

## Comparison of shear bond strength of different bonding systems on bleached enamel

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### 국문초록

수종의 치질 접착제의 미백 처리된 법랑질에 대한 전단접착강도 비교

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치아 미백술은 치아의 심미성을 향상시키는 가장 보존적인 방법의 하나로 인식되어져 왔으며 최근의 심미치과에 대한 관심의 증가와 함께 그 빈도가 급격히 증가하고 있는 술식의 하나이다. 일반적으로 치아 미백술 후 바로 접착수복을 할 경우 결합력이 감소하는 것으로 알려져 있으며 이를 해소하기 위해 일정시간 경과 후 접착수복 술식을 시행할 것을 권장하고 있다. 자가산부식형(self-etching primer system) 접착제는 기존의 접착제와 다른 성분으로 인해 치아 미백제에 의한 영향에 대해 잘 알려져 있지 않은 상태이다.

이에 본 실험에서는 미백술을 시행한 법랑질 상에서 서로 다른 세 가지의 접착 시스템을 이용하여 미백술 후의 지연 시간이 결합력에 미치는 영향을 비교하고자 하였다.

발거한 대구치 68개를 물기가 있는 상태에서 근원심으로 절단하고 치관부를 자가중합 레진에 식립하여 시편을 제작하였다. 세 가지 접착제로 One-step®, Clearfil SE Bond primer®, One-up Bond F® 를 이용하였다. 각 접착제를 미백을 시행하지 않은 대조군과 미백 시행 후 바로 접착한 즉시 접착군, 그리고 2주간 생리식염수에 보관한 후에 접착한 지연군으로 나누어 총 9개의 실험군으로 나누었다. 접착제를 처리한 면에 Clearfil AP-X® 복합레진을 2mm 충전하고, 40초간 광중합을 시행하였다. 24시간 후 전단접착강도를 측정하였으며, 그 결과는 다음과 같이 나타났다.

One-step®의 경우, 즉시 접착군에서 지연 접착군보다 유의성 있게 낮은 접착강도를 나타내었다.

Clearfil SE Bond®의 경우, 즉시 접착군과 미백을 시행하지 않은 군간에는 접착 강도에 유의한 차가 없었으나, 지연 접착군에서는 낮은 강도를 나타내었다.

One-Up Bond F®의 경우, 즉시 접착군에서 유의성 있게 낮은 전단접착강도를 나타내었고, 전반적인 접착 강도가 다른 두 접착제에 비해서 유의성 있게 낮은 값을 보였다.

One-step®을 사용할 경우 지연접착을 하는 것이 추천되며, Clearfil SE Bond®의 경우에는 즉시 접착을 시행하더라도 영향을 적게 받는 것으로 나타났으며, One-Up Bond F®의 경우 미백술 후 접착수복 과정에 사용에 제한이 있는 것으로 나타났다. [J Kor Acad Cons Dent 29(1):30-35, 2004]

**주요어 :** Bleaching, Enamel bonding, Bonding agents, Self-etching system

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### I . INTRODUCTION

Since 1877, the first tooth bleaching was reported by Chapple, many different agents have been used and one of them, nightguard vital bleaching

has become an effective and predictable method to lighten discolored teeth. Both 30 % hydrogen peroxide-based systems and 10 to 15 % carbamide peroxide-based systems have been used successfully in lightening teeth. However, carbamide peroxide has been reported to be less acidic because of the presence of ammonia and carbon dioxide, both of which are by-products of the urea breakdown cycle<sup>1)</sup>. For these reasons, carbamide peroxide bleaching has been suggested as a safer alternative to the harsher hydrogen peroxide-based system<sup>2)</sup>.

Although preresorative carbamide peroxide bleaching is gaining popularity, its effect on the bond strength to composite has been inconclusive. Carbamide peroxide, major component of current bleaching system, has been implicated in adversely affecting the bond strength of composite to enamel and dentin. There have been reports regarding the interaction between bleaching agents and bond strength of composite materials to enamel; some authors have reported a severe decrease in the average shear bond strength of composite to bleached enamel compared with unbleached enamel<sup>3,4)</sup>. Among the strategies of recovery of the reduced bond strength after bleaching process, delayed bonding method have suggested<sup>5,6)</sup>.

