The comparison on micro-tensile bond strengths of variable adhesive systems to Class V cavity

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I. INTRODUCTION

With the recent developments in preventive dentistry and periodontology, senior citizens have more and more retained teeth, so the incidence of root caries and non carious cervical lesions have increased, and the demand for restoration of cervical lesions, root dentin defects such as wedge-shaped cervical defects and root caries has increased.

Resin-modified glass ionomers were introduced in 1988 by adding resin ingredients to the glass ionomer formulation. The indication area of glass ionomers diverse, as there existed conventional and resin-modified glass ionomer formulations for temporary, permanent filling, luting of indirect restorations, crowns & bridges, and brackets for orthodontic treatment, sealing of pits and fis-

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sures, and for obturating root canals.

And the recently introduced polyacid-modified resins, compomer have become popular for Class V restorations because of their fluoride release and good handling and esthetic properties. It was developed as a light curing, one component restorative with fluoride release, that combined the major benefits of both resin composite and glass ionomer chemistry. However, recent studies have revealed that the bond strength of compomer to enamel is not satisfactory, with the result being margin discoloration of the enamel.

Since Buonocore introduced the acid-etching technique, although adhesion to phosphoric acid-etched enamel is reliable and long-lasting, adhesion to dentin has been far more challenging because of the complex mineral and organic phases of dentin. The bonding mechanism of adhesive resins to dentin proposed by Nakabayashi was described as micro-mechanical due to the impregnation and polymerization in situ of monomers into exposed collagen of demineralized dentin surfaces, creating a hybrid layer.

Modern dental adhesive systems utilized two different methods to achieve micromechanical retention between resin and dentin. The first method attempted to remove the smear layer completely via acid etching and rinsing, total-etching technique. The second approach aimed at preserving the smear layer, self-etching/primer technique. Contemporary self-etching primers have been developed by replacing the separate acid-conditioning step with increased concentration of acidic resin monomers. Self-etching primers with different degrees of aggressiveness may either completely dissolve or preserve the smear layer. Despite the presence of a thin hybrid layer of about $0.5 \sim 1 \mu m$ in thickness high initial bond strength has been reported for sound dentin.

Conventional testing methods for adhesion require relatively large surface areas for adhesion, which makes it difficult to evaluate the difference of regional bond strengths. A new bond-testing procedure called micro-tensile bond strength test has been developed recently that permits the measurement of small cross-sectional bonded areas. The procedure allows the testing of irregular surfaces such as Class 1, II and V restorations. Since this method can measure the bond strength of a relatively small surfaces, it has been widely used to test different dentin substrates. In this study, this testing method was used to evaluate the regional bond strength of occlusal and gingival floors of cervical wedge-shaped cavities.

The purpose of this study was to compare the micro-tensile bond strengths and scanning electron microscopy (SEM) appearances of adhesive bonds made to occlusal vs. gingival surfaces of wedge-shaped defects of variable adhesive systems. The null hypotheses were that there was difference in the micro-tensile bond strengths of these two regions and between adhesive systems.

II. MATERIALS AND METHODS

One resin-modified glass ionomer: Fuji II LC(GC, Tokyo, Japan), one compomer: Dyract AP(Dentsply, Milford, USA), and two dentin bonding systems and one composite resin: Single Bond(3M, St.Paul, USA), SE Bond(Kuraray, Osaka, Japan), Clearfil AP-X(Kuraray, Osaka, Japan), were used. The materials, components, manufacturers used in this study listed in Table 1.

Sixty extracted sound human premolars were used for micro-tensile bond testing, and five additional teeth were used for SEM examination, which had been stored in normal saline at 4℃. Wedge-shaped cervical cavities, approximately 5mm wide, 3mm long, 3mm deep, were prepared in the buccal cervical cooling(Fig. 1-A).

