Tensile Bond Strength of a Flowable Composite Resin to Er:YAG-Laser-Treated Dentin

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Background and Objectives: This in vitro study evaluated the influence of a flowable composite resin (FCR) on the tensile bond strength of resin to dentin treated with the Er:YAG Laser (L) and diamond bur (DB).

Study Design/Materials and Methods: Ninety dentin surfaces obtained from 45 third molars were ground and randomly divided into six groups (n = 15): G1–DB, G2–DB + FCR, G3–L (100 mJ, 10 Hz, 37.04 J/cm²), G4–L (100 mJ, 10 Hz, 37.04 J/cm²) + FCR, G5–L (250 mJ, 2 Hz, 92.60 J/cm²), and G6–L (250 mJ, 2 Hz, 92.60 J/cm²) + FCR. After surface etching with 37% phosphoric acid and the application of an adhesive system, inverted conical specimens were prepared with a hybrid composite resin. In groups G2, G4, and G6 a FCR was placed before the hybrid composite resin. After 24 hours-storage in distilled water, the tensile test was performed in a universal testing machine (0.5 mm/minute, 500 N).

Results: Data were submitted to Kruskal Wallis test (P = 0.01). The mean bond strength values (MPa±SD) were: G1–13.54 (±2.99), G2–14.67 (±2.32), G3–9.49 (±3.09), G4–14.60 (±2.76), G5–8.97 (±3.89), and G6–13.02 (±2.18). Groups G1 and G2 presented the highest bond strength values, which were statistically similar to those of G4 and G6. The groups treated with laser and without the FCR (G3 and G5) showed the lowest shear bond strength values.

Conclusions: FCR can increase the adhesion to dentin treated with Er:YAG laser within different parameters.

INTRODUCTION

During the last few years, the demand for esthetic procedures and for preserving healthy tooth structure, has lead to the development and improvement of adhesive materials that can fulfill their function of bonding to enamel and dentin. These procedures were originally developed to act on the tooth substrate prepared by conventional techniques. However, new investigations point to alternative tools that could better prepare the dentin surface for future bonding procedures. Among these innovations for dentin surface treatment, the use of lasers has been widely disseminated.

Described as a safe and effective tool for the removal of dental hard tissues [1], Er:YAG laser works at a wavelength of 2.94 μm, which coincides with the absorption peak of water and hydroxapatite [1–3] and can ablate the enamel and dentin effectively. Some studies have pointed to the feasibility of using an Er:YAG laser in conjunction with a water spray to remove hard dental tissues without compromising the laser cutting efficiency. With a fine water mist, the temperature can be suppressed and the irradiation can be performed with no damage to the pulp [4–7].

The cavities prepared with Er:YAG laser do not present a smooth surface; their surfaces have opened dentinal tubules without smear layer production and microscopically rough surfaces [2,8,9]. These dentin characteristics are expected to favor resin bonding, so that most of these cavities are filled with adhesive materials.

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Some in vitro studies reported that bond strength to Er:YAG-laser-treated dentin is lower than that of dentin that had not been laser treated [10–13]. In order to achieve greater bonding results, some authors have evaluated the use of different surface treatments and irradiation parameters [11–13].

Recently, a new class of low-viscosity resin composites, called flowable composites, was introduced in the market and these are widely used by professionals. Some of their characteristics are: low viscosity [14], low elasticity modulus [14–16], and easy application [17]. Despite its ability to reduce dentinal cervical sensitivity [18], this new adhesive material has been reported to provide better adaptation to dentin [15,17]. At present, little is known about the use of low-viscosity composite resin to increase the adhesion of hybrid composite resin to laser-treated dentin [13,19]. It is expected that this restorative material associated to an adequate adhesive system [13], can show a superior adaptation to the irregularities of the laser treated dentin.

This study aimed to determine whether the use of a flowable composite resin (FCR) on dentin treated with Er:YAG laser affects the tensile bond strength between dentin and composite resin.

