

## Bonding efficacy of cured or uncured dentin adhesives in indirect resin

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## ABSTRACT

**Objectives:** This study examined the effect of the uncured dentin adhesives on the bond interface between the resin inlay and dentin.

**Materials and Methods:** Dentin surface was exposed in 24 extracted human molars and the teeth were assigned to indirect and direct resin restoration group. For indirect resin groups, exposed dentin surfaces were temporized with provisional resin. The provisional restoration was removed after 1 wk and the teeth were divided further into 4 groups which used dentin adhesives (OptiBond FL, Kerr; One-Step, Bisco) with or without light-curing, respectively (Group OB-C, OB-NC, OS-C and OS-NC). Pre-fabricated resin blocks were cemented on the entire surfaces with resin cement. For the direct resin restoration groups, the dentin surfaces were treated with dentin adhesives (Group OB-D and OS-D), followed by restoring composite resin. After 24 hr, the teeth were assigned to microtensile bond strength ( $\mu$ TBS) and confocal laser scanning microscopy (CLSM), respectively.

**Results:** The indirect resin restoration groups showed a lower  $\mu$ TBS than the direct resin restoration groups. The  $\mu$ TBS values of the light cured dentin adhesive groups were higher than those of the uncured dentin adhesive groups ( $p < 0.05$ ). CLSM analysis of the light cured dentin adhesive groups revealed definite and homogenous hybrid layers. However, the uncured dentin adhesive groups showed uncertain or even no hybrid layer.

**Conclusions:** Light-curing of the dentin adhesive prior to the application of the cementing material in luting a resin inlay to dentin resulted in definite, homogenous hybrid layer formation, which may improve the bond strength. [J Kor Acad Cons Dent 2011;36(6):490-497.]

**Key words:** Bond interface; Confocal microscopy; Dentin adhesive; Microtensile bond strength; Resin inlay

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## INTRODUCTION

Posterior composite restorations have risen in popularity as a result of the development of improved resin composites, bonding systems and operating techniques.<sup>1</sup> Direct composite restorations are pre-

ferred to indirect composite restorations because they require minimal intervention during cavity preparation.<sup>2</sup> A major limitation of direct restoration is the difficulty of controlling the degree of conversion and polymerization shrinkage.<sup>3</sup> To overcome these limitations, the indirect fabrication of a composite inlay

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and cementation with a resin cement has been advocated. Unfortunately, the current available resin cements with indirect restorations do not always bond to dentin as strongly as dentin adhesive systems bond with direct resin composite restorations.<sup>4,5</sup> A relatively weak bond to the tooth structure leads to a host of problems: marginal gap formation, postoperative sensitivity, premature failure, and secondary caries formation.<sup>5,6</sup>

Regarding an indirect composite restoration, successful dentin bonding is of particular clinical importance for inlays because the final strength of the tooth-restoration complex is highly dependent on the dentin adhesive procedures.<sup>7-10</sup> For the cementation of an indirect composite restoration, several techniques have been proposed for a dentin bonding procedure. In the most commonly used method, the application of a dentin adhesive with light-curing on the treated dentin surface is followed by the application of resin cement. Nevertheless, the cured dentin adhesive could pool at the margin or inner line angles of the cavity due to the effect of gravity and the relatively low viscosity, which could hinder seating of the restoration.<sup>11-14</sup> These concerns regarding the possible detrimental effects of pre-curing of dentin adhesive have led to another method, where the dentin adhesive is not polymerized beforehand but is polymerized with resin cement.<sup>13-15</sup> In comparative studies of the bond strength, the tooth-restoration complex by dentin adhesive pre-curing was reported to have superior bond strength compared to when the dentin adhesive and resin cement had been cured simultaneously.<sup>11,12,16,17</sup> Only a small number of reports have explained the reason for the higher bond strength of the former method, even though there are many comparative reports of the bond strength according to

the application technique of dentin adhesive.

