Effectiveness of an Er:YAG Laser in Etching the Enamel Surface for Orthodontic Bracket Retention

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The purpose of this study was to test the effectiveness of an Er:YAG laser in etching the enamel surface for orthodontic treatment. Bovine incisors were either acid-etched or laser-treated. An orthodontic bracket was attached on each treated surface using one-step dentin adhesive and self-curing resin. Tensile bond strength was then evaluated. In addition, the surface morphology of specimens treated with phosphoric acid/laser and self-etching primer, as well as the cross-section of enamel-primer-resin interfaces, were observed. One-Up Bond F-treated specimens after Er:YAG laser ablation showed statistically similar tensile bond strength (9.9±1.3 MPa) to that of phosphoric acid-etched specimens (11.8±1.7 MPa). Surface roughness and thickness of the enamel-primer-resin interfaces did not much affect the tensile bond strength of the tested specimens. In conclusion, Er:YAG laser ablation achieved clinically acceptable level of tensile bond strength when used with One-Up Bond F.

Key words: Tensile bond strength, One-step adhesive, Er:YAG laser, SEM

INTRODUCTION

Among the types of laser available, Er:YAG laser is one of the most promising lasers for the treatment of dental hard tissues¹⁻⁵. The promising capability of Er:YAG laser lies in its wavelength. Er:YAG emits a laser at 2.94µm wavelength which coincides with a very strong water absorption peak, and which is well absorbed by the OH⁻ group in hydroxyapatite⁶⁻⁷. When this laser is irradiated on the tooth, temperature and pressure rise rapidly within the tooth and initiate the ablation process, the latter being driven by the explosive vaporization of water within the tissues. Since Er:YAG laser ablation is induced by the explosive vaporization of water, the surface is generally flaky with irregularly serrated and microfissured structure, and one which is usually free from melting and carbonization⁶.

Recently, lasers have been introduced to the orthodontic field to test their feasibility in etching the tooth surface instead of phosphoric acid⁹⁻¹¹. Acid etching is a conventional technique that produces micromechanically retentive structure on enamel by the preferential dissolution of inorganic structure, and then facilitates the penetration of monomers to create resin tags in enamel¹²⁻¹⁵. On the other hand, some lasers etch (ablate) enamel by making the surface microscopically rough and uneven without chemical process. These microirregularities make the enamel surface microretentive, thereby promoting adhesion. However, surface roughness does not correlate much with the bond strength of composites to tooth¹⁶⁻¹⁹. In addition to morphological modification, laser irradiation on enamel is known to produce structural modification due to the phase transformation or melting of inorganic substances, expansion of the organic matrix, as well as the subsequent blocking of ion diffusion pathways²⁰,²¹. In other words, acid resistance can be simply achieved by laser treatment. Despite these attractive advantages of laser treatment, in most cases including the Er:YAG laser treatment, the resin bond strength of laser-ablated specimens is lower than that of acid-etched specimens⁹⁻¹⁵.

Recently, one-step self-etching and priming dentin adhesives are commercially available in the market²²,²³. With these adhesives, etching and priming are performed simultaneously using weak acidic agents without rinsing. By eliminating the acid-conditioning step, this system simplifies (and usually shortens) the bonding procedure as well as reduces technique sensitivity. As such, it shows much potential and promise as a dental adhesive system although more details about bonding to teeth need to be evaluated²⁴,²⁵.

The purpose of this study was to test the effectiveness of an Er:YAG laser in etching the enamel surface for orthodontic bracket retention. Two one-step dentin adhesives were introduced to test their efficacy in enhancing the bond strength of laser-treated enamel.

MATERIALS AND METHODS

Tooth specimens and laser treatment

Fresh and non-caries bovine incisors were obtained from the local slaughterhouse and stored in 0.1% thymol solution at 4°C after extraction. Using
bovine incisors for experiments is beneficial because they are relatively easy to standardize as compared to human teeth. The labial side of the specimens was polished using SiC papers (#600, #1500) and diamond pastes (6 μm, 3 μm), and sonicated in distilled water for three minutes. The enamel surface was irradiated using Er:YAG laser (Twinlight, Fotona, Slovenia) at 2.94 μm wavelength. Diameter of the spot was about 1.2 mm, and irradiation energy was 380 mJ/pulse (33 J/cm²/pulse) with a repetition rate of 2 Hz. The laser beam guided through an articulated-arm probe scanned across the specimen surface in a non-contact mode. Only one laser pulse was irradiated on each spot. Surface of the laser-ablated specimens was then cleaned using running water without brushing and dried in air.

**Tensile bond strength**

Specimens were divided into three groups according to their surface condition: control (acid-etched), dentin adhesive-treated, and laser-treated. Laser-treated specimens were further divided into three groups according to the dental adhesive used. Two different dentin adhesives were chosen for two groups: AQ Bond (Sun Medical Co., Japan) and One-Up Bond F (Tokuyama Corp., Japan). Compositions of adhesive systems used in this study are given in Table 1. For the experiments, specimens were prepared as follows.

