Microtensile Bond Strength of Two Adhesives Bonded to Laser Treated Dentin.

A. HAFEZ¹, A. ABO EL NAGA², O. BARAKAT², and H. EL-SHENAWY³

¹Ass. Prof. Dental Biomaterials, Faculty of Dentistry, Mansoura University, Egypt.
²Ass. Prof. of operative Dentistry, Ibn Sina National College for Medical Studies, Jeddah, Saudi Arabia.
³Lecturer and Researcher in Medical & Biological Application of Laser Science .National Research Centre, Cairo, Egypt. Corresponding author: Olarhm@hotmail.com

Abstract: Objectives: Evaluation of the effect of laser and etching time on the microtensile bond strength of two adhesives applied to dentin surfaces. Methods: Occlusal surfaces of forty molars were ground to obtain flat dentin surfaces. The teeth were divided into two groups (n=20 in each). One group subjected to Er.YAG laser. Each group was divided into four subgroups according to the used adhesive system and the etching time: 1) Prime &Bond NT etched for 15 seconds. 2) Adper Scotchbond Multi-Purpose Plus etched for 15 seconds. 3) Prime &Bond NT etched for 60 seconds. 4) Adper Scotchbond-Multi-Purpose Plus etched for 60 seconds. For each treated specimen, Feltik Supreme Plus composite was incrementally applied to form composite build up. After 24 hours distilled water storage at 37°C, the bonded specimens were vertically sectioned into 0.8mm² sticks. Two central sticks were randomly chosen from each specimen forming a total of 10 sticks per subgroup. The sticks were subjected to a tensile load using Lloyd universal testing machine at crosshead speed of 0.5 mm/minute. The data were statistically analyzed using One-way ANOVA and Tukey’s test (P≤ 0.05). Results: There was no statistically significant difference in the mean microtensile bond strength values between subgroup 2 of non laser treated dentin (39.62 MPa) and subgroup 4 of Laser treated dentin (41.54 M Pa). This was followed by subgroup 1 for non Laser treated dentin which showed lower values (30.39). There was no statistically significant difference in the mean microtensile bond strength values between subgroups; 1 (11.97 MPa), 2 (14.59 MPa) and 3 (14.62 MPa) for laser treated dentin surfaces. Conclusion: Both tested adhesives showed decreased bond strength when the dentin surfaces were laser treated and etched for 15 seconds. Etching of laser treated dentin surfaces for 60 seconds improved microtensile bond strength.

Key words: adhesive system, Laser, microtensile bond

1.Introduction: During the last years, an expressive number of studies, Visuri et al.,(1996); Cozean et al.,(1997); Keller et al.,(1998); Hossain et al.,(1999); Borsatto et al.,(2001); Ceballos et al.,(2001); Corona et al.,(2001); Roebuck et al.,(2001) have been focused on investigating the efficiency of erbium: yttrium aluminium garnet (Er:YAG) laser for potential dental applications, such as removal of carious lesions, cavity preparation, surface conditioning, endodontic procedures, and others. Emitted at a 2.94-mm wave length, Er laser is strongly absorbed by water and is well absorbed by hydroxyapatite. The ablation of tooth structure is achieved via a thermo mechanical interaction and since the tissue is not completely vaporized, but only disintegrated into fragments. The majority of incident radiation is consumed in the ablation process, leaving very little residual energy for adverse thermal interactions to the pulp and surrounding soft and/or hard structures, Hibst and Keller(1989); Visuri et al.,(1996); Cozean et al.,(1997); Keller et al.,(1998); Hossain et al.,(1999); Borsatto et al.,(2001); Ceballos et al.,(2001); Corona et al.,(2001); Roebuck et al.,(2001) .

The effect of lasers on dentin is caused mainly by the changes in temperature that can be extremely high at irradiated spot even for a short action time. Consequently, the dentin melts, vaporizes, and a crater is formed at the irradiation site. Laser energy causes a quick local temperature rise and prompts melting, recrystallization, and decomposition of the apatite crystals. Heat conduction should lead to a thermal equalization between the pronouncedly heated components and the surrounding tissues when a laser pulse of the order of a few micro seconds is used. The phase transformation and structural changes of the enamel at different temperature intervals have been investigated, Kuroda and Flower (1984). However, similar changes in the dentin are less well known. According to the study of Kantola (1973)
recrystallization of the dentin occurred during CO$_2$ laser irradiation. Simultaneously, growth in the crystal size was observed, and dentin of a low order of crystalline changed structurally in such a way that it came to closely resemble the crystalline structure of the hydroxyapatite of normal enamel.

