Comparative evaluation of micro-shear bond strength between two different luting methods of resin cement to dentin

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ABSTRACT

The purpose of this study was to evaluate the effect of dual bonding technique by comparing micro-shear bond strength between two different luting methods of resin cement to tooth dentin.

Three dentin bonding systems(All-Bond 2, One-Step, Clearfil SE Bond), two temporary cements (Propac, Freegenol) were used in this study.

In groups used conventional luting procedure, dentin surfaces were left untreated. In groups used dual bonding technique, three dentin bonding systems were applied to each dentin surface. All specimens were covered with each temporary cement. The temporary cements were removed and each group was treated using one of three different dentin bonding system. A resin cement was applied to the glass cylinder surface and the cylinder was bonded to the dentin surface. Then, micro-shear bond strength test was performed. For the evaluation of the morphology at the resin/dentin interface, SEM examination was also performed.

1. Conventional luting procedure showed higher micro-shear bond strengths than dual boning technique. However, there were no significant differences.
2. Freegenol showed higher micro-shear bond strengths than Propac, but there were no significant differences.
3. In groups used dual bonding technique, SE Bond showed significantly higher micro-shear bond strengths in One-Step and All-Bond 2 ($p < 0.05$), but there was no significant difference between One-Step and All-Bond 2.
4. In SEM observation, with the use of All-Bond 2 and One-Step, very long and numerous resin tags were observed.

This study suggests that there were no findings that the dual bonding technique would be better than the conventional luting procedure. [J Kor Acad Cons Dent 30(4):283-293, 2005]

Key words: Dual bonding technique, Resin cement, Conventional luting procedure, Micro-shear bond strength

- Received 2004.9.21, revised 2005.4.6, accepted 2005.4.14 -

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Ⅰ. INTRODUCTION

In tooth-colored posterior restorations, direct composite restorations are the preferred treatment over indirect restorations because they require minimal intervention and cavity preparation[1).

However, a major limitation of direct composite restorations is the inability to control polymerization shrinkage and depth of cure[2]. These may lead to marginal gap formation and microleakage resulting in secondary caries and subsequent failure of the restoration[3]. But Indirect composite restorations could be reduced polymerization shrinkage stress and increased degree of conversion. These results in less stress in the restoration/cementation/tooth interfaces, fewer marginal voids, less microleakage, and reduced postoperative sensitivity. Therefore, indirect restorations are usually recommended when teeth should be required large restorations.

Clinical success with indirect restorations has been assisted by the ability to develop a reliable bond of resin cement to dental tissue[4]. However, the resin cements do not bond as strongly to dentin as do resin adhesives that are designed for direct composite restorations[5]. So this luting resin cement requires several additional steps to secure optimal adhesion.

Dentin bonding has become more successful with the development of new dentin bonding systems over the last 10 years[6-9]. Recently, dentin bonding systems with self-etching primers have been introduced, yielding major improvements in bonding to tooth structures. The use of these bonding systems is a result of attempts to improve the bonding quality while reducing the number of necessary procedures[10].

In indirect tooth-colored restorations, the conventional luting procedure consists in delaying the application of dentin bonding system until luting the final restoration. After tooth preparation, cavity is taken impression and a provisional restoration using a temporary cement is placed. In conventional luting procedure, the contamination of prepared dentin surfaces with temporary filling materials, blood, and saliva during laboratory procedure for indirect restoration may deteriorate bond strengths drastically and it may adversely affect the longevity of the restorations[11,12]. A relatively weak bond may lead to gap formation, producing post-operative sensitivity that results in premature failure of the indirect restorations[13].

