Effect of Water Storage on the Micro-shear Bond Strength of Two Self-etch Adhesives to Enamel and Dentin

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Abstract:
Objective: This study evaluated the influence of storage time on micro-shear bond strength of two self-etching materials to enamel and dentin.

Materials and Methods: Human third molar teeth were sectioned to 1.5 mm thick beams and randomly divided into 2 groups. In group I, SE Bond and in group II, Tri-S Bond was used to bond a composite rod (AP-X) to each treated surface. Specimens were prepared according to manufacturer instructions. Each group was further divided into three subgroups according to water storage time; 1 day, 6 and 12 months. Micro-shear bond strengths were determined under a crosshead speed of 1mm/min using a universal testing machine and expressed in MPa. Data was statistically analyzed by ANOVA and Dunnett post hoc test.

Results: Micro-shear bond strength of two adhesives to enamel and dentin showed a slight but not significant decrease over time (P>0.5). After one day, the mean bond strength of enamel in groups I and II were 39.47 and 34.65 MPa and in dentin were 45.20 and 36.0 MPa respectively. There was no statistically significant difference between two materials (P=0.190, P=0.082). After six months the bond strength in group I and II was 35.93 and 35.18 MPa for enamel, and 38.27 and 35.19 MPa for dentin respectively, these differences was not statistically significant (P=0.520, P=0.179). After one year, the bond strength of enamel in groups I and II, were 34.47 and 29.91 MPa, and in dentin were 33.86 and 32.53 MPa respectively which was not statistically significant (P=0.609, P=0.991).

Conclusion: The micro-shear bond strength of both adhesives to enamel and dentin decreased slightly over time; however these decreases were not statistically significant.

Key Words: Self etch adhesive; Durability; Micro-shear bond strength; Enamel; Dentin

INTRODUCTION
In the past two decades, the traditional mechanical retention methods for restorative materials have been replaced by tooth-conserving adhesive methods. The ultimate goal of a dental bonding system is to achieve a durable bond between restorative materials and enamel or dentin [1]. In the past decade, many dental adhesives have been commercialized with the aim of increasing the bonding efficacy and simplifying the bonding process. The durability of these bonds between adhesive resins and tooth structures are of critical importance for the longevity of bonded restorations. Most of the currently marketed adhesive resins achieve high bond strength immediately after polymerization of resin. The longevity of this bond is an important criterion to determine
their effectiveness; and adhesive bonds durability is one of the areas of current interest in adhesive dentistry. Several studies demonstrated a decrease in bond strength after one year water storage [2-4], some others reported no significant changes [5,6]; however, in one study an increase in bond strength was observed [7].

The aim of this study was to evaluate the influence of water storage on micro-shear bond strength of two self-etching adhesive system to enamel and dentin.

MATERIALS AND METHODS
In this experimental study, two self-etching adhesive systems were evaluated. Details of composite resin and two adhesive systems used in present study are listed in Table I.

For evaluating the micro-shear bond strength, extracted human third molars were selected and stored in normal saline solution. After cleaning and disinfection by 0.05% thymol solution, teeth were sectioned (mesiodistally) to 1.5 mm thick slices with a low speed diamond saw (Buehler Ltd, Lake Bluff, IL), these slices were ground flat with 600-grit silicon carbide sandpapers under water coolant. All specimens were randomly divided into two groups; in group I (n=30), Clearfil SE Bond (CSEB) was applied, and the specimens in group II (n=30) were treated with Clearfil Tri S Bond (CTSB) according to the manufacturer’s instruction.

Clearfil AP-X resin composite was bonded to the treated surfaces using hollow Tygon tubes (0.75×1mm, Tygon, Norton performance Plastic, Co. Cleveland, OH, USA) to make a cylinder, and was light cured for 40 seconds. All specimens were stored in water at 37°C, and were further divided into three subgroups according to the water storage time: 24 hours, 6 and 12 months storage for subgroups 1, 2 and 3 respectively. Teeth in each group were evaluated under a stereomicroscope (Olympus, Tokyo, Japan) for possible defects. Specimens which showed apparent defects were excluded and were substituted to ensure of 10 specimens in each group.