The introduction of confocal laser scanning microscopy (CLSM) provides a valuable new technique for visualizing the bonding structures, such as the hybrid layer and resin tags in dentin. The advantages of confocal microscopy include a non-destructive examination because the layer visualized can be up to 100  $\mu\text{m}$  below the surface and drying of samples is unnecessary, meaning there is less risk of shrinkage or other drying artifacts.<sup>17-21</sup>

This study examined the effect of the light-curing of dentin adhesive on the bond interface when luting a resin inlay to dentin using microtensile bond strength tests and confocal microscopy.

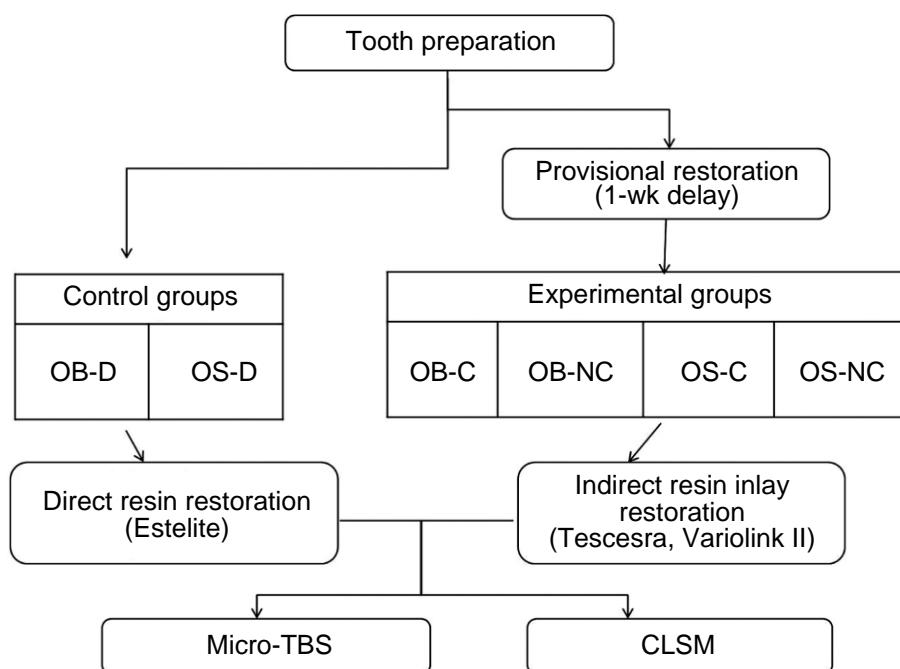
## MATERIALS AND METHODS

Twenty four extracted permanent molars without cavities, cracks or other defects were stored in a physiological saline solution. After embedding the teeth in a self-cure acrylic resin, the occlusal surfaces were ground flat to expose the dentin surface under water irrigation using a low speed diamond saw (MetSAW, MSH-04-112, R&B Inc., Daejeon, Korea). The hydrophilic primer of 3-step etch-and-rinse adhesive system (OptiBond FL, Kerr, Orange, CA, USA) and the bonding agent of 2-step etch-and-rinse adhesive system (One-Step, Bisco, Schaumburg, IL, USA) were labeled with 0.1% Rhodamine B isothiocyanate (R1755, batch No. 114F0376, Sigma, St. Louis, MO, USA) (Table 1). The fluorescent dye was applied directly to the supplied packaged bottles. The teeth were divided into 2 groups, according to the restoration methods: direct and indirect resin restoration (Figure 1).

For direct resin restoration groups, which served as

**Table 1.** Materials used in this study

| Material    | Manufacturer               | Composition   |
|-------------|----------------------------|---|
| OptiBond FL | Kerr, Orange, CA, USA      | Primer: HEMA, GPDM, Bis-GMA, MMEP, Ethanol, Water, Initiators<br>Bond: HEMA, GPDM, Bis-GMA, Disodium hexa-fluoro-silicate, Barium-aluminum borosilicate glass, Fumed silica |
| One-Step    | Bisco, Schaumburg, IL, USA | HEMA, BPDM, Bis-GMA, Acetone  |



**Figure 1.** Experimental groups and sequence.

the control groups, the exposed dentin surface was etched with 37% phosphoric acid (ETCH-37, Bisco) for 20 seconds, washed with water for 15 seconds and the surface was gently air dried. Subsequently, dentin adhesive was applied according to the manufacturer's recommendation. The entire surfaces were built with composite resin (Estelite $\Sigma$ , Tokuyama dental Corp, Tokyo, Japan) up to 3 mm, and each increment of 1 mm thick was light polymerized (Demetron LC, Kerr) at 600 mW/cm<sup>2</sup> for 20 seconds.