By applying a dentin bonding system immediately following cavity preparation prior to taking an impression, the prepared tooth surface is sealed to protect the pulp from mechanical trauma, thermal stimuli and bacterial invasion during impression taking, temporization, and final cementation. This modified luting method is so called the "dual bonding technique"[14]. In a first step of the dual bonding technique, the dentin bonding agent is applied and cured right after tooth preparation. After sealing the dentin surface, cavity is taken impression and a provisional restoration using a temporary cement is placed. At the time of the completion of the final restoration, the provisional restoration is removed, and the dentin surface is cleaned with pumice. For the second step of that, the same dentin bonding agent is applied again on the prepared dentin surface. The dual bonding technique is likely to improve the early bond strength of resin cement to dentin. Previous studies have shown that tensile bond strengths of resin cements to dentin could be successfully increased by applying a dentin bonding system on the dentin surface after cavity preparation[15,16].

Indirect restorations usually require a provisional restoration that is luted to the prepared cavity with a temporary cement before final restoration. An adverse effect of temporary cements may be affected either due to changes in wettability and reactivity of the dentin or due to remnants of the material on the surface[17,18]. Additionally, residual contents of the temporary cements may interact with the setting resin composites, and therefore they leads to impair polymerization[19]. Eugenol-containing temporary materials are cheap and easily removable, so therefore are widely used in the dental practice. Furthermore, many clinicians also use their materials with their sedative effect on sensitive teeth. However, eugenol has been suggested as having the most deleterious effect to the polymerization process of resin composites, because it is known to be a radical scavenger. While some authors have described the inhibition of composite polymerization or a reduction of hardness, as well as an alteration of the cured resin composites surface[20]. But Powell and Huget[21] did not find any influence of eugenol-containing materials on the compressive strain and the tensile stress of resin composites. Woody and Davis[22] also demonstrated that eugenol-containing temporary cements had no influence on the microleakage of resin-luted inlays.

Shear testing has become a very popular among the variety of bond strength testing method for evaluation of adhesive effect of materials[23-25]. Shear stress is believed to be major stresses involved in in-vivo bonding failures of restorative materials[24,25]. Recently, Sano et al.[26] developed micro-tensile bond test, permitting the measurement as small bounded area as 1 ㎟, and leads a uniform stress distribution, so that most bond failures occur interfacially(adhesive failure). In this study, bond strengths were assessed by means of a micro-shear bond test that measured bonding to small areas of substrate.

The purpose of this study was to evaluate the effect of dual bonding technique by comparing micro-shear bond strength to two different luting methods of resin cement to tooth dentin. In addition, adhesive-substrate interfaces were examined by SEM to evaluate bonding efficacy.

Ⅱ. MATERIALS AND METHODS

Three dentin bonding systems (All-Bond 2 (BISCO, USA), One-Step (BISCO, USA), Clearfil SE Bond (Kuraray, Japan)), two temporary cements (Propac (GC, Japan), Freegenol (GC, Japan)), and one resin cement (Choice (BISCO, USA)) were used in this study, and their components, manufacturers were listed in Table 1.

Table 1. Materials used in this study

Materials	Component	Composition	Manufacturer
All-Bond 2	Conditioner Primer A Primer B Adhesive	37% Phosphoric acid 2% NTG-GMA 16% BPDM Bis-GMA, UDMA, HEMA	BISCO. Inc. (IL, USA)
One-Step	Conditioner Adhesive	37% Phosphoric acid Bis-GMA, UDMA, HEMA, Initiator, acetone	BISCO. Inc. (IL, USA)
Clearfil SE Bond (SE Bond)	Primer Adhesive	MDP, HEMA, water MDP, HEMA, dimethacrylate, microfiller	Kuraray Co., (Osaka, Japan)
Propac	Base Accelerator	Zinc oxide, olive oil, turpentine oil Eugenol, Rosin, Carnaba wax	GC Co. (Tokyo, Japan)
Freegenol	Base Accelerator	Zinc oxide, olive oil, Vaseline Polymer-fatty acid, estergum, beeswax, oleic acid	GC Co. (Tokyo, Japan)
Choice	Adhesive paste Dual-cure catalyst paste	Strontium glass, amorphous silica, Bis-GMA, UDMA, photoinitiator Amorphous silica, Bis-GMA, TEGDMA, benzoyl peroxide	BISCO. Inc. (IL, USA)

