

# Microshear Bond Strength of OptiBond All-in-One Self-adhesive Agent to Er:YAG Laser Treated Enamel After Thermocycling and Water Storage



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## Introduction

Despite recent advances in the manufacture of composite resins, polymerization shrinkage and subsequent reduction in bond strength remain the major drawbacks of composite restorations.<sup>1</sup> The acid etch technique was first introduced by Buonocore in 1955 as a standard technique for enamel surface preparation to enable mechanical interlocking and bonding of resin restorations to enamel. The formation of resin tags enhances the bond of resin materials to tooth structures in this technique.<sup>2</sup>

In the past decade, self-etch bonding systems were introduced to enable simple bond to enamel and dentin simultaneously.<sup>3-5</sup> Self-etch adhesives have less technical sensitivity than etch and rinse systems since in the former, conditioning and priming of the enamel and dentin surfaces are performed concomitantly.<sup>3,5,6</sup> The most re-

## Abstract

**Introduction:** This study aimed to compare the microshear bond strength of composite to enamel treated with Erbium-Doped Yttrium Aluminum Garnet (Er:YAG) laser using a self-etch one step bonding agent.

**Methods:** Seventy-six enamel surfaces were prepared from 38 sound human third molar teeth. Specimens were randomly divided into four groups of 18. The enamel surface in half the specimens was irradiated with Er:YAG laser. One extra specimen from each group was evaluated under a scanning electron microscope (SEM). Composite micro-cylinders were bonded to the specimen surfaces using OptiBond All-In-One (OB) adhesive agent and stored in distilled water for 24 hours. Half the specimens were thermocycled (2000 cycles) and stored in distilled water at 37°C for three months (TW). The microshear bond strength of composite to enamel was measured using a universal testing machine at a crosshead speed of 1 mm/min. The fractured surfaces were evaluated under a stereomicroscope at ×40 magnification to determine the mode of failure. Data were analyzed using repeated measures analysis of variance (ANOVA) and *t* test.

**Results:** The mean values (±standard deviation) were 17.96±2.92 MPa in OB group, 22.29±4.25 MPa in laser+OB group, 18.11±3.52 MPa in laser+OB+TW group and 9.42±2.47 MPa in OB+TW group. Repeated measures ANOVA showed that laser irradiation increased the microshear bond strength ( $P<0.001$ ). Bond strength decreased when the samples were thermocycled and stored for three months ( $P<0.001$ ). The interaction effect of water storage and laser treatment on bond strength was significant ( $P<0.05$ ).

**Conclusion:** Enamel surface preparation with Er:YAG laser is recommended to enhance the durability of the bond of self-etch bonding systems to enamel.

**Keywords:** thermocycling; shear bond strength; morphology; adhesives; lasers; water storage

cent generation of self-etch adhesive systems introduced to the dental market is the all-in-one seventh generation bonding agents; in which, all phases of etching, priming and bonding have been combined into one single step. All-in-one adhesives are believed to provide lower bond strength values and have higher technical sensitivity than two-step self-etch systems. However, these one step adhesive agents are more appealing to dentists due to the simplicity of use and shorter chairside time.<sup>7,8</sup>

Long-term water storage and thermocycling can decrease the bond strength of these systems.<sup>9,10</sup> Alternative techniques such as air abrasion and laser irradiation have been recommended for conditioning enamel and dentin surfaces without compromising the tooth structure.<sup>6</sup> The mechanism by which laser effects the bond to enamel is via causing physical and structural changes in the enamel

surface by increasing its mineral content, removing the smear layer and forming a new compound via the process of recrystallization.<sup>11,12</sup> Enamel etching with laser creates an irregular surface ideal for composite bond. Some studies have reported enhanced composite bond to permanent teeth after laser irradiation due to increased micromechanical retention.<sup>13-16</sup> It has been reported that laser-etched surfaces are resistant to acid attacks because laser changes the calcium to phosphorous ratio and decreases the carbonate to phosphate ratio. Consequently, the solubility of enamel decreases and it becomes more resistant to acid attacks and development of secondary caries.<sup>17,18</sup>

Results of previous studies are controversial about the effects of thermocycling and water storage on the bond strength of one-step self-etch adhesives to enamel. Some studies stated that long-term storage significantly decreased the bond strength<sup>9,10</sup> while some others found no significant difference in this regard.<sup>19-21</sup>

This study aimed to compare the microshear bond strength of a seventh generation bonding system to laser etched enamel following thermocycling and three months of water storage. The results of this study may help improve the durability and clinical service of restorations bonded with single-step seventh generation bonding systems.