**MATERIALS AND METHODS**

**Specimen Preparation**

This study protocol was reviewed and approved by the Ethical Committee of the University of São Paulo. Forty-five recently extracted third molar teeth were cleaned and stored in distilled water (37°C ± 1) up to the beginning of the experiment (1 month). The crowns were removed approximately to the cementoenamel junction and were longitudinally sectioned with a double-faced diamond disk (KG Sorensen, Barueri, SP, Brazil) used in a low-speed microtome (LABC UT 1010/Extec Enfield, CT, USA). Two samples were obtained from each tooth. Samples with stains or cracks, observed under a stereomicroscope used at 30× (Meiji Techno EMZ series, Saitama, Japan), were discarded. Ninety crowns were embedded individually in a self-curing polyester resin (Uceflex UC 2120, Redelease Produtos, Brazil) in a polypropylene ring mould 2.0 cm in diameter, so that the external surface of the crown (enamel from the buccal or lingual surfaces) was exposed, and then left to polymerize.

**Preparation of the Dentin Surface and Laser Irradiation**

After the polyester resin polymerization, the samples were ground in a water-cooled polishing machine (Politriz Ecomet 3/Buehler) with 120, 240, and 400-grit silicon carbide paper (Struers Company, Ballerup, Denmark) to remove the overlying enamel and to provide uniform superficial dentin surfaces. After that, the dentin surface of each sample was polished with a 600-grit silicon carbide paper and immediately washed with an ultrasonic system in order to remove the smear layer.

The dentin specimens were randomly assigned to six groups (n = 15), as shown in Table 1. The samples from G1 and G2 were not submitted to laser irradiation and were considered as positive and negative controls, respectively. A round diamond bur (DB) (#1012, KG Sorensen) in a high-speed drill, under air-water spray, was used to prepare the dentin surface of the samples from groups G1 and G2.

The samples from the experimental groups G3, G4, G5, and G6 were then irradiated with the Er:YAG laser (Key Laser II, KaVo, Biberach, Germany) emitting photons at a wavelength of 2.94 μm. The output power and repetition rate of this equipment range from 60 to 500 mJ and 1 to 15 Hz, respectively. The beam diameter at the focal area for the handpiece #2051 (non contact) was 0.63 mm. The handpiece #2051 was positioned 12 mm from the dentin surface. The samples were irradiated with the energy depicted in the equipment display and corresponded to the energy delivered from the handpiece. The energy density used for the laser irradiation on groups G3/G4 and G5/G6 was 37.04 J/cm² and 92.60 J/cm², respectively. The handpiece was positioned perpendicular to the dentin surface and the samples were irradiated scanning once in each direction, horizontally and vertically, in order to promote homogeneous irradiation and to cover the entire sample area. The irradiation was performed with a water-cooled spray (5.0 ml/minute).

**Bonding Procedures**

In order to demarcate the dentin bonding area, a piece of insulating tape with a 2-mm diameter central hole was attached to the specimen surface. After the surface treatment, the samples were submitted to the bonding procedures. The materials with their compositions, speci-
The bonding and restorative procedures strictly followed the manufacturer’s instructions. Prior to the use of a bonding agent, the dentin was etched with 37% phosphoric acid (Total Etch®; IVOCLAR-VIVADENT, Schaan, Liechtenstein) for 10 seconds. After that, the surface was rinsed for 15 seconds and gently air dried for 5 seconds. The bonding system used was the excite system (IVOCLAR-VIVADENT), according to the manufacturer instructions. Immediately after the use of a bonding agent, a thin layer of the tested FCR (Tetric Flow/IVOCLAR-VIVADENT) was placed onto the dentin disks from groups G2, G4, and G6 with disposable brush tips and light cured for 20 seconds.