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

Eighty-four freshly extracted caries- and restoration-free human third molars were used in this study. Initially, the teeth were embedded in epoxy resin using acrylic ring (2 ㎝ diameter, 1.5 ㎝ height). Then, flat superficial occlusal dentin surfaces were obtained, initially using a low speed diamond saw (ISOMET; Buhler, USA), and exposed dentin surface was ground up to #600-grit SiC paper serially with copious water. Thereafter, the teeth were randomly divided into twelve groups according to performing conventional or dual bonding technique shown as Table 2.

1. Specimen preparation

The specimens were made by the occlusal dentin surfaces which received with the following dentin treatments prior to taking of impressions;

In Group CPA, CPO, CPS, CFA, CFO, and CFS, conventional luting procedure was used, the dentin surfaces were left untreated prior to taking of impression.

In Group DPA, DPA, DPS, DFA, DFO, and DFS, dual bonding technique was used, three dentin bonding systems (All-Bond 2, One-Step, Clearfil SE Bond) were applied on the dentin surfaces of specimens according to manufacturer's instruction (Table 3). Then, the dentin surfaces of all 84 specimens were taken impression with silicon impression material (EXAFINE, GC, Japan) and covered with each temporary cement (Propac (eugenol-containing), Freegenol (eugenol-free)) respectively. After setting of the temporary cements, all of the specimens were stored in distilled water at room temperature for 5 days before test.

Then, the temporary cement on the dentin surface was mechanically removed and the dentin surface was cleaned with pumice. Each specimen of 12 groups was treated using one of three dentin bonding system respectively, according to manufacturer's instruction (Table 3).

The standardized circular surface (diameter: 1.1 ㎜) on each tooth was isolated to limit and standardize the bond area. An adhesive tape (thickness: 50 ㎛) with 1.1 ㎜ diameter hole punched was placed on the dentin surface. The resulting holes were 1.1 ㎜ in diameter, and two to four holes were placed on each tooth.

The glass cylinders (diameter: 3 ㎜) were sectioned with the low speed diamond saw to produce 3 ㎜ in height. The surface of the glass cylin-

Table 2. Experimental groups classified by luting methods, temporary cement, and dentin bonding system

Luting method	Temporary cement	Dentin bonding system	Group
Conventional luting procedure (C)	Propac (P)	All-Bond 2 (A)	CPA
		One-Step (O)	CPO
		Clearfil SE Bond (S)	CPS
	Freegenol (F)	All-Bond 2 (A)	CFA
		One-Step (O)	CFO
		Clearfil SE Bond (S)	CFS
Dual bonding technique (D)	Propac (P)	All-Bond 2 (A)	DPA
		One-Step (O)	DPO
		Clearfil SE Bond (S)	DPS
	Freegenol (F)	All-Bond 2 (A)	DFA
		One-Step (O)	DFO
		Clearfil SE Bond (S)	DFS

Table 3. Application manner of Dentin Bonding System (DBS)

DBS	Manner of application to denin surface
All-Bond 2	Etching 15 sec, Priming - mixed Primer A and B (five times), air dry 5 sec Adhesive, light-cure 20 sec
One-Step	Etching 15 sec Adhesive (two coat), air dry 5 sec Light-cure 10 sec
SE Bond	Primer 20 sec, air dry Adhesive, light-cure 10 sec

ders were sandblasted, cleaned with 37% phosphoric acid and treated with a silane agent (Porcelain Primer, BISCO, USA). The glass cylinder was bonded to the punched dentin surface with the resin cement (Choice, Bisco, USA) and light-cured with Spectrum 800 (Dentsply, USA) for 60 sec.