Today, growing efforts are made to simplify and shorten the bonding procedure, by combining the function of conditioner, primer and adhesive. The use of strongly acidic primers introduced the concept of "self-etching" primers not only to dentin but also to enamel, which eliminates the necessity of a separate conditioning step<sup>7)</sup>. The self-etching primer provide comparable bond strengths without the time consuming process of applying and rinsing the etchant<sup>8)</sup>. Fewer steps in the bonding process mean fewer operator errors. Because the monomers that cause the etching are also responsible for bonding, the depth of penetration of the monomers to be polymerized is exactly the same as the depth of demineralization, resulting in a complete hybrid layer. Perdigao et al.<sup>9)</sup> investigated the effect of calcium on enamel and dentin

bond strength. They suggested that calcium removal appears to be less detrimental for self-etching primers compared to total-etch adhesives. Self-etching primer showed different etching pattern and the length of resin tag on enamel compared to those etched with phosphoric acid.

Those differences of bonding mechanism might lead to different bond strength on bleached enamel and clinical investigations are necessary to evaluate the potential of recently developed all-in-one self-etching products which combine the functions of conditioner, primer, and adhesive.

The objective of this study is to compare (1) the effect of type of bonding agents; conventional and self-etching adhesive systems and (2) immediate and delayed bonding of hybrid composite resin on the 10% carbamide peroxide bleached enamel.

## II. MATERIALS AND METHODS

Sixty eight extracted, noncarious human molars stored at 4°C in isotonic saline were used in this study. The last 24 hours before beginning the experiment, they were kept in distilled water. The crowns of 68 teeth were cut at the cemento-enamel junction (CEJ) using a low speed diamond saw under copious water cooling then the crowns were cut in half in mesio-distal direction. Each section was embedded in auto-polymerizing acrylic resin with enamel surface facing up (Orthodontic Resin, Dentsply/Detray, Konstanz, Germany) so that the prepared enamel surfaces were 2 mm above the acrylic resin cylinders, and placed in tap water to reduce the temperature rise from the exothermic polymerization reaction.

After the resin had completely polymerized, the enamel surfaces were grounded parallel to the long axis of the tooth on a water-cooled, model trimming wheel and #800 abrasive paper to create flat enamel surface and to make uniform bonding condition.

The teeth were randomly divided into nine groups of 11 specimens each and treated in the

manner Table 1.

Bleaching material, Opalescence® (Ultradent Product, Inc, Salt Lake City, UT 84095, USA) with 10% of carbamide peroxide gel was used. Control groups were not bleached and stored in saline for 10 days. Bleached groups were exposed to daily application of bleaching gel for six hours during 10 consecutive days. After 6 hours of bleaching gel application, the samples were washed and stored in saline, until the next application. After the bleaching procedure was done, the surface was cleaned with pumice and pre-

pared to bonding composite.

Bonding materials used were One-step® (Bisco., Inc., Schaumburg, Illinois, USA), Clearfil SE Bond® (Kuraray Co., Ltd., Osaka, Japan), and One-up Bond F® (Tokuyama Co., Ltd., Shibutaku, Tokyo, Japan) (Table 2). All bonding materials were applied following the manufacturer's instructions.

Hybrid composite (Clearfil AP-X, Kuraray Co., Ltd., Osaka, Japan) was packed into the Ultradent mount jig mold (Ultradent Product Inc., South Jordan, Utah, USA) and light-cured

**Table 1.** Experimental groups following bonding condition of enamel surface

Group	Treatment	Bonding agent
OS 1	No bleaching, 10 days storage in saline	One step®
OS 2	Bleaching, immediate bonding	
OS 3	Bleaching, delayed bonding(2week later)	
SE 1	No bleaching, 10 days storage in saline	Clearfil SE Bond®
SE 2	Bleaching, immediate bonding	
SE 3	Bleaching, delayed bonding(2week later)	
OU 1	No bleaching, 10 days storage in saline	One-up Bond F®
OU 2	Bleaching, immediate bonding	
OU 3	Bleaching, delayed bonding(2week later)	

