Influence of environmental conditions on orthodontic bracket bonding of self-etching systems

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The influence of environmental conditions on the bond strength of orthodontic adhesives was investigated. Two self-etching/composite type, one acid-etching/composite type, and one acid-etching/PMMA type of adhesives were examined under different temperature and RH conditions. Orthodontic brackets were bonded to bovine enamel, and shear bond strength test was performed at a crosshead speed of 1.0 mm/min after 24-hour storage in 37°C water. Data were analyzed by Tukey’s HSD test. Each specimen was assigned an ARI score. All the materials tested exhibited their highest bond strength under room conditions at a range of 10.4–17.0 MPa. Conversely, when under a lower RH condition, bond strength for all the systems ranked the lowest within a range of 6.8–12.0 MPa, with bond failure at the bracket–adhesive interface. These results demonstrated that care should be exercised when using orthodontic adhesive systems in the oral environment.

Key words: Bracket bonding, Oral condition, Bond strength

INTRODUCTION

Considerable efforts have been made to develop restorative materials that bond to dentin in the oral environment without requiring complicated clinical procedures. Since the initial introduction of phosphoric acid as an etching solution for enamel bonding, several other agents have been investigated for potential applications in clinical dentistry. One such example is a recently developed self-etching primer, which contains acidic functional monomers that demineralize the enamel surface while simultaneously improving the resin monomer penetration. Using this approach, an orthodontic adhesive is then applied to the porous surface to create a stable bond between the orthodontic bracket and the demineralized enamel. This combined etching and priming procedure has allowed clinicians to avoid technique-sensitive factors that may influence bonding efficacy. Moreover, with simplified clinical procedures, patient comfort is improved.

The screening of orthodontic adhesive materials is usually carried out by in vitro adhesion testing and microleakage evaluation under ambient conditions of 23°C and 50% relative humidity (RH). However, environmental factors are known to have an important influence on the bonding strength of adhesive systems used for orthodontic restorations. Resin composites are thought to be sensitive to environmental moisture, which has been shown to decrease bond strength. In a high-humidity environment (35°C and 95% RH), Plasmans et al. found that the influence of extrinsic dentin wetness differed among bonding systems, with some being more sensitive than the others. The presence of moisture, whether from intrinsic or extrinsic sources, has a crucial effect on the bonding of orthodontic brackets to the enamel surface. Although the use of a rubber dam can protect the tooth surface from extrinsic moisture, this approach is not practical during orthodontic treatment.

The current study investigated the influence of temperature and RH on the enamel bond strength of orthodontic adhesives including self-etching systems. The hypothesis tested was that environmental conditions have no significant effect on the shear bond strength of orthodontic adhesives. The parameters investigated were the shear bond strength and site of bond failure according to the adhesive remnant index (ARI).

MATERIALS AND METHODS

Materials used

The four orthodontic adhesives used in this study were Beauty Ortho Bond (Shofu Inc., Kyoto, Japan), Transbond Plus Self Etching Primer (3M Unitek, Monrovia, CA, USA), Kurasper F (Kuraray Medical, Tokyo, Japan), and Superbond Orthomite (Sun Medical, Moriyama, Japan), as shown in Table 1. All orthodontic adhesive systems were used according to the respective manufacturer’s instructions.
Japan) used in this study had a surface area of 15.56 mm². An Optilux 501 (SDS Kerr, Danbury, CT, USA) curing unit was employed, with a light intensity of 800 mW/cm² measured using a Model 100 dental radiometer (SDS Kerr).

Tooth specimen preparation
Mandibular incisors from cattle aged 2–3 years, which had been stored at −20°C for up to two weeks after extraction, were used as substitutes for human teeth. After removing the roots with an Isomet low-speed saw (Buehler, Lake Bluff, IL, USA), the pulp was extracted and the pulp chamber of each tooth was filled with cotton to avoid penetration of the embedding medium.

Each tooth was mounted in a self-curing acrylic Tray Resin II (Shofu Inc., Kyoto, Japan). To facilitate the bonding procedure, the labial surfaces of the bovine incisors were exposed whereby they were positioned level with, and parallel to, the ring of the mounting aluminum mold. They were then placed in tap water to reduce the temperature increase caused by exothermic polymerization. Final finish was accomplished by grinding the tooth on wet 600-grit silicon carbide (SiC) paper to obtain a flat surface. After ultrasonic cleaning with distilled water for three minutes to remove excess debris, the surfaces were washed and dried with oil-free compressed air.

