An effect of immediate dentin sealing on the shear bond strength of resin cement to porcelain restoration

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PURPOSE. The aim of this study was to determine differences in shear bond strength to human dentin using immediate dentin sealing (IDS) technique compared to delayed dentin sealing (DDS). MATERIALS AND METHODS. Forty extracted human molars were divided into 4 groups with 10 teeth each. The control group was light-cured after application of dentin bonding agent (Excite® DSC) and cemented with Variolink® II resin cement. IDS/SE (immediate dentin sealing, Clearfil™ SE Bond) and IDS/SB (immediate dentin sealing, AdapterTM Single Bond 2) were light-cured after application of dentin bonding agent (Clearfil™ SE Bond and AdapterTM Sing Bond 2, respectively), whereas DDS specimens were not treated with any dentin bonding agent. Specimens were cemented with Variolink® II resin cement. Dentin bonding agent (Excite® DSC) was left unpolymerized until the application of porcelain restoration. Shear strength was measured using a universal testing machine at a speed of 5 mm/min and evaluated of fracture using an optical microscope. RESULTS. The mean shear bond strengths of control group and IDS/SE group were not statistically different from another at 14.86 and 11.18 MPa. Bond strength of IDS/SE group had a significantly higher mean than DDS group (3.14 MPa) (P < .05). There were no significance in the mean shear bond strength between IDS/SB (4.11 MPa) and DDS group. Evaluation of failure patterns indicates that most failures in the control group and IDS/SE groups were mixed, whereas failures in the DDS were interfacial. CONCLUSION. When preparing teeth for indirect ceramic restoration, IDS with Clearfil™ SE Bond results in improved shear bond strength compared with DDS. [J Adv Prosthodont 2010;2:39-45]

KEY WORDS. Immediate dentin sealing, Delayed dentin sealing, Dentin bonding agent, Shear bond strength, Ceramic restoration, Resin cement

INTRODUCTION. Immediate dentin sealing, Delayed dentin sealing, Dentin bonding agent, Shear bond strength, Ceramic restoration, Resin cement

In recent years, interests in porcelain restorations such as laminate veneer have increased as people aspire more for esthetics. Conservative tooth preparation is the major advantage of laminate veneer. However, there is a high possibility of dentin exposure when teeth are convex or crowdely aligned despite of confining to enamel surface in preparation. When dentin is exposed, applying dentin bonding agent (DBA) is necessary for retention of laminate.

In conventional dentin bonding procedure, dentin bonding agent is applied when laminate is seated to tooth in cementation. Clinically, to avoid incomplete seating of the restoration, it is recommended to maintain resin adhesive unpolymerized before laminate veneer is placed.¹² The reason is that the thickness of polymerized dentin adhesive varies from 60 - 80 μm to 200 - 300 μm depending on the structure of tooth surfaces, although less than 40 μm thickness is recommended before setting of restoration.¹³ Additionally, since the thickness of oxygen inhibition layer which plays an important role in bonding with resin reaches up to 40 μm, making dentin adhesive excessively thin may weaken the bond strength between bonding agent and resin.¹ In other words, the thick film could interfere with the complete seating of the restoration when dentin bonding agent is light-cured before placement of laminate veneer.