For the indirect resin restoration groups, the exposed dentin surfaces were restored using a provisional restoration material (Quicks, Denkist, Seoul, Korea), 2 mm in height, and light polymerized for 20 seconds. After 1 week storage in saline, the provisional restoration was removed and dentin was cleaned with pumice. The experimental groups were varied according to the sequence and mode of application of the dentin adhesives.

In Groups OB-C and OB-NC, the exposed dentin surface was etched and washed in the same manner as in the control groups. The OptiBond FL primer was applied to the dentin surface, and air dried thoroughly for 5 seconds to evaporate the solvent. The

adhesive resin was applied and air dried slightly. Light-curing of the adhesive resin was followed for 20 seconds for Group OB-C. On the other hand, Group OB-NC, the applied adhesive resin was left uncured until the application of the luting material. In Group OS-C and OS-NC, the exposed dentin surface was treated in a similar manner to previous groups. One-Step was applied to the surface, and air dried thoroughly for 5 seconds. Light-curing of the bonding agent was followed for 20 seconds for Group OS-C. In Group OS-NC, the applied bonding agent was left uncured until the luting material was applied (Table 2).

Sixteen indirect resin blocks, 3 mm in height and 10 mm in diameter (Tescera, Bisco), were prepared to simulate the laboratory-processed resin composite restorations. The surface of each composite disc was sandblasted with 50  $\mu$ m aluminum oxide particles for 10 seconds from a distance of approximately 5 mm. The composite disc surface to be cemented was silanized with a Monobond-S (Ivoclar vivadent) for 1 minute, and then air dried. Variolink II (Ivoclar vivadent) luting resin cement was mixed according to the manufacturer's instructions, and applied to the

tooth specimen with the composite disc. The excess resin cement was removed with a probe. The resin cement was light polymerized for 60 seconds.

The test specimens were then stored in 100% humidity for 24 hours at 37°C. The teeth of each group were divided randomly into two sub-groups for microtensile bond strength ( $\mu$ TBS) testing and CLSM (LSM510, Carl Zeiss, Oberkochen, Germany) observations.

#### Microtensile bond strength test

After storing in distilled water for 24 hours, the teeth were sectioned in the X and Y perpendicular directions with a low speed diamond saw to obtain 20 specimens in each group, 1 × 1 mm in size and 8 mm in length. The ends of each specimen were fixed to the Microtensile tester (Bisco) with cyanoacrylate adhesive (ZAPIT base, Dental Ventures of America Inc., Corona, CA, USA) plus an accelerator (ZAPIT accelerator, Dental Ventures of America Inc.). The results were obtained at the moment of the specimen fracture and calculated in MPa.

One-way ANOVA followed by Student-Newman-

Keuls test was used to determine any differences between the groups using same material. The differences between the whole experimental groups were statistically analyzed with the two-way ANOVA and SNK test at a 5% level of significance.

#### Confocal laser scanning microscope analysis

After 24 hours water immersion, three 500- $\mu$ m thick bucco-lingual sectioned samples were obtained from each teeth by sectioning in the perpendicular directions with a low speed diamond saw (MetSAW, MSH-04-112). The sliced specimens were mounted on glass slides and analyzed. The resin/dentin interfacial image examination was performed with a Leica TCS SP5 II microscope (Leica, Heidelberg, Germany) using a 50× epiplan objective.

## RESULTS

The results of the study are displayed in Table 3. The direct resin restoration groups, Groups OB-D and OS-D, showed higher  $\mu$ TBS than the indirect resin restoration groups. The mean  $\mu$ TBSs within the

**Table 2.** Experimental groups

| Group | Type of restoration        | DBA         | Light-curing of DBA |
|-------|----------------------------|-------------|---------------------|
| OB-D  | Direct resin restoration   | OptiBond FL | Yes                 |
| OS-D  |                            | One-Step    | Yes                 |
| OB-C  | Indirect resin restoration | OptiBond FL | Yes                 |
| OB-NC |                            | OptiBond FL | No                  |
| OS-C  |                            | One-Step    | Yes                 |
| OS-NC |                            | One-Step    | No                  |

DBA, dentin bonding agent; OB, OptiBond FL; OS, One-Step; C, cured; NC, not cured.