2. Micro-shear bond strength test

All specimens of 12 groups were stored for 24 hours in distilled water at room temperature. Thereafter, each specimen was placed in a testing machine (EZ-test, Shimadzu, Japan) for micro-shear bond testing. First, the specimen was placed in the shear jig. A thin wire was looped around the glass cylinder, making contact through half of its circumference, and was gently held flush against the dentin-glass interface. A shear force was applied to each specimen at a cross-head speed of 1 ㎜/min until failure occurred.

3. SEM Evaluation

For the evaluation of the resin-dentin interface, specimens were bonded to receive the same treatment as the micro-shear bond strength test. After twenty-four hours, the specimens were sectioned perpendicular to bonding surface by means of the low speed diamond saw, and then embedded in epoxy resin. The sectioned surfaces were serially ground to #2000-grit SiC papers, and highly polished with a diamond paste. The specimens were subjected to 10% phosphoric acid treatment for 3 - 5 sec[27]. Then the 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 specimens were air dried, gold-sputter-coated, and examined in SEM (S-2300; Hitachi Co., Japan).

4. Statistical analysis

The maximum shear force was divided by the area of the specimen and the measured micro-shear bond strength values were analysed using one-way ANOVA / Scheffe's post-hoc test at 95% significance level.

Ⅲ. RESULTS

1. Micro-shear bond strength

The means and standard deviations of the micro-shear bond strengths of each group are shown in Table 4.

In Group CPA, CFA, DPA, and DFA, conventional luting procedure showed higher micro-shear bond strengths than dual boning technique, but there were no significant differences between four groups ($p > 0.05$). Also Freegenol showed higher micro-shear bond strengths than Propac, but there were no significant differences between four

Table 4. Micro-shear bond strength of experimental group (Unit: ㎫, Mean ± S.D.)

Luting method	Temporary cement	Dentin bonding system		
		All-Bond (A)	One-Step (O)	SE Bond (S)
Conventional (C)	Propac (P)	34.99 ± 8.34	41.13 ± 10.29	42.74 ± 6.45
	Freegenol (F)	37.27 ± 8.35	38.38 ± 10.30	47.23 ± 8.78
Dual bonding (D)	Propac (P)	27.31 ± 8.75	31.38 ± 7.88	45.52 ± 9.46
	Freegenol (F)	28.19 ± 8.10	32.76 ± 8.71	44.94 ± 12.54

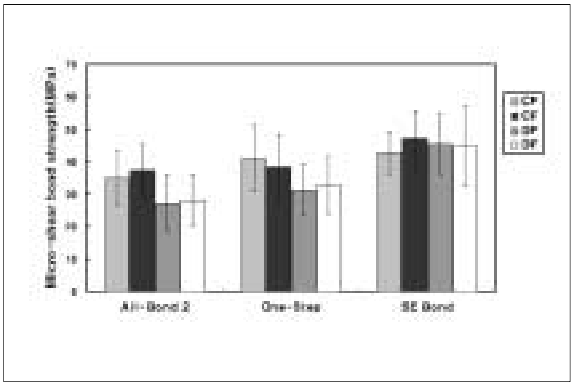

Figure 1. Micro-shear bond strengths of luting method according to dentin bonding system.

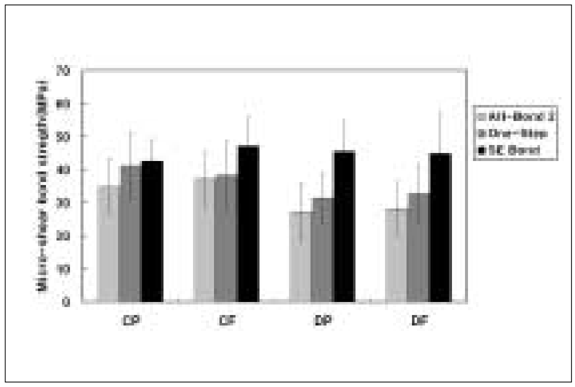

Figure 2. Micro-shear bond strengths of dentin bonding system according to luting method.

groups ($p > 0.05$).