## Methods

This *in vitro* experimental study was conducted on 36 surgically extracted sound human third molars. The teeth were immersed in 10% buffered formalin (Shahid Ghazi Co., Tabriz, Iran) for four months prior to the study. Then, they were cleaned with a prophylactic brush and a mixture of water and pumice paste and were randomly divided into four groups of nine. Teeth with cracks, abrasion, caries, restorations or dental anomalies were excluded and replaced with sound teeth. Teeth crowns were cut and separated from the roots at the cemento-enamel junction using a rotary diamond disc under cooling water. The crowns were then cut into halves mesiodistally and 72 enamel buccal and lingual pieces were prepared. Using silicon molds, each half-crown was embedded in auto-polymerizing acrylic resin (Marlic Med Co., Tehran, Iran). After setting acrylic resin, the acrylic surface was ground parallel to the horizontal plane to obtain an exposed enamel surface measuring 4×4 mm. To uniform the surface of specimens and standardize the smear layer, the enamel surface was ground using a 600 grit abrasive paper under running water.

In the control group (OB), the enamel surface of specimens was conditioned using OptiBond All-In-One (Kerr, Orange, CA, USA) self-etch one-step bonding system according to the manufacturer's instructions. Adequate amount of bonding agent was applied to the enamel surface in two consecutive steps of 20 seconds each and dried with gentle air spray for five seconds. A plastic cylinder with an internal diameter of 0.8 mm and height of 1mm was placed on prepared enamel surfaces and light

cured for 10 seconds using Demi LED Light Curing System (Kerr Corp, Orange, CA, USA). A3 shade of Clearfil APX composite resin (Kuraray Co., Tokayama, Japan) was applied into the microtubes by a periodontal probe and light-cured for 40 seconds. The plastic microtubes were carefully separated from the composite cylinder after one hour and specimens were immersed in distilled water at room temperature for 24 hours.

In laser-etched enamel plus OptiBond All-In-One adhesive system (Er:YAG + OB), the same steps were followed as in OB control group except that prior to the application of adhesive agent, enamel surfaces were etched with Erbium-Doped Yttrium Aluminum Garnet (Er:YAG) laser at a wavelength of 2.94  $\mu\text{m}$  irradiated with Fotona laser device (Fotona, Fidelis3 Plus, Ljubljana, Slovenia) with 10 Hz frequency, 100  $\mu\text{s}$  pulse duration, 100 mJ pulse energy, 1 W output power and 6/4 water to air ratio. Laser was manually irradiated using a R14 handpiece with Sapphire tip (0.8 mm diameter) from 0.5 mm distance almost perpendicular to the surface at a speed of 1 mm/s and horizontally scanned the entire surface by a sweeping motion. In Er:YAG + OB + TW and OB + TW groups, the same steps were followed as in the control and Er:YAG + OB groups. The only difference was that after curing the composite resin, the specimens were thermocycled for 2000 cycles and stored in water at 37°C for three months. Each thermal cycle included immersion in a hot bath at 55±1°C for 30 seconds and cold bath at 5±1°C for 30 seconds with a 30-second dwell time at room temperature.

A mechanical universal testing machine (SANTAM, SMT-20, Iran) was used to measure the microshear bond strength of composite to enamel. The specimens were fixed to the jaw of the testing machine. A fine brass wire with 0.2 mm diameter was looped around each composite cylinder in such a way that the metal loop embraced the lower half of the composite resin cylinder and was in contact with the tooth surface. The specimens were subjected to shear stress at a crosshead speed of 1mm/min until fracture. The microshear bond strength of each specimen was recorded in MPa. Data were analyzed using repeated measures analysis of variance (ANOVA). The significance level was set at  $P=0.05$ . Paired *t* test was used for pairwise comparison of groups ( $\alpha=0.02$ ). The fractured surfaces were evaluated under a stereomicroscope (SZ240, Olympus, Tokyo, Japan) at ×40 magnification to determine the mode of failure (adhesive, cohesive in dentin, cohesive in composite resin and mixed) by one operator. Four extra teeth were selected and prepared for micro-morphological assessment prior to applying composite. Teeth crowns were cut, the teeth were sectioned mesiodistally and four enamel buccal pieces were selected for evaluation under a scanning electron microscope (SEM). Each buccal half-crown was embedded in auto-polymerizing acrylic resin. The enamel surface of non-treated specimen was ground by a disc and abrasive paper. This surface received no conditioning. Next, the specimens were rinsed for 10 seconds with acetone as an organic solvent followed by 10 seconds of rinsing with 96% alco-

hol. For the purpose of desiccation, the specimen were immersed in 50%, 70%, 95% and 100% concentrations of alcohol, respectively for 30 seconds followed by 30 seconds of drying with air spray.<sup>22</sup> This desiccation protocol was also performed for other specimens. The surface of specimen in OB group was conditioned by OptiBond All-In-One bonding agent according to the manufacturer's instructions; but no light curing was done. Rinsing and drying were performed as described earlier. Er:YAG laser was employed for lasing and etching of the enamel surface of specimen in Er:YAG laser group with the aforementioned parameters. In Er:YAG + OB group after laser conditioning of the enamel surface, the bonding agent was applied according to the manufacturer's instructions. The specimen was rinsed as described earlier and air-dried. Next, the surface of specimens was gold-coated using SBC12 sputter coater (KYKY Technology Development Ltd. China) and observed under an electron microscope at  $\times 1000$ ,  $\times 2000$  and  $\times 4000$  magnifications (TESCAN, VEGAII, XMU, Czech Republic).

**Results**

As seen in Table 1, the highest and the lowest mean microshear bond strength values belonged to Er:YAG + OB ( $22.29 \pm 4.25$  MPa) and OB + TW ( $9.42 \pm 2.47$  MPa) groups, respectively. Repeated measures ANOVA showed that laser increased ( $P < 0.001$ ) and thermocycling and water storage decreased ( $P < 0.001$ ) the microshear bond strength of specimens. The interaction effect of laser and water storage on microshear bond strength was statistically significant ( $P = 0.019$ ). Therefore, paired t-test was performed for pairwise comparison of groups. The adjusted *P* value for the family error was 0.02 for paired *t* test (Table 1). Paired *t* test revealed no significant difference between

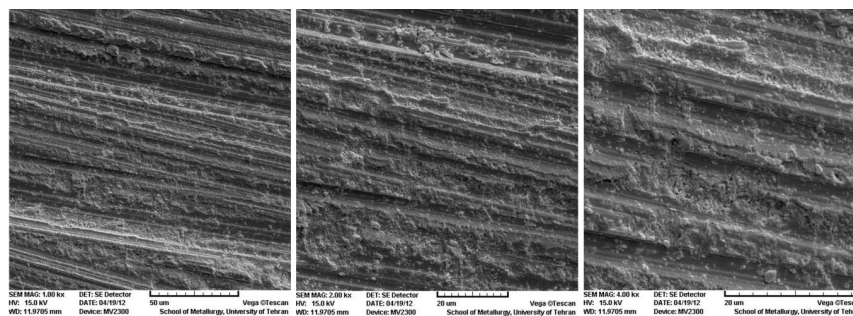
Er:YAG + OB and OB groups ( $P = 0.88$ ); however, the differences between other groups were statistically significant ( $P < 0.001$ ). Table 1 shows the frequency distribution of the modes of failure for specimens in each group after microshear bond strength testing. Figures 1 to 4 show the SEM micrographs obtained from the specimens.

**Discussion**

The assessment of the bonding ability of restorative materials to tooth structures and the durability of the bond under in vitro settings are extremely important since the clinical setting can be simulated as such. Poor adhesion between tooth structures and restorative materials results in gap formation, marginal microleakage, marginal discoloration and caries recurrence.<sup>23</sup>

The current study showed that the irradiation of enamel surface with Er:YAG laser prior to the application of OptiBond All-In-One one-step self-etch adhesive significantly increased the microshear bond strength of composite to enamel ( $P < 0.001$ ). Some other studies have also confirmed this finding.<sup>24-28</sup>

Lasing the enamel surface with erbium lasers creates an irregularly rough surface and allows the penetration of adhesive resin into these porosities and undercuts, resulting in the formation of resin tags and subsequent increased micromechanical retention. Previous studies suggested surface roughening by laser irradiation as an alternative to acid etch technique.<sup>13-16, 29, 30</sup> It has been stated that laser irradiation may provide the micromechanical retention necessary for the bond of adhesives to enamel. This phenomenon is known as the laser etching effect.<sup>13-16, 31</sup> Previous morphological electron microscopic studies reported enhanced micromechanical retention following laser irradiation, as well as increased surface roughness and enamel-resin interface surface area.<sup>16, 27, 31, 32</sup>



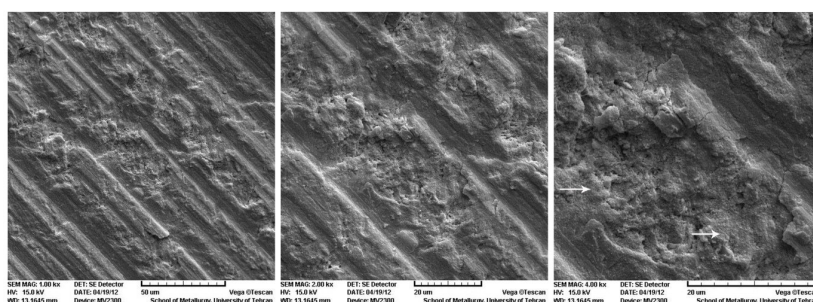
**Figure 1.** SEM Micrographs of Specimens Ground by an Abrasive Disc ( $\times 1000$ ,  $\times 2000$  and  $\times 4000$  Magnifications). Parallel abrasion lines can be seen on the superficial layer of enamel.

**Table 1.** The Mean and SD Values of Microshear Bond Strength (MPa) in the Study Groups (n=18)

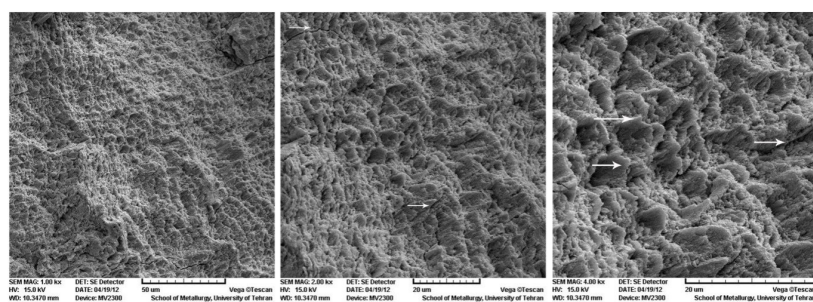
Groups	Laser Surface Conditioning	Thermocycling & Water Storage	Mean	SD	Mode of Failure A/M/C
Control (OB)	No	No	17.96 <sup>A</sup>	2.99	14/4/0
OB + TW		Yes	9.42	2.47	17/1/0
Er + OB	Yes	No	22.29	4.25	14/4/0
Er + OB + TW		Yes	18.11 <sup>A</sup>	3.52	12/6/0

Abbreviations: SD, standard deviation; A, adhesive; M, mixed; C, cohesive. Values with the same uppercase letters indicate no significant difference according to paired *t* test.

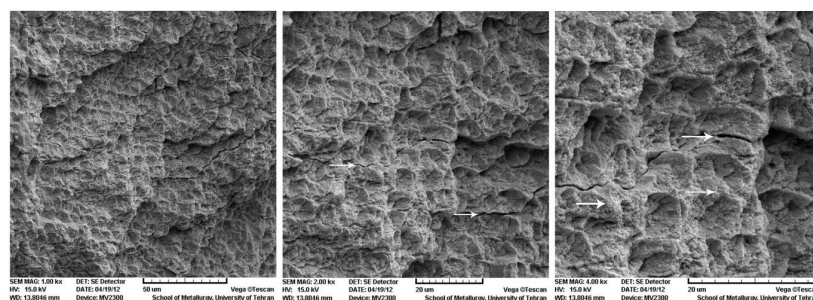




**Figure 2.** The specimen surface was conditioned using OptiBond All-In-One. The parallel abrasion lines were somehow smoothed by the application of OptiBond self-adhesive agent. Microretentive areas are seen in the enamel surface ( $\times 1000$ ,  $\times 2000$  and  $\times 4000$  magnifications).



