Micro-shear Bond Strength of Current Resin Cements to Enamel and Dentin

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ABSTRACT

Background & Aim: The purpose of this study was to evaluate the micro-shear bond strength of two kinds of resin-based cements with fluoride release with the usage of five adhesive systems to enamel and dentin.

Materials & methods: Bovine flat enamel or dentin surfaces were used in this study. Micro cylinders of resin cement with Xeno Cem Plus (XCP) or Panavia Fluoro Cement (PFC) were bonded to enamel or dentin as followed: XCP or PFC as instructions prior to the usage of their own adhesive systems, Xeno CF II+XCP, SE Bond+PFC, or Clearfil Liner Bond II (CLB) + PFC. After the storage in 37°C water for 24 hrs, micro-shear bond test was applied for all the test groups. The data were statistically analyzed using two-way analysis of variance (ANOVA). After two-way ANOVA, one-way ANOVA and multiple comparisons with Fisher’s PLSD test were made.

Results: Are given as follow (enamel and dentin, mean±SD in MPa); XCP, 13.7±4.0 and 15.5±9.2; PFC, 23.7±10.6 and 15.0±7.3; XCP+XCF II, 35.4±11.5 and 17.1±4.0; PFC+SE, 54.2±9.8 and 41.7±12.0; PFCCLB, 31.39±3.09 and 15.09±4.35. ANOVA (α=0.05) analyze revealed the usage of SE Bond under PFC significantly increased the bond strength to both enamel and dentin. Bonding of Xeno CFII was only improved the bonding to enamel with the application of XCP.

Conclusion: Light-cure adhesive systems significantly improve the bonding of current resin cements.

INTRODUCTION

Today, resin-based cement has an important role in adhesive dentistry. (1,2)

Current resin cement can be divided in 3 groups depending on their polymerization: chemically activated, photocured, and the “dual-cure” materials. (2) Chemically activated cements have a short working time, but their use is not limited by the thickness of resin or porcelain restorations. (2) Dual-cure resin cements have in their composition both photo-initiator and chemical activation components to achieve the best working and setting characteristics. However, it has been reported that the maximum bond strength of dual-cure type cements is achieved only when photoactivation is properly made. (2,3)

Since preventive aspect of new dentistry needs materials with protective effects, resin-based cements with fluoride release are increasingly used. Low solubility, high physical properties, reduction in micro leakage, increase the resistance of the teeth, and also positive effect of fluoride release on secondary caries are several advantages of these cements. (1,4)

However, current resin cements do not
always provide good bonding performance compared with dentin bonding systems with direct composite. (5) Recently, a resin coat techniques has been developed, which enable the coverage and protection of prepared tooth surface immediately after cutting, and provide high bond strength of resin cement with light cure bonding systems. (6) Therefore, with acceptance of shear testing in the literature, (7,8) this study was designed to investigate the bond strength of two kinds of resin base cement (chemically activated and dual cure), with fluoride release and usage of five adhesive systems to enamel and dentin.

**MATERIALS & METHODS**

Materials used in this study were listed in table 1.

One hundred and thirty (130) mandibular bovine teeth kept frozen in distilled water for no more than three months were used in this study. Before the bonding procedure, the crown were cleaned and placed under the running water for 24 hrs.

**Table 1. Materials used.**

<table>
<thead>
<tr>
<th>Materials</th>
<th>Manufacture</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zeno Cem Plus</td>
<td>Sankin</td>
<td>Fluoride releasing adhesive cement Powders (Lot no. 357-103)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Liquid (Lot no. 357-708)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>CEM Plus Primer (Lot no. 357-907)</td>
</tr>
<tr>
<td>Xeno CF II Bond</td>
<td>Sankin</td>
<td>Light cure fluoride releasing bonding agent Universal (Lot no. 357-01)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Catalyst (Lot no. 357-02)</td>
</tr>
<tr>
<td>Panavia Fluoro Cement</td>
<td>Kuraray</td>
<td>Dual cure dental adhesive system MDP. Bis phenol A. silanated silica</td>
</tr>
<tr>
<td>A paste</td>
<td></td>
<td>Benzoyl peroxide. Photo initiator (Lot no. 00067A)</td>
</tr>
<tr>
<td>B paste</td>
<td></td>
<td>Bisphenol A barium glass. Sodium fluoride Aromatic sulfins (Lot no. 00038 A)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ED Primer II A (Lot no. 00128B)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ED Primer II B (Lot no. 00015 C)</td>
</tr>
<tr>
<td>Clearfil SE Bond</td>
<td>Kuraray</td>
<td>Primer : water. MDP. HEMA. Hydrophilic dimetaclylate (Lot no. 00207 A)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Bond : MDP. Bis-GMA. HEMA. Hydrophilic diMetaclylate. colloidal silica (Lot no. 00211 A)</td>
</tr>
<tr>
<td>Clearfil Liner Bond</td>
<td>Kuraray</td>
<td>Primer : Liquid A (Lot no. 00086 C)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Bond II Liquid B (Lot no. 00086 A)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Water. MDP. HEMA. Hydrophilic diMetaclylate. Bond : Liquid A (Lot no. 000147 A)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Liquid B (Lot no. 00015 A)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MDP. Bis-GMA. HEMA. Hydrophilic diMetaclylate. Coloidal silica</td>
</tr>
</tbody>
</table>

