Microtensile bond strength of different adhesive systems in dentin irradiated with Er:YAG laser

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Abstract: The objective of this study was to evaluate in vitro the bond strength of two etch-and-rise and one self-etching adhesive system after dentin irradiation with Er:YAG (erbium: yttrium aluminum garnet) laser using microtensile test. The results revealed that the groups treated with laser Er:YAG presented less tensile bond strength, independently of the adhesive system used. The prompt L-pop adhesive presented less microtensile bond strength compared to the other adhesives evaluated. There was no difference between single bond and excite groups. The adhesive failures were predominant in all the experimental groups. The Er:YAG laser influenced negatively bond strength values of adhesive systems tested in dental substrate.

Microtensile bond strength of different adhesive systems in dentin irradiated with Er:YAG laser

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1. Introduction

The need of complete sealing between the restorative material and the dental substrate led to a better understanding of adhesion mechanisms, stimulating new technologies, such as Er:YAG laser application for dentin surface treatment. The Er:YAG laser is a very promising laser system for dental use since the emitted 2940 nm wavelength of coincides with the main absorption peak of water, and it is also well absorbed by hydroxyapatite [1,2].

Dental adhesive systems have also been studied and implemented. A variety of dentin bonding systems has been developed for clinical use [3]. In great part of currently available adhesive systems, primer and bonding agent are combined in a single bottle, still preceded by a separate etching step. Self-etching adhesive systems were introduced to reduce the number of handling steps and make the applicability of such products more practical than that of multiple-bottle bonding agents. These self-etching adhesive systems are advantageous because they abbreviate the application time and the errors that can occur at each bonding step [4]. It is important to emphasize that all adhesive systems and procedures were originally developed to act on tooth substrate prepared and treated by conventional techniques. Therefore, the interaction of the lasers with newly developed dental materials is not fully understood [5–7].

It is of extreme interest for adhesive dentistry to investigate the morphology of the dentin/adhesive systems interface when different kinds of surface treatment are accomplished (laser and/or acid conditioning). The aim of this study was to evaluate in vitro the bond strength of two etch-and-rise and one self-etching adhesive systems, after the dentinal tissue irradiation with Er:YAG laser, as well as, to analyze the microstructure of resin-dentin interface.

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Table 1 Experimental groups

<table>
<thead>
<tr>
<th>Groups</th>
<th>Er:YAG laser</th>
<th>PA</th>
<th>Ad</th>
<th>CR</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>+ (260 mJ, 10 Hz, 20 s)</td>
<td>+</td>
<td>+ (excite)</td>
<td>+ (tetric ceram)</td>
</tr>
<tr>
<td>G2</td>
<td>–</td>
<td>+</td>
<td>+ (excite)</td>
<td>+ (tetric ceram)</td>
</tr>
<tr>
<td>G3</td>
<td>+ (260 mJ, 10 Hz, 20 s)</td>
<td>–</td>
<td>+ (prompt L-pop)</td>
<td>+ (tetric ceram)</td>
</tr>
<tr>
<td>G4</td>
<td>–</td>
<td>–</td>
<td>+ (prompt L-pop)</td>
<td>+ (tetric ceram)</td>
</tr>
<tr>
<td>G5</td>
<td>+ (260 mJ, 10 Hz, 20 s)</td>
<td>+</td>
<td>+ (single bond)</td>
<td>+ (tetric ceram)</td>
</tr>
<tr>
<td>G6</td>
<td>–</td>
<td>+</td>
<td>+ (single bond)</td>
<td>+ (tetric ceram)</td>
</tr>
</tbody>
</table>

PA – 37% phosphoric acid; Ad – adhesive; CR – composite resin

Table 2 Technical specifications of the Er:YAG laser

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wavelength</td>
<td>2.94 µm</td>
</tr>
<tr>
<td>Pulse width</td>
<td>200–450 µs</td>
</tr>
<tr>
<td>Focus diameter</td>
<td>0.77 mm</td>
</tr>
<tr>
<td>Focus radius</td>
<td>0.385 mm</td>
</tr>
<tr>
<td>Spot area</td>
<td>0.466 mm²</td>
</tr>
<tr>
<td>Energies per pulse</td>
<td>260 mJ</td>
</tr>
<tr>
<td>Frequency</td>
<td>10 Hz</td>
</tr>
<tr>
<td>Mean intensity</td>
<td>5.6 W/cm²</td>
</tr>
<tr>
<td>Fluencies</td>
<td>55 J/cm²</td>
</tr>
<tr>
<td>Focal distance</td>
<td>13 mm</td>
</tr>
<tr>
<td>Irradiation mode</td>
<td>Focused in scanning</td>
</tr>
</tbody>
</table>

2. Materials and methods

This study was approved by the Ethics Research Committee of the School of Dentistry of Araraquara (UNESP – State University of São Paulo). Thirty human molars teeth recently extracted caries-free and non-restored were cleansed and stored in distilled water and 0.2% thymol [8] until the experiment was carried out, for a period not longer than three months. The whole experiment followed the methodology for microtensile bond strength test [9].