The prepared teeth were randomly divided into five experimental groups with twelve teeth each. The identification of the experimental groups by adhesive systems are listed in Table 2.
Table 1. Materials used in this study

| Products                  | Main Components                                                                 | Manufacturer                      |
|---------------------------|---------------------------------------------------------------------------------|-----------------------------------|
| **Resin modified glass ionomer** |                                                                                 |                                   |
| Fuji II LC Dentin Conditioner | fluoroaluminum silicate glass, polyacrylic acid, HEMA                         | GC Co. (Tokyo, Japan)             |
| **Compomer**              |                                                                                 |                                   |
| Dyract AP Etchant         | resin, strontium-fluoro silicate glass                                         | Dentsply Caulk (Milford, USA)     |
| Prime & Bond NT Adhesive  | 36% Phosphoric acid, UDMA, PENTA, nanofiller, acetone, trimethacrylate resin   |                                   |
| Non Rinse Conditioner     | itaconic acid, maleic acid                                                     |                                   |
| **Self-etching system**   |                                                                                 |                                   |
| Clearfil SE Bond Primer   | MDP, HEMA, water, dimethacrylate, microfiller, MDP, HEMA                       | Kuraray Co. (Osaka, Japan)        |
| Clearfil SE Bond Adhesive |                                                                                 |                                   |
| **Self-priming system**   |                                                                                 |                                   |
| Single Bond Etchant       | 37% Phosphoric acid, HEMA, Bis-GMA, ethanol, water, Polyalkenoic acid copolymer | 3M Co. (St. Paul, MN, USA)        |
| Single Bond Adhesive      |                                                                                 |                                   |
| **Resin composite**       |                                                                                 |                                   |
| Clearfil AP-X Primer      | Barium glass, silicone dioxide, 3.0§ (0.1−15§), 84.5wt%                       | Kuraray Co. (Osaka, Japan)        |
| Clearfil AP-X Adhesive    |                                                                                 |                                   |

Bis-GMA = Bisphenol-A glycidyl methacrylate
HEMA = Hydroxethylmethacrylate
MDP = methacryloyloxydecyl dihydrogen phosphate

Table 2. Five experimental groups

| Group | Materials                        |
|-------|----------------------------------|
| GI    | Fuji II LC Dentin Conditioner    |
| DE    | Dyract AP Prime&Bond NT(etch)    |
| DN    | Dyract AP Prime&Bond NT(NRC)     |
| SE    | Clearfil AP-X Clearfil SE Bond   |
| SB    | Clearfil AP-X Single Bond        |

1. Specimen preparation

In the resin-modified glass ionomer, Fuji II LC(GC Co., Tokyo, Japan), the 20% polyacrylic acid dentin conditioner was applied for 10sec-ond(sec) and the prepared cavities were rinsed for 10sec and gently blot-dried. Fuji II LC(GI) was applied in the prepared cavities as the procedure recommended by the manufactures.

Compomer, Dyract-AP(Dentsply, Milford, USA), in the ‘total-etch’ protocol(DE), the cavities were conditioned with the 36% phosphoric acid gel for 15sec, rinsed for 10sec and dried briefly to keep the dentinal surfaces visibly moist. Then, the self-primed adhesive, P&B NT(Dentsply, Milford, USA) was applied onto the etched surface and evaporated the excess of solvent and wait for 20sec. In the Non-Rinse Conditioning protocol (DN), the cavity was first conditioned with the...
Non-Rinse Conditioner (Dentsply, Milford, USA) for 20sec, and then bonded with P&B NT (Dentsply, Milford, USA), without rinsing. Then, the adhesive layer was light cured for 10sec. After the bonding procedures, each cavity was restored with Dyract-AP in one increment (i.e., bulk-filled).

The self-etching primer system (Clearfil SE Bond, SE; Kuraray Co., Osaka, Japan) was applied on the cavity surface of all specimens according to the manufacturer’s instruction. The composite resin bulk-filled of Clearfil AP-X. Composite resin was light-cured (Spectrum 800: Dentsply, Milford, USA) for 40sec.