However, it is reported that curing dentin adhesive and resin cement individually in order showed greater bonding strength than curing both simultaneously.² ³ This comes from the fact that unpolymerized dentin-resin hybrid layer collapses during the placement of restoration.¹⁶ Therefore, new attempts have been carried out to optimize the application of dentin bonding agent.¹⁷ ²³ The procedure where adhesive is applied right before the impression taking and after tooth preparation is called immediate dentin bonding or immediate dentin sealing (IDS).¹ In addition, to demarcate conventional dentin adhesive system from IDS, it is called delayed dentin sealing (DDS).³ By carrying out IDS, restorations can be correctly placed because impression is taken after the complete polymerization of the dentin adhesive. It is reported that bonding strength is improved by protecting dentin-resin hybrid layer.¹³ ³⁸ ¹⁰⁻¹² Paul and Shärer⁰ stated that bonding strength increased...
when dentin adhesive was applied immediately after tooth preparation. In addition, the study by Magne and Douglas on bonding strength depending on different methods of adhesive application, showed that when adhesive was applied between tooth preparation and impression taking rather than right before the restoration settlement, higher bonding strength was gained. Moreover, by using adhesive containing fillers in IDS, more stable and homogeneous dentin-resin hybrid layer was acquired, resulting in improved bonding strength. Besides, it is reported that immediate IDS after tooth preparation decreases microleakage between restoration and dentin, contamination of bacteria and pulpal sensitivity.

The purpose of this study is to compare bonding strength and fracture patterns between resin cement and dentin using IDS or DDS when applying resin which is widely used for indirect esthetic bonding restoration such as laminate veneer. Additionally, bonding strength and fracture patterns for Clearfil™ SE Bond or Adapter™ Single Bond 2 in IDS were compared throughout this study.

MATERIALS AND METHODS

1. Materials

Table 1. Dentin bonding agents used in this study

| Material             | Manufacturer                     | Composition                                                   |
|----------------------|----------------------------------|---------------------------------------------------------------|
| Clearfil™ SE Bond    | Kuraray Co., Ltd., Tokyo, Japan   | Primer: dimethacrylate monomer, MDP, HEMA, water, catalyst    |
|                      |                                  | Bond: MDP, HEMA, dimethacrylate monomer, microfiller, catalyst|
| Adapter™ SingleBond 2| 3M ESPE, Seefeld, Germany         | Etchant: 35% phosphoric acid, silica thickener                |
|                      |                                  | Bond: HEMA, Bis-GMA, dimethacrylates, methacrylate, functional copolymer of polyacrylic and poly itaconic acid, ethanol, water, nanofiller |

MDP: 10-Metacryloyloxydecyl dihydrogen phosphate  
HEMA: hydroxyethyl methacrylate  
Bis-GMA: bisphenol A diglycidylether methacrylate

Table 2. Resin cement used in this study

| Material         | Manufacturer                  | Composition                                                                                     |
|------------------|------------------------------|-------------------------------------------------------------------------------------------------|
| Variolink™ II    | Ivoclar Vivadent, Schaan, Liechtenstein | Etchant (Total Etch™): 37% phosphoric acid  
Primer (Exite™ DSC): HEMA, DMA, phosphoric acid acrylate, silica, ethanol, initiators  
Adhesive Luting resin (Variolink II low viscosity)  
Base paste: Bis-GMA, UDMA, TGDMA, fillers, pigments and stabilizers  
Catalyst paste/low viscosity: Bis-GMA, UDMA, TGDMA, fillers, pigments, stabilizers and catalysts  
IPS Ceramic etching gel: 5% HF  
Monobond S: 3-methacryloxypropyl-trimethoxysilane, water/ethanol solution containing acetic acid set up to pH 4 |

HEMA: hydroxyethyl methacrylate  
DMA: dimethacrylate  
Bis-GMA: bisphenol A diglycidylether methacrylate  
UDMA: urethane dimethacrylate.  
TGDMA: triethylene glycol dimethacrylate  
HF: hydrofluoric acid
faces were reduced with Model trimmer (Osung, Kimpo, Korea) exposing more than 5 mm of dentinal layer. By attaching cellophane tapes with holes punched in uniform diameter (2.44 mm), uniform bonding area and cementing width were gained.