**Tensile Bond Strength**

After the bonding procedures, all samples (G1–G6) were individually fixed in a metallic clamping device, keeping the dentin surface parallel to a flat base. A split bisected polytetrafluoro-ethylene jig was positioned on the sample surface, thus providing an inverted conical cavity with the smaller diameter coinciding with the delimited 2-mm diameter bonding site. A hybrid light-cured composite resin (Tetric Ceram/IVOCLAR-VIVADENT) was inserted into the jig in three increments (1.5 mm thick), each polymerized for 40 seconds. As the cavity was filled, the sample was removed from the metallic device, leaving an inverted cone with 4-mm diameter tapering to a 2-mm diameter and 4.5 mm high adhering to the delimited area. After 24 hours storage in distilled water at 37 °C, the cone-shaped samples were submitted to the tensile bond strength evaluation, using a universal testing machine (Mini Instron 4442), running at a cross-head speed of 0.5 mm/minute and a 500 N load cell until fracture.

**RESULTS**

Statistical analysis was made by a parametric method using the one-way analysis of variance (ANOVA) and Kruskal–Wallis test (α = 1%). Mean tensile bond strengths and standard deviations are shown in Table 3. Means varied in a range from 8.97 to 14.67 MPa.

The one-way ANOVA revealed statistically significant difference in tensile bond strength within the experimental groups. The Kruskal–Wallis test showed that there was no statistically significant difference between the non-irradiated groups (G1 and G2) and the laser-treated dentin restored with a FCR (G4 and G6). The lowest TBS values were obtained with dentin irradiated with the Er:YAG laser, without the use of a FCR (groups G3 and G5 with TBS of 9.49 and 8.97 MPa, respectively).

**DISCUSSION**

The results of the present study suggest that the use of a FCR can increase the adhesion of a hybrid composite resin to dentin treated with Er:YAG laser within different parameters. Flowable composite were created by retaining the same small particle size of traditional hybrid composites, but reducing the filler content and allowing the increased resin to reduce the viscosity of the mixture [15,16]. The flowability of low-viscosity composite resins is achieved mainly by increasing the proportion of monomer in the composite formulation [16]. These characteristics are expected to reduce the shrinkage stress generated during the placement of a composite restoration and may preserve the integrity of the adhesive interface [20]. However, the application of a lightly filled resin at the margin of a restoration may lead to increased wear of this region and can compromise the quality of the bonding margins [20]. Clinically, FCRs are expected to be applied to all the cavity walls, excluding the cavity margins. In the present study, the FCR was applied to the dentin surface in order to promote a thin layer capable of reducing stress shrinkage.

Considering the decrease in the filler content, FCRs are claimed to increase adhesion to dentin, since its monomer content can better integrate to the one present in the adhesive system and result in a more homogeneous layer with demineralized dentin tubules penetrated by the adhesive materials.

<table>
<thead>
<tr>
<th>TABLE 2. Restorative Systems Used and Their Compositions, Specifications, and Manufacturers</th>
</tr>
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<tbody>
<tr>
<td><strong>Material</strong></td>
</tr>
<tr>
<td>Adhesive system, excite</td>
</tr>
<tr>
<td>Flowable composite, tetric flow</td>
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<tr>
<td>Hybrid composite resin, tetric ceram</td>
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<tr>
<th>Materials</th>
<th>Composition</th>
<th>Manufacturers</th>
</tr>
</thead>
<tbody>
<tr>
<td>Adhesive system, excite</td>
<td>Phosphonic acid, HEMA, dimethacrylates, alcohol</td>
<td>IVOCLAR VIVADENT</td>
</tr>
<tr>
<td>Flowable composite, tetric flow</td>
<td>BIS-GMA, triethylene glycol dimethacrylate, urethandimethacrylate</td>
<td>IVOCLAR VIVADENT</td>
</tr>
<tr>
<td>Hybrid composite resin, tetric ceram</td>
<td>Monomer matrix: BIS-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate (20.2% weight); inorganic fillers: barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, and silicon dioxide, spheroid mixed oxide (79.0% weight)</td>
<td>IVOCLAR VIVADENT</td>
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**TABLE 3. Tensile Bond Strength Data (Means ± SD in MPa) of the Dentin Submitted to Different Restorative Treatments**