Several studies have shown that the use of Er:YAG laser irradiation on root surface makes some thermal changes, like melting the cementum and root dentine which may partially or totally obliterate dentinal tubules. In addition, side effects such as carbonization, craters, and micro cracks, Morlock et al.,(1992); Lan and Liu(1996). Several authors have reported that laser irradiation on dentine surface can occlude dentinal tubules. However, some of them have emphasized that the severity and kind of root surface changes are related to power of laser, Lan et al.,(1999); Gaspric and Skaleric (2001); Lan et al.,(2004); Magalhaes et al.,(2004) . Post-operative sensitivity is frequently encountered with the use of adhesives that require conditioning of the dentin, Akpata and Sadiq (2001); Unemori et al., (2001).Incomplete sealing and continuous transduction of dentinal fluid through open dentinal tubules before polymerization of the adhesive may result in entrapment of water-filled blisters along the adhesive interface, Tay et al.,(1996) Compression of these blisters during mastication may cause, within the dentinal tubules, rapid fluid movement that activates the intradental nerve fibers, which results in postoperative sensitivity Barnstorm and Astrom(1972); Narhi et al.,(1994)

The aim of this study is to evaluate the effect of laser and etching time on the microtensile bond strength of two adhesives applied to dentin surfaces.

2.Materials and Methods:
2.1.Materials:
2.1.1.samples:
A total of forty freshly extracted sound human molars were collected. Teeth were cleaned by removing the remaining soft tissues and stored in physiologic saline solution until testing. Each tooth was embedded in an acrylic resin up to cement-enamel junction (CEJ) using a special designed mould. Dimension of the mould was 20 mm width x 20 mm length x 15mm height. The occlusal third of the crown of each tooth was removed by sectioning the crown perpendicular to its long axis using a low speed diamond saw (Buehler- Isomat, LakeBulff, IL, USA) under copious amount of water. The exposed dentin were finished using 600 Grit Wet Silicone Carbide abrasive papers in circular motion under water coolant to create a uniform, clinically relevant smear layer. The teeth were then rinsed, dried and the exposed dentin surfaces were inspected under a stereomicroscope to ensure removal of all enamel remnants.

The teeth were randomly divided into two groups (n=20):

**Group one** subjected to Er:YAG laser (60mJ, 15Hz, and 0.9W) for 1 minute,

**Group two** did not subjected to laser.

Each group then divided into four subgroups (n=5) according to the adhesive used and etching time:
1) Two-step, Prime&Bond NT, (Dentsply) (PBNT) etched for 15seconds.
2) Three-step, Adper Scotchbond Multi-Purpose Plus, (3M/ESPE) (SBMP) etched for 15seconds.
3) PBNT etched for 60seconds.
4) SBMP etched for 60seconds. The adhesive systems were applied according to manufacturer’s instructions. Table (1) shows the composition and manufacturers’ instructions of the used adhesive systems.

2.2.Methods:
2.2.1.scheme of the work:
Each composite cylinder was light cured for 40 seconds using visible-light curing unit (PRO-DENT systems, Inc. Portland, USA) at intensity of 500 m W/ cm$^2$. Light intensity output was monitored using visible curing light meter (Cure Rite, EFOS Inc; Ontario, Canada). After 24 hours distilled water storage at 37°C, the bonded specimens vertically sectioned into 0.8mm$^2$ sticks.

Two central sticks were randomly chosen from each specimen forming a total of 10 sticks per each subgroup. Sticks were subjected to micro tensile load using a universal testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) at crosshead speed of 0.5 mm/minute. Data were statistically analyzed using One-way ANOVA and Turkey’s test (P≤ 0.05).

2.2.2.Statistical analysis:
Data were statistically analyzed using One-way ANOVA and Turkey’s test (P≤ 0.05).

3.Results:
The results of Microtensile bond strength means and slandered deviations for two adhesive systems bonded to laser treated or non laser treated dentin surfaces are shown in table 2. Regarding to the non laser treated dentin, it was found that three step
SBMP) etched for 15 s recorded the highest mean value (39.62 MPa) followed by (SBMP) etched for 60 s (31.80 MPa), then (PBNT) etched for 15 s (30.39 MPa). While (PBNT) etched for 60 s recorded the lowest mean value (26.38 MPa).

Regarding to laser treated dentin (SBMP) etched 60s recorded the highest mean value (41.59 MPa) followed by (PBNT) 60s (14.62 MPa) then (SBMP) 15s (14.59 MPa), while (PBNT) 15 s recorded the lowest mean value (11.97MPa).