In Group CPO, CFO, DPO, and DFO, conventional luting procedure showed higher micro-shear bond strengths than dual boning technique, but there were no significant differences between four groups ($p > 0.05$).

In Group CPS, CFS, DPS, and DFS, the micro-shear bond strengths decreased as followed ; CFS ≥ DPS ≥ DFS ≥ CPS. However, there were no significant differences between four groups ($p > 0.05$).

Group CFS showed the highest micro-shear bond strengths among CPA, CPO, CPS, CFA, CFO, and CFS. However, there were no significant differences between six groups ($p > 0.05$).

In Group DPA, DPO, DPS, DFA, DFO, and DFS, SE Bond showed significantly higher micro-shear bond strengths in One-Step and All-Bond 2 ($p < 0.05$), but there was no significant difference between One-Step and All-Bond 2 ($p > 0.05$).

The micro-shear bond strengths according to dentin bonding systems and to luting methods are showed in Figure 1 and 2.

2. SEM Evaluation

In SEM observation, there were several notable differences in dentin bonding systems. For All-Bond 2, the hybrid layer thickness ranged from between 4 - 5 ㎛. Very long and numerous resin tags observed. Resin tags were clearly observed with the typical funnel shape at the top of the tubules and more than 30 ㎛ in length. One-Step exhibited similar pattern to All-Bond 2.

For SE Bond, the thickness of the hybrid layer was measured between 2 - 3 ㎛. Resin tags of SE bond were fewer and shorter than All-Bond 2 and One-step.

In this study, there were no differences of the bonding layer thickness between dual bonding technique and conventional luting procedure.

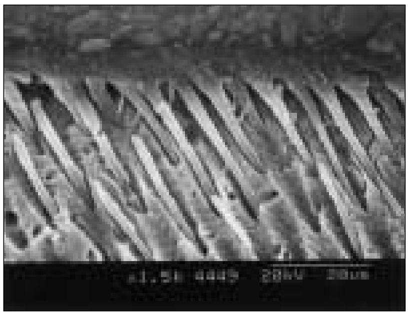

Figure 3. SEM photograph of the adhesive interface of CPA group (×1500).

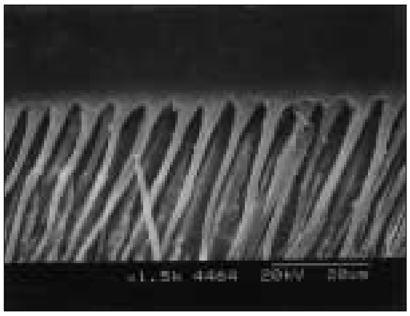

Figure 4. SEM photograph of the adhesive interface of CPO group (×1500).

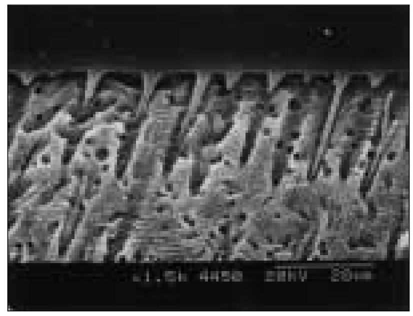

Figure 5. SEM photograph of the adhesive interface of CPS group (×1500).

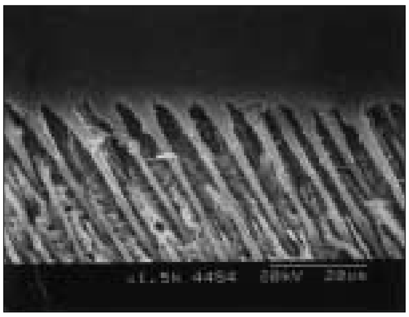

Figure 6. SEM photograph of the adhesive interface of CFA group (×1500).