Bleaching: apply Opalescence® 10%, 6 hours per day for 10 days

**Table 2.** Chemical ingredients of the bonding agents used in this study

Bonding agent	Chemical composition	
One-step	Etching	Primer
	32% Phosphoric acid	Biphenyl dimethacrylate(BPDM), Hydroxyethyl methacrylate(HEMA) Bis-phenol A diglycidylmethacrylate(Bis-GMA), acetone
	Primer	Bonding
Clearfil SE Bond	10-methacryloyloxyl dihydrogenphosphate(MDP), 2-hydroxyethyl methacrylate, hydrophilic dimethacrylate, dl-camphorquinone, N,N-diethanol-p-toluidine, water	MDP, Bis-GMA di-camphorquinone, N,N-diethanol-p-toluidine, silanated colloidal silica
	Primer A & B	
	MAC-10, HEMA, PRG filler, fluoroalluminosilicate glass, acetone, water, initiator	

**Table 3.** Shear bond strength of resin composite to bleached enamel

Group	n	Shear bond strength	Tukey test
OS 1	11	30.8±4.4	A
OS 2	11	24.8±2.6	B
OS 3	11	26.5±5.1	AB
CS 1	11	24.7±3.6	B
CS 2	11	23.5±3.4	BC
CS 3	11	16.2±5.2	D
OU 1	10	18.6±2.6	CD
OU 2	10	11.6±3.9	E
OU 3	11	16.1±2.4	DE

· Means with different letters are significantly different at  $p < 0.05$ .

· OS: One-step®, CS: Clearfil SE Bond®, OU: One-up Bond F®

for 40 seconds. After polymerization, the alignment tube and mold were removed and the specimens were placed in 37°C distilled water for 24 hours.

Control and immediate bonding groups were tested in shear mode using a chisel-shaped rod in an Instron testing machine (Type 4202, Instron Corp., Canton, Massachusetts, USA) at a cross-head speed of 1 mm/minute. After two-week storage, the groups of delayed bonding specimens were bonded and tested in the same mode.

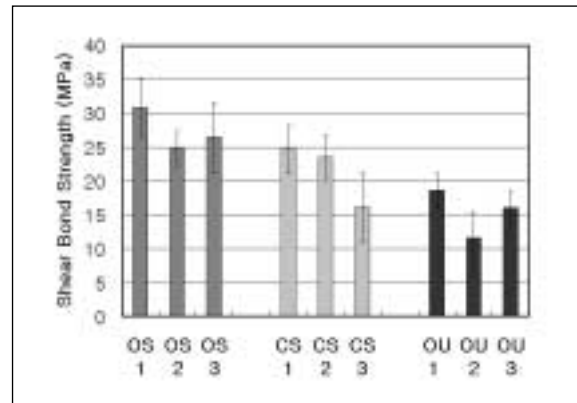
The data for each group were subjected to one-way ANOVA followed by Tukey's test at  $p < 0.05$  to make comparisons among the groups.

### III. RESULTS

The results of the shear bond strength tests to bleached enamel are shown in table 3 and Fig.1.

In One-step® and One-up Bond F® groups, the immediate bonding group showed lower bond strength than control and delayed groups.

In Clearfil SE Bond® group, there were no statistical difference of shear bond strength in immediate bonding group compared to control group, but the bond strength of delayed group was lower than the others.

**Fig. 1.** Shear bond strength of resin composite to bleached enamel surface.

Among bonding agents, One-up Bond F® showed significantly lower bond strength than One-step® and Clearfil SE Bond®.

### IV. DISCUSSION

Although prerestorative carbamide peroxide bleaching is gaining popularity as a more conservative and economical method of improving the esthetics, its detrimental effect on the bond strength to composite has been suggested. Some authors have reported a severe decrease in the average bond strength of composite to bleached enamel compared with unbleached enamel<sup>2-6,10</sup>.

The exact mechanism of the decreased bond strength after bleaching is not certain, but there were some speculated reasons. One of the reason is that changes in enamel structures resulting from the loss of mineral content, or increased porosity as manifested by an "over-etched" appearance with loss of prismatic form<sup>10</sup>.

