Bonding of Self-etching and Total-etch Systems to Er:YAG Laser-irradiated Dentin. Tensile Bond Strength and Scanning Electron Microscopy

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This study investigated the effect of Er:YAG laser on bonding to dentin and the interaction pattern of different adhesive systems with the lased substrate. Tensile bond strength of a self-etching [Clearfil SE Bond (CSEB)] and two total-etch [Single Bond (SB) and Gluma One Bond (GOB)] systems to lased and non-lased dentin was evaluated and the adhesive interface morphology was examined by SEM. Dentin was either treated following the manufacturers’ instructions (A) or submitted to Er:YAG lasing (80 mJ; 2 Hz) + adhesive protocol (B). Resin cones were bonded to demarcated dentin sites and tested for tensile strength. For SEM, dentin discs were obtained, bisected and the halves were treated (A or B). The adhesive interfaces were examined. Means of tensile bond strength (in MPa) were: CSEB: (A) 20.65 ± 1.81, (B) 14.06 ± 1.88; SB: (A) 18.36 ± 1.48, (B) 16.19 ± 1.90; GOB: (A) 16.58 ± 1.94, (B) 14.07 ± 2.13. ANOVA and Tukey tests showed that lasing of dentin resulted in a significant decrease in bond strength (p<0.05). In the non-lased subgroups, CSEB had higher bond strength than the total-etch adhesives (p<0.05). Conversely, in laser-ablated specimens, CSEB had the lowest bond strength, while SB had the highest values (p<0.05). Consistent hybrid layers were observed for conventionally treated specimens, whereas either absent or scarce hybridization zones were viewed for lased subgroups. Er:YAG laser irradiation severely undermined the formation of consistent resin-dentin hybridization zones and yielded lower bond strengths. CSEB self-etching primer appeared to be the most affected by the laser ablation on the dentin substrate, resulting in the weakest adhesion.

Key Words: Er:YAG laser, dentin, tensile bond strength, scanning electron microscopy.

INTRODUCTION

Due to its highly efficient absorption by both water and hydroxyapatite (1,2), the erbium:yttrium:aluminum garnet (Er:YAG) laser has been reported to effectively remove dental hard tissues with minimal injury to the pulp and without causing severe thermal side effects, such as cracking, melting or charring of the remaining tooth structure and/or surrounding tissues (1-3). Over the previous and current decades, bonding of adhesive materials to lased tooth substrates has become a subject of increasing interest for dental laser research. A large number of studies (4-9) have focused on investigating the potential applicability of Er:YAG laser in dentistry and, moreover, the feasibility of performing the adhesive protocols on lased dentin surfaces.

Because of its high water content, dentin is a target tissue that has a strong interaction with the Er:YAG laser beam, which emits a 2.94-micrometer wavelength in the mid-infrared range, close to the main absorption peak of water. During irradiation, the incident energy is readily absorbed by water molecules present in dentin crystalline structures and organic components (mainly the intratubular fluid and collagen network), thus causing sudden heating and water vaporization. The resulting high-stream pressure within...
the lased tissue leads to the occurrence of multiple micro-explosions that constitute the major principle of Er:YAG laser ablation and produce a non-uniform destruction of tooth structure and the ejection of both organic and inorganic tissue particles (1,2,10).

Er:YAG laser irradiation of dentin has been reported to yield a scaly, anfractuous surface with open dentinal tubules and no smear layer (1-3,10). These characteristics led some authors (4,10) to assume that the etching pattern created by the laser ablation would be favorable for bonding procedures. Nevertheless, since the morphological appearance of lased dentin strongly differs from that of the conventionally acid-etched substrate (3,6,10,11), there has been a major interest to investigate the interaction pattern between the currently available adhesive systems and the laser-irradiated dentin; the concern being the quality and integrity of the resin-dentin interface affected. Indeed, a review of the literature on the effects of irradiating dental surfaces with Er:YAG laser prior to bonding procedures reveals controversial outcomes (4,5-12). These divergent results can be ascribed not only to differences in the methodologies and laser equipment, but also to the specificities inherent to the various adhesive systems tested. It seems reasonable to assume that the characteristics of the evaluated materials in different studies (taking into account the great variety of compositions, bonding mechanisms and pretreatments) might have compromised to a great extent their performance on lased dental substrates.