**Shear bond test**

One hundred (100) teeth were chosen from 130 teeth and flat enamel or dentin surfaces were prepared on labial surfaces by wet grinding using 600 grit silicon carbide papers. For dentin bonding site, care was taken to ensure that only super facial dentin was exposed. The enamel and dentin surfaces were randomly divided into 5 subgroups of ten samples each and following categories were applied:

Group 1: apply Xeno Cem Plus (XCP) Primer\(^1\) on enamel for 20 second as instruction.

Group 2: like group 1, but on dentin.

Group 3: apply the mixing of universal and catalyst of Xeno CF II Bond1 on enamel for 20 second, dry and light-cure for 10 second with Optilux 500\(^2\).

Group 4: like group 3, but on dentin.

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\(^1\)Sankin. Tokyo. Jaman

\(^2\)Kerr. Orange. CA. USA
Group 5: apply the mixing of ED Primer A and B of Panavia Fluoro Cement (PFC) on enamel for 30 seconds and air drying.

Group 6: like Group 5, but on dentin.

Group 7: apply SE Primer on enamel for 20 seconds, dry and apply SE Bond3, air thinning and light-cure for 10 seconds.

Group 8: like Group 7, but on dentin.

Group 9: after apply the mixing of primer A and B of Clearfil Liner Bond II (CLB II) on enamel for 30 seconds, followed by drying and application of mixed Bond A and B and air thinning.

Group 10: like group 9, but applying on dentin.

One of the freshly mixed resin cement, XCP or PFC, were then used to fill an iris that was cut from micro bore tygon tubing (TYG_030) with an internal diameter of 0.80mm and a height of approximately 0.5mm. In this manner, very small cylinders of cement approximately 0.80 mm diameter and 0.50mm in height were bonded to surface of samples. For groups 1, 2, 3 and 4, XCP was used to make the cylinders and protected by Oxy Guard for 4 minutes. For groups 5, 6, 7, 8, 9, and 10, PFC was used and cured for 20 seconds and protected by Oxy Guard for 4 minutes.

After 24 hours storage in water at 37°C incubator, tygon tubing was removed and the tooth with the cylinder was adhered to micro-shear testing device (Bencor-Multi-T) with a cyanoacrylate adhesive (Zapit) which in turn was placed in a Universal testing machine (Ez-test-500N) for shear bond testing (Fig 1). Then a thin wire (diameter 0.20mm) was looped around the resin cylinder, making contact through half its circumference and was gently held flush against the cement/tooth interface. Shear force was applied to each specimen at a crosshead speed of 1.0 mm/min until failure occurred.

The data were statistically analyzed using two-way analysis of variance (ANOVA). After two-way ANOVA, one-way ANOVA and multiple comparisons with Fisher’s PLSD test were made. The adhesive systems used and tooth substrates (enamel and dentin) were the two factors. Statistical significance was defined as P<0.05.

SEM observation

Following shear testing, all fracture interfaces were examined under stereomicroscope and scanning electron microscope (JSM-5310LV) to determine the mode of fracture. Also remaining 20 teeth were randomly assigned to 10 groups of 2 teeth each and were bonded same as the shear bond testing and prepared for interfacial analyzes with SEM. Then sectioned for three to four cut across the bonding surface with diamond saw approximately 2mm samples embedded in epoxy resin and polished with increasingly fine diamond pastes of decreasing particle size down to 0.25µm. The polished surfaces were cleaned ultrasonically, air dried and placed in desiccators for 24 hours then were subjected to argon-ion beam etching (EIS-1E) for 10 minutes, under operating conditions of an accelerating voltage of 1kV and ion current density of 0.2 mA/cm², with the ion beam directed perpendicular to the polished surface, the surface were then sputter coated with gold and observed under SEM.