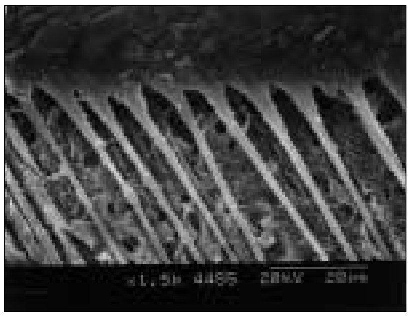

Figure 7. SEM photograph of the adhesive interface of CFO group (×1500).

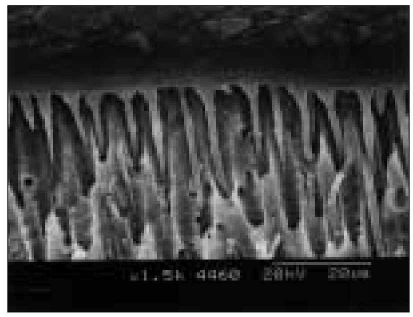

Figure 8. SEM photograph of the adhesive interface of CFS group (×1500).

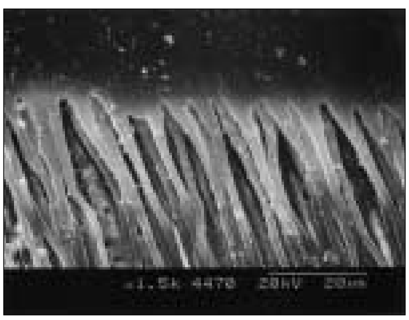

Figure 9. SEM photograph of the adhesive interface of DPA group (×1500).

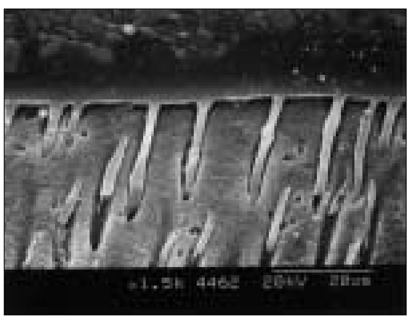

Figure 10. SEM photograph of the adhesive interface of DPO group (×1500).

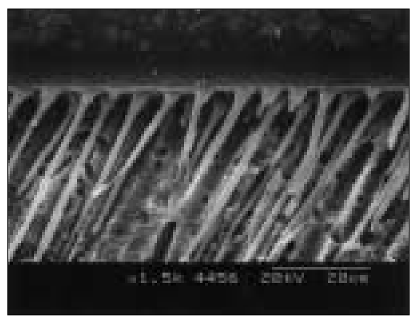

Figure 11. SEM photograph of the adhesive interface of DPS group (×1500).

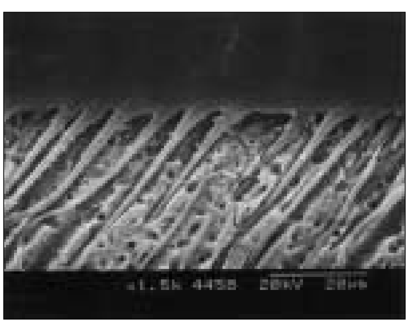

Figure 12. SEM photograph of the adhesive interface of DFA group (×1500).

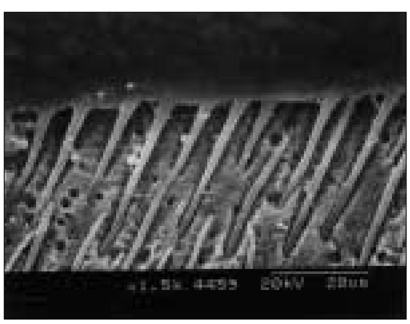

Figure 13. SEM photograph of the adhesive interface of DFO group (×1500).

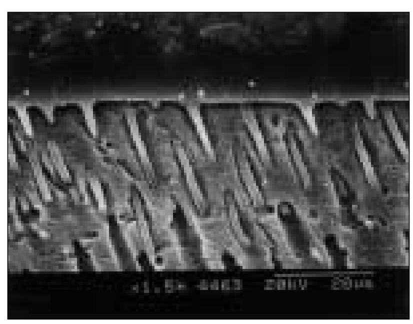

Figure 14. SEM photograph of the adhesive interface of DFS group (×1500).