In spite of being an established and predictable clinical procedure, acid etching of dentin has always concerned both clinicians and researchers, and studies have extensively described features inherent to dentin conditioning that can influence the bonding performance of adhesives. Moisture control under clinical conditions has proven to be a particularly critical and definite factor for the quality of adhesion (13). Moreover, inadvertent over-drying of etched dentin after acid rinsing substantially increases the risk of collapse of the collagen mesh, which restricts the diffusivity of the resin monomers throughout the intertubular dentin (13). In addition, excessive etching of dentin may produce weak bonding due to the possibility that the resin monomers may not be able to penetrate into the open dentinal tubules and diffuse across the hydrated demineralized collagen network as deep as the etchant agent. Thus, this lack of penetration leaves behind non-impregnated or poorly infiltrated, unsupported areas at the base of the hybrid layer, which are more prone to micro- and nano-leakage, collagen hydrolysis and degradation of the interface over time (13,14).

These shortcomings prompted further developments in adhesive dentistry, and the concept of utilizing the smear layer as a bonding substrate was reintroduced to discussion. The goals of the so-called self-etching primer adhesive systems were to simplify the bonding procedure and reduce the technique sensitivity of the adhesive protocol by eliminating the need for acid-conditioning, rinsing and drying of etched dental substrate. This promising approach to adhesion involves the use of primers with acidic monomers that slightly demineralize the dentin surface by partially dissolving the mineral crystals around the collagen fibrils and simultaneously provide resin infiltration beyond the smear-covered surface into the underlying dentin matrix. Therefore, a hybrid layer is formed with the smear layer incorporated to it (15). Since etching and priming occur simultaneously, the risk of incomplete resin impregnation of the demineralized dentin is dramatically minimized. Improved bonding performance of self-etching systems to dentin has been reported (16).

It is important to emphasize that dental materials are originally developed to be applied onto tooth surfaces prepared/treated by rotating instruments and conventional techniques. Thus, in view of the widespread appeal for adhesive dentistry and the increasing approach of laser technology in dental practice, it seems relevant to investigate the effect of laser ablation on adhesion and assess the interaction pattern of the adhesive systems with lased dentin substrate, due to its physiological dynamics, heterogeneous composition and complex tubular structure.

Therefore, the aims of this study were to assess the tensile bond strength of one self-etching and two total-etch adhesive systems to Er:YAG laser-irradiated dentin and to examine the morphology of resin-dentin interfaces under scanning electron microscopy. The null hypothesis tested was that there is no difference in the bonding of self-etching and total-etch adhesives to either sound dentin or laser-ablated dentin.

MATERIAL AND METHODS

Fifty-seven sound human molars, extracted within a six-month period, were selected, cleaned and stored
in 0.9% saline solution with 0.4% sodium azide at 4°C.

**Tensile Bond Strength**

For tensile bond strength tests, 45 teeth were used. Roots were sectioned 2 mm below the cemento-enamel junction and crowns were bisected longitudinally in a mesiodistal direction with a water-cooled diamond saw (Minitom, Struers A/S, Copenhagen, Denmark), thus providing 90 halves. Surfaces were identified to avoid both buccal and lingual halves of the tooth being assigned to the same experimental group. Halves were individually embedded in polyester resin (Milflex Indústria Química Ltda., São Bernardo do Campo, SP, Brazil) using PVC rings (2.1 cm diameter; 1.1 cm high) as molds (Figure 1A). After resin polymerization, the rings were removed and the tooth/resin blocks were ground in a water-cooled polishing machine (Politriz DP-9U2, Struers, A/S) with #320- and #400-grit silicon carbide (SiC) paper (Buehler Ltd., Lake Bluff, IL, USA) to remove the overlying enamel and expose superficial dentin (Figure 1B). Viewing the ground surfaces under a 20X magnifier ensured complete removal of enamel. The flat, smooth dentin surfaces were polished with #600-grit SiC paper for 30 s to produce a standardized smear layer.