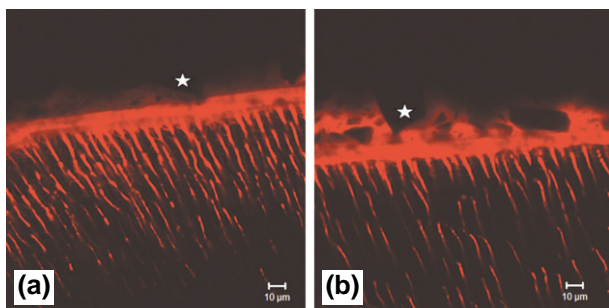
**Table 3.** Microtensile bond strength

(n = 20)

| Group                                     | Mean (MPa) $\pm$ SD                 |
|---|-------------------------------------|
| Control groups: direct restoration        | OB-D 24.37 $\pm$ 3.23 <sup>a</sup>  |
|   | OS-D 21.94 $\pm$ 2.86 <sup>a</sup>  |
| Experimental groups: indirect restoration | OB-C 19.86 $\pm$ 4.11 <sup>b</sup>  |
|   | OB-NC 17.11 $\pm$ 4.98 <sup>c</sup> |
|   | OS-C 16.33 $\pm$ 4.87 <sup>c</sup>  |
|   | OS-NC 12.34 $\pm$ 4.93 <sup>d</sup> |

OB, OptiBond FL; OS, One-Step; C, cured; NC, not cured.

Mean values followed by the same superscript.

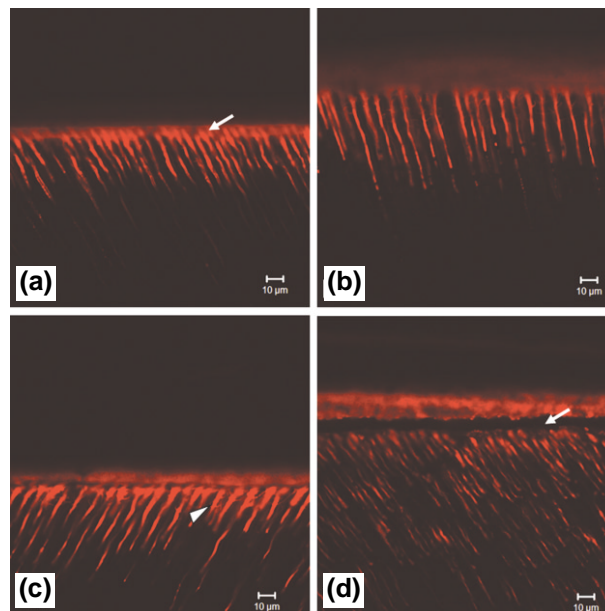


**Figure 2.** CLSM images of the direct resin restoration groups. (a) Group OB-D, dentin adhesive, OptiBond FL. Obvious hybrid layer is observed. (b) Group OS-D, dentin adhesive, OneStep. Filler particles of composite resin (EsteliteΣ) were seen in both groups (asterisk).

direct restoration groups (Groups OB-D and OS-D) were not statistically different, 24.37 and 21.94 MPa, respectively. The hybrid layer was definite and uniform, and some filler particles of the composite were also observed in these groups (Figure 2).

Light-curing of dentin adhesives and the use of three-step etch-and-rinse adhesives exhibited significantly higher  $\mu$ TBS in the indirect restoration groups ( $p < 0.05$ ). Group OB-C, which showed the highest  $\mu$ TBS, contained a typical resin dentin interdiffusion zone (RDIZ, Figure 3a). The primer of OptiBond FL and fluorescent dye infiltrated the demineralized inter-tubular dentin, creating an evident and homogenous hybrid layer. Moreover, it is likely that the primer infiltrated a small layer of intertubular dentin from the lateral direction. Group OS-C also showed a clearly distinguishable hybrid layer but had a thinner hybrid layer than Group OB-C (Figure 3c). In this group, multiple lateral branches were observed along with resin tags as well.

