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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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

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. 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.

In the past decade, self-etch bonding systems were introduced to enable simple bond to enamel and dentin simultaneously. 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. The most recent 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.

Long-term water storage and thermocycling can decrease the bond strength of these systems. Alternative techniques such as air abrasion and laser irradiation have been recommended for conditioning enamel and dentin surfaces without compromising the tooth structure. 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.\textsuperscript{11,12} 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.\textsuperscript{13-18} 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.\textsuperscript{17,18}

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\textsuperscript{19-21} while some others found no significant difference in this regard.\textsuperscript{19-21}

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.

\section*{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, the teeth were sectioned mediodistally and 72 enamel buccal and lingual pieces were prepared. Using silicon molds, each half-crown was embedded in auto-polymerizing acrylic resin. The enamel surface of non-treated enamel was conditioned using OptiBond All-In-One (Kerr, Orange, CA, USA) for 30 seconds and cold bath at 5±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 morphological assessment prior to applying composite. Teeth crowns were cut, the teeth were sectioned mediodistally 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. The 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-
For the purpose of desiccation, the specimens were immersed in 50%, 70%, 95% and 100% concentrations of alcohol, respectively for 30 seconds followed by 30 seconds of drying with air spray. This desiccation protocol was also performed for other specimens. The surface of specimens 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 ×1000, ×2000 and ×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 ± 4.25 MPa) and OB + TW (9.42 ± 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.

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. 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. Previous morphological electron microscopic studies reported enhanced micromechanical retention following laser irradiation, as well as increased surface roughness and enamel-resin interface surface area.

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

<table>
<thead>
<tr>
<th>Groups</th>
<th>Laser Surface Conditioning</th>
<th>Thermocycling &amp; Water Storage</th>
<th>Mean</th>
<th>SD</th>
<th>Mode of Failure A/M/C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control (OB)</td>
<td>No</td>
<td>No</td>
<td>17.96</td>
<td>2.99</td>
<td>14/4/0</td>
</tr>
<tr>
<td>OB + TW</td>
<td>No</td>
<td>Yes</td>
<td>22.29</td>
<td>3.52</td>
<td>12/6/0</td>
</tr>
<tr>
<td>Er + OB</td>
<td>Yes</td>
<td>No</td>
<td>18.11</td>
<td>3.52</td>
<td>12/6/0</td>
</tr>
<tr>
<td>Er + OB + TW</td>
<td>Yes</td>
<td>Yes</td>
<td>18.11</td>
<td>2.47</td>
<td>17/1/0</td>
</tr>
</tbody>
</table>

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.
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. 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. 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. 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. They attributed the low bond strength to the presence of small cracks and destruction of the underlying enamel layers. 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. 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. An optimal bond also depends on laser parameters such as power, pulse en-
ergy, frequency, pulse duration, water cooling during las-
ing and method of laser application to the enamel surface.
Considering the advances in laser systems and their en-
hanced 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 dis-
cussed in some other studies.\textsuperscript{23,34} 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.\textsuperscript{35}
Staninec et al noticed that narrow pulse width (35 µs)
compared to wider pulse width (250-500 µs) caused dif-
ferent morphological changes and the bond strength in the
group with narrow pulse width was similar to that in
non-lased groups.\textsuperscript{36} 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 pro-
vided significantly lower bond strength.\textsuperscript{34} 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 is the durability of
the enamel substrates and these systems have been compared with
total etch systems in terms of bond strength in many stud-
ies.\textsuperscript{5,9,37} Laser irradiation appears to be efficient prior to
the application of self-etch adhesives to enamel, because
laser irradiation creates an etched pattern, increases sur-
face roughness and improves mechanical retention.\textsuperscript{38}
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 en-
vironment is the ultimate environment to test and predict restorations' behavior, in vitro methods such as thermo-
cycling and long-term storage in aqueous media can sim-
ulate the in vivo settings and explain the mechanism of resin-tooth bond disintegration.\textsuperscript{23} 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 reduc-
tion in bond strength of one-step self-etch systems even
after short-term water storage.\textsuperscript{7,26} During thermocycling,
as result of the difference in the modulus of materials
thermal expansion, stresses are created at the tooth-res-
toration interface.\textsuperscript{38} 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 set-
ting and suggested conduction of 1000 thermal cycles.\textsuperscript{39,40}
Water penetration into the bonding agent-enamel inter-
face leads to swelling and plasticization of bonding resin,
and during thermal cycles, hot water accelerates the pro-
cess of resin hydrolysis.\textsuperscript{41} Long-term water storage results
in penetration of water into the bonding interface, caus-
ing nanoleakage and subsequent disintegration and hy-
drolysis of bonding components. Water diffusion into the
bonding layer softens the polymer matrix and decreases
its mechanical properties.\textsuperscript{39} Some studies have reported
optimal bond strength for teeth subjected to laser irra-
diation, thermal cycling and water storage, and a previ-
ous study reported that six months of water storage and
12000 thermal cycles had no significant effect on bond
strength.\textsuperscript{23} The enamel surfaces subjected to different conditioning
techniques were morphologically analyzed under SEM in
the current study. As seen on SEM micrographs, acidic non-lased specimens (Figure 1). However, laser etching of enamel created a honey combing and cra-
ter-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 den-
tal substrate has yet to be completely understood and con-
troversial 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.\textsuperscript{35} More-
over, the micro-abrasive mechanism of Er:YAG laser re-
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.\textsuperscript{23,25,27}
Such morphological changes increase the bonding surface area (which probably requires more time for disintegra-
tion) and enhance the durability of bond.\textsuperscript{23} The stereomicroscopic assessment of specimens (to de-
termine 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 frequen-
cy compared to control specimens. This finding was in
accord with the bond strength results. It appears that la-
sar 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 ad-
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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