(2) Fabrication of porcelain specimen and surface modification
To fabricate the mold for porcelain specimen, temporary stopping (DiaDent, Cheong-ju, Korea) with diameter of approximately 2.8 mm was cut into 1.5 mm width and put into investment (Lamina vest, Shofu Inc., Kyoto, Germany). Temporary stopping was eliminated from the mold and porcelain with A1 shade (Super Porcelain EX-3, Noritake Kizai Co., Ltd., Nagoya, Japan) was inserted into the space. Then the mold was plasticized in porcelain furnace (Vacumat 40®, Vident™, Brea, USA). Investment was removed briefly by sparkling with 50 μm aluminium oxide under 3 atmospheric pressure. After spraying water on the porcelain surface from which investment material had been removed, it was polished with silicon carbide, using number 220 to 320 in regular sequence to gain width of 1.2 ± 0.2 mm. Then the porcelain specimens were stored in distilled water, rinsed by ultrasonic cleanser for 5 minutes and dried in the air. As a next step, dried porcelain samples were etched with IPS Ceramic etching gel® for one minute, and Monobond S, the type of silane composite, was applied on the samples and the samples were dried at the room temperature.

(3) The modification of dentinal surface and porcelain bonding
Forty teeth were randomly divided into 4 groups of 10 specimens each. The specimens are processed as stated in Table 3.

Regarding Group 1 as the control group, surface-treated porcelain samples were bonded to exposed dentinal surface, using Variolink® II resin cement after tooth preparation. The procedures were done by the following order: reduced dentinal surface was etched for 15 seconds using Total Etch™ and dried. Then Excite® DSC was applied on adhesive layer between dentin and the porcelain specimen and light-cured. The processed porcelain samples were bonded to dentinal surface using base paste of Variolink® II cement. While processing bonding, excess cement was removed during initial cement hardening time (2 - 3 seconds) and the sample was light-cured for 8 seconds. The distance between curing-unit and specimen during curing procedure was approximately same as thickness of the cellophane panel (circa 0.1 mm).

As for the samples in Group 2 and 3, Clearfil™ SE Bond or Adapter™ Single Bond 2 was applied on the prepared dentinal surface of each group, following manufacturer’s manual, then light-cured, then neutral Liquid Strip (Ivoclar vivadent, Schaan, Liechtenstein)-glycerine gel was applied and light cured for 10 more seconds in order to cure oxygen inhibition layer. Afterward, samples were put into thermal cycler (HA-K178, Tokyo Giken, Tokyo, Japan) and thermo-cycled 500 times for 30 seconds in 5 - 55 ℃ condition. Porcelain was cemented by Variolink® II but Excite® DSC was not light-cured at this moment.

Teeth from group 4 had no dentinal surface treatment at all and thermo-cycled 500 times like in group 2 and 3. Variolink® II was used for bonding without Excite® DSC light-curing.

(4) Measurement of shear bonding strength
After storing all teeth specimens in 37 ℃ distilled water for 24 hours, omnipotent tester (Kyung Sung Testing Machine Co., Ltd., Puchun, Korea) was used to measure bonding strength of each group. By cross-head speed with the speed of 5 mm/min, loading was applied vertically against the specimens in order to load around binding area of dentin and porcelain sample. Maximum loading (kg), which porcelain sample separates, was found out and these values were transferred into stress unit (MPa). The results from previous procedure were statistically analyzed with SPSS® Version 12.0 (SPSS Inc., Chicago, IL, USA) for Windows. One-way ANOVA was processed to

![Table 3. Classification of experimental groups](image-url)

| Group      | N  | DBA application | Thermo-cycling | Variolink II application |
|------------|----|----------------|----------------|-------------------------|
|            |    |                |                | Etching | Adhesive application | Adhesive light curing | Cementation |
| 1 (Control)| 10 | x              | x              | o       | o                    | o                  | o          |
| 2 (IDS, SE)| 10 | o (SE)         | x              | o       | o                    | x                  | o          |
| 3 (IDS, SB)| 10 | o (SB)         | x              | o       | o                    | x                  | o          |
| 4 (DDS)    | 10 | x              | o              | o       | o                    | x                  | o          |

C: Control
DBA: dentin bonding agent
IDS: immediate dentin sealing
DDS: delayed dentin sealing
SE: Clearfil™ SE bond
SB: Adapter™ Single Bond 2
define statistical significance between each group. Henceforward, Scheffe’s test was used for post-censor in the significance level of 95%.