<table>
<thead>
<tr>
<th>Groups</th>
<th>Treatment</th>
<th>TBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>DB</td>
<td>13.54 ± 2.99 a</td>
</tr>
<tr>
<td>G2</td>
<td>DB + FC</td>
<td>14.67 ± 2.32 a</td>
</tr>
<tr>
<td>G3</td>
<td>L (100 mJ/10 Hz)</td>
<td>9.49 ± 3.09 b</td>
</tr>
<tr>
<td>G4</td>
<td>L (100 mJ/10 Hz) + FC</td>
<td>14.60 ± 2.76 a</td>
</tr>
<tr>
<td>G5</td>
<td>L (250 mJ/2 Hz)</td>
<td>8.97 ± 3.89 b</td>
</tr>
<tr>
<td>G6</td>
<td>L (250 mJ/2 Hz) + FC</td>
<td>13.02 ± 2.18 a</td>
</tr>
</tbody>
</table>

Same letters indicate statistical similarity (P = 0.01).
The infiltration of the resin into the surrounding de-mineralized dentin is important for achieving a favorable resin bonding, since it attaches to and integrates with the resin tags [21]. This may possibly justify the findings of the present study. When using the FCR (G2, G4, and G6), the tensile bond strength was higher than that of the other groups, except for G1. The result of group G1 was expected, because dentin preparation with DB leads to the formation of a smear layer that can be removed by surface etching [22]. After the acid etching, both the intertubular and peritubular dentin can be demineralized and contribute to the interdiffusion of the adhesive system. Therefore, the use of a FCR on cavities prepared with DB was not shown to enhance bonding quality.

Er:YAG laser presents a great interaction with the hard dental tissues and is able to produce a dentin surface without smear layer, with a microretentive pattern, revealing tubule openings [1,2,19]. This characteristic is supposed to improve the bond strength of resins to dentin [19,23]. However, some studies have reported the increase of tooth acid resistance after the laser treatment. Consequently, the collagen matrix may not be totally exposed by the 37% phosphoric acid, mainly in the peritubular region [12], and adhesion to composite resin can be compromised by the lack of resin infiltration into the demineralized dentin [21].

The laser-treated dentin with the flowable composite (G4 and G6) presented the highest bond strength, similar to the results obtained with the control groups (G1 and G2). The finding that the combination of laser and flowable composite were equal to the DB suggests that flowable composite seems to be the help for achieving high bond strength values of resin to irradiated dentin. Although, Er:YAG laser can increase dentin acid resistance and create a dentin surface without peritubular dentin demineralization [13], the use of a FCR seems to revert these adverse effects by better adapting to the irregularities of the laser treated dentin surface due to its flowability.

Furthermore, in the present study the decrease in the energy applied and the increase of the repetition rate (G4) was shown to present similar tensile bond strength values compared to the group with higher energy (G6), with the advantage of decreasing the possibility of temperature damage to the pulp when using water cooling. An in vitro study conducted by Glockner et al. (1998) [4] revealed no temperature increase when using laser with water cooling, while the use of a high-speed handpiece can lead to temperature rises of over 60°C. Gouw-Soares et al. (2001) [24] showed temperature increases of less than 3°C in cavity preparations with energy density of 108.28 J/cm² and concluded that these parameters are safe and effective for clinical application. Therefore, the dentin treatment with the energy densities evaluated in the present study can be considered a safe and effective alternative for cavity preparation.

Despite the possibility of abrating dental hard tissue and increasing the adhesion of composite resin to dentin without damage to the pulpal tissue [19], another important characteristic of laser treatment is its antimicrobial property [25–27]. Even if there is no difference between the irradiated and non-irradiated dentin with respect to its bond strength, the cavities prepared with the laser device are expected to undergo microbial reduction in the infected dentin.

Even when the current effectiveness of the conventionally prepared dentin with DBs is acknowledged, the concept of laser-treated dentin is promising and deserves increasing attention in the future. Further studies should be conducted in order to elucidate the interaction of different restorative materials with laser-treated dentin and bring to light a new pattern of interaction for bonding to Er:YAG irradiated dentin.

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