To demarcate the dentin bonding site, a piece of insulating tape with a 3-mm-diameter central hole, made by means of a modified Ainsworth rubber-dam punch, was attached to specimen surface (Figure 1C). Demarcation of the bonding site had a double aim: to define a fixed test surface, so that the bond strength recorded would be solely related to the evaluated area and to ensure that the truncated resin composite cone would be precisely adhered to dentin surface, thus avoiding accidental adhesion to the surrounding enamel or to non-lased dentin (for Er:YAG lased specimens).

The specimens were randomly assigned to 3 groups of equal size (n=30), according to the adhesive system: Clearfil SE Bond (CSEB); Single Bond (SB); Gluma One Bond (GOB). The tested materials with their compositions, specifications and manufacturers are listed in Table 1. Each group was divided into two subgroups (n=15), depending on the surface treatment carried out: (A) conventional bonding protocol, as recommended by the manufacturers; (B) the dentin site was irradiated with a 2.94-µm wavelength Er:YAG laser, with an 80 mJ pulse energy and a 2 Hz repetition rate (frequency), and then the bonding protocol was performed. The experimental groups and subgroups are detailed in Table 2. Since various studies (6,7-9,11,12) have shown that tooth preparation/conditioning by the laser only (i.e., without further acid etching) yields markedly low bond strengths, a subgroup irradiated with Er:YAG laser alone was not included in our study.

**Table 1. Adhesive systems - compositions, specifications and manufacturers.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Clearfil SE Bond</th>
<th>Single Bond</th>
<th>Gluma One Bond</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Type</strong></td>
<td>Self-etching primer adhesive system</td>
<td>Total-etch adhesive system</td>
<td>Total-etch adhesive system</td>
</tr>
<tr>
<td><strong>Principle Ingredients</strong></td>
<td>Primer: HEMA, MDP, water; Bond: MDP, Bis-GMA, HEMA, silanated colloidal silica</td>
<td>Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, ethanol/water</td>
<td>HEMA, 4-META, UDMA, acetone</td>
</tr>
<tr>
<td><strong>Batch No.</strong></td>
<td>Primer: 00157A 9DC 135603</td>
<td>9DC 135603</td>
<td>135603</td>
</tr>
<tr>
<td><strong>Manufacturer</strong></td>
<td>Kuraray Co., Ltd. Osaka, Japan</td>
<td>3M ESPE St Paul, MN, USA</td>
<td>Heraeus Kulzer Dormagen, Germany</td>
</tr>
</tbody>
</table>

MDP = 10-methacryloyloxydecyl dihydrogen phosphate.

**Table 2. Experimental groups and subgroups.**

<table>
<thead>
<tr>
<th>Subgroup</th>
<th>Clearfil SE Bond</th>
<th>Single Bond</th>
<th>Gluma One Bond</th>
</tr>
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<tbody>
<tr>
<td>A</td>
<td>Self-etching primer</td>
<td>35% phosphoric acid etching</td>
<td>20% phosphoric acid etching</td>
</tr>
<tr>
<td>B</td>
<td>Er:YAG laser + self-etching primer</td>
<td>Er:YAG laser + acid etching</td>
<td>Er:YAG laser + acid etching</td>
</tr>
</tbody>
</table>