Group 1: Polished specimens (n=15) were etched using a 37% phosphoric acid, rinsed and dried for 30 seconds respectively. Both phosphoric acid-etched surface and base of the chosen bracket were pasted with ORTHO-ONE primer (Bisco, Schaumburg, IL). Self-curing resin was uniformly applied on the base of the bracket (142-10, Tomy Inc., Japan) and pressed strongly for 10 seconds. Remnant resin around the margin of the attached bracket was then removed.

Group 2: Both laser-treated surface and base of the chosen bracket were pasted with ORTHO-ONE primer. Self-curing resin was uniformly applied on the base of the bracket and pressed strongly for 10 seconds. Remnant resin around the margin of the attached bracket was then removed.

Group 3 and 4: Laser-treated surface was pasted with dentin adhesive (Group 3: AQ Bond, Group 4: ONE-UP Bond F) following the manufacturer's instructions and polymerized for 10 seconds using a light-curing unit (CuringLight XL3000, 3M, St. Paul, MN). Then, both the treated surface and base of the chosen bracket, as well as the rest of the treatment procedure, were as per described above. Finally, one-step dentin adhesive was applied to test whether it can enhance the tensile bond strength of laser-treated specimens.

Group 5 and 6: AQ Bond (Group 5)/One-Up Bond F (Group 6) was applied on the polished specimen surface following the manufacturer's instructions and polymerized for 10 seconds using a light-curing unit. Then, both the treated surface and base of the chosen bracket, as well as the rest of the treatment procedure, were as per described above.

All procedures are summarized in Table 2.

### Table 1: Compositions of adhesive systems used

<table>
<thead>
<tr>
<th>ORTHO-ONE</th>
<th>Etchant</th>
<th>37% phosphoric acid</th>
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</thead>
<tbody>
<tr>
<td>Primer</td>
<td>BisGMA, TEGDMA</td>
<td></td>
</tr>
<tr>
<td>Paste</td>
<td>Fused silica, BisGMA, TEGDMA</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>AQ Bond</th>
<th>Base</th>
<th>Methacrylate monomers (MMA, 4-META, UDMA)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sponge</td>
<td>Polyurethane foam, p-TSNa</td>
<td></td>
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</table>

<table>
<thead>
<tr>
<th>One-Up Bond F</th>
<th>Liquid A</th>
<th>Phosphoric acid monomer, MAC-10, MMA, Bisphenol A polyethoxymethacrylate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Liquid B</td>
<td>2-Hydroxyethyl methacrylate, MMA, Borate catalyst (photoinitiator), Fluoroluminosilicate glass filler</td>
</tr>
</tbody>
</table>

BisGMA: Bisphenol A diglycidylmethacrylate; MAC-10: 11-Methacryloyloxy-1,1-un-decanedicarboxylic acid; 4-META: 4-methacryloxyethyltrimellitic acid anhydride; MMA: Methyl methacrylate; p-TSNa: Sodium p-toluenesulfinate; TEGDMA: Triethyleneglycoldimethacrylate; UDMA: Urethane dimethacrylate

### Table 2: Test conditions for tensile bond strength

<table>
<thead>
<tr>
<th>Group</th>
<th>Bonding procedure</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>Phosphoric acid-etched + ORTHO-ONE primer + ORTHO-ONE SC resin + Bracket</td>
</tr>
<tr>
<td>2</td>
<td>Er:YAG laser-treated + ORTHO-ONE primer + ORTHO-ONE SC resin + Bracket</td>
</tr>
<tr>
<td>3</td>
<td>Er:YAG laser-treated + AQ BOND + ORTHO-ONE primer + ORTHO-ONE SC resin + Bracket</td>
</tr>
<tr>
<td>4</td>
<td>Er:YAG laser-treated + ONE-UP BOND F + ORTHO-ONE primer + ORTHO-ONE SC resin + Bracket</td>
</tr>
<tr>
<td>5</td>
<td>AQ BOND + ORTHO-ONE primer + ORTHO-ONE SC resin + Bracket</td>
</tr>
<tr>
<td>6</td>
<td>ONE-UP BOND F + ORTHO-ONE primer + ORTHO-ONE SC resin + Bracket</td>
</tr>
</tbody>
</table>
test specimens were stored in 37°C distilled water for 24 hours. Finally, tensile bond strength test was conducted using a universal testing machine (Model 4202, Instron-Satec Systems, Grove City, PA) at a cross-head speed of 1 mm/min.

Scanning electron microscope (SEM) examination
The surface morphology of phosphoric acid-etched and laser-treated specimens was observed using an SEM (S4200, Hitachi, Japan). To observe the surface morphology of AQ Bond- and One-Up Bond F-treated specimens, tooth surface (enamel) was polished and treated with AQ Bond or One-Up Bond F. Each treated surface was rinsed with acetone and running water before light curing, and then dried in air. Bracket-attached specimen was imbedded in an epoxy resin, dried fully, and cross-sectioned using high speed saw. The cross-sectioned surface was polished using SiC papers and alumina pastes as mentioned above, and washed with running water. Specimens were desiccated in a dryer, and the surface was gold-coated for SEM examination. Observation was performed at the enamel-primer-resin interfaces.

Statistical analysis
Acquired data from the tensile bond strength measurements for six different groups were analyzed by one-way ANOVA followed by Tukey's studentized range test for variable at a p value of 0.05.

RESULTS
Bracket tensile bond strengths (MPa) obtained from the six different treatment conditions are shown in Table 3. The tensile bond strength of the phosphoric acid-etched specimens showed the highest value, followed by One-Up Bond F-treated specimens on the polished and laser-treated surfaces. However, there were no statistical differences among them. On the laser-treated surface, application of One-Up Bond F significantly enhanced tensile bond strength as compared to the no dentin adhesive-treated but only laser-treated specimen. One-Up Bond F-treated specimens yielded higher bond strength than AQ Bond-

Table 3 Bracket tensile strengths (MPa) obtained from different groups

<table>
<thead>
<tr>
<th>Group</th>
<th>Tensile bond strength*</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>11.8 ± 1.7</td>
</tr>
<tr>
<td>2</td>
<td>6.9 ± 1.24</td>
</tr>
<tr>
<td>3</td>
<td>7.8 ± 1.0</td>
</tr>
<tr>
<td>4</td>
<td>9.9 ± 1.3</td>
</tr>
<tr>
<td>5</td>
<td>7.8 ± 1.4</td>
</tr>
<tr>
<td>6</td>
<td>10.1 ± 2.2</td>
</tr>
</tbody>
</table>

*Significantly different (one-way ANOVA, p<0.05).

Within the column, values marked with the different letters were significantly different (Tukey's studentized range test, p<0.0001).

Fig. 1 Surface morphology of (a) untreated, (b) phosphoric acid-etched, and (c) laser-treated enamel surfaces.

treated specimens. For the same dentin adhesive, surface condition did not affect tensile bond strength.
Fig. 2 AQ Bond- (a,b) and One-Up Bond F-treated (c,d) enamel surfaces observed under different magnifications: ×5,000 (a,c) and ×50,000 (b,d).

Fig. 3 Cross-section of enamel-primer-resin interfaces for different groups: (a) phosphoric acid-etched, (b) laser-treated, (c) laser-treated + AQ Bond-treated, and (d) laser-treated + One-Up Bond F-treated + ORTHO-ONE primer + ORTHO-ONE SC resin-treated specimens. Upper part is SC resin and the lower part is the treated enamel surface. The dark-color part between SC resin and enamel is primer + dentin adhesive. Arrows in the figures indicate microcracks or poorly attached microfragments. These microdefects were produced during laser treatment.
regularly rough and left a unique etching pattern. Exposed enamel rods were easily distinguishable. The Er:YAG laser-treated surface was quite different from the others, where the treated surface was scaly and irregularly serrated. However, no surface melting and carbonization were observed.

Fig. 2 shows the AQ Bond- and One-Up Bond F-treated enamel surfaces. Unlike the phosphoric acid-etched case, the two dentin adhesive-treated surfaces looked smooth and did not show any significant differences in their morphology (×5,000 magnification), even though enamel crystals were unevenly exposed as a result of self-etching (×50,000 magnification). The difference in enamel crystal size could be due to the difference of the tooth specimens used for SEM observation.

Fig. 3 shows the cross-section of enamel-primer-resin interfaces for Groups 1 to 4. In Figs. 3(a) and (b), the layer between SC resin and enamel surface was of the minimum thickness, whereas One-Up Bond F-treated specimen yielded the highest. Cross-section of the laser-treated surfaces looked like waves. Microcracks and poorly attached microfragments were visible on the laser-treated surfaces.

**DISCUSSION**

Results obtained from the tensile bond strength test showed that the use of Er:YAG laser for orthodontic treatment can be a useful substitution of the conventional phosphoric acid etching if Er:YAG laser were used with a proper dentin adhesive. The goals of enamel etching are numerous: to clean the enamel surface, to remove the enamel smear layer, to increase microscopic roughness by preferential removal of interprismatic mineral crystals, to increase the surface free energy of enamel by producing enough monomer infiltration to seal the enamel surface with resin, and to contribute to the retention of resin composites. When enamel is acid-etched, it is transformed from a smooth and solid surface to a regularly rough and extensively microfissured structure by a preferential dissolution of interprismatic crystallites. According to a previous report, the microfissures on the surface were not deep — the depth did not exceed 12 μm. Overall cross-sectional area of the etched surface — which is closely related to the microfissures — affects resin-enamel bond strength at the time when resin monomers fill into these microfissures.