The self-priming system, Single Bond (SB, 3M Dental Products, MN, USA), the cavity was acid etched for 15sec with the 37% phosphoric acid gel, rinsed for 10sec and dried briefly to keep the dentinal surfaces visibly moist. Then, the self-primed adhesive was applied two successive coats onto the etched surface and evaporated the excess of solvent with 2–5sec air blast. Then, the adhesive layer was light cured for 10sec. Composite bulk-filled of Clearfil AP-X. Resin composite was applied as previously described.

Additional resin composite was then applied onto the buccal surface of the tooth covering the restoration and cured for mounting on the micro-tensile testing zig. This procedure is a prerequisite to provide sufficient bulk for micro-tensile bond strength testing.

2. Micro-tensile bond strength test

All prepared specimens were stored in water at 37°C for 24hrs and then embedded in the acrylic ring (Diameter-20mm, Height-15mm) with self-curing epoxy resin before testing and mounted in a cut-of assembly of slow speed diamond saw (ISOMET, Buehler, Lake Bluff, USA) for sectioning.

The bonded specimens were then serially sectioned into two slices approximately 1.0mm thick parallel to the long axis of the tooth using a low-speed diamond saw under water cooling (Fig. 1-C).

These sections were then trimmed and shaped to form an hour-glass shape with the narrowest portion at the adhesive interface using a superfine diamond point (FG #104R, Shofu, Japan) mounted in a high-speed handpiece under copious water spray (Fig. 1-D). Alternate sections were trimmed to test either the occlusal or gingival walls of each bonded specimen. The adhesive interface trimmed to a cross sectional area, which ranged from 0.95 to 1.05mm$^2$, was calculated before testing by measuring the diameter and thickness of each specimen. These specimens were then attached to the micro-tensile testing zig with a cyanoacrylate adhesive (Zapit, DVA, Lewis Cts. Corona, USA).
which, in turn, was placed in a Testing Machine (EZ-Tester, USA) for tensile testing at a crosshead speed of 1mm/min. 

3. SEM Examination

For the SEM observation of the resin-dentin interface, a cervical wedge-shaped defect was produced on each tooth in same manner as the micro-tensile bond strength test. Each cavity was treated to the bonding procedures mentioned above. The bonded samples were embedded in epoxy resin, then sectioned into two specimens, parallel to the longitudinal axis to the tooth using a low-speed diamond saw. Then the cut surfaces were ground with a series of increasingly finer silicon carbide abrasive papers and highly polished with a diamond paste. The specimens were subjected to 10% phosphoric acid treatment for 3 to 5 sec. Then specimens were rinsed with water for 15 sec and treated with 5% hypochlorite solution for 5 min. After being extensively rinsed with water, the treated specimens were air dried, gold-sputter-coated, and observed by SEM (S-2300, Hitachi Co., Tokyo, Japan) at 20kvp.

4. Statistical analysis

Overall means and standard deviations (S.D.) of the micro-tensile bond strength were calculated for each region: Occlusal wall, Gingival wall. Statistical analysis of the tensile bond strengths was performed using a one-way ANOVA and Duncan’s test and independent t-test at a 95% level of confidence (p<0.05).

Ⅲ. RESULTS

1. Micro-tensile bond strength

The mean micro-tensile bond strengths and standard deviations for each adhesive systems and cavity location are shown in Table 3.

The highest bond strengths measured to both occlusal and gingival wall were obtained with SB group (36.47 MPa and 30.20 MPa), and the lowest bond strengths were obtained with GI group (23.27 MPa and 15.09 MPa).