**Figure 3.** Er:YAG laser-treated Specimen at  $\times 1000$ ,  $\times 2000$  and  $\times 4000$  Magnifications. A rough, irregular enamel surface and honeycomb pattern with sharp marginal porosities created by the application of laser can be seen. At  $\times 4000$  magnification, the crater-like pattern and microcracks are evident.



**Figure 4.** Er:YAG Laser-Treated and OptiBond All-In-One Adhesive-Conditioned Surface. The honeycomb and crater-like patterns are clearly visible. The microcracks created by laser irradiation are seen at  $\times 2000$  and  $\times 4000$  magnifications. In these specimens, the sharp marginal porosities were smoothed by the effect of acidic properties of self-etch bonding system.

As seen in the SEM micrographs in the current study, laser irradiation of enamel created a honeycomb and crater-like pattern and significantly increased the surface roughness. However, microcracks were also seen on SEM micrographs, which are important in two aspects: these microcracks may serve as undercuts and increase mechanical retention if they are not very deep. Nevertheless, frequent and deep enamel cracks may undermine the enamel and compromise the bond strength.<sup>31</sup> The effect of laser parameters on the number of microcracks formed may explain the reason why some studies reported a reduction or no change in bond strength following laser irradiation.<sup>7,27,31</sup>

Kameyama et al in 2008 reported that Er:YAG laser irradiation had no effect on the bond strength of one-step self-etch adhesive systems to enamel. However, the type

of bonding system and laser parameters in their study were different from our settings.<sup>7</sup> De Munck et al, in 2002 compared the application of laser, bur and two types of self-etch and total etch adhesives to enamel and dentin, and reported that laser had no significant effect on bond strength.<sup>33</sup> They attributed the low bond strength to the presence of small cracks and destruction of the underlying enamel layers.<sup>33</sup>

The controversial results regarding the effect of Er:YAG laser on bond strength of composite to enamel may also be attributed to differences in laser parameters and the variability of the adhesive systems.<sup>27,33</sup> Acid application has a significant effect on the etched pattern of the lased enamel surface and the use of 37% phosphoric acid smoothens the effect of enamel surface lasing.<sup>31,33</sup> An optimal bond also depends on laser parameters such as power, pulse en-

ergy, frequency, pulse duration, water cooling during lasing and method of laser application to the enamel surface. Considering the advances in laser systems and their enhanced efficacy, high variability is seen in the adjustment of laser parameters in different studies, which makes the comparison of results difficult. This has also been discussed in some other studies.<sup>23,34</sup> Laser parameters in the current study were adjusted according to the laser device manufacturer's instructions.

Some studies have recommended that low energy laser (200 mJ) is adequate for enamel surface conditioning and the shear bond strength of composite to laser-etched enamel is similar to that for acid-etched enamel surfaces.<sup>35</sup> Staninec et al noticed that narrow pulse width (35  $\mu$ s) compared to wider pulse width (250-500  $\mu$ s) caused different morphological changes and the bond strength in the group with narrow pulse width was similar to that in non-lased groups.<sup>36</sup> Raji et al in 2012 compared 100 and 150 mJ laser energy with acid etching and stated that the shear bond strength of specimens lased with 150 mJ laser was not significantly different from that of surfaces etched with phosphoric acid. However, 100 mJ pulse energy provided significantly lower bond strength.<sup>34</sup> Future studies are required to further assess the effect of laser parameters on bond strength and find the ideal exposure settings that lead to an optimal bond strength to tooth structures.

The type of adhesive system also plays an important role in the strength and the durability of bond. The bond of one-step, self-etch adhesive systems to enamel has always been a concern for clinicians considering their mild etching property and these systems have been compared with total etch systems in terms of bond strength in many studies.<sup>5,9,37</sup> Laser irradiation appears to be efficient prior to the application of self-etch adhesives to enamel, because laser irradiation creates an etched pattern, increases surface roughness and improves mechanical retention.<sup>27</sup>

Another important issue regarding the application of one-step, self-etch systems to enamel is the durability of the bond created. The most commonly used methods for assessment of the durability of materials *in vitro* are thermocycling and water storage, which directly simulate the clinical service of restorations. Although the oral environment is the ultimate environment to test and predict restorations' behavior, *in vitro* methods such as thermocycling and long-term storage in aqueous media can simulate the *in vivo* settings and explain the mechanism of resin-tooth bond disintegration.<sup>23</sup> Although, in the current study thermocycling and three months of water storage decreased the bond strength in all groups ( $P=0.0001$ ), lased specimens had generally higher bond strength than non-lased specimens ( $P=0.0001$ ), and reduction in bond strength following thermocycling and water storage was significantly greater in non-lased groups.