(5) Analyzing fracture pattern
For studying fracture pattern, light microscope (SZ-TP, Olympus, Tokyo, Japan) was used with ×40 magnifications. After drying samples, they were sorted into 3 groups by fracture pattern of dentinal surface which was as following. The fracture where resin cement is removed from dentinal surface without residual debris were called adhesive failure pattern; when fracture occurs inside the resin cement - cohesive failure pattern; and lastly, the one with both previous aspects was named as mixed failure pattern. Later, significance between the groups was observed using Mann-Whitney U Test in the confidence level of 95% with significance at the 5% level.

RESULTS

1. Bonding strength

The result of bonding strength measurement is listed at Table 4. The mean bonding strength measured in group 1 is 14.86 MPa, 11.18 MPa in group 2, 4.11 MPa in group 3, and 3.14 MPa in group 4, which shows that group 1 has the highest results and group 4 - the lowest. The analysis of measured bonding strength from each group by one-way ANOVA and Scheffe’s test shows that group 1 and 2 have significantly higher bonding strength than group 3 and 4 (P < .05) (Table 5). Group 2 and 1 shows no significant difference. On the other hand, group 3 shows slight more higher bonding strength than group 4 without significance (P < .05) (Table 5).

2. Fracture patterns

Adhesive fracture occurred mostly in group 1, 2 and combined fracture in group 4 (Table 6). When they are tested with Mann-Whitney U test, significant difference can be seen between the groups-group 1 and 4, group 2 and 4 (P < .05) (Table 7).

DISCUSSION

The traditional way of bonding indirect porcelain restoration with resin cement has been carried out by delayed dentin sealing in which dentinal cementation is done with final restorations after tooth preparation, and temporary restorative period. This method, however, does not provide the optimum condition for bonding because dentin is contaminated before it is bonded and dentin-resin hybrid layer easily collapses before it is light-cured that the bonding strength between the restoration and the dentin diminishes. The bonding strength of porcelain restorations such as a laminate veneer can affect the longevity of the restoration to a considerable degree that new approach has been giving it a try in search of the optimum cementation. Bertschinge et al. and Paul et al. said the bonding strength could be improved by bonding dentin layer right after preparing the tooth and before taking the impression. Magne et al. also reported that the bonding strength could be improved more when bonding with the immediate dentin sealing after preparing the tooth than when bonding with the delayed dentin sealing. They reported that the immediate dentin sealing with direct restoration showed high bonding strength with relatively insignificant difference and that improved retention enabled a more conservative tooth prepa-
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...reported that the immediate dentin bonding recorded higher bonding strength than the delayed dentin bonding when bonding the porcelain restoration.

The immediate dentin bonding has been reported to decrease the risk of bacterial penetration and hypersensitivity of dentin.1,8,12 An experiment with the finite element analysis and the injection electronic microscope demonstrated that the immediate dentin sealing was able to withstand the heat and mechanical loading for a longer period of time and also improved the marginal adaptation between the restoration and the dentin.1

This experiment followed the immediate dentin bonding procedure as Magne et al.4 reported. As the immediate dentin sealing requires removal of the uncured layer on the adhesive interface so as to prevent an interaction between the dentin adhesive and the impression material, the same has been done for this experiment after applying Clearfil™ SE Bond and Adapter™ Single Bond 2 followed by application of Liquid Strip on top of dentin adhesive and light curing for ten seconds.

