resin을 적용하였다. 그러나 일반적 보철 치료에서는 임시 보철물을 필요로 하고, 임시 보철물은 임시 접착제를 사용하여 접착하고 1주일 이상의 사용기간을 가지게 된다. 따라서 실험군은 임시접착제를 사용하는 1주일 동안의 저장 기간을 설계하였다. 1군은 Temp-Bond® 도포 후 1주간의 저장 후 이를 제거하고 상아질 접착제 도포 후 composite resin을 축성하였고, 2군은 상아질 접착제를 도포하고 Temp-Bond®를 도포 한 후 저장 기간 후에 composite resin을 축성하였다. 3군은 임시 접착제 사용 없이 저장 기간 전 후로 상아질 접착제를 도포하였으며, 4군은 Temp-Bond® 전, 후로 상아질 접착제를 도포하는 dual application 방법을 사용하였고, 5군은 4군과 과정은 같으나 amphiphilic copolymer를 두 번째 상아질 접착제 전에 사용하여 두 상아질 접착제 간의 결합력을 증진하고자 하였다. 모든 군의 상아질 면은 임시 접착제 제거를 위해 불소를 함유하지 않은 pumice와 chlorehexidine으로 닦아내었다.

결과는 대조군과 3군이 가장 높은 인장강도를 보였으며, 2군은 가장 낮은 인장 강도를 가졌다. 2군에서 사용된 상아질 처리방법은 1군에서 사용된 통상적인 도포방법에 비해 매우 낮은 결합강도를 보였으므로 임상적으로 사용하기에 추천될 만 하지 않으며, 4군에서 사용된 one-bottle dentin bonding agents의 dual application 방법은 상아질과 composite resin의 결합강도에 도움이 되지 않았다. 5군에서 사용된 amphiphilic copolymer의 작용은 실험상에서 확인할 수 없었다. 유지놀 함유 임시 접착제를 사용한 경우 레진의 결합강도가 저하되었는데, 이는 유지놀의 효과인지 임시 접착제의 잔사에 의한 것인지 확인할 수 없었다.

Key words : 콤포지트 레진, one-bottle dentin bonding agents, 인장 결합 강도, 유지놀 함유 임시 접착제. dual application 방법, amphiphilic copolymer.