In the uncured dentin adhesive groups, Groups OB-NC and OS-NC, the structural features of the RDIZ were different from those observed in the cured dentin adhesive groups. Group OB-NC showed an uncertain and blurred hybrid layer in RDIZ (Figure 3b). In Group OS-C, the hybrid layer was often discontinuous or was even absent (Figure 3d).



**Figure 3.** CLSM images of the indirect resin restoration groups. (a) Group OB-C, cured dentin adhesive, OptiBond FL. Evident and homogenous hybrid layer (white arrow) is seen in resin-dentin interdiffusion zone (RDIZ). (b) Group OB-NC, uncured dentin adhesive, OptiBond FL. (c) Group OS-C, cured dentin adhesive, OneStep. Lateral branching of adhesive tubule tags (white arrowhead) are presented. (d) Group OS-NC, uncured dentin adhesive, OneStep. Note the discontinuous and ruptured hybrid layer (white arrow) in RDIZ.

## DISCUSSIONS

This *in vitro* study was designed to clarify the effect of light-curing of dentin adhesive when luting a resin inlay on the bond strength and micromorphological appearance of a RDIZ. The mean  $\mu$ TBSs of the cured dentin adhesives groups was higher than those of the uncured dentin adhesives, which is consistent with previous studies.<sup>11-15,22</sup> The RDIZ of the cured adhesive groups, particularly Group OB-C (Figure 3a) showed an evident and homogenous hybrid layer along with the margin of the resin cement layer. Pre-curing of dentin adhesive prior to luting the resin inlay to dentin might improve the bond strength, and might be related to the formation of a thicker and more definite hybrid layer.

The  $\mu$ TBS of Group OB-NC was lower than that of Group OB-C. The CLSM image of this group showed a blurred and uncertain hybrid layer in RDIZ. This suggests that the uncured dentin adhesive might have spread and mixed with the resin cement during its application.

Group OS-NC exhibited the lowest bond strength (12.34 MPa) among the experimental groups, which was approximately half value of the control direct restoration groups (24.3 and 21.9 MPa). Surprisingly, the most common micromorphologic image of this group was debonding at the top of the hybrid layer in RDIZ (Figure 3d). It could be assumed that these defects and the discontinuity of the hybrid layer contribute to the lower bond strength compared to the other groups. Lee and Park reported that when dentin adhesives are not light cured prior to cementation, the exposed, decalcified collagen could collapse during the cementing procedure as a consequence of the pressure applied during the luting process, which can lead to the failure of an indirect restoration.<sup>12</sup> However, from a practical point of view, it is recommended that the adhesive resin be kept unpolymerized before the restoration is fully seated because of interference of complete insertion, as mentioned previously. Considering the above, Magne *et al.* suggested the immediate dentin sealing (IDS) technique, that applying a dentin bonding agent (DBA) prior to impression taking.<sup>8,13</sup> IDS technique provides the ideal condition for dentin bonding that has no contamination by temporary restoration and the freshly cut and clean dentin. Further, recent studies exhibited that prepolymerization of the DBA results in improved bond strength.<sup>1,8,12,13,23</sup>

The debonding of uncured dentin adhesive is also due to polymerization shrinkage of resin cement, which occurred during the light-curing procedure. Being unfilled or less highly-filled, the magnitude of polymerization shrinkage of resin cement is expected to be higher than direct restorative composites. This result confirmed previous reports that the bond between the resin inlay and resin cement was the weak link in indirect composite restorations.<sup>24,25</sup> It is possible that (1) the uneven and ruptured hybrid layer and resin tags in the indirect resin restoration groups contributed to the lower bond strength, (2)

polymerization shrinkage of resin cement occurring while light-curing weaken the bond strength. Variolink II used in the present study showed significant superior  $\mu$ TBS compared to the other composite luting cement, such as self-adhesive resin cement (e.g. Rely X Unicem). Variolink II luting cement is a dual-cure composite system that requires a total etching and bonding procedure. Owing to this distinctive dentin bonding procedure, it might exhibit an evident hybrid layer and thick resin tags (Figure 3a) when following the manufacturer's instruction.<sup>26-28</sup>