The result of the experiment demonstrated that the group 2 using Clearfil™ SE Bond showed shear bond strength with relatively insignificant difference compared to the experimental group, which showed higher shear bond strength than bonding with the delayed dentin sealing. Also, group 3 which used Adapter™ Single Bond 2 exhibited higher bonding strength than using the delayed dentin sealing with insignificant difference. The following causes could have led to such results:

First of all, dentin right after it had been prepared could have provided better condition for dentin bonding.3,14 In this experiment, the shear bonding strength demonstrated the highest bonding strength in group 1 which did not go through the heat circulation procedure, while group 4 with no surface treatment after tooth preparation showed the lowest bonding strength. Group 2 in which dentin bonding agent was applied immediately exhibited higher bonding strength than group 4. Group 2, which used Clearfil™ SE Bond for the immediate dentin sealing showed no significant difference with group 1 without contamination of the dentin after tooth preparation due to application of the dentin bonding agent after tooth preparation, supposedly had prevented the contamination of the dentin. In a recent study, Magne et al.4 said that the immediate dentin sealing could prevent the contamination of dentin while maintaining the bonding strength for 12 weeks of temporary restoration. Although this experiment involved an alternative submersion in a tank containing distilled water. However, the actual oral cavity is more exposed to contamination due to food intake that the delayed dentin bonding procedure is expected to have less bonding strength.

Secondly, the increased bonding strength of immediate dentin sealing can be explained by the fact that early polymerization of the dentin bonding agent would heighten the bonding strength. McCabe et al.6 and Dietzchi et al.5 mentioned that curing resin cement or a composite resin restoration after curing dentin bonding agent would perform better bonding strength than curing dentin bonding agent, resin cement and composite resin restoration at the same time. This is because placing the restoration or applying composite resin would exert pressure on unpolymerized dentin-resin hybrid layer beneath, causing it to collapse.6 In this experiment, all groups except for group 1, which was the control group, also bonded porcelain with cement without polymerizing Excite® DSC, dentin bonding agent of Variolink® II, and then light-cured dentin bonding agent and cement together. However, light-curing after tooth preparation followed by immediate dentin sealing increased the bonding strength that group 2 did not show any evidence of significant difference with group 1.

On the other hand, this study also compared the shear bonding strength of Clearfil SE™ Bond and Adapter™ Single Bond 2 in which Adapter™ Single Bond contained 5 nm of nanofillers. As the result demonstrates, the groups using Clearfil™ SE Bond had higher bonding strength than the groups with Adapter™ Single Bond 2. Assuming that fillers could have been the cause of such result, Jayasooriya et al.11 in previous studies reported that filler-containing Clearfil™ SE Bond would demonstrate higher bonding strength than unfilled Adapter™ Single Bond in the immediate dentin sealing. Also, Magne et al.4 stated the use of filled dentin bonding agent would form more continuous and uniform layer. Many dentin bonding agents of nowadays contain microfillers whereby adhesive resin is usually dissolved in a volatile solvent and the solvent vaporizes after application that only adhesive resin remains to be lightcured. Then, the fillers would enable a formation of the uniform layer of adhesive resin with relatively low viscosity and stabilize the dentin-resin hybrid layer.22 Also, filler-containing adhesives would have increased viscosity forming a thick adhesive layer would form and be able to absorb the internal or the external stress which might occur while curing composite resin or while in function.22 But the increase of the bonding strength due to the presence of fillers is controversial. If fillers can penetrate deep enough through collagen fibers, it can increase the compressive strength of the dentin-resin hybrid layer, and therefore maintain strong bond. Lee et al.23 also said that filler-containing dentin bonding agent would have less amount of decrease in bonding strength in an experiment simulating an environment of the oral cavity. However, when fillers cannot penetrate through collagen fibers and are clogged or remain as a bulk, it would not help in bonding. Also, if the amount of fillers exceeds 10%wt, the penetration of monomer is likely to decrease, forming vapour, and resulting in decrease of bonding strength.24 Therefore, the amount of fillers penetrating collagen fibers to stabilize the dentin-resin hybrid layer can be an influencing factor for the bonding strength of dentin bonding agent.
In this experiment, both of the dentin bonding agents contained fillers, so the difference in bonding strength depended on dentin bonding systems rather than types of fillers. As for Adapter™ Single Bond 2, a one-bottle system dentin bonding agent, which combines the use of primer and adhesive resin, it is inevitable to maintain humid environment for collagen fibers not to collapse, and for resin to penetrate after washing out the etchant. In this case, excessive drying or wetness would cause demineralized collagen to collapse, resulting in decrease in bonding strength. On the other hand, Clearfil SE™ Bond is a self-etching dentin bonding agent which simplifies the etching and priming procedure, followed by the application of the bonding agent. In this case, the primer would be acidic in nature that as far as the primer can penetrate so can the etchant, meaning that the etched layer and the resin-penetrating layer would be the same and this would prevent the dehydration of the collagen and make it easy for wet-bonding. However, in this experiment extracted teeth were used, and the application of Adapter™ Single Bond 2 had led to low bonding strength since acceptable humid environment was hard to achieve in the process of drying after etching. Tay et al. also reported that one-bottle system could be very sensitive to humidity of a tooth.