2.1. Dentinal surface preparation

The teeth were sectioned 2 mm from the occlusal (Isomet 1000, Buehler Ltd., Lake Bluff, IL, USA), and a diamond disk series 15Lc (T=1/2”, with 6” dia/0.020, # 11.4276), under water coolant. Dentinal surfaces were ground during 1 minute with granulated sandpapers 300, 400, and 600 (DF Vasconcelus S.A. – São Paulo – Brasil) in a DP-10 polishing machine, (Panamba Industral e Técnica S.A. – São Paulo – Brasil) under water coolant, to completely eliminate the residual enamel (sandpapers 300 and 400) and to create standardized smear layer (sandpaper 600). The elimination of the enamel islets were confirmed using an optical microscope (DF Vasconcelus S.A. – São Paulo – Brasil), in a 30× magnification. Samples were randomly divided into six groups (n=5), as shown in Table 1, and stored in distilled water for 24 hours in room temperature.

The samples from the G2, G4, and G6 groups (control groups) were not submitted to laser irradiation. The samples from G1, G3, and G5 groups were irradiated with Er:YAG laser (Twinlight, Fotona Medical Lasers – Slovenia), under air and water coolant. The Er:YAG laser hand-piece was fixed perpendicularly to the dentinal surface, at a 13 mm distance, with the aid of a translator with 3 axes x–y–z (Model A LH Starret – USA). The tooth was manually moved in vertical and horizontal directions so that a homogeneous irradiation was provided for the whole area of exposed dentin. The appliance specifications as well as the irradiation parameters are shown in Table 2.

2.2. Bonding procedures

The samples were submitted to the adhesive procedures in the whole dentinal surface. The materials composition and the manufacturers are listed in Table 3. All the restoration procedures were performed according to the manufacturers’ instructions. The samples from G1, G2, G5, and G6 Groups were etched with 37% phosphoric acid for 15 s before the adhesive agent application. The specimens from G3 and G4 Groups were etched with self-etching adhesive system Adper Prompt L-pop. The teeth were filled with tetric ceram composite resin, shade A3, in 1.5 mm increments and light-activated separately for 20 s. The halogen light equipment (KM-200R/DMC, Equipamentos Ltda.EPP-São Carlos-SP-Brasil) used for resin activation was tested for light intensity with a radiometer (LM-10, Coherent HTD, USA) coupled to a multimeter (Fieldmaster, Coherent, USA), where the minimal values were always equal or higher than 450 mW/cm². A metallic matrix was used to provide a cylindrical restoration with 5 mm of height and 6 mm of diameter. After storage in distilled water at 37°C for 24 h, the samples were thermocycled (model 521.4-serie 95) (550 cycles of 10±2°C and 50±2°C).

2.3. Microtensile bonding test

All the samples were sectioned in specimens of 1 mm²±1 mm² of transversal section and height of ap-
Table 3 Materials used in the study

Table 4 Data of the microtensile bond strength to dentin (mean ± standard deviation in MPa)

Figure 1 (online color at www.lphys.org) Mean values of microtensile bond strength of dentin, MPa

3. Results

Bonding strength averages and standard deviation are shown in Table 4 and Fig. 1. The analysis of data showed a significantly lower decrease in the bond strength when dentin surface was treated by Er:YAG laser irradiation prior to accomplishing the adhesive procedure (p < 0.05).

After testing failure mostly occurred involving the specimen/adhesive interface (adhesive failure) for the all groups (Table 5).

The resin-dentin interfaces after debonding for the three adhesive systems tested are illustrated in the scanning electron micrographs in Fig. 2.
Table 5 Modes of failure

<table>
<thead>
<tr>
<th>Groups</th>
<th>Adhesive failure</th>
<th>Mixed failure</th>
<th>Cohesive failure</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1 (laser + EX)</td>
<td>73.07% (19)</td>
<td>23.07% (6)</td>
<td>3.84% (1)</td>
<td>100% (26)</td>
</tr>
<tr>
<td>G2 (EX)</td>
<td>82.60% (19)</td>
<td>8.69% (3)</td>
<td>4.34% (1)</td>
<td>100% (23)</td>
</tr>
<tr>
<td>G3 (laser + PR)</td>
<td>89.49% (17)</td>
<td>10.51% (2)</td>
<td>–</td>
<td>100% (19)</td>
</tr>
<tr>
<td>G4 (PR)</td>
<td>86.21% (25)</td>
<td>13.79% (4)</td>
<td>–</td>
<td>100% (29)</td>
</tr>
<tr>
<td>G5 (laser + SB)</td>
<td>95% (19)</td>
<td>5% (1)</td>
<td>–</td>
<td>100% (20)</td>
</tr>
<tr>
<td>G6 (SB)</td>
<td>92% (23)</td>
<td>4% (1)</td>
<td>4% (1)</td>
<td>100% (25)</td>
</tr>
</tbody>
</table>

4. Discussion

Considering the interaction between the bonding agents and the surface treatment accomplished, it was observed that the use of all adhesive systems on Er:YAG lased dentin resulted in the lowest bond strength mean. The major mechanism of bonding to dentin surface relies directly upon the entanglement of hydrophilic monomers to the exposed collagen web and thereby depends on the availability and integrity of the fiber mesh [10]. A possible cause for such results would be that the greater heterogeneity of lased substrate (mostly arising from the union of microcraters derived from laser procedure) coupled with the less uniform etching pattern produced by irradiation (Fig. 2a and Fig. 2b). The dentinal substrate is a target tissue with a stronger interaction with Er:YAG laser irradiation due to the great water content in its composition. The incident radiation is highly absorbed by water molecules in dentin components and structures, mainly the intratubular fluid and collagen network, leading to sudden heating and water evaporation. The resulting high-stream pressure leads to the occurrence of successive microexplosions with ejection of tissue particles, which are characteristic of the ablation process and determine the microcrater like appearance of lased surfaces [10–12].

Previous studies [6,13,14] reported that Er:YAG lasing of dentin leads to fractures and fissures not fully impregnated by the bonding resin during the adhesive technique, thus creating a weakened zone just below the interface. Based on the microscopic observations, presence of microcraters, scaly and flaky surface appearance (Fig. 2a and Fig. 2b), we are able to affirm those characteristics are responsible for poor performance to adhesive systems on Er:YAG lased dentin, resulting lowest bond strength mean. The results of this study agree with several previous studies (Ramos et al. (2002) [10], Aoki et al. (1998) [13], Dunn et al. (2005), [14] Martinez-Insua et al. (2000) [15], Ceballos et al. (2002) [16], Brulat et al. (2007) [17], and partially with Gurgan et al. (2007) [18] that suggested that adhesion on dentin surface treated with Er:YAG depends of the adhesive system used. However, disagree of the studies of Visuri et al. (1996) [19], Bertrand et al. (2004) [20], Botta et al. (2007) [21], Malta et al. (2007) [22], Marraccini et al. [23], Marraccini et al. [24] and Jelinkova et al. [25]. The works of Marraccini et al. [24] and Jelinkova et al. [25] accomplished only morphological evaluation. These studies reported that Er:YAG lasing of dentin leads to rough surface, open dentinal tubules[24], and strong adhesion to the composite filling material [25]. However, these studies do not performed evaluation of the bond strength.

Ceballos et al. (2002) [16] reported that there existed 3–4 µm of dentin surface subsurface that is denatured, with
cross-banding lost and were fused together, in Er:YAG irradiated dentin, decreasing interfibrillar spaces that would impair primer infiltration, and, thus, avoiding the formation of the hybrid layer. It is well known that a hybrid layer will be adequately formed only in the presence of preserved collagen structure [26], otherwise monomer penetration would not happen.

The dentin with no laser irradiation presented higher bond strength values for etch-and-rise adhesive systems. As these adhesive systems are ethanol and water based, it permeates the demineralized dentin supported collagen network that will be infiltrated by monomers and form a hybrid layer, as well as resin tags [26], which could be responsible for the higher bond strength observed in the unirradiated groups. On the other hand, the lower values of microtensile bond strength presented by self-etching adhesive system on dentin surfaces irradiated and no irradiated could be due to high concentration of hydrophilic acid molecules that could increase a absorption of water by the adhesive resin and form hydrophilic zones, called of “water trees”, before polymerization, interfering with the mechanical properties [27]. This phenomenon could partially explain the lower adhesion values presented by Adper Prompt L-pop adhesive system.

Concisely, the values of bond strength in dental substrate irradiated with Er:YAG found in literature are still imprecise and conflicting, due to the differences of employed methodology. The studies present variation of energy, frequency and time of application of the Er:YAG laser, which can explain the different results obtained in this study. It may be concluded that the Er:YAG laser had a negative influence in of the bond strength of adhesive systems tested in dental substrate. The etch-and-rise adhesive systems presented higher values of bond strength, independently of the experimental conditions. Further investigations are necessary to determine which adhesive protocol and irradiation parameters should be used to yield an optimal bonding to laser-treated dental substrate, before Er:YAG laser use can be established as a reliable technology in restorative dentistry.

References