Ⅳ. DISCUSSION

It is desirable to seal mechanically exposed dentin immediately after tooth preparation, before the impression is made, and in order to prevent bacterial microleakage[29]. The application of a dentin bonding system on the prepared cavity before taking impression has been claimed to protect the exposed dentin and prevent postoperative sensitivity[30,31]. That has also been proved to be reduced the interfacial gap formation, and improve bond strengths ceramic inlay to prepared cavity[15,32].

Bertschinger et al.[16] reported that dual application of dentin bonding system significantly increased the shear bond strength to dentin. Paul and Scharer[14] also presented the dual bonding technique as a modified luting method. Magne and Douglas[33] applied dentin bonding system to freshly prepared dentin, and polymerized it before taking the impression. In both studies this technique resulted in a considerable increase in the bond strength value. However, in this study, the micro-shear bond strengths of dual bonding technique and conventional luting procedure are no significant differences. As a result, there were no findings that the dual bonding technique would be better than the conventional luting procedure. This result could be influenced that the thickness of the oxygen inhibition layer would be greater for dentin bonding system. Also damage to the adhesive layer at the time of removal of the temporary cement may have contributed toward this result. In SEM observation, there were no differences of the bonding layer thickness between dual bonding technique and conventional luting procedure.

The Clearfil SE Bond resulted in higher micro-shear bond strength of resin cement to dentin compared with All-Bond 2 and One Step. Several factors could explain this difference between wet bonding system (All-Bond 2 and One Step) and self-etching system (Clearfil SE Bond). The self-etching system (Clearfil SE Bond) used on dry dentin contains an acidic self-etching primer, which simultaneously conditions both enamel and dentin followed by application of an bonding resin to the conditioned tooth surface[10]. On the other hand, the conventional bonding system (All-Bond 2) and self-priming system (One Step) used with the wet bonding technique require etching with phosphoric acid, followed by application of a primer or one- bottle adhesive to moist dentin[34]. The technical sensitivity of All-Bond 2 and One-Step, owing to difficulty in obtaining an adequately moist surface, may have resulted in lower micro-shear bond strength for these systems[35]. Nakabayashi and Saimi[36] suggested that the self-etching primer was very effective in creating diffusion channels while simultaneously promoting monomer impregnation into dentin at the same depth in their study. They also reported that hybridization created by this system was free from defects and was continuous from resin to calcium-rich dentin. However, it is still now unclear if these materials can produce strong, durable bonds.

The temporary cements used in this study represent the eugenol-containing temporary cement (Propac) and eugenol-free temporary cement (Freegenol). There were no significant differences in micro-shear bond strength between the groups treated with Propac and Freegenol. This result suggests that the use of eugenol- containing temporary cement had no adverse effect on micro-shear bond strength of a dual- curing composite luting cement to dentin. One possible side effect of eugenol may be the inhibition of the polymerization of the composite due to the radical scavenger properties of the phenols[17]. However, these effects do not seem to be strong enough to alter tensile strength as well as compressive strength of the bulk material[21]. Ganss and Jung[37] reported that pretreatment of dentin with eugenol-containing or eugenol-free temporary cements had no adverse effect on shear bond strength of dual- cured luting cement.

In this study, the thickness of hybrid layer or the length of resin tag have little affected the bond strength, supporting previous study that there was no correlation between bond strength and hybrid layer thickness[38]. No differences in thickness of hybrid layer and adhesive layer could be found between the groups used with conventional luting procedure and dual bonding technique.

Sealing dentin after cavity preparation is important and necessary in order to maintain dentin vitality, pulpal health, and patient comfort. Dentin exposure means a potential increase of risk of pulpal injuries, since dentin tubules can represent channels for the diffusion of injurious substances, and triggered a pulpal inflammatory response[39]. Moreover, exposed dentin can be sensitive to mechanical, thermal, tactile or osmotic stimuli, causing the clinical symptom of dentin hypersensitivity.