A comparison of fracture through microscopic analysis revealed that the control group and group 2 which performed the immediate dentin bonding with Clearfil SE™ Bond showed more of combined fracture status than Group 4 which carried out the delayed dentin sealing. This demonstrates that the control group and group 2 showed higher shear bonding strength with relative significance than group 4. Magne et al. also said that the immediate dentin bonding group had more bonding fractures than the delayed dentin bonding group, while McCabe et al. reported increased cohesive fractures and combined fractures during the immediate dentin bonding. In this experiment, fracture between the cement and the laminate porcelain did not occur as the porcelain had increased bonding strength by etching and silane treatment. Although there is a report mentioning that the dentin bonding agent exposed to oral cavity would absorb water and it would likely weaken the bond between the bonding agent and the ceramic, while the immediate dentin sealing was reported to have no gap in between.

This study faced some experimental limitation in a way that this experiment used posterior teeth whereas the actual cases of esthetic restoration would usually involve anterior teeth and more research is to be done. Also, the application of dentin bonding agent after extraction provided unfavorable environment for one-bottle system for wet bonding and it was hard to achieve an objective result for Clearfil SE™ Bond and Adapter™ Single Bond 2 which were using different bonding systems. Further study will have to be done using anterior tooth and dentin bonding procedure before extraction.

CONCLUSION

This study compared the shear bonding strength and the fracture of immediate dentin sealing with those of delayed dentin bonding when bonding the porcelain restoration with resin cement. The following conclusions were acquired.

1. For all groups comparing their shear bonding strength, the control group, in which dentin bonding agent was light-cured and porcelain was bonded immediately after tooth preparation, showed the highest bonding strength.
2. As for comparing the shear bonding strength of the immediate dentin sealing with that of the delayed dentin sealing: the immediate dentin sealing with Clearfil SE™ Bond demonstrated higher bonding strength than the delayed dentin sealing with significance (P < .05), while the immediate dentin sealing with Adapter™ Single Bond 2 demonstrated higher bonding strength than the delayed dentin sealing but without relative significance.
3. As for comparing the shear bonding strength according to the use of different dentin bonding agents for the immediate dentin sealing, using Clearfil SE™ Bond demonstrated higher bonding strength than using Adapter™ Single Bond 2 with relative significance (P < .05).
4. Comparing the fracture modality, the immediate dentin sealing with Clearfil SE™ Bond demonstrated more of combined fractures than the delayed dentin sealing (P < .05).

The abovementioned result shows that the bonding strength of porcelain restoration acquired is appropriate when the immediate dentin sealing is applied immediately after tooth preparation is done. However, research using anterior teeth as in actual cases and an experiment using the same dentin bonding agent mentioned above are thought to be required.

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