Most previous studies have reported a significant reduction in bond strength of one-step self-etch systems even after short-term water storage.<sup>7,9,38</sup> During thermocycling, as result of the difference in the modulus of materials thermal expansion, stresses are created at the tooth-res-

toration interface.<sup>38</sup> According to ISO TR 11450 (1994) standard, specimens must be subjected to 500 thermal cycles in water between 5-55°C for simulation of clinical service. However, some other studies have stated that 500 thermal cycles are not enough to simulate the clinical setting and suggested conduction of 1000 thermal cycles.<sup>39,40</sup> Water penetration into the bonding agent-enamel interface leads to swelling and plasticization of bonding resin, and during thermal cycles, hot water accelerates the process of resin hydrolysis.<sup>41</sup> Long-term water storage results in penetration of water into the bonding interface, causing nanoleakage and subsequent disintegration and hydrolysis of bonding components. Water diffusion into the bonding layer softens the polymer matrix and decreases its mechanical properties.<sup>19</sup> Some studies have reported optimal bond strength for teeth subjected to laser irradiation, thermal cycling and water storage, and a previous study reported that six months of water storage and 12000 thermal cycles had no significant effect on bond strength.<sup>23</sup>

The enamel surfaces subjected to different conditioning techniques were morphologically analyzed under SEM in the current study. As seen on SEM micrographs, acidic monomers in the bonding agent slightly smoothed the grooves created by polishing discs and created micro-retentive areas on the enamel surface (Figure 1). However, laser etching of enamel created a honey combing and crater-like pattern; although microcracks were also observed in the surface. The creation of such rough and irregular enamel surface can increase the composite enamel bond (Figure 2).

The effect of laser irradiation on the morphology of dental substrate has yet to be completely understood and controversial results have been reported in this regard. Some researchers have reported that Er:YAG laser irradiation of dental surfaces causes specific topographic changes in the surface of dental substrates i.e. removal of smear layer and no melting or carbonization of enamel surface.<sup>33</sup> Moreover, the micro-abrasive mechanism of Er:YAG laser results in the evaporation of water and organic content and creation of crater-like areas in the dental surface, which play a significant role in resin restorations bonding.<sup>23,25-27</sup> Such morphological changes increase the bonding surface area (which probably requires more time for disintegration) and enhance the durability of bond.<sup>23</sup>

The stereomicroscopic assessment of specimens (to determine the mode of failure) showed that the mode of failure for most specimens was the adhesive type (Table 1). However, in specimens subjected to water storage and thermocycling, the adhesive failure had a higher frequency compared to control specimens. This finding was in accord with the bond strength results. It appears that laser irradiation is somehow responsible for changing the mode of failure from adhesive to mixed. Mixed failures had a higher frequency in lased specimens. Considering the daily application of laser systems in dentistry and advances in bonding systems, further studies are required to find new techniques for enhancing the bond and clinical

success of composite restorations.

### Conclusion

Based on the results of this study, the irradiation of Er:YAG laser increases the microshear bond strength of one-step self-etch OptiBond All-In-One adhesive system to enamel. Thermocycling and three months of water storage at 37°C decreased the composite bond strength to enamel and this reduction in microshear bond strength was greater in non-lased groups. Thus, enamel surface conditioning by Er:YAG laser irradiation is recommended to improve the bond strength and durability of one-step self-etch adhesive systems to enamel.

### Ethical Considerations

This study have been approved by ethical committee of the Vice Chancellor of Research, Hamadan University of Medical Sciences.

### Conflict of Interests

All of the named authors have been involved in the work leading to the publication of the paper and have read the paper before its submission for publication. The authors declare that they have no conflict of interest.

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