Unlike the acid-etched surface, Er:YAG laser treatment created an irregularly serrated surface with some loosely attached microfragments and a surface free of smear layer. Although each treatment created morphologically different structures, the correlation between etch depth and enamel-resin bond strength was weak, and as other reports had indicated surface roughness did not much affect the bond strength of composites to enamel. When compared to the phosphoric acid-etched specimens (Group 1), those in the Er:YAG laser treatment group (Group 2) yielded a higher tensile bond strength — which could be a result of the microdefects formed in the latter specimens. Since the tested dentin adhesives had lower viscosity than ORTHO-ONE primer (ORTHO-ONE primer flowed more slowly than AQ Bond and One-Up Bond F), dentin adhesives could have effectively interacted with the formed microdefects and improved the restoration (Groups 3 and 4 versus Group 2). Indeed, following dentin adhesive treatment, enhancement of tensile bond strength could have been achieved through this interaction. Moreover, due to the lower viscosity of the dentin adhesives, surface quality (whether it was rough without microdefects (Groups 5 and 6) or rough with microdefects (Groups 3 and 4)) did not decisively affect tensile bond strength. The same dentin adhesive yielded almost the same bond strength whether the specimen surface was treated with laser or not.

Apart from morphological differences, the Er:YAG laser-treated surface would be expected to have a lower water content than the original tooth because Er:YAG laser-induced photothermal effect is based on the explosive vaporization of water in enamel. The observed difference in tensile bond strength between the phosphoric acid-etched and laser-treated specimens would be partially due to surface wetness or monomer viscosity. The lower water content on the surface would reduce hydrophilic monomer (such as HEMA included in One-Up Bond F) solubility, thereby resulting in reduced bond strength on the laser-treated surface. Monomer diffusion might be limited due to the loss of water channel from the dried tooth surface, and insufficient resin tags would reduce bond strength. Photothermal effect modifies surface morphology and tooth composition. As a result of these changes, a laser-treated tooth inherits acid resistance. Such advantage can compensate the weak bond strength of a laser-treated tooth so that orthodontic treatment can be performed on a tooth weak against caries.

The two one-step dentin adhesives used in this study were of the self-etching and self-priming system, where both etching and priming steps were combined to treat enamel. The advantage of this system lies in reduced chairtime — which is chiefly attributed to a simplified and usually shortened bonding procedure. One-Up Bond F included a hydrophilic monomer that is extremely effective in wetting tooth surfaces. On the other hand, 4-META — an acidic monomer included in AQ Bond — has an excellent spreading property which aids in improving the ability of the monomer to wet tooth surfaces. Wetting the enamel surface is a necessary, initial step in bonding. The acidic monomers or acid
solution in these systems etch the tooth surface by interacting with the mineral substances, and thereby enhance monomer infiltration\(^2\). Infiltrated monomers then form a resin-enamel hybrid layer with the applied resin adhesive\(^3\). From the images of the various treated enamel surfaces, the modified surface after self-etching system treatment did not show a deep etching pattern (Fig. 2) commonly observed in phosphoric acid-etched specimens (Fig. 1(b)). The bond strengths of dentin adhesive-treated specimens, however, did not show much difference compared to the phosphoric acid-treated specimens even though their surface qualities were quite different. One-Up Bond F-treated specimens yielded a bond strength similar to the phosphoric acid-etched specimens, whereas AQ Bond-treated specimens yielded a lower bond strength. As for the same laser-ablated specimens, the reason for their bond strength differences was not clear too. We speculated that differences in the active ingredients of the products might have contributed to the variation in enamel bond strength. Unfortunately, little was known about the effect of the ingredients on enamel bond strength, which means that further investigations are needed. In any case, however, laser-treated enamel with one of the one-step dentin adhesives achieved a much higher bond strength than that of the laser-treated enamel without such a dentin adhesive treatment.

CONCLUSION

The effectiveness of an Er:YAG laser in etching the enamel surface for orthodontic purpose was tested by applying two one-step dentin adhesives. When One-Up Bond F was used, bracket bond strength (about 10 MPa) of either the polished or laser-treated specimen was not statistically different from that of the 37% phosphoric acid-etched specimen (about 12 MPa). AQ Bond-treated specimens, however, yielded significantly lower bracket bond strength than that of One-Up Bond F-treated and phosphoric acid-etched specimens. When Er:YAG laser treatment was used instead of phosphoric acid etching, One-Up Bond F treatment was effective in enhancing the tensile bond strength.

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