In this study, there were no findings that the dual bonding technique would be better than the conventional luting procedure for the test of the micro-shear bond strength. In this study, Clearfil SE Bond (self-etching system) showed higher micro-shear bond strength of resin cement to dentin compared with All-Bond 2 and One-Step (wet bonding system). Also in this study, the eugenol may be no adverse effect on the micro-shear bond strength of dentin bonding systems and resin cement to dentin. In clinical situation, for sealing dentin after cavity preparation in order to prevent contamination of exposed dentin surface, dual bonding technique may be considered.

Ⅴ. CONCLUSION

This study was designed to evaluate the effect of dual bonding technique by comparing the micro-shear bond strength between two different luting methods (conventional luting procedure, dual boning technique) of resin cement to dentin. Three dentin bonding systems (All-Bond 2 (BISCO, USA), One-Step (BISCO, USA), Clearfil SE Bond (Kuraray, Japan)), two temporary cements (Propac GC, Japan), Freegenol (GC, Japan)), and one resin cement (Choice (BISCO, USA)) were used in this study From the results of this study. we can conclude as follows;

1. Conventional luting procedure showed higher micro-shear bond strengths than dual boning technique. However, there were no significant differences ($p > 0.05$).
2. Freegenol showed higher micro-shear bond strengths than Propac, but there were no significant differences ($p > 0.05$).
3. The micro-shear bond strengths of two luting methods were decreased in order of SE Bond, One-Step and All-bond 2. In groups used dual bonding technique, SE Bond showed significantly higher micro- shear bond strengths in One-Step and All-Bond 2 ($p < 0.05$), but there was no significant difference between One-Step and All-Bond 2 ($p > 0.05$).
4. In SEM observation, with the use of total etching procedure (All-Bond 2 and One-Step), very long and numerous resin tags were observed.

This study suggests that there were no findings that the dual bonding technique would be better than the conventional luting procedure. In clinical situation, for sealing dentin after cavity preparation in order to prevent contamination of exposed dentin surface, dual bonding technique may be considered.

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국문초록

합착 술식에 따른 레진 합착제의 상아질에 대한 미세전단결합강도의 비교 연구

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본 연구는 합착 술식에 따른 레진 합착제의 상아질에 대한 미세전단결합강도를 비교 연구하여 이중 접착 술식의 유용성을 평가하고자 시행되었다.

합착 술식은 전통 합착 술식과 이중 접착 술식, 임시 합착제는 Propac과 Freegenol, 상아질 접착제는 All-Bond 2, One-Step, Clearfil SE Bond를 사용하였다.

이중 접착 술식을 적용한 군에서만 상아질 접착제 처리 후, 모든 시편에 임시 합착제를 도포하였다. 이후 임시 합착제를 제거하고 상아질 접착제 적용 후 유리봉에 레진 합착제를 도포하여 상아질 면에 접착하였다. 미세전단결합강도를 측정하고 접착 계면을 주사전자현미경으로 관찰하였다.

1. 전통 합착 술식이 이중 접착 술식보다 높은 미세전단결합강도를 보였으나 통계학적 유의차가 없었다.
2. Freegenol이 Propac보다 높은 미세전단결합강도를 보였으나 유의차가 없었다.
3. 미세전단결합강도는 이중 접착 술식을 적용한 경우 Clearfil SE Bond가 One-step, All-Bond 2보다 유의성 있게 높았으나($p < 0.05$), One-step, All-Bond 2 간 유의차는 없었다.
4. 전자현미경 소견에서 All-Bond 2와 One-Step을 사용한 군은 길고 수많은 resin tag가 관찰되었다.

본 연구 결과 전통 합착 술식과 비교하여 이중 접착 술식의 우수함을 확인하지 못하였다.

주요어: 레진 합착제, 미세전단결합강도, 이중 접착 술식, 전통 합착 술식