By comparison of the bond strengths between the adhesive systems, two dentin bonding systems (SB and SE) were higher than resin-modified glass ionomer (GI) on the occlusal wall in bond strengths (p<0.05), and they were higher than resin-modified glass ionomer and compomer (GI and DE) on the gingival wall in bond strengths (p<0.05). In the all groups, the bond strengths to occlusal wall were higher than those to gingival wall. For GI, DE and SE groups, there were statistically significant differences (p<0.05) when the bond strengths were compared between occlusal and gingival wall. But, for DN and SB groups, there were no statistically significant differences (p>0.05).

There was no significant difference to the condi-

| Table 3. Micro-tensile bond strength of experimental groups (Unit : MPa±SD) |
|---------------------|---------------------|---------------------|
| Group | Occlusal wall | Gingival wall |
|-------|----------------|---------------|
| GI    | 23.27±8.63\textsuperscript{a} | 15.09±5.10\textsuperscript{a} |
| DE    | 29.84±10.54\textsuperscript{ab} | 16.52±4.14\textsuperscript{a} |
| DN    | 29.63±9.94\textsuperscript{A} | 22.53±11.90\textsuperscript{A} |
| SE    | 36.34±11.11\textsuperscript{b} | 27.51±7.99\textsuperscript{b} |
| SB    | 36.47±13.39\textsuperscript{bA} | 30.20±12.40\textsuperscript{bA} |

Mean values with the same uppercase and lowercase superscript letters are not statistically different (p>0.05).
tioning protocol on the occlusal wall between DE and DN (p > 0.05), in contrast there was significant difference on the gingival wall in bond strengths (p < 0.05).

2. SEM Examination

The direction of the dentinal tubules for the occlusal wall was almost parallel to the interface (Fig. 5-9, A), while for the gingival wall, it was almost perpendicular to the interface (Fig. 5-9, B).

For resin-modified glass ionomer, Fuji II LC (GI) has adapted well to the conditioned dentin surface. A hybrid-like layer was formed between the resin-modified glass ionomer and dentin surface, this layer is approximately 2-5㎛ thick (Fig. 5A and 5B).

For the Compomer, Dyract-AP, in the etching protocol (DE), formed intertubular and peritubular hybrid layers 2-3㎛ thick (Fig. 6A and 6B). The resin tags exhibited the typical reverse cone shaped appearance indicating that both intertubular and peritubular dentin were decalcified. Lateral branches were also present. In the Non-Rinse Conditioning protocol (DN), a very distinct but thin hybrid layer approximately 1-3㎛ thick was observed (Fig. 7A and 7B). The quality of hybrid layer was poor showing a porous zone along the whole interface of the dentin. The resin tags were thinner and had a less funneled appearance indicating the milder decalcification caused by the etchant. Lateral branches were also formed.

For the SE group, the acidic primer removed most of the smear layer and smear plugs and demineralised the superficial layer of dentine. For occlusal wall, the thickness of the hybrid layer was about 0.5-3㎛, and some areas showed the penetration of resin tags into some tubules (Fig. 8A). For gingival wall, the thickness of the hybrid layer was uniform, at about 4-5㎛, and penetration of resin tags into lateral branches of the tubules was also observed, although they were very thin (Fig. 8B).
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Fig. 5A. SEM photograph of the adhesive interface of the GI group on the occlusal wall.

Fig. 5B. SEM photograph of the adhesive interface of the GI group on the gingival wall.

Fig. 6A. SEM photograph of the adhesive interface of the DE group on the occlusal wall.

Fig. 6B. SEM photograph of the adhesive interface of the DE group on the gingival wall.

Fig. 7A. SEM photograph of the adhesive interface of the DN group on the occlusal wall.

Fig. 7B. SEM photograph of the adhesive interface of the DN group on the gingival wall.
For the SB group to the occlusal wall, the thickness of the hybrid layer was about 5㎛, and some areas observed showed resin tags in some tubules. The hybrid layer for specimens bonded parallel to tubule axis appeared to be more uniform than that for specimens bonded perpendicular to the tubules (Fig. 9A). For the SB group to the gingival wall, a hybrid layer and resin tags could be clearly observed. The thickness of the hybrid layer was about 1~2㎛. The penetration of resin tags into lateral branches of the tubules could be observed (Fig. 9B).

