Effect of dentin laser irradiation at different pulse settings on microtensile bond strength of flowable resin

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The aim of this study was to compare the effect of Er:YAG laser irradiation and adhesive systems on the microtensile bond strength (μTBS) of flowable resin to dentin. The flat dentin surfaces of 30 wisdom molar teeth were randomly divided into 3 groups (n=10): no laser etching (control), laser etching using QSP and MSP modes. After the application of the flowable and the composite resin, and 5,000 thermal aging, the μTBS results were analyzed with two-way analysis of variance and Tukey’s honest standard of difference (HSD) tests. There were no significant differences between the μTBS of the self-etch group and the total-etch groups for MSP (p>0.05). MSP mode is a useful pulse mode for dentin surface treatment due to elimination of the acid-etching step.

Keywords: Microtensile bond strength, Flowable resin, Laser etching

INTRODUCTION

Adhesive restorative procedures are an important step in the bonding protocol. The clinical success of dental restorations depends on the chemistry of the adhesive, clinical application of the material, and knowledge of any present morphological changes.

Current adhesive systems follow one of two approaches: the total-etch approach or the self-etch approach. Total-etch systems have been shown to be an efficient strategy for smear layer removal, exposing open dentinal tubules and a thin superficial layer of demineralized intertubular dentin. Nevertheless, a disadvantage attributed to acid etching is the demineralization of tooth structures, making them more permeable and prone to acid attacks, especially if the demineralized substrates are not completely filled by adhesive resins. In order to overcome this limitation, new investigations are underway into alternative techniques that could produce better effects than those produced by acids. Among these innovations for dentinal surface treatment, the use of lasers has been widely advocated.

The erbium: yttrium-aluminum-garnet (Er:YAG) laser is one of the most useful types of lasers for dental hard tissues and emits a wavelength (2.94 μm) coincident with the main absorption band of water (3.0 μm). The Er:YAG is also well absorbed in hydroxyapatite and effectively treats the dentin surface by removing the smear layer in a way that is similar to acid etching, opening dentinal tubules and creating a microscopically rough surface with a micromechanical retention pattern, which is apparently ideal for adhesion. Moreover, cavity pretreatment with Er:YAG laser has been proposed as an alternative to acid etching of enamel and dentin.

Er:YAG laser irradiation parameters define the exact interaction of the laser on the target tissue. Pulse duration is a factor affecting the surface properties. Active electronic control of laser pulse duration and amplitude is possible today with the development of Variable Square Pulse (VSP) technology (Fotona d.d., Ljubljana, Slovenia). With VSP, the duration of pulses can be adjusted from 50 μs (super-short pulse; SSP) to 100 μs (medium-short pulse; MSP), 300 μs (short pulse; SP), 600 μs (long pulse; LP), and 1,000 μs (very long pulse; VLP). The energy loss through heat is lower due to its higher energy in the shorter pulses. Eventually, ablation becomes more effective and a thermal effect is not evident on the tissue.

The quantum square pulse (QSP) mode (Fotona d.d.) has also recently been introduced in Er:YAG laser technology. The QSP pulse consisted of five pulselets (quantas) of 50 μs pulse duration, that follow each other at an optimally fast rate. In this way, absorption and scattering of the laser beam is avoided and undesirable thermal effects are decreased for the tissues. Cavities having high surface quality are sharp and well defined. Lasers operating at this mode are reported to provide fast and precise hard dental tissue preparation.

Recently, a new class of low-viscosity resin composites called flowable composites has become widely used by physicians. These materials have low viscosity, low elasticity modulus, and easy application. They are indicated for minimally invasive cavity restorations, small and non-stress-bearing occlusal restorations, Class III and V restorations, base/liner under direct restorations, undercut blockout, and repair of resin and acrylic temporary materials.

Another important factor for achieving favorable resin bonding is the infiltration of the resin into the
surrounding demineralized dentin, since it attaches to and integrates with the resin tags. The filler content of flowable composites is decreased, so it is considered to increase adhesion to dentin as its monomer content can better integrate to the content present in the adhesive system. This better integration results in a more homogeneous layer, with demineralized dentin tubules penetrated by the adhesive materials. Increasing the monomer in the composite formulation would be expected to reduce the shrinkage stress generated during placement of a composite restoration and may preserve the integrity of the adhesive interface.