The micro-shear bond strength values for each group were determined using a universal testing machine (EZ-test 500N, Shimazu CO, Kyoto, Japan) at a crosshead speed of 1mm/min. The maximum load at the time of failure was reported as Newton, and then bond strength was calculated in MPa.

All the fractured surfaces were examined under a stereomicroscope (Olympus, Tokyo, Japan) at ×30 magnifications to determine the mode of failure, and were classified as adhesive, cohesive and mixed failures.

Collected data were statistically analyzed by two-way and one-way ANOVA and Dunnett post hoc test.

RESULTS
Descriptive statistics of micro-shear bond strength of each adhesive group to enamel and dentin after 24 hours, 6 months, and one year are summarized in Table II.

The results of the study showed that mean

<table>
<thead>
<tr>
<th>Table I: The Materials used in this study.</th>
<th>Manufacture</th>
<th>Compositions</th>
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<tbody>
<tr>
<td>Clearfil SE Bond</td>
<td>Kuraray Medical Inc., Tokyo, Japan</td>
<td>Hydrophilic dimethacrylate, N, N-diethanol P-toluidine, di-Camphorquinone, N, N-diethanol P-toluidine, silanated colloidal Silica, MDP(10-methacryloyloxydecyl dihydrogen phosphate), HEMA Water, MDP, Bis-GMA, HEMA, hydrophobic, dimethacrylate, di-Camphorquinone, Ethanol, Water, Silanated, Colloidal silica.</td>
</tr>
<tr>
<td>Clearfil Tri - S Bond</td>
<td>Kuraray Medical Inc., Tokyo, Japan</td>
<td>Water, MDP, Bis-GMA, HEMA, hydrophobic, dimethacrylate, di-Camphorquinone, Ethanol, Water, Silanated, Colloidal silica.</td>
</tr>
<tr>
<td>Clearfil AP-X</td>
<td>Kuraray Medical Inc., Tokyo, Japan</td>
<td>hybrid Light-curing Composite</td>
</tr>
</tbody>
</table>
bond strength of two adhesives to enamel and dentin has a slight but not significant decrease over time (P>0.5).

Although after one day water storage, the mean bond strength for enamel and dentin in group I was more than that in group II, the difference was not statistically significant (P=0.190, and P=0.082 for enamel and dentin respectively).

After 6 months, the micro-shear bond strength for enamel and dentin in group I was more than group II, however these differences was not statistically significant (P=0.529, and P=0.179 respectively).

Micro-shear bond strength measurement after one year water storage for enamel and dentin showed higher values for group I. These differences was not statistically significant between the two groups (P=0.609, and P=0.991 respectively).

The fracture modes are presented in Table III and included adhesive, cohesive and mixed failures. The failure modes for CSEB specimens included all three patterns. In the CTSB group the fracture mode was mostly adhesive failure. Comparison of the failure mode of the two adhesives systems in enamel and dentin shows that this difference was statistically significant (P<0.036, and P<0.001 respectively).

DISCUSSION
The purpose of this study was to evaluate the micro-shear bond strength of two self-etch adhesives to enamel and dentin after storing in water for one-day, 6-months and one-year. Clearfil SE Bond is a mild two-step self-etching adhesive (pH=2). In this system collagen fibrils are not completely deprived of hydroxyapatite. This residual hydroxyapatite may then chemically interact with specific carboxyl or phosphate groups of functional monomers [1,8]. Theses monomers may be able to interact more intimately with hydroxyapatite-coated collagen that otherwise would have almost completely lost its hydroxyapatite coating with a rather aggressive total-etching technique [6,8,9]. Clearfil Tri-S Bond is a new product. It is one-step self-etching adhesive, which is made for simplifying the procedure and lessen the application time.

The micro-shear bond strength test was chosen due to its advantages compared to other bond strength evaluation methods and its relative easiness of sample preparation and suitability for enamel bonding tests [10-12].

Results of the present study showed that mean bond strength of two adhesives to enamel and dentin has a slight but not significant decrease over time, which was similar to other studies [13-15].