Since various studies (6,7-9,11,12) have shown that tooth preparation/conditioning by the laser only (i.e., without further acid etching) yields markedly low bond strengths, a subgroup irradiated with Er:YAG laser alone was not included in our study.
Figure 1. Schematic illustration of specimen preparation and tensile bond strength testing. A: Hemi-crown embedded in polyester resin; B: dentin surface exposed after grinding; C: 3-mm-diameter dentin bonding site demarcated with insulating tape; D: split bisected polytetrafluoroethylene jig; E: metallic clamping device; F: resin/tooth block and polytetrafluoroethylene jig positioned in the clamping device (lateral and upper views); G: inverted, truncated resin composite cone adhered to the demarcated dentin site; H: apparatus used for tensile bond testing.
The Er:YAG laser device used was the Kavo Key Laser 2 model (Kavo Dental GmbH & Co., Biberach, Germany). The laser beam was delivered on non-contact, defocused mode, with a fine water mist at 5 mL/min. A 2051 handpiece, attached to the flexible fiber delivery system, was used. The irradiation distance was standardized using a custom designed apparatus consisting of a holder that positioned the handpiece in such a way that the laser beam was delivered perpendicular to the specimen surface, at a constant working distance of 17 mm from the target site, and a semi-adjustable base, on which the specimen was fixed with wax. Two previously trained operators manipulated the apparatus micrometer screws, in such a way that the semi-adjustable base with the specimen was alternately moved in right-to-left and forward-to-back directions, thereby allowing the laser beam to provide an accurate irradiation of the entire dentin site. The time of irradiation was an average of 40 s.

The tested adhesive systems were carefully applied with disposable tips to avoid excess and pooling of adhesive along the edges of the insulating tape that could compromise the distribution of tension during the tensile test and hence the validity of results. For group CSEB (A and B), Clearfil SE Bond self-etching primer adhesive system was used. Primer component was applied to dentin, left undisturbed for 20 s and gently air-dried to evaporate the volatile ingredients. No subsequent rinsing was carried out. Then, a uniform layer of Bond component was applied to the entire surface, slightly thinned with a mild, oil-free air stream and light-cured for 10 s, with a visible light curing unit (XL 3000; 3M/ESPE) with a 450 mW/cm² output. Group SB (A and B) was bonded with Single Bond, an ethanol- and water-based, total-etch single-bottle bonding agent. The dentin surface was etched with a 35% phosphoric acid gel (Scotchbond etchant, 3M/ESPE) for 15 s, rinsed thoroughly for 15 s and gently dried with absorbent paper to remove excess water and keep the surface moist. Two consecutive coats of Single Bond were applied to the entire surface, slightly thinned with a mild, oil-free air stream and light-cured for 10 s, with a visible light curing unit (XL 3000; 3M/ESPE) with a 450 mW/cm² output. Group GOB (A and B) was bonded with Gluma One Bond. The dentin surface was etched with a 20% phosphoric acid gel (Gluma Etch 20 gel, Heraeus Kulzer Inc.) for 20 s, rinsed thoroughly and gently dried. The adhesive system was applied to the etched surface, left for 10 s and a second coat was richly applied. GOB was spread over the surface with a gentle air-stream to evaporate the solvent and the residual moisture and light-cured for 20 s.

After the bonding protocols were completed, specimens were individually fixed in a metallic clamping device (developed at the Houston Biomaterials Research Center and manufactured at the Precision Workshop at the School of Dentistry of Ribeirão Preto), keeping the dentin surface parallel to a flat base (Figure 1E). A split bisected polytetrafluoroethylene jig (Figure 1D) was positioned on the tooth/resin block surface, thus providing an inverted conical cavity with the smaller diameter coincident with the demarcated 3-mm-diameter bonding site (Figure 1F). A hybrid light-curing resin composite (Z250, 3M/ESPE) was inserted into the jig in three increments, each polymerized for 40 s. As the cavity was filled, the specimen was removed from the clamping device and the jig was opened, leaving adhered to the demarcated dentin surface an inverted, truncated resin composite cone with a 6-mm-diameter tapering to a 3-mm-diameter and 4 mm high (Figure 1G).

After 24-h storage in distilled water at 37°C, the cone-shaped composite/polyester resin block was placed into an apparatus with an internal taper, corresponding to the resin cones’ shape. This configuration was loaded in tension, using a universal testing machine (MEM 2000, EMIC Ltda., São José dos Pinhais, PR, Brazil), running at a cross-head speed of 0.5 mm/min and a 50 kgf load cell until fracture (Figure 1H).

Bond strengths were recorded in kgf and converted into MPa. Mean and standard deviation were calculated and data were analyzed by two-way ANOVA using a factorial design with adhesive system and surface treatment as independent variables. Multiple comparisons were done using a Tukey statistical test at a 0.05 significance level. Fractured specimens were observed with a 40X stereomicroscope to assess the failure modes, which were classified as adhesive, cohesive or mixed.

Scanning Electron Microscopy

The remaining 12 molars were utilized in this part of the study. Using a sectioning machine, the occlusal overlying enamel surface of the teeth was
eliminated with a water-cooled diamond saw at low-speed to prevent fracturing or overheating the tooth structure. To warrant complete removal of enamel, the exposed dentin surfaces were always viewed under a 20X magnifier. Then, for each tooth, another cut was done perpendicular to the long axis of the tooth, thus providing a dentin disc 1.0 ± 0.1 mm thick per tooth.

The discs were randomly assigned to 3 groups (n=4), according to the adhesive system (Clearfil SE Bond, Single Bond, Gluma One Bond), and then bisected in a buccal-lingual direction. The resulting hemi-discs had their pulp surface isolated with two layers of a colorless nail varnish to prevent the release of organic solvents through dentinal tubules. The dentin surface corresponding to the occlusal side of the hemi-discs was polished with #600-grit SiC papers for 30 s to produce a standardized smear layer and then treated following the same experimental conditions (i.e., non-lasing or lasing) and the Er:YAG laser parameters (i.e., energy, frequency, irradiation distance) stated for bond strength tests. The working distance (17 mm) was standardized using the same custom-designed apparatus described for tensile strength specimens. The irradiation length was 90 s, on average. It is important to highlight that different laser irradiation times are found in our study because we utilized specimens with different shapes and dimensions for tensile bond strength tests and SEM analysis. Since the dentin discs used for SEM examinations had greater dimensions than the 3-mm-diameter dentin bonding sites, increased laser irradiation time was required to perform a complete, uniform lasing of the entire surface.

Afterwards, a layer of Z250 light-cured resin composite was placed on the dentin surface to facilitate the subsequent sectioning of the specimens. The restored halves were bisected perpendicular to the resin/tooth interface, and the resulting fragments were hand polished with #600- to #4000-grit SiC paper and treated according to the following protocol: first, the resin/dentin interface was etched with a 37% phosphoric acid gel for 5 s, rinsed and the samples were ultra-sonicated (Ultrasonic Cleaner T-1449-D, Odontobrás Ind. e Com., Ribeirão Preto, SP, Brazil) for 10 min, thoroughly washed with distilled water and immediately immersed in 2.5% glutaraldehyde (Merck KGaA, Darmstadt, Germany) in 0.1 M sodium cacodylate buffer at pH 7.4 (Merck KGaA), for 12 h at 4°C. After fixation, the samples were rinsed with 0.1 M sodium cacodylate buffer several times, sequentially dehydrated in an ascending ethanol series (25% for 20 min; 50% for 20 min; 75% for 20 min; 90% for 30 min; 100% for 60 min), then immersed in 100% hexamethyldisilazane (HMDS) (Merck KGaA), for 10 min, placed on absorbent paper inside glass plates and left drying in an exhaust system. Specimens were mounted on stubs with their treated surfaces up-faced and sputter-coated with gold. The adhesive-dentin interfaces were examined with a JSM T330 scanning electron microscope (JSM T330A, JEOL, Tokyo, Japan), operating at 15 kV, for the formation or not of a hybrid layer, focusing on its integrity and homogeneity along the interface, as well as on the arrangement, uniformity of size and hybridization characteristics of resin tags.

RESULTS

Tensile Bond Strength

Bond strength data for non-lased and lased subgroups are shown on Table 3.

As regards the factor adhesive system, not taking into account the surface treatment performed, CSEB showed the highest bond strength to dentin and GOB had the lowest bond strength (p<0.05). As to the variable surface treatment, two-way analysis of variance showed a significant decrease in bond strength when the dentin surface was irradiated with Er:YAG laser before the adhesive protocols were carried out (p<0.05). This decrease was the largest for Clearfil SE Bond.

Multiple comparison tests assessed the interactions between the variables adhesive system and surface treatment and revealed that CSEB self-etching system applied on conventionally treated dentin surfaces yielded the highest bond strength (20.65 MPa) (p<0.05). No statistically significant difference (p>0.05) was found between the bond strengths recorded for the

<table>
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<tr>
<th></th>
<th>Clearfil SE Bond</th>
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<th>Gluma One Bond</th>
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<tbody>
<tr>
<td>A</td>
<td>20.65 ± 1.81</td>
<td>18.36 ± 1.48</td>
<td>16.58 ± 1.94bc</td>
</tr>
<tr>
<td>B</td>
<td>14.06 ± 1.88d</td>
<td>16.19 ± 1.90c</td>
<td>14.07 ± 2.13d</td>
</tr>
</tbody>
</table>

Same letters indicate statistical similarity.
total-etch systems in non-lased specimens (18.36 MPa and 16.58 MPa for Single Bond and Gluma One Bond, respectively). On the other hand, comparing the performance of the adhesive systems on Er:YAG laser-ablated substrates, CSEB yielded the lowest mean bond strength (14.06 MPa) (p<0.05), while SB obtained the best results (16.19 MPa) and was statistically different from the 2 other systems (p<0.05).

The analysis of bonding sites after tensile strength tests revealed that, for non-lased fractured specimens, an adhesive-failure mode was predominantly observed (SB: 73%; GOB: 70%; CSEB: 58%). The total-etch systems showed a limited number of cohesive failures in dentin (7%), which were not observed for the self-etching system. However, when the dentin surface was irradiated with Er:YAG laser prior to the bonding protocols, there was a notable alteration in the fracture pattern and mostly mixed failures occurred. For all systems, there was a significant decrease in the number of adhesive failures and a remarkable increase in the number of cohesive failures in dentin. For the self-etching system, a significant number of cohesive failures in dentin (27%) was observed among the Er:YAG laser-irradiated specimens.

Figure 2. SEM photomicrographs showing the interfacial morphology of Clearfil SE Bond with conventionally treated dentin (A; B) and Er:YAG laser-irradiated dentin (C; D). A: A hybrid layer (HL) apparently thinner than that produced by the total-etch systems was observed. However, it was consistent and continuous throughout the adhesive interface. R = resin composite. B: The resin tags (T), even though shorter than those formed by the total-etch adhesives, exhibited consistent triangular-shaped hybridization at the transition between the peritubular and intertubular dentin, sealing the tubules’ openings (*). C: Conversely, notice the absence of a typical resin-dentin interdiffusion zone or hybrid layer along the adhesive interface (arrowheads). Intratubular and intertubular lateral resin branches (RB) were seen. D: In most specimens, consistent resin/dentin hybridization zones (HL) were seldom identified (arrowheads).
**Scanning Electron Microscopy**

Analysis of the SEM micrographs revealed that, in non-irradiated specimens (Figures 2AB, 3AB, 4AB), the signs of hybridization were evident, with the formation of consistent, homogeneous hybrid layers, extending along the interface. Also, there were resin tags of varying lengths and conical shape, with the larger base at the tubule openings, funneling into dentinal tubules. In contrast, in the Er:YAG laser-ablated specimens (Figures 2CD, 3CD, 4CD), areas of consistent hybridization were seldom identified. The formation of ill-defined, irregular hybrid layers, with various interruptions along the resin-dentin interface was mostly observed. In several specimens, consistent resin-dentin interdiffusion zones were absent, especially for the self-etching system. Resin tags, even though present in great number, appeared much thinner, and did not exhibit the triangular-shaped hybridization at tubule openings. Intratubular and intertubular lateral resin branches were occasionally found, probably due to the cratered nature of laser-ablated dentin (Figures 2C, 4C).

Figure 3. SEM photomicrographs showing the interfacial morphology of Single Bond with conventionally treated dentin (A; B) and Er:YAG laser-irradiated dentin (C; D). **A**: Consistent, well-defined resin-dentin interdiffusion zone or hybrid layer (HL), extending along the interface (arrowheads). **A** = adhesive layer. **B**: Cone-shaped resin tags (T), with the larger base at the dentinal tubule entrances funneling into them, were observed throughout. Evident signs of hybridization at the transition between the peritubular and the intertubular dentin were often seen (*). **C**: In contrast, the zones of consistent hybridization were rarely identified and, instead, the formation of ill-defined, irregular hybrid layers with various interruptions along the resin-dentin interface was mainly observed. The asterisks (*) indicate areas where hybridization possibly occurred, while the arrowheads point to regions with apparent absence of hybrid layer formation. **D**: The resin tags (T), even though present in a good number, appeared much thinner and did not exhibit the triangular-shaped hybridization at tubule openings.
DISCUSSION

The outcomes of this research seem to corroborate the assumption that the Er:YAG laser exerts a deleterious effect on both mineral and organic components of dentin and thereby impairs the optimal interaction of the adhesive systems to laser-etched substrate. Our results disclosed that the irradiation of dentin surface with an Er:YAG laser, prior to the application of self-etching and total-etch adhesive systems, adversely affected the interaction pattern of the adhesive systems with the lased dentin and yielded a remarkable decrease in bond strength. Similar results were reported in other studies (5,6,7-9,11,12), in which bond strength of different adhesive systems to laser-treated dentin was tested to failure in tensile, micro-tensile or shear and the interfacial morphology was observed under SEM and TEM. As a rule, these authors advocate that it seems unlikely that the formation of an interdiffusion zone between the resin monomers and the irradiated intertubular dentin, i.e., a hybrid layer, occurred with the same characteristics observed on conventionally...
treated dentin surfaces. Er:YAG laser irradiation has been reported to produce a laser-modified superficial layer with a peculiar morphological pattern, in which collagen fibers are partially denatured, fused and/or melted, poorly attached to the underlying dentin substrate and having lost part of their cross-banding (12). According to these authors, this amorphous collagen layer, which is completely devoid of interfibrillar spaces, probably restricts resin interdiffusion into the subsurface intertubular dentin, thus undermining the formation of an authentic, typical hybrid layer (12). Rather, in view of the anfractuous, cratered nature of Er:YAG laser-ablated surfaces, adhesion to lased dentin appears to develop mainly from the mechanical retention provided by resin tag formation into open dentinal tubules and by the infiltration and entanglement of the bonding agent into the micro-irregularities and micro-craters created during irradiation (4,12). We assume that all these events were responsible for the lower bond strength observed for the laser-ablated specimens.

In the present work, Clearfil SE Bond self-etching primer produced bond strength significantly higher than those of the total-etch systems on conventionally treated subgroups. Recent investigations (16,17) evaluating Clearfil SE Bond and various adhesive systems reached similar results. It has been advocated that, in spite of forming thinner hybrid layers (0.5-1.0 µm) than those formed by the total-etch systems (2.0-5.0 µm), the self-etching primers may provide bond strengths to dentin comparable or even superior to those obtained with adhesive systems that advise the acid-etching as a separate step of the bonding protocol (15-17).

In contrast to its performance on non-lased specimens, the self-etching primer adhesive system yielded the lowest bond strength in the Er:YAG-lased subgroups, and confirmed the outcomes of previously reported studies (7,8). The limited effectiveness of self-etching primers on laser-treated dentin could be ascribed to the limited capacity of the acidic monomer to demineralize the laser-modified superficial layer and alter the morphological pattern resulting from it. According to previous studies (18), the degradation of dentin organic substances and the changes in size and ultrastructure ofapatite crystals resulting from laser irradiation substantially increase the acid-resistance of lased dentin. Additionally, it has been reported (19) that Er:YAG laser radiation modifies calcium-to-phosphorus ratio, reduces carbon-to-phosphorus ratio and leads to the formation of more stable and less acid-soluble compounds, thus reducing dentin susceptibility to acid attack. Therefore, it seems feasible to speculate that an etchant agent with stronger acid potential, such as 35% phosphoric acid (which is part of Single Bond adhesive protocol), is expected to present higher efficiency in removing the dentin layer modified by the laser than an etchant agent with weaker acid potential, such as the acidic monomer (Clearfil SE Bond self-etching primer) or even 20% phosphoric acid, as recommended for Gluma One Bond.