Lida et al. reported that the use of a flowable resin composite as an adhesive liner produced a significantly greater gap-free resin-dentin interface in CEREC inlay and direct resin composite restorations. Ktiyama et al. reported that resin coating with a combination of a dentin-bonding system and a flowable resin composite may be indicated (for Class II cavities) prior to impressing teeth with computer-aided design/computer-aided manufacturing (CAD/CAM) ceramic inlays in order to reduce microleakage at the tooth-resin interface.

This study planned to determine the effect of surface etching using the QSP and MSP modes of the Er:YAG laser on µTBS between the dentin and flowable composite resin. The null hypothesis of this study was that laser treatment and different adhesive systems would not affect the bond strength between flowable composite resin and dentin.

### Table 1 The materials used in this study

<table>
<thead>
<tr>
<th>Materials</th>
<th>Lot number</th>
<th>Manufacturer</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prime-Dent Blue Etchant Gel</td>
<td>YJ12Q</td>
<td>Prime Dental Manufacturing, IL, USA</td>
<td>37% phosphoric acid</td>
</tr>
<tr>
<td>Adeziv 200T</td>
<td>N449863</td>
<td>3M ESPE, St. Paul, MN, USA</td>
<td>5 nm colloidal filler (10%) HEMA, Bis-GMA, dimethacrylates, ethyl alcohol, water</td>
</tr>
<tr>
<td>Single Bond Universal</td>
<td>567594</td>
<td>3M Deutschland, Neuss, Germany</td>
<td>MDP, HEMA, dimethacrylate resins, vitrebond copolimer, fillers, ethyl alcohol, water, initiators, silane</td>
</tr>
<tr>
<td>Filtek™ Ultimate Flowable Restorative</td>
<td>N514397</td>
<td>3M ESPE</td>
<td>Bis-GMA, TEGDMA and procrylat resins. The ytterbium trifluoride filler (0.1 to 5.0 microns), silica filler, zirconia/silica cluster filler. (The inorganic filler 46% by volume)</td>
</tr>
<tr>
<td>Universal Restorative 200</td>
<td>N335613</td>
<td>3M ESPE</td>
<td>Bis-GMA, UDMA, Bis-EMA resins, zirconia/silica fillers. (The inorganic filler 60% by volume; without silane treatment)</td>
</tr>
</tbody>
</table>

Bis-GMA: bisphenol A diglycidyl ethermethacrylate, HEMA: hydroxyethyl methacrylate, MDP: 10- methacyroyloxydecyl dihydrogen phosphate, UDMA: urethane dimethylacrylate, Bis-EMA: ethoxylated bisphenol A dimethacrylate, TEGDMA: triethylene glycol dimethacrylate
etched with 37% phosphoric acid (Prime-Dent, Prime Dental Manufacturing, IL, USA) and rinsed off with water prior to application of total-etch adhesive on the dentin surfaces.

A layer of flowable resin (Filtek™ Ultimate Flowable Restorative, 3M ESPE) was placed at a thickness of 1 mm and light-cured on top of the adhesive layer and composite (Universal Restorative 200, 3M ESPE) was built up incrementally to a thickness of 4 mm in all specimens. The finished specimens were transferred to distilled water and stored at 37°C for 24 h. In this study, lot numbers, manufacturers and compositions of materials are presented in Table 1.

**Thermocycling and bond strength testing**

All specimens were subjected to thermocycling for 5,000 cycles between 5–55°C with a dwelling time of 20 s in each bath and a transfer time of 10 s according to the standards published by the International Organization for Standardization (ISO)23.

Twenty microbars (1×1 mm) from each specimen were prepared. Each microbar was bonded to the attachment area using a cyanoacrylate adhesive system (Pattex, Turk Henkel AŞ, Turkey). The composite-dentin interface was centered at the free space between the jaws of the attachment unit. μTBS testing was performed on a microtensile tester machine (The Microtensile Tester, BISCO, Schaumburg, IL, USA). The load was applied using a crosshead speed of 0.5 mm/min. μTBS bonding values were calculated via the following equation: performed (N)/ bonding area (mm²).

**Stereomicroscope analysis**

Fractured surfaces of the dentin after the μTBS test were examined with the naked eye and under a stereomicroscope (M165C, Leica Microsystems, Wetzlar, Germany) at ×45 magnification. Failure modes were classified as adhesive (at the dentin-flowable resin interface), cohesive (at within the flowable resin), or mixed. In cases of mixed failure, the surface of the dentin was partly covered by the remaining flowable resin.

**Scanning electron microscopy (SEM) analysis**

Dentin specimens were prepared for no surface treatment, acid-etching, and laser-and acid-etching groups. The surfaces of drying dentin specimens were sputter-coated (Polaron, Emitech, Kent, England) with a thin layer of gold-palladium under high-vacuum conditions. The dentin surfaces after surface treatments and fractured surfaces of the dentin after the μTBS test were examined (Jeol 6390, Jeol, Tokyo, Japan). Photomicrographs were taken with a magnification of ×80–1,500.

**Statistical analysis**

Data were analyzed by using two-way ANOVA and Tukey’s HSD tests. The groups were compared to verify the differences at a significance level set at p<0.05. The calculations were handled using the SPSS for Windows version 22.0 (SPSS, Chicago, IL, USA).

**RESULTS**

**Microtensile bond strength results**

Statistically significant differences were found between the μTBS of the total-etch and self-etch specimens (p<0.001) (Tables 2 and 3). There were no statistically significant differences between the μTBS of the different surface treatments (p>0.05).

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### Table 2 Results of the two-way ANOVA test

<table>
<thead>
<tr>
<th>Source</th>
<th>Type III sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive system</td>
<td>1,258.92</td>
<td>1</td>
<td>1,258.92</td>
<td>32.205</td>
<td>0.000</td>
</tr>
<tr>
<td>Surface treatment</td>
<td>47.09</td>
<td>2</td>
<td>23.54</td>
<td>0.602</td>
<td>0.549</td>
</tr>
<tr>
<td>Adhesive system *Surface treatment</td>
<td>362.23</td>
<td>2</td>
<td>181.11</td>
<td>4.633</td>
<td>0.012</td>
</tr>
</tbody>
</table>

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### Table 3 Results of the μTBS test (MPa)

<table>
<thead>
<tr>
<th>Surface treatment</th>
<th>Adhesive system</th>
<th>Mean (Standard deviation)</th>
<th>Failure mode</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>A</td>
<td>C</td>
</tr>
<tr>
<td>No laser</td>
<td>Self-etch</td>
<td>16.04 (6.26)</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>Total-etch</td>
<td>24.23 (8.21)</td>
<td>2</td>
</tr>
<tr>
<td>QSP mode</td>
<td>Self-etch</td>
<td>16.83 (5.03)</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>Total-etch</td>
<td>26.04 (5.86)</td>
<td>0</td>
</tr>
<tr>
<td>MSP mode</td>
<td>Self-etch</td>
<td>20.64 (7.52)</td>
<td>4</td>
</tr>
<tr>
<td></td>
<td>Total-etch</td>
<td>22.20 (3.40)</td>
<td>0</td>
</tr>
</tbody>
</table>

*abc: Same letters were not significantly different at p>0.05.
The interaction between surface treatment and adhesive system factor levels was statistically significant \((p<0.01)\). Total-etch specimens yielded a higher \(\mu\)TBS than did self-etch specimens for the control (no laser) and QSP groups \((p<0.05)\). There were no significant differences between the \(\mu\)TBS of the self-etch group and the total-etch groups for MSP \((p>0.05)\).

**Stereomicroscope analysis**
Regarding the types of failure in fractured specimens, mix failure was the predominant failure mode for all the groups (Table 3).

**SEM analysis**
Figure 1 shows no treated dentin surface; it can be observed that the dentinal tubules are closed by smear layer. Figure 2 shows acid-treated dentin surface. The dentinal tubules are open, with no deposits or smear layers on the surface. The surface morphology was the same in the laser-treated groups. Rough, irradiated dentin surface can be observed in both specimens treated with the MSP (Fig. 3) and QSP (Fig. 5) modes. No recrystallization or melting surfaces were noted. The surface morphology was similar in laser and acid-treated groups (Figs. 4 and 6).

Fractured surfaces of the dentin after the \(\mu\)TBS test were examined with SEM (Figs. 7–9). Flowable resin application produced partial or total cohesive failure in the low-viscosity resin layer and within the adhesive system layer or dentin surface. In both laser irradiated groups, resin shows more integration to the dentin surface in total etch groups than self etch groups. There were similar images for MSP and QSP modes.
Fig. 5  SEM image of the dentin surface after application of the Er:YAG laser (QSP mode).

Fig. 6  SEM image of the dentin surface after application of the Er:YAG laser (QSP mode) and acid etching.

Fig. 7  A: SEM image of the debonded dentin surface after self etching; B: SEM image of the debonded dentin surface after total etching (D: Dentin, AL: Adhesive layer, F: Flowable resin).