The application of a self-etching primer on enamel results in a shallow etching pattern. This might be due to the mild decalcification effect of self-etching primer or the etching might decrease gradually as a result of neutral-

<table>
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<tr>
<th>Table II: Mean and standard deviation (values in parentheses) of shear bond strength (MPa) of two adhesive systems.</th>
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<tbody>
<tr>
<td>CSEB</td>
</tr>
<tr>
<td>Enamel</td>
</tr>
<tr>
<td>---</td>
</tr>
<tr>
<td>24h ours</td>
</tr>
<tr>
<td>39.47 (5.34)</td>
</tr>
<tr>
<td>6 Months</td>
</tr>
<tr>
<td>35.93 (7.13)</td>
</tr>
<tr>
<td>One year</td>
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<td>34.47 (8.44)</td>
</tr>
</tbody>
</table>

Table III: Modes of failure of two adhesive systems after 24 hours, six months and one year water storage (A=adhesive, C=cohesive and M=mixed failures).

<table>
<thead>
<tr>
<th>Material</th>
<th>Enamel</th>
<th>Dentin</th>
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<tbody>
<tr>
<td>CSEB 24 hours</td>
<td>4</td>
<td>3</td>
</tr>
<tr>
<td>CTSB 24 hours</td>
<td>7</td>
<td>1</td>
</tr>
<tr>
<td>CSEB 6 Months</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>CTSB 6 Months</td>
<td>5</td>
<td>3</td>
</tr>
<tr>
<td>CSEB One year</td>
<td>7</td>
<td>3</td>
</tr>
<tr>
<td>CTSB One year</td>
<td>4</td>
<td>4</td>
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</tbody>
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lization of the acidity of primer on the enamel surface [16-18], or it may be a result of selective decalcification of the interprismatic region due to weak acidity of self-etching primer [18]. Decrease in bond strength in enamel after one year storage could also be due to formation of shallow resin tags [16]. In addition, water diffusion into the bonding interface may result in composite resin swelling and plasticizing of the resin [16]. The primer agent of the CSEB system includes a hydrophilic acidic monomer of MDP as the functional monomer, HEMA, DMA, and photo initiator dl-camphorquinone, while the bonding agent contains filler and bis-GMA in addition to primer ingredients, except for the water. The all-in-one system, CTSB contains all the aforementioned components in a single bottle. Acidic monomers are the main ingredients of the self-etching primer systems and besides participating in the polymer network, have various functions which contribute to the bonding performance to the dentin and enamel substrates [1,19].

The etching effects of both systems used in the present study are due to the acidic effect of the water-MDP solution. MDP is also responsible for the dentin priming through wetting the dentinal collagen network via infiltration into the dentin [19]. Moreover, MDP has been reported to chemically interact with the hydroxyapatite and form low soluble calcium salts [19]. Many researchers have studied the hydrolytic stability of acidic monomers. In acidic methacrylate phosphate MEP (methacryloxy ethyl dihydrogen phosphate), both the methacrylate and phosphate ester bonds were hydrolyzed and resulted in the formation of methacrylic acid (MAA), phosphoric acid or other derivatives. The phosphate ester bond in MDP was found to be significantly more stable than MEP that has a shorter alkylene spacer [19]. Salz et al [19] observed a significant reduction in the concentration of the MDP in an aqueous solution after 16 weeks of storage at 37 °C.

The results of present study is also in agreement with the results of studies conducted by Sano et al [2], Okuda et al [5], and, Steven et al [7], which showed a decrease in bond strength of self-etch adhesives after long-term storage. Most of which hypothesized that a decrease in bond strength over long period of time is due to hydrolytic degradation of ester bonds of polymerized resin within the hybrid layer which gradually increase as water diffused through nano-leakage channels which become larger over the time, resulting in lower bond strengths and interfacial failure [5]. Fracture modes results showed that the mode of failure for CSEB specimens included all three various patterns. The fracture modes in CTSB specimens were mainly classified as adhesive failure, especially in dentin substrate which all specimens showed adhesive failure over one year water storage. The results are in agreement with Wang et al [16] that the fracture toughness of adhesive may decrease.

CONCLUSION

Within the limitations of this study, it was concluded that although the micro-shear bond strength of both adhesives to enamel and dentin seems to decrease slightly within time, the difference was not statistically significant.

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