Another ten bovine enamel or dentin surfaces were prepared to observe the etch pattern of primers and assigned for each test.
groups. The primers were applied in the same manner as employed in ten groups without bonding or curing process. For removing of primer and any other residual material, the specimens were rinsed with acetone for 2 minutes and after drying process coated with gold and seen with use of SEM.

### Table 2. Micro-shear bond strength (MPa) and statistical analyzing results.

<table>
<thead>
<tr>
<th></th>
<th>Enamel Mean (SD)</th>
<th>N</th>
<th>Dentin Mean (SD)</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>XCP</td>
<td>13.66 (3.97)</td>
<td>10</td>
<td>a</td>
<td>15.53 (9.19)</td>
</tr>
<tr>
<td>XCP + XCF</td>
<td>35.40 (11.50)</td>
<td>10</td>
<td>b</td>
<td>17.06 (4.01)</td>
</tr>
<tr>
<td>PFC</td>
<td>23.73 (10.57)</td>
<td>10</td>
<td>a</td>
<td>15.00 (7.26)</td>
</tr>
<tr>
<td>PFC + SE</td>
<td>54.21 (9.77)</td>
<td>10</td>
<td></td>
<td>41.73 (12.0)</td>
</tr>
<tr>
<td>PFC + CLB</td>
<td>31.39 (3.09)</td>
<td>10</td>
<td>b</td>
<td>15.09 (4.35)</td>
</tr>
</tbody>
</table>

Groups with same letter did not show significant difference

**RESULTS**

**Shear bond test**

The mean shear bond strength and standard deviations in MPa are shown in Table 2. ANOVA analyze revealed that there was a statistically significance of the obtained bond strength values between the groups. The usage of SE Bond significantly increased the bond strength of PFC to both enamel and dentin. Xeno CFII only improved the bond to enamel of Xeno Cem Plus and the effect on dentin was not obvious.

**SEM observations.**

SEM photomicrographs of the sheared test sites for the highest and lowest groups are shown in Fig.2. Almost all groups showed adhesive fractures in adhesive substrate interface. However, no statistically significant differences of mode of failure were found among the bonding systems used. In groups 3, 7, 8, and 9, a few samples were mixed with cohesive in substrate. Especially, after the shearing of XCP with its own primer showed cement particles remained on both enamel and dentin (Figs.2c,d).

Enamel or dentin surfaces after priming are shown in Fig3. The application of self-etching primer almost removed the smear layer from the surface of both enamel and dentin Fig3. Nonetheless, SE Bond Primer and Liner Bond II primer showed less demineralization of enamel or dentin than ED Primer or Xeno CF II.

Adhesive interface between the resin cement and enamel/dentin are shown in Fig. 4. When Xeno CF II was applied to dentin, the etched dentin was observed with the thickness ranged 3-4µm in depth (Fig4f). While the etching of XCP Primer was not so evident and etched dentin was approximately 2µm in depth. In SE Bond or CLB II system, the effect of etching was not clear.

In the case of enamel-cement interface, the formation of etched enamel tag for micromechanical retention of resin was not evident in all the systems used (Fig4).
Fig2c. Xeno cem plus mode of fail × 100.

Fig2d. Xeno cem plus mode of fail × 500.

Fig3a. ED Primer 30 second etch pattern in enamel.

Fig3b. ED Primer 30 second on dentin. Note smear was removed.

Fig3c. Xeno Cem plus primer 20 second etch pattern in enamel.

Fig3d. Xeno CF II bond 20 second etch pattern in enamel.

Fig3e. Clearfil liner bond II mixed primer 30 second etch pattern.