Fig. 8  A: SEM image of the debonded dentin surface after the application of the Er:YAG laser (QSP mode) and self etching; B: SEM image of the debonded dentin surface after the application of the Er:YAG laser (QSP mode) and total etching (D: Dentin, AL: Adhesive layer, F: Flowable resin).
DISCUSSION

In the present study, we have tested the μTBS of commercially available flowable resin to dentin with different surface treatments. Different pulse settings did not affect the strength of the bond. However, the total-etch resulted in better μTBS values for all groups. Based on the results of the study, the null hypothesis was partially accepted.

Use of the Er:YAG laser has been recommended to increase the adhesion of restorative materials to dental hard tissues. However, the effectiveness of laser treatment in the literature is controversial; while some researchers support the preparation or etching ability of laser to dentin24,25), others believe the method is not effective26,27). It is important to choose parameters to ablate the tooth tissue. Undesirable modifications in dentin collagen after laser irradiation can occur, which would affect bond strength between restorative materials and the tooth negatively28). It is not easy to compare our results with other studies, as different laser parameters for items such as output and distance can alter surface treatments and QSP is a novel pulse mode duration.

Only a few reports that evaluate Er:YAG laser surface treatments of different pulse durations exist in the literature. Baraba et al. reported that the use of the SP and MSP modes of the Er:YAG laser with one-step self-etch adhesive did not improve the μTBS in dentin29). Altunsoy et al. reported that the highest μTBS was recorded when Vertise Flow (self-adhesive flowable resin) was applied with acid etching, with no statistically significant differences between the QSP and MSP modes. However, in the same study, the highest μTBS for Fusio Liquid Dentin (self-adhesive flowable resin) was recorded with the MSP mode29).

Filtek™ Ultimate Flowable Restorative was used in the current study. This material is a low-viscosity, visible light-cured, radiopaque flowable nanocomposite. The use of dental adhesive systems, either total-etch or self-etch (both of which are designed to be compatible with methacrylate composites), leads to bonding to the tooth structure. Adeziv 200T was used as a total-etch adhesive and Single Bond Universal were used as a self-etch adhesive. Total-etch specimens served a higher μTBS than did the self-etch specimens for the control (no laser) and QSP groups. The difference was not statistically significant in groups in which a surface treatment was applied in the MSP mode. Self-etch adhesives are not able to remove smear layer as well as total etch systems due to mild acidity of the primer of self etch adhesives. However after etching with MSP mode, smear layer was removed and a more rough dentin surface was obtained (Fig. 3). This surface enhanced the μTBS values of the bonding agent. However, total etching following MSP mode laser etching resulted in more roughness in peritubular dentin when compared to total etching following the QSP mode laser etching (Figs. 4 and 6). Therefore, the authors of the present study claim that while self etching adhesives used with MSP mode laser etching increased the bond strength values, total etch+MSP mode laser leaded in lesser values.

Sagir et al. reported that Er:YAG laser etching of enamel with the MSP and QSP modes presented a successful alternative to acid etching by providing higher or comparable shear bond strength values31). Differences in results across studies may be due to the evaluation of the connection with dentin in the present study. Dentinal tubules on an untreated dentin surface were closed by the smear layer. The opened dentinal tubules were observed in dentin surface etched with acid. SEM views of the laser-treated dentin surfaces in the MSP and QSP modes are similar. The laser- and acid-etched specimens served more regular surfaces than only laser-etch groups and were similar with those of the acid-etch group. The SEM views of the present study are aligned with the study of Altunsoy et al.30). When we evaluate the fractured surfaces after debonding in both laser irradiated groups, resin shows more integration to the dentin surface in total etch groups than self etch groups.
Controlled clinical studies are necessary to evaluate the success of treatment procedures such as those described in this study. Restored teeth are subject to temperature changes, moisture, chewing forces, and chemical attacks in the oral cavity. The bond between dental material and the hard tissues of the teeth is consequently weakened. However, such controlled studies are expensive and time-consuming. In the present study, specimens were subjected to thermocycling. Clinical loading conditions should be performed in future studies.

CONCLUSION

Under the limitations of this in vitro study, total-etch specimens yielded a higher μTBS than did self-etch specimens for the no-laser and QSP groups. No significant differences were observed in the μTBS between the self-etch and total-etch groups in MSP mode. Self-etch system showed higher bond strength when dentin was treated with the MSP mode compared with that with the QSP mode. Additional studies are required to confirm the benefits of flowable resins and laser surface treatments.

REFERENCES