Ⅳ. DISCUSSION

The bond strengths for SB and SE, two dentin bonding systems and composite resin were significantly higher than those reported for GI, resin-modified glass ionomer on the occlusal wall. While, the bond strengths for GI and DE were significantly lower than those reported for SE and SB on the gingival wall.

The resin-modified glass ionomers were developed by combining conventional glass ionomer fillers with resin composite and set by means of an acid-base reaction with polymerization of methacrylate functional groups. The mechanical
properties of these materials are superior to conventional glass ionomers but not as strong as composite resins. To improve the adhesive property, dentin conditioning with polyacrylic acid was recommended. Polyacrylic acid is a weak etchant and it removes the smear layer but does not remove smear plugs in the dentinal tubule. It may permit the HEMA(2-hydroxyethyl methacrylate) in the resin-modified glass ionomers to penetrate the collagen fiber network in the conditioned dentin and also improves the wetting and adaptation of the materials to dentin\textsuperscript{23}. When bonded to dentin, the chelation reactions occur between ions around the collagen fibers and the polyacrylic acid molecules diffuse\textsuperscript{24}. So, a collagen-glass ionomer hybrid materials may be formed. According to previously reported, ‘hybrid-like layer’ was observed in the resin-modified glass ionomer and dentin interfaces\textsuperscript{25}. In this study, Fuji II LC showed the lower bond strengths than those of other adhesive systems. But, some previous studies suggest that these materials release at least as much as fluoride as conventional glass ionomers.

And the recently introduced compomer combines the major benefits of both resin composite and glass ionomer chemistry. However, recent studies have revealed that the bond strength of compomer to the teeth is not satisfactory, with the result being margin discoloration and compomer released significantly less fluoride ions than did the resin-modified glass ionomer and dentin interfaces\textsuperscript{25}. In this study, compomers(DE and DN) show similar or lower bond strength than two dentin bonding systems with composites.

By the way, the bond strength of DN was significantly higher than those of DE on the gingival wall. It is possible that with Non-Rinse Conditioning(NRC) chemical bonding had occurred due to the presence of itaconic acid, which contains carboxylic groups that can adhere to calcium ions of the tooth, thereby contributing to the strength of the bond. Because NRC is not rinsed, the incorporation of the etching debris as fillers might have increased the bond strength\textsuperscript{29}. However, there was no significant difference between the bond strengths of DE and DN on the occlusal wall.

In some previous studies, self-etching systems showed high initial bond strengths to sound dentin despite of the presence of a thin hybrid layer\textsuperscript{13}. It is known that self-etching primer generally produces a shallow depth of demineralization than the systems with a separate etching phase\textsuperscript{26}.

The self-priming adhesive system, SB showed slightly higher bond strength than those of the self-etching system, SE in the occlusal wall but there were no significant differences among two systems.

In this study, the bond strengths to occlusal wall were significantly higher than those to gingival wall, for GI, DE and SE. There were also statistically significant difference when the bond strengths for each adhesive system were compared between occlusal and gingival wall. But, for DN and SB, there were no statistically significant difference. Phrukkanon et al.\textsuperscript{28} compared the micro-tensile bond strengths of the Single Bond and an experimental adhesives to dentin as a function of tubule orientation. They suggested that bonding of a self-etching primer to sound dentin is independent of the tubular orientation. Yoshiyama et al.\textsuperscript{12} measured the regional bond strength of LB(Clearfil Liner Bond) in natural and artificial wedge-shaped defects of extracted human teeth. They reported no significant differences between bond strength to the occlusal walls and to the gingival walls. However, Ogata et al.\textsuperscript{30} reported that the micro-tensile bond strengths of two self-etching primer systems were lower at the gingival wall than at the occlusal wall of cervical cavities. They hypothesized that these differences might be explained by the different dentinal tubule orientations.