However, Dishman et al.<sup>11</sup> argued that the quality of composite bond is compromised through a decreased number of resin tags present, suggesting that there may be some kind of polymerization inhibition taking place. Kalili et al.<sup>12</sup> proposed that such inhibition could result from bleaching agents that cause oxygen to penetrate and concentrate on the surface of enamel, thus inhibiting the cure of some resin tags.

Perdigao et al.<sup>13</sup> reported that vital bleaching

with a commercial 10 % carbamide peroxide gel did not change the oxygen concentration in the surface of bleached enamel. And they found that carbamide peroxide bleached enamel showed relatively lower concentration of Ca and P.

To recover the bond strength, some authors have advised of delays in bonding one or more weeks after bleaching because the reduction of composite bond strength to freshly bleached enamel has been transient<sup>3,14,15)</sup>. Similarly, removal of the superficial layer, pretreatment of bleached enamel with alcohol, and use of adhesives containing organic solvents have been also suggested to result in complete reversal of the reduced enamel bonds<sup>16-18)</sup>.

Recently used bonding agents can be divided in two groups by their surface conditioning method. One is conventional total etching system and the other is self-etching system.

Although self-etching primers had lower acidity than conventional etchants, they showed comparable shear bond strengths to conventional types<sup>19-21)</sup>. They explain the reason that the length of resin tags has been shown to contribute little to the bond strength of resin to enamel and bonding is mainly attributable to the ability of the resin to penetrate between the enamel crystallites and rods. If the resin monomers adequately penetrate into the superficially etched surface, that is enough to obtain proper bond strength<sup>20)</sup>.

In control groups, following the results of this study, the conventional acid etching system showed higher bond strength than the self-etching systems and the One-up Bond F®, all-in-one type bonding system, was the lowest.

In the immediate bond strength of OS 2 and CS 2 was comparable but OU 2 group was much lower than the other groups. This can be explained that the lower acidity (higher pH) of self-etching primer. In total etching system, the depletion of calcium from enamel surface could reduce bond strength<sup>22,23)</sup>, but in self-etching system calcium removal from the tooth surface was not detrimental.

The latter system may less influence on the hardness of already weakened enamel structure.

In case of using self-etching primer after bleaching, there may be no need to delay bonding steps.

In the delayed bonding groups, the bond strength of OS 3 and OU 3 were recovered to the control group level, but CS 3 was different. We have not known the reason of these results, and more study may be need to reveal those reasons.

In case of One-up Bond F®, there were significantly lower shear bond strength in all test groups than two groups. So it may be used cautiously.

## V. CONCLUSION

The objective of this study is to compare (1) the effect of type of bonding agents; conventional and self-etching adhesive systems and (2) immediate and delayed bonding of hybrid composite resin on the 10 % carbamide peroxide bleached enamel.

68 extracted, noncarious human molars were used in this study. With enamel surface facing up, sectioned teeth were embedded in auto-polymerizing acrylic resin. The teeth were randomly divided into nine groups of 11 specimen each. Bleaching material, Opalescence® with 10% of carbamide peroxide gel was used. Control groups were not bleached and after 10-day storage, those were tested. Experimental groups were exposed to one daily application of bleaching gel for six hours during 10 consecutive days. Used bonding materials were One-step®, Clearfil SE Bond®, One-up Bond F®. All bonding materials were applied per manufacturer's instructions. The immediate bonding groups were bonded the composite right after the final bleaching procedure and delayed groups were immersed in saline and bonded composite. 24 hours after bonding shear bond strength were measured by Instron testing machine at a crosshead speed of 1 mm/minute.

The data for each group were subjected to One-way ANOVA followed by Tukey's test at  $p < 0.05$  to make comparisons among the groups

The results are :

In One-step® groups, the immediate bonding group showed lower bond strength than control and delayed groups.

In Clearfil SE Bond® groups, there were no statistical difference of shear bond strength between the control and immediate groups, but delayed group showed lower bond strength.

In One-up Bond F® groups, the immediate bonding group showed lower bond strength than control and delayed groups. Among bonding agents, One-up Bond F showed significantly lower bond strength than One-step and Clearfil SE Bond.

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