The analysis of the adhesive interfaces by scanning electron microscopy revealed the formation of consistent, well-defined hybrid layers, continuous along the adhesive interface, when the bonding protocols were performed on non-lased dentin substrates. Additionally, there were tags of varying lengths and conical shape, with the larger base sealing the tubule entrances and funneling into them. This indicates demineralization of peritubular dentin and suggests hybridization of resin tags to tubule walls (16). Conversely, for the Er:YAG laser-irradiated subgroups, the formation of ill-defined hybrid layers exhibiting an irregular and discontinuous pattern along the interface was observed. In several specimens, mainly those in which the self-etching primer was applied, a typical resin-dentin interdiffusion zone was hardly identified. These findings agree with those of previous studies (5,6,8,11,12). Also observed was the formation of thinner, less pronounced resin tags, with parallel walls and diameter similar to that of the original tubule lumen. Consistent signs of hybridization at the transition between peritubular and intertubular dentin were rarely seen. Since hybridization of the lateral walls of dentinal tubules was lacking, lower tensile bond strengths were expected for lased specimens. Similar results and conclusions were reported by previous investigations (5,6,9,12).

As regards the types of failure observed in the fractured specimens, an adhesive-failure mode was predominantly observed in the non-lased specimens. These findings indicate that failure after testing mostly occurred at the interface between the adhesive system and dentin substrate. In contrast, in the laser-irradiated specimens, there was an alteration in the fracture pattern, with a greater number of mixed failures and a significant increase in the number of cohesive failures in dentin, mostly for the self-etching adhesive system. The findings of an earlier investigation (6) disclosed...
that surfaces treated by Er:YAG laser fractured cohesively in dentin, probably due to the formation of a degraded and structurally modified dentin surface layer, constituting an altered zone, weakly adhered to dentin substrate, assumed to be more susceptible to a cohesive-failure mode. The predominance of mixed failures in the irradiated subgroups may be attributed to the fact that the Er:YAG laser beam does not provide a uniform, homogeneous etching pattern (20), leaving non-lased areas between pulses. It may be, then, speculated that failures could have first occurred in laser-ablated areas (probably cohesive failures in dentin, due to the aforementioned reasons) during the tensile strength testing. Next, adhesive or cohesive failures in resin could have occurred in the areas not reached by the laser beam, in which bonding to dentin substrate is expected to be stronger, thereby characterizing a mixed-failure mode.

In view of its potential for ablating dental substances, the Er:YAG laser has been pointed out as a technology of outstanding applicability in operative dentistry and appears as a promising alternative to replace high- and low-speed handpieces. Nevertheless, there is still too much to be investigated on the ultimate effect of lasing on tooth structure before the use of laser devices becomes routine in dental practice. The questions raised and the diverging standpoints presented in this study stress the fact that the reports in the literature concerning the application of Er:YAG laser in dentistry are conflicting in several aspects, mainly those regarding the feasibility of performing adhesive procedures on irradiated surfaces. Furthermore, the great variety of current adhesive systems is a crucial feature to be considered. Depending on the recommended bonding protocol, a specific interaction pattern with the lased substrate should be expected. Therefore, it is mandatory that further studies be developed with the aim of investigating the structural alterations that Er:YAG laser ablation produces on dentin, mainly its effects on dentinal organic contents, to assess the type of interaction that occurs between the adhesive systems and the lased substrate and thus foresee, with some degree of reliability, the quality of the adhesion obtained.

Based on the findings of this research, the null hypothesis was rejected. The irradiation of dentin with an Er:YAG laser adversely affected the interaction pattern of total-etch and self-etching adhesive systems with the lased substrate, i.e., severely undermined the formation of consistent resin-dentin hybridization zones and yielded a significant decrease in bond strength. Clearfil SE Bond self-etching system appeared to be the most affected by the micro-structural alterations produced by the laser on the dentin substrate and resulted in the weakest adhesion.

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