Since reliable dentin bonding requires optimal hybridization both to intertubular and peritubular dentin, the direction and density of the tubules at the bonding site may affect the quality of the bond\textsuperscript{30}. Another factor may be the thickness of
the hybrid layer\textsuperscript{31}. With self-etching primer systems, the hybrid layer is very thin due to the relatively mild dentin demineralization. In contrast, dentin demineralization by phosphoric acid etching is deeper and the result was that hybrid layer is thicker. However, it has been suggested that the thickness of the hybrid layer is not correlated with bond strength\textsuperscript{32-35}. On the other hand, the quality of hybridization is probably the key factor for a bonding between dentin and the resin restorative. Some authors reported that a sound hybrid layer may act as a stress-absorbing layer when polymerization contraction stress loads the bonding interface\textsuperscript{35-37}. The hybrid layer of comonomers (DE and DN) contained more porous areas than two dentin bonding systems with composites, which may affect the strength of the bonded interface.

Many factors can influence the bonding of adhesive systems to dentin. These factors are the dentin substrate, the handling of the material, and the testing methods. Until recently, shear bond tests were routinely used to measure the bonding performance of adhesive systems. Such tests involve the preparation of flat surfaces of dentin with diameters ranging between 3 and 10 mm. However, large human dentin surfaces can be only prepared from crown segments of molars, and molars have great variability in dentin structure and composition. Moreover, the most convenient source of human teeth is unerupted, young third molars, which mainly consist of highly permeable dentin. Bonding procedure on such surfaces will therefore include different substrates resulting in combined bonding patterns. The micro-tensile bond strength testing method has recently been developed by Sano et al.\textsuperscript{18}. Pashley et al.\textsuperscript{38} have stated a number of potential advantages for this methodology: (1) more adhesive failures, fewer cohesive failures; (2) higher interfacial bond strengths can be measured; (3) the ability to measure regional bond strengths; (4) means and variances can be calculated for single teeth; (5) it permits testing of bonds to irregular surfaces; (6) it permits testing of very small areas; and (7) it facilitates examination of the failed bonds by scanning electron microscopy (SEM).

With this study, it became clear that bond strengths of dentin bonding systems and composite resin (SB and SE) to cervical wedge-shaped cavity were higher than those reported for resin-modified glass ionomer and compomer (GI, DE, and DN) and that the bond strengths to occlusal wall were higher than those to gingival wall.

V. CONCLUSION

This study was designed to compare on the micro-tensile bond strength ($\mu$-TBS) of variable adhesive systems to Class $V$ cavity, resin-modified glass ionomer (GI), compomer (DE and DN), and dentin bonding systems and composite resin (SE and SB). From the results of this study, it can be concluded as follows:

1. The $\mu$-TBSs for two dentin bonding systems and composite resin (SB and SE) showed higher than resin-modified glass ionomer (GI) ($p < 0.05$).

2. The bond strengths to the occlusal wall were significantly higher than those to the gingival wall in the GI, DE and SE ($p < 0.05$), while, for DN and SB. There were no statistically significant differences between the occlusal and gingival wall ($p > 0.05$).

3. There was no significant difference to the conditioning protocol on the occlusal wall between DE and DN, in contrast there was significant difference on the gingival wall ($p < 0.05$).

4. On SEM observation, the direction of the dentinal tubules for the occlusal wall was almost parallel to the interface, while for the gingival wall, it was almost perpendicular to the interface.

In this study the micro-tensile bond strength of resin-modified glass ionomer is lower than that of composite resin, when caries are thoroughly removed and the cavity is isolated from oral fluid. If the teeth restored with adhesive resin composites, they should be used with their superior
physical properties and excellent bond strengths to tooth tissue.

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