Bonding procedure
The embedded teeth were transferred to a controlled temperature and humidity chamber (Type V-85, Atom Corp., Tokyo, Japan). As the temperatures of the samples differed from those inside the chamber, they were left for 10 minutes to equilibrate to the four different sets of environmental conditions tested (25 ± 1°C and 50 ± 5% RH; 25 ± 1°C and 95 ± 5% RH; 37 ± 0.5°C and 50 ± 5% RH; and 37 ± 1°C and 95 ± 5% RH).

For Beauty Ortho Bond and Transbond Plus adhesives, a mixed solution of the primer was applied to the enamel surface with a disposable applicator, left for three seconds, and then dried with oil-free compressed air. The paste was applied to the base of the metal bracket and then pressed firmly onto the tooth surface. Excess paste was removed from around the base of the bracket, and the adhesive was light-cured for 10 seconds on each interproximal side.

For the Kurasper F adhesive, the enamel surface was etched with 40% phosphoric acid gel for 30 seconds, washed for 20 seconds, and then dried with oil-free compressed air. The bonding agent was applied and light-cured for 10 seconds. The brackets were then bonded with paste and light-cured for 20 seconds on each interproximal side.
For the Superbond Orthomite adhesive, the enamel surface was etched with Red Activator for 30 seconds, washed for 20 seconds, and then dried with oil-free compressed air. A partly oxidized tri-n-butyl borane (TBB) catalyst was added to activate the monomer liquid. The polymer powder and activated monomer liquid were then mixed. The resulting mixture was used to bond the metal brackets to the enamel via the brush-dip technique.

**Shear bond strength test**

After bonding, the finished specimens were transferred to distilled water at 37°C and stored for 24 hours. The samples (10 per group) were tested in a shear mode with a Type 4204 universal testing machine (Instron Corp., Canton, MA, USA) at a crosshead speed of 1.0 mm/min. Shear bond strength values (MPa) were calculated based on the peak load at failure divided by the bracket surface area.

**Statistical analysis**

For each material, the mean bond strength and standard deviation (SD) values were subjected to Tukey’s honestly significant difference (HSD) test to identify significant differences among the environmental conditions tested. All statistical tests were performed at 5% significance level for each adhesive system.

**Bond failure analysis**

After shear bond strength test, each specimen was examined under an SZH-131 optical microscope (Olympus, Tokyo, Japan) at a magnification of ×8 to identify the bond failure location using fiber optic transillumination. An analysis of the residual adhesive on the tooth surface was performed visually after shearing the bracket. Each specimen was assigned an ARI score according to the amount of adhesive remaining on the tooth surface as follows: 0 = no adhesive remaining; 1 = less than 50% of the adhesive remaining; 2 = more than 50% of the adhesive remaining; and 3 = all of the adhesive remaining with a distinct impression of the bracket base. The Chi-square test was used to analyze differences in the distribution of the ARI scores.

Following shear bond strength measurements, fractured specimens were dehydrated in ascending concentrations of tert-butanol (50% for 20 minutes, 75% for 20 minutes, 95% for 20 minutes, and 100% for two hours), and then transferred to a critical point dryer for 30 minutes. The surfaces were coated in a vacuum evaporator (Quick Coater Type SC-701, Sanyu Denshi Inc., Tokyo, Japan) with a thin film of gold. Ultrastructural observation of the enamel surfaces was performed using field emission SEM (ERA 8800FE, Elionix Ltd., Tokyo, Japan).

**RESULTS**

Table 2 shows the shear bond strength results under the different environmental conditions. All the materials tested exhibited their highest bond strength under the conditions of 25 ± 1°C and 50 ± 5% RH. The bond strength values of Beauty Ortho Bond, Transbond Plus, Kurasper F, and Superbond Orthomite were 12.1 ± 1.8, 10.4 ± 3.7, 11.2 ± 3.2, and 17.0 ± 4.5 MPa respectively. Under a higher RH condition at 37 ± 1°C and 95 ± 5% RH, the bond strength values ranked the lowest and were significantly reduced to 8.0 ± 2.1, 6.8 ± 1.8, 7.7 ± 2.6, and 12.0 ± 2.8 MPa for Beauty Ortho Bond, Transbond Plus, Kurasper F, and Superbond Orthomite respectively.

Figure 1 shows the representative SEM images of the enamel surfaces after shear bond testing. Under higher RH conditions, bond failure frequently occurred at the bracket-adhesive interface. The ARI scores for the adhesive systems are presented in Table 3. Chi-square analysis revealed significantly different ARI score distributions for the different environmental conditions tested ($\chi^2$=27.273, $P<0.001$).

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>25 ± 1</th>
<th>25 ± 1</th>
<th>37 ± 1</th>
<th>37 ± 1</th>
</tr>
</thead>
<tbody>
<tr>
<td>RH (%)</td>
<td>50 ± 5</td>
<td>95 ± 5</td>
<td>50 ± 5</td>
<td>95 ± 5</td>
</tr>
<tr>
<td>Beauty Ortho Bond</td>
<td>12.1 (1.8)$^a$</td>
<td>9.4 (2.3)$^{b,c}$</td>
<td>10.6 (2.1)$^{a,b}$</td>
<td>8.0 (2.1)$^c$</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>10.4 (3.7)$^d$</td>
<td>8.5 (1.6)$^{e,f}$</td>
<td>9.1 (1.6)$^{d,e}$</td>
<td>6.8 (1.8)$^f$</td>
</tr>
<tr>
<td>Kurasper F</td>
<td>11.2 (3.2)$^g$</td>
<td>9.3 (4.5)$^g$</td>
<td>9.8 (2.2)$^{g,h}$</td>
<td>7.7 (2.6)$^h$</td>
</tr>
<tr>
<td>Superbond Orthomite</td>
<td>17.0 (4.5)$^i$</td>
<td>14.4 (2.2)$^i$</td>
<td>15.8 (2.7)$^{i,j}$</td>
<td>12.0 (2.8)$^j$</td>
</tr>
</tbody>
</table>

Values in parentheses indicate standard deviations ($n = 10$).

Values with the same superscript letters indicate no significant differences within each adhesive system according to Tukey’s HSD test ($p>0.05$).
DISCUSSION

Although the ideal material for this type of study is human maxillary teeth, bovine teeth were used here as a substitute. Bovine teeth are more easily obtainable and have been reported to act as a reliable substitute for human teeth in bonding studies. Bovine teeth also have a relatively flat bonding surface, thereby avoiding the difficulties of fitting a bracket base to a curved surface.

The current study investigated different adhesive systems utilizing phosphoric acid and self-etching primers, which combine etching and priming into a single step. During the etching procedure, the surface enamel is permanently lost from the tooth surface, and the extent of this effect depends on the type of acid used. Subsequently, the adhesive resin is able to infiltrate the etched and roughened enamel to establish a stable bond with the orthodontic bracket. After infiltrating the etched

![Fig. 1 SEM images of enamel surfaces after bond strength tests.](image)

(A) A small amount of adhesive remained around the edge of bracket (ARI score: 1). (B) Less than 50% of the adhesive remained on the tooth surface (ARI score: 1). (C) Almost more than 50% of the adhesive remained on the tooth surface (ARI score: 2). (D) Not all, but most of the adhesive remained on the tooth surface (ARI score: 2).

Table 3  ARI scores at various temperature and RH conditions.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Relative humidity (%)</th>
<th>ARI scores</th>
<th>Beauty Ortho Bond</th>
<th>Transbond Plus</th>
<th>Kurasper F</th>
<th>Superbond Orthomite</th>
</tr>
</thead>
<tbody>
<tr>
<td>25 ± 1</td>
<td>50 ± 5</td>
<td>0 1 2 3</td>
<td>0 3 7 0</td>
<td>0 3 7 0</td>
<td>0 4 6 0</td>
<td>0 1 9 0</td>
</tr>
<tr>
<td>25 ± 1</td>
<td>95 ± 5</td>
<td>0 1 2 3</td>
<td>0 5 5 0</td>
<td>0 6 4 0</td>
<td>0 7 3 0</td>
<td>0 0 10 0</td>
</tr>
<tr>
<td>37 ± 1</td>
<td>50 ± 5</td>
<td>0 1 2 3</td>
<td>0 6 4 0</td>
<td>0 6 4 0</td>
<td>0 8 2 0</td>
<td>0 3 7 0</td>
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<tr>
<td>37 ± 1</td>
<td>95 ± 5</td>
<td>0 1 2 3</td>
<td>0 7 3 0</td>
<td>0 7 3 0</td>
<td>0 4 6 0</td>
<td>0 3 7 0</td>
</tr>
</tbody>
</table>

ARI categories: 0 = no adhesive remained on the tooth surface; 1 = less than 50% of adhesive remained on the tooth surface; 2 = more than 50% of adhesive remained on the tooth surface; 3 = all adhesive remained on the tooth surface (n = 10).
enamel, the adhesive resin must undergo polymerization to produce a durable bond that is capable of withstanding external stresses. A commonly encountered problem during orthodontic treatment is bond failure. This is because bonding procedures require multiple clinical steps, and that success with these adhesive systems can depend on technique-sensitive and material-related factors. On this note, the environmental temperature and RH conditions during the restorative procedure are additional clinically relevant factors. In the current study, specimens produced under 95% RH conditions showed reduced bond strengths with all the adhesive systems tested.