Two-way ANOVA showed a non significant difference in mean of microtensile bond strength value between subgroups 1, 2 and 3 for laser treated dentin. Tukey’s test indicated that the highest microtensile bond strength was found with Adper Scotch bond Multipurpose adhesive applied for 60 sec for laser treated dentin

Table 1: The manufacturers, composition and manufacturers’ instructions of the used adhesive systems.

<table>
<thead>
<tr>
<th>Adhesive System</th>
<th>Classification</th>
<th>Composition</th>
<th>Instruction for use</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prime &amp; Bond NT Dentsply Caulk, Milford Del.</td>
<td>Two-step technique</td>
<td>Etchant (Gel): 34% phosphoric acid with silica Adhesive: PENTA, Urethane dimethacrylate and T- resin (cross linking) and D- resin (small hydrophilic molecule).</td>
<td>1-Etchant surface with 34% tooth conditioning gel for 15 sec. 2-Rinse thoroughly for 10 sec. 3-Blot dry; dentin should be moist and brushed with Prime &amp; Bond NT adhesive to wet the surface 4- Light activation (10s-60smW/cm2)</td>
</tr>
<tr>
<td>Adper Scotchbond Multi-Purpose Adhesive (MP) 3M ESPE Dental Products, St. Paul, MN USA</td>
<td>Three step technique</td>
<td>37% phosphoric acid Primer-Aqueous solution of HEMA, Adhesive –BIS-GMA,HEMA Dimethacrylate and initiator</td>
<td>1 – Acid etching (15 s), rinsing (15 s) and Air drying(10 s); 2 – Application of 2 coats of the primer (10s with slight agitation); 3 – Air-drying (10 s at 20 cm); 4 – Application of 1 coat of the adhesive (10 s with slight agitation); 5 – Air-drying (10 s at 20 cm); 6 – Light-activation (10 s - 600 m W/cm2)</td>
</tr>
</tbody>
</table>

Table 2: The mean of Microtensile bond strength for two adhesive systems bonded to laser treated or non laser treated dentin surfaces.

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Non laser treated dentin</th>
<th>Laser treated dentin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Two-step, Prime&amp;Bond NT, (PBNT) etched for 15seconds.</td>
<td>Mean: 30.39 MPa SD: 2.72</td>
<td>Mean: 11.97MPa SD: 1.79</td>
</tr>
<tr>
<td>Two-step, Prime&amp;Bond NT, (PBNT) etched for 60seconds</td>
<td>Mean: 26.38 MPa SD: 2.48</td>
<td>Mean: 14.6 MPa SD: 1.39</td>
</tr>
<tr>
<td>Three-step, Adper Scotchbond Multi-Purpose Plus, (SBMP) etched for 60seconds.</td>
<td>Mean: 31.8 MPa SD: 2.03</td>
<td>Mean: 41.59 MPa SD: 4.76</td>
</tr>
</tbody>
</table>

4. Discussion:
The ability of Er:YAG laser to effectively ablate dental hard tissues is ascribed to its 2.94-mm wavelength emission, which is coincident to the main absorption band of water (3.0 mm) and is also well absorbed by OH groups in hydroxyapatite, Kuroda and Flower(1984); Hibst and Keller(1989); Cozean et al.,(1997). Due to the great water content in its composition, dentinal substrate is a target tissue with a stronger interaction with Er: YAG laser irradiation. 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 micro explosions with ejection of tissue particles, which are characteristic of the ablation process and determine the micro crater-like appearance of lased surfaces.

Table 3: The mean, SD values and results of comparison between the groups

<table>
<thead>
<tr>
<th>Group</th>
<th>Mean</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>No laser (2 steps 15 Sec)</td>
<td>30.39</td>
<td>2.72</td>
</tr>
<tr>
<td>No laser (3 steps 15 Sec)</td>
<td>39.62</td>
<td>4.52</td>
</tr>
<tr>
<td>No laser (2 steps 60 Sec)</td>
<td>26.38</td>
<td>2.48</td>
</tr>
<tr>
<td>No laser (3 steps 60 Sec)</td>
<td>31.80</td>
<td>2.03</td>
</tr>
<tr>
<td>Laser (2 steps 15 Sec)</td>
<td>11.97</td>
<td>1.76</td>
</tr>
<tr>
<td>Laser (3 steps 15 Sec)</td>
<td>14.59</td>
<td>1.30</td>
</tr>
<tr>
<td>Laser (2 steps 60 Sec)</td>
<td>14.62</td>
<td>1.39</td>
</tr>
<tr>
<td>Laser (3 steps 60 Sec)</td>
<td>41.59</td>
<td>4.76</td>
</tr>
</tbody>
</table>