In general, two-step etch-and-rinse adhesives performed less favorable clinically than conventional three-step etch-and-rinse adhesives, which is consistent with previous studies.<sup>29,30</sup> Since simplified etch-and-rinse adhesive contain higher percentages of hydrophilic monomers, such as HEMA, compared to three-step adhesive, they exhibit more permeability after polymerization, which facilitates the presence of water-contained areas within the hybrid layer.<sup>31,32</sup> On the other hand, in three-step adhesive, the hydrophobic coat of bonding resin may in part overcome the water movement, preserving the adhesive interface from hydrolysis. OptiBond FL, three step etch-and-rinse adhesive, also contains a barium glass filler with approximately 0.6  $\mu$ m sized particles in its composition. The barium glass filler provides the bond gel consistency, which according to the theory of an 'elastic cavity wall', improves the bond strength and reduces polymerization shrinkage of the adhesive resin.

## CONCLUSIONS

The  $\mu$ TBS of a resin inlay to dentin is improved by the light-curing of the dentin adhesive prior to the application of the cementing material. The hybrid layer was definite, homogenous in the RDIZ when the dentin adhesive was light cured before luting the resin inlay but not when it was cured with the resin cement simultaneously.

Conflict of Interest: No potential conflict of interest relevant to this article was reported.

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## 국문초록

## 간접 레진수복시 상아질 접착제의 중합 여부에 따른 결합 효능

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**연구목적:** 본 연구는 간접 레진수복시 상아질 접착제의 중합 여부가 레진인레이와 상아질간의 미세인장결합강도와 결합계면의 형태에 미치는 영향을 공초점 현미경 관찰을 통하여 평가하고자 하였다.

**연구 재료 및 방법:** 24개의 발거된 대구치를 절단하여 교합면 상아질을 노출시켰다. 수복 방법에 따라 크게 간접 레진수복과 직접 레진수복의 두 군으로 나누었다. 간접 레진수복군은 임시 수복하여 1주일 후, 상아질 접착 과정에 따라 4군(OB-C와 OB-NC군, OS-C와 OS-NC군)으로 나누었다. 간접 레진수복군은 산부식 처리된 상아질면에 OptiBond FL (Kerr) 또는 One-Step (Bisco) 상아질 접착제를 중합 여부에 따라 나누어 적용하였다. 처리된 상아질면에 제작된 3 mm 높이의 레진 인레이(Tescera, Bisco)를 Variolink II (Ivoclar Vivadent)를 사용하여 제조사의 지시에 따라 합착하였다. 직접 레진수복군은 산부식 처리된 상아질면에 상아질 접착 후, 복합레진 (Estelite $\Sigma$ , Tokuyama)으로 즉시 수복하였다(OB-D군, OS-D군). 수복을 마친 모든 치아는 24시간 후, 미세인장 결합 강도 평가와 공초점 레이저 주사 현미경 관찰(CLSM)을 시행하였다.

**결과:** 간접 레진수복군은 직접 레진수복군에 비하여 낮은 미세인장결합강도를 나타내었으며, 상아질 접착제를 광중합한 군(OB-C군, OS-C군)이 광중합을 시행하지 않은 군(OB-NC군, OS-NC군)에 비하여 높은 미세인장결합강도를 나타내었다. 접착 계면에 대한 CLSM 관찰에서 상아질 접착제를 광중합한 군에서 더 분명하고 균일한 혼성층이 관찰되었다. 상아질 접착제를 광중합하지 않은 군에서는 혼성층이 불분명하거나 거의 관찰되지 않았다.

**결론:** 간접 레진수복물의 합착시 레진시멘트의 적용 전에 상아질 접착제의 광중합은 분명하고 균일한 혼성층의 형성하며, 이는 결합강도의 향상에 기여한다.

**주요단어:** 공초점 현미경; 레진인레이; 미세인장결합강도; 상아질 접착제; 접착계면