Self-etching primers are applied to the tooth surface before the adhesive to improve both the monomer penetration into the porous surface and wettability. These events in turn ensure optimum bonding. After applying the self-etching primer, the enamel surface should be air-dried because primers contain solvents such as water, ethanol, and acetone. Under extreme environmental conditions, dilution of the self-etching primer components can occur due to vapor condensation on the enamel surface. Self-etching primers contain acidic functional monomers that demineralize the tooth surface. The rate of water evaporation from a water-resin monomer mixture was previously reported to be inversely related to the environmental RH. Under higher RH conditions, a decrease in the net rate of diffusion of water molecules from the liquid to the gas phase is thought to occur as the latter approaches saturation. A large amount of residual water in the self-etching primers could induce the dispersion of resin molecules and limit the propagation of the polymerization reaction of adhesive cement. The strength of a cured adhesive is dependent on its composition, the extent of conversion, and the length of the polymer chain. Any unreacted resin monomer remaining in the adhesive might alter its mechanical properties.

Enamel surface contamination can occur at two critical points during the bonding procedure: before the application of the enamel conditioner and before the application of adhesive cement. Water remaining on the enamel surface might have an adverse effect on the polymerization of the adhesive cement applied after the priming step. A similar situation might occur in a system that utilizes phosphoric acid etching before adhesive cement application. Under high-RH conditions, it might be difficult for all the water to evaporate from the enamel surface. Moreover, under high-temperature conditions, the molecular kinetic energy is greater. This means that more molecules can escape from the surface and the saturated vapor pressure becomes correspondingly higher. Consequently, more moisture is in contact with the surface. The acid-treated enamel surface might therefore be expected to contain extrinsic water even after air-drying, thereby reducing enamel bond strength.

In the present study, the evaluation of the ARI scores indicated that specimens made under higher temperature with higher RH condition had the lower ARI scores, due to the hydrophilic properties of the primer and composite. Most specimens of composite-type orthodontic adhesives in the present study had ARI scores of 1 and 2, indicating that about half of the adhesive remained on the tooth specimens. This might be due to the inherent hydrophilic properties of the resin and the possibility of primer dilution with moisture, owing to the higher RH condition. Superbond Orthomite with phosphoric acid etching showed the highest ARI score. The amount of residual composite might not be related to shear bond strength, but was governed by factors caused by bracket base design and properties of the adhesive.

Clinically adequate tensile bond strengths between metal brackets and enamel reportedly range from 6 to 8 MPa. However, there is no consensus regarding the minimum clinically acceptable values for orthodontic adhesives to achieve optimum bond strength for archwire placement. Brackets were previously shown to be able to withstand static loads during the tying in of archwires at 15 minutes after bonding, without adversely affecting the shear bond strength of the adhesive. The bond strengths of the orthodontic adhesives used in this study ranged from 6.8 to 12.0 MPa, and these were thought to be acceptable values for clinical orthodontic procedures. Although there is no definitive indication of when the bond strengths will reach a plateau, the storage period is a critical factor in evaluating the efficacy of orthodontic adhesive systems.

The current in vitro study suggested that extrinsic water derived from oral humidity levels above a critical threshold might adversely affect the orthodontic bracket bonding of adhesive systems. Care must therefore be exercised when using orthodontic adhesive systems in the oral environment, even with systems that employ hydrophilic components in the primer or adhesive.

ACKNOWLEDGEMENTS

This work was supported, in part, by a Grant-in-aid for Scientific Research (C) No. 19592211, by Grants-in-aid for Young Scientists (B) Nos. 18791411 and 19791415 from the Japan Society for the Promotion of Science, by a Grant from the Dental Research Center, Nihon University School of Dentistry, Japan, by the Uemura Fund, and by the Sato Fund, Nihon University School of Dentistry.
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