Fig3f. Clearfil liner bond II mixed primer 30 second on dentin. Note smear was removed.
DISCUSSION

In this study bovine instead of human teeth were used because they provide larger and more consistent samples, especially in dentin.\(^\text{(10,11)}\) With improvement of cohesive strength of adhesive resin, we can apply the shear force to resin cylinder without any interface.\(^\text{(7,12)}\) Since other reports about failure analysis revealed mixed or cohesive fracture near both interface of cement\(^\text{(13)}\) or even adhesive failure near the dentin with indirect restorations and adhesive cement.\(^\text{(14)}\)

The results of the shear bond testing indicated that SE Bond as a light-cure self-etch primer system significantly increased the bond strength of PFC to both dentin (41.73MPa) and enamel (54.21MPa). The result of SE Bond is approximately equal with several studies, done with micro-shear test,\(^\text{(15,16)}\) but is higher than the other test results.\(^\text{(17,18)}\) The discrepancy is probably because of the difference on the test method of shear measurement.

Our interfacial image of SE Bond showed 0.5 to 1\(\mu\)m hybrid layer on dentin surface (Fig4e), which is consistent with another observation\(^\text{(19)}\) with PFC and SE Bond. Nonetheless, it has been reported that bond strength do not related with hybrid layer thickness.\(^\text{(20)}\) The reason of high bond strength values of SE Bond might be because the sufficient penetration of resin monomer into the etched dentin,\(^\text{(21)}\) as the quality of hybrid layer, rather than thickness, is believed to be the most important factor for obtaining high bond strength.\(^\text{(22)}\)

It was considered that, the usage of a light-cure self-etch primer system significantly enhanced the bond strength of resin cement. Regardless of the compositional similarity of SE Bond and CLB II, SE Bond significantly improved the bonding of PFC. It is probably because the rate of polymerization in light-cure resin is more than that of the self-cure, in accordance with previous studies.\(^\text{(23,24,25)}\)

Another variable that must be considered is the effect of difference of substrate as an adherent on bonding. It means that the bonding to enamel of SE Bond, even with the acidity of this primer is lower than current phosphoric acid gel, is significantly higher than to dentin, which is accepted by other research \(^\text{(17,26)}\). Also some type of self-etching primers show the formation of hybrid like layer of enamel and resin\(^\text{(27,28)}\) or micro-tags,\(^\text{(29)}\) that seemed to contribute to effective bonding to enamel.

Nakaoki et al reported that 27.8MPa of micro-shear bond strength to dentin of Xenon CF Bond system. Even though our results are lower than previous study, this gap would be because of difference of material used, as previous study used resin composite whereas this study used resin cement, XCP.\(^\text{(16)}\)

The enamel bonding of XPC was significantly increased with the application of XCF II (35.4MPa), where no effect of XCF II was seen on dentin bonding. It is highly likely that the etching effect of XCF II is enough in enamel bonding (Fig3d), whereas lower bond in dentin due to specific properties of dentin such as tubular structure and its intrinsic wetness bonding to dentin seem to be more difficult to achieve and inconsistent than bonding to enamel.\(^\text{(30-33)}\)

SEM findings showed that XCP exhibit high porosity (Fig4d) whereas PFC did not. This is probably because the former is a powder-liquid mixture, while the latter is a paste-paste mixture. The pore may act as stress concentrators, resulting in lower bonding of XCP.\(^\text{(34)}\)

Interfacial images of XCP and PFC showed de-bonding from dentinal surface (Figs4a, b) that indicated the low bond for dentin or cohesive fracture in adhesive (Fig4a).
Fig4a. Xeno cem plus (de-bonding) cohesive fracture in primer.

Fig4b. Panavia fluoro cement with ED Primer (de-bonding) adhesive fracture.

Fig4c. SE bond enamel (proper adaptation).

Fig4d. Xeno CF II bond enamel (proper adaptation). Note voids in Xeno Cem plus.

Fig4e. SE bond dentin. Note 0.5 to 1 µm hybrid layer on dentinal surface.

Fig4f. Xeno CF II bond dentin. Note approximately 3 to 4 µm hybrid layer on dentinal surface.

With the results of this research and profits of resin coat technique, light curing of adhesive for resin cement can be used instead of self-cure in order to increase the bonding on tooth surface. However, additional in vitro or in vivo tests such as various cementing time, microleakage and durability and clinical trial were needed in order to effective bonding technique.

CONCLUSION

Within the limitation of this in vitro study, the following conclusions were drawn:
1. The effect of chemical-cure self-etch primer system on bonding of resin cement was lower than that of the light-cure system.
2. SE Bond significantly increased PFC bond to dentin and enamel, compared with instructed system in the package.
3. Xeno CF II Bond increased only Xeno Cem Plus bond to enamel.
REFERENCES