*: Significant at p≤ 0.05, different letters indicate statistically significant according to Tukey's test

A number of new adhesive systems have been developed in an attempt to reduce the steps and simplify clinical bonding procedures. Two major simplified bonding approaches have developed, Van Meerbeek et al., (1998). The first utilities the total – etching technique to simultaneously remove the smear layers from both enamel and dentine surfaces, followed by application of one –bottle agent that combines the primer and the adhesive in on solution, Li et al.,(2000). As the demineralised collagen fibril mesh is used as the bonding substrate, a wet bonding technique is required to insure its full expansion, Gwinnett (1992); Kanka (1992). The need for moist dentine surface in complex cavity preparations often create over wet and under wet regions in the same tooth, making bonding to dentine with adhesives very sensitive technique, Tay et al.,(1996); Van Meerbeek et al.,(1998); Miyazaki et al.,(2000) .The second approach is the use of self etching primer, Bouillaguet et al.,(2001) .Their bonding mechanism is based upon the simultaneous etching and priming the seam layer covered to dentin using an acidic primer, Hayakawa et al.,(1995); Nakabayahi and Saimi (1996); Inoue et al.,(2000), followed by the application of an adhesive resin. Self etching primers eliminate the separate acid –etching and rinsing steps, simplifying bonding techniques and reducing the technique sensitivity, Tay and Pashley(2001). The results of this study showed that dentin treated with Er: YAG laser before etching for 15 second for both adhesive system 2 step and 3 step techniques reduce the microtensile bond strength. This may be due to the changes in collagen fibre and dentin surface with the laser beam and also the time of etching (15 second) is not sufficient to produce changes in the dentin surface. These results were in agreement with Barnstorm et al ..,(1972); Tay et al.,,(1996) who concluded that the potential impact of the Er: YAG laser on collagen network has not yet been clearly disclosed. It remains unclear if the microstructural alteration and microrupture of collagen fibres caused by laser irradiation could actually compromise the interaction of adhesive systems with lased dentin substrate, which would inherently affect the resulting bond strength. This speculation is based on the fact that 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 fibre mesh. Therefore, as the literature has strongly emphasized, Barnstorm and Astrom(1972); Tay et al.,(1996) if the structure of the collagen net somehow collapses or is altered, the penetration of primer monomers and hence the adhesive protocol is hampered and incomplete.

The findings of an earlier investigation, Kataumi and Nakajima (1998) showed that Er:YAG laser irradiation followed by acid-etching led to the formation of hybrid layer and resin tags equivalent in size and depth to those obtained with acid-conditioning
only. Dentin surfaces solely lased showed no hybrid layer formation and the tags were much thinner as compared to other groups. Nevertheless, no significant difference was observed among the tested groups regarding bond strength to dentin. According to the authors, a reasonable explanation for this similarity would be that, although laser did not lead to a hybrid layer formation, the adhesive system reached the open dentin tubules and superficial micro-cracks resulting from irradiation, thus providing good micromechanical retention even without subsequent acid conditioning. Meanwhile, another study Kameyama et al.,(2008) investigated the tensile bond strengths of three step adhesive and two step self etch adhesive to both Er:YAG laser –irradiated and non irradiated enamel and concluded that no significant differences were noted between Er:YAG laser –irradiated and non irradiated enamel for each adhesive tested.

Results of this study revealed that there was no statistical differences in the mean microtensile bond strength values between subgroup 4 of laser treated dentin (3 step with 60 sec etching) and subgroup 2 of non laser treated dentin (3 step with 15 second etching) .This may be due to the hard changes in the dentin surface after laser treated needed to increase the time of etching with 37% phosphoric acid to improve the surface tension, conditioning and open the dentinal tubules.

However, for three-step adhesives system, it has been reported that phosphoric acid etching for 60 second increases micro tensile bond strength to laser treated dentin. While two-step self-etch adhesive system exhibited the highest micro tensile bond strengths with non-treated dentin. This was probably due to the acidity of two steps self etching adhesive was milder than phosphoric acid in the three step techniques.

Recommendation: For surface treated dentin with laser we prefer to use 3 step adhesive system and increase the time of etching to 60 second to obtain the highest values for microtensile bond strength.

Conclusion:
Under the test conditions, both tested adhesives showed decreased bond strength when the dentin surfaces were laser treated and etched for 15 seconds. After 60 seconds etching of laser treated dentin might improve microtensile bond strength of three-step adhesives.

5. References: