Comparative Evaluation of Shear Bond Strength of Flowable Composite Bonded to Teeth Enamel with Laser Surface Treatment.

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Abstract
Background: Acid etching of enamel appears to improve retention by selectively eroding certain hydroxyapatite formations and facilitating penetration by the development of resin tags. Aim of study: To evaluate shear bond strength (SBS) of flowable composite to non-lased and lased groups, compare the (SBS) of flowable composite between Total and Self-etch in non-lased and lased groups, and failure mode analysis.

Materials and Methods: Forty bovine incisors were used, enamel surfaces was grounded by silicon carbide paper and samples were randomly assigned into two main groups (Group A: Non lased n=20, Group B: lased prepared n=20). Each main group was subdivided randomly according to the adhesive systems that used which are: total-etch adhesive and self-etch adhesive, which were applied to the prepared enamel surface according to manufacture instruction. Composite rod applied over the bonded area and cured. Samples were stored in distilled water at 370 for 24 hours. The evaluation of shear bond strength was employed by the use of universal testing machine. Results: The results of Student’s t-test showed that there was no significant difference between the lased and non-lased enamel groups (P= 0.311). One–way ANOVA test and Duncan's Multiple Range test that compare shear bond strength between total and self-etch adhesives showed no significant difference in both lased and non-lased enamel groups. Conclusion: The mean shear bond strength and enamel surface etching obtained with an Er,Cr:YSGG laser was not comparable to that obtained with non- lased enamel surface.

Keywords: Bovine Enamel, Er,Cr:YSGG laser, adhesives, composites, Shear bond strength.

Introduction
Despite improvements to resin composite formulations over the years, polymerization shrinkage of the resin is still considered highly relevant to the failure of direct resin composite restoration (Goracci et al. 1995). Therefore, pretreatment of the tooth surface is essential for establishing a strong bond between the resin and both the enamel and dentin (Swift et al. 1995).

Acid etching has been the standard approach for enamel pretreatment since the publication of Buonocore’s report. Effective adhesion to enamel has been achieved with relative ease and has repeatedly proven to be a durable and reliable clinical procedure for routine applications in modern adhesive restorative dentistry (Buonocore 1955).

A recent laser system which considered effective for the ablation of dental hard tissues is the Er,Cr:YSGG laser (waterlase YSGG;BIOLASE Technology), which emits light in the mid-infrared region at 2.78μm with the assistance of an air and water spray (Dela et al. 2004).

The results of previous studies on the application of laser etching for increasing bond strengths of restorative materials have been controversial. Investigation of enamel surface roughness showed that laser irradiation yielded a comparable or smaller amount of surface roughness than acid etching (Hess 1990, Arcoria et al. 1993). With regard to bond strengths of restorative materials, some studies indicated that acid-etched teeth had significantly more bond strength than laser-Laser etch teeth (Ariyaratnam et al. 1997), whereas other demonstrated that laser etching (Melendez et al. 1992, Walsh et al. 1994, Shahabi et al. 2014,Lorenzo et al. 2015). These variations could be attributed to the different types of lasers or different irradiation parameters used because the laser-hard tissue interaction is dependent on wavelength and irradiation energy.
because the laser-hard tissue interaction is dependent on wavelength and irradiation energy. With the advancement in adhesive dentistry, various adhesive systems were introduced. Total etch adhesive system is one of the commonest adhesive systems used. It contains hydrophilic/hydrophobic primers with the ability to penetrate into the demineralized enamel and dentin created after phosphoric acid etching and smear layer removal. Further development led to what is called self-etch adhesive system to overcome drawback associated with total etch system due to collapse in collagen fibrils following dryness. This system supplied in either one or two step of application. Current two-step self-etching primers or single-step self-etching adhesives (all-in-one systems) produce simultaneous conditioning and priming effects on dental tissues (Loguercio et al. 2008).

The hypothesis tested in this study is that there is no significant difference in shear bond strength between Er,Cr:YSGG lased and non-lased enamel, different adhesives when enamel is etched by Er,Cr:YSGG laser, and different adhesives in non-lased group.

**Aim of the study**
The aims of this study were to: (1) Evaluate shear bond strength (SBS) of flowable composite to non-lased and lased groups. (2) Compare the (SBS) of flowable composite between Total and Self-etch in non-lased and lased groups. (3) Evaluate the failure mode of experimental groups.

**Material and Methods**
Forty bovine incisors, frozen to maintain freshness and defrosted immediately before specimen preparation, were used. Roots were sectioned 2mm below the cemento-enamel junction and the teeth were embedded in acrylic resin (Ivoclarvivadent, Liechtenstein) with the labial surface of the crown exposed and parallel to the base of the resin mold. The labial surfaces of bovine incisors were polished with 320-600 grit size wet silicon carbide papers using a grinding/polishing machine (Struers, Denmark). under a continuous stream of water to obtain flat enamel surface. The teeth were randomly divided into two groups (non-lased and lased) according to the surface treatment. For group (A) non-lased enamel and (B) lased enamel, flowable composite resin (DMG, Hamborg, Germany) used. Then for each group two types of adhesive system used (10 samples for each type): self-etch adhesive (Contax, DMG, Hamborg, Germany) and total etch adhesive (Teco, DMG, Hamborg, Germany).

In all samples of non-lased and lased groups adhesive tape with a central hole of 4mm diameter was attached to the flattened enamel surface. For the lased groups the laser system used was Er,Cr:YSGG (Waterlaseiplus, BIOLASE Technology, USA) in department of oral surgery, Mosul university. The laser energy was delivered through fiber-optic system to a sapphire tip terminal (MZ6) with 600 μm in diameter. Samples were lased for 20 seconds in the non-contact mode perpendicular to the flat surface with a 1-mm fixed distance from the laser tip. A sweeping motion was used to achieve an even coverage of the tested surface by overlapping the laser impacts. The following parameters (2780 nm wavelength, a pulse duration 200 μs, a pulse repetition rate of 10 pulses per second (10 Hz), 2.5 W power, energy: 250 mJ, 60% air spray and 80% water spray) were used. After that the adhesive agents were applied to a standardized enamel bonding site for all samples followed by light curing unit (LEDition, Ivoclarvivadent; Austria) according to the manufacturer’s instruction. After bonding, a translucent standardized plastic straw with an internal diameter of 4mm and height of 4mm was positioned exactly over the hole of the adhesive tape and fixed with a sticky wax. The straw was filled with resin composite (Table1) and light cured for 40 seconds.
Samples were stored in distilled water at 37°C for 24 hours then subjected to a thermal cycling of 500 cycles between 5°C (±2°C) and 55°C (±2°C) using water baths with 30 seconds in each bath. After which, shear bond strength (SBS) test was carried out using a Universal Testing Machine (WP universal material tester, gunt-Hamburg, Germany) at a cross-head speed of 1.0 mm/min. The load was applied to the composite/ tooth surface interface. Maximum load to failure was recorded (digitally) in newton (N) for each sample and then shear bond strength was expressed in megapascals (MPa), in order to respect the unit commonly used in similar research, which is derived by dividing the load at failure (Newtons) by the bonded surface area (12.56 mm²) (Bahrami et al., 2011).

S=\frac{F}{A}

Where: S= Shear bond strength (Mpa),
F= load at failure (N) \quad A= \pi r^2 (12.56 \text{ mm}^2). \pi = 3.14
r= radius of bonding area (2mm)

Following testing procedure, the mode of failures between composite and enamel surface were examined with optical microscope of the enamel surface at X40 magnification using a Stereomicroscope (Motic, Italy). The failure modes were classified as follow: Adhesive failure (enamel exposed); Cohesive failure (composite or adhesive observed on enamel); and Mixed failure (combination of adhesive and cohesive).

**Table 1. Adhesive systems and composite resin composition, batch numbers, and the application modes. according to manufacturer’s instructions.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Application Mode</th>
</tr>
</thead>
<tbody>
<tr>
<td>Teco one bottle 2-step total-etch Adhesive, DMG German; Batch#: 684040</td>
<td>Bis-GMA-based resin matrix, starter, additive, pigments, DMG Etching Gel: O-phosphoric acid in water (37%), aerosol.</td>
<td>Acid etching (20 seconds). Rinse (15 seconds) and air-dry. Apply two coats of adhesive. Air-dry (15 seconds). Light-cure (20 seconds).</td>
</tr>
<tr>
<td>Ecusphere-Flow composite Resin; DMG, Germany Batch#: 698833</td>
<td>Dental glass in an optimized matrix of Bis-GMA; 63% by weight - 41% by volume inorganic filler (0.02-3 μm).</td>
<td>Straw filled with resin composite. Light cured for 40 seconds.</td>
</tr>
</tbody>
</table>

Bis-GMA- Bisphenol A-Glycidylmethacrylate
Results
The results of SBS means and standard deviations in non-lased and lased groups are shown in (Table 2) and (figure 1). Student’s t-test revealed that there was no significant difference between the non-lased and lased enamel groups (p=

<table>
<thead>
<tr>
<th>Groups</th>
<th>Type</th>
<th>Mean</th>
<th>N</th>
<th>Std. Deviation</th>
<th>Std Mean</th>
<th>T</th>
<th>df</th>
<th>Sig. (2-tailed)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group A</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-Lased</td>
<td>Contax</td>
<td>7.64</td>
<td>10</td>
<td>3.94</td>
<td>1.25</td>
<td>-</td>
<td>1.665</td>
<td>(0.132) N.S.</td>
</tr>
<tr>
<td></td>
<td>Teco</td>
<td>10.83</td>
<td>10</td>
<td>3.87</td>
<td>1.22</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Group B</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-Lased</td>
<td>Contax</td>
<td>6.45</td>
<td>10</td>
<td>2.61</td>
<td>0.83</td>
<td>-</td>
<td>1.794</td>
<td>(0.106) N.S.</td>
</tr>
<tr>
<td></td>
<td>Teco</td>
<td>9.48</td>
<td>10</td>
<td>4.12</td>
<td>1.30</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Contax= Self-etch; Teco=Total etch; N.S.= Not significant.

Table 2. The results of Paired Samples t-test for SBS.

Figure 1. Results of SBS (MPa) of test groups
There was no significant difference between self-etch (Contax) and total-etch (Teco) adhesives in non-lased enamel group (p= 0.132). There was no significant difference between self-etch and total-etch adhesives in lased enamel group (p= 0.106). Student’s t-test showed no significant difference in self-etch adhesive between non-lased group and lased enamel group (p= 0.435) and no significant difference in total-etch adhesive between non-lased and lased enamel group (p= 0.459).

One-way ANOVA and Duncan's Multiple Range test did not indicate any significant differences among the non-lased and lased enamel groups (F= 2.769, p= 0.056). Total etch (Teco) showed the highest bond strength in both non-lased and lased groups.

Fracture mode: The analysis of bonding sites after shear strength tests revealed that, for non- lased fractured specimens, cohesive failure mode was predominantly observed followed by adhesive failure mode. The total-etch systems showed a limited number of in enamel (8%), which were not observed for the self-etch system. However, for lased fractured specimens, mostly mixed failures occurred. For the self- etching system, a significant number of cohesive failures in enamel was observed (28%).

Discussion

According to ISO 11405/TS bovine incisors can be used as a substitute for human enamel. Unlike bovine dentine, which is not recommended as a substitute for human dentine, (Retief et al. 1990) bovine enamel exhibits very similar bonding characteristics to human teeth (Oesterle 1998) and has often been used as a replacement (Chung et al. 2000, Czochrowska 1999).

Laser etching of enamel surfaces is popular because of the potential disadvantages of acid etching. Acid etching results in chemical changes that can modify calcium-toposphorus ratio, reduces the carbonate-to-phosphate ratio, and leads to the formation of more stable and less acid-soluble compounds. As a result of this demineralization, enamel becomes more susceptible to caries attack (Sungurtekin, Oztas 2010, Martinez-Insua et al. 2000) Recently, Er:YAG and Er:Cr:YSGG lasers were introduced in dentistry. For physical and medical reasons, they are used for the treatment of hard tissue. The advantage of an Erbium wave is that it is well-absorbed by water and dental hard tissue. These lasers can also be used for etching of enamel surfaces for the purpose of bonding the composite resin to enamel surface (Yazici AR et al. 2013).

The structural changes of the enamel tissue as a result of laser treatment using Er:YAG and Er:Cr:YSGG is ablation process without formation of the smear layer. This results in surface irregularities and enamel prisms exposure. These changes are thought to increase the bond strength of resin composites (Keller et al. 1993) but in fact, these morphological changes created by laser ablation do not have the same ideal pattern obtained by acid phosphoric application. Therefore, this heterogeneous structure in lased enamel affects the bond strength of resin composite (Christensen et al. 1996). Parhami et al. (2014) reported that Er:YAG laser treatment reduced shear bond strength of flowable resin composite to enamel compared to the ones treated by conventional technique using a bur previous to acid etching.

The parameter used in this study was 2.5W output power with water according to the manufacturer of Er:Cr:YSGG lasers that recommended 2.25 to 2.5 W for laser etching but other studies used 2 to 3 W referring from their results of a pilot study to obtain the proper effects (Galişkanyd'in et al. 2010).

A plethora of flowable composite products are commercially available as bonding agents because of these favorable properties: non-stickiness and injectability (Elaut et al. 2002). There are some studies evaluating the shear bond strength of resin composites to lased dentin, (Visuri et al. 1996, Armengol et al. 1999) but there are not enough studies about the shear bond strength of flowable composite to lased enamel. Therefore this study was aimed to evaluate and compare the shear bond strength of flowable composite to enamel etched with Er:Cr:YSGG laser.

Pretreatment with 37% phosphoric acid increases bond strength because thick outer enamel layer may prevent the permeation of self-etch primers and bonding agents, thus leaving some areas partially unetched. This then results in formation of shorter and poorly defined resin tags. Removing the outer enamel with phosphoric acid etching, longer resin tags are formed and thus bond strength is increased (Kanemura et al.2009, Scougall et al. 2009). On the other hand, self-etch primers and bonding agents have some advantages over the phosphoric acid etchant. Self-etch primers simplify the clinical handling of adhesive systems by combining the conditioning and priming agents into a single product (Bishara et al. 2004). Self-etch bonding agents prevent aggressive decalcification and bulk enamel loss which are characteristics of phosphoric acid etching (Arhun et al. 2006). This means that they reduce the risk of enamel damage due to their reduced ability to sufficiently etch and penetrate the enamel surface (Eminkahyagil et al. 2005). Most self-etch adhesives did not etch enamel as deeply as the phosphoric acid etchants did, but the shallow etching pattern compromised bonding to enamel (Kim et al. 2005).
It has been reported that the morphological changes of the intact and ground enamel surfaces after interaction of the self-etching adhesive depend on the pH classification of the self-etching adhesives (Chakmakchi. 2005). Demineralization effects of self-etch primers and bonding agents are proportional to their acidity. The lower the pH, the higher the acidity, the deeper the etch; and the pH values of self-etch adhesive systems are higher than that of phosphoric acid etchant (Di Frances et al. 2007).

In this study, although there was no significant differences between Er,Cr:YSGG lased and non-lased enamel groups, the lower bond strength was achieved when enamel surface etched by Er,Cr:YSGG laser, and this finding presented in both types of adhesive systems that used ( Teco, two step total etch and contax, two step self etch adhesives). Therefore the null hypothesis is that there is no significant difference between lased and non-lased etched enamel was accepted.

Several of the findings concerning the use of lasers for enamel etching are contradictory. Von Fraunhofer et al. (1993) and Usumez et al. (2003) have stated that laser irradiation is not capable of etching enamel. In contrast, Visuri et al. (1996) and Hossain et al., (2003) who reported that laser irradiation may be used to etch enamel. These contradictory findings are due to the different outputs and experimental designs of the studies.

Our results disclosed that the irradiation of enamel surface with an Er,Cr;YSGG 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 enamel and yielded a decrease in bond strength. Similar results were reported in other studies. Martines-Insua et al. (2000) found weaker adhesion forces in an Er: YAG laser-etched enamel surface than an acid etched enamel surface. This was related to sub-surface cracks observed in SEM images. Dunn et al. (2005) reported a decrease in bond strength to Er: YAG laser irradiated dental hard tissue. Sungurtekin and Oztas (2010) noted that Er,Cr:YSGG laser etching did not eliminate the need for acid etching. Moreover, they found that when a Er,Cr:YSGG laser and acid etching were combined, Er,Cr:YSGG was as effective as the conventional acid etching technique. However, the laser irradiation of enamel surfaces produced surface fissures and a union or blending of a distinctive etch pattern normally seen in acid-etched enamel. This blending effect likely prevented the penetration of resin into enamel, resulting in lower enamel bond strength values. Delme et al. (2006) who postulated that the acid-etching procedure is essential after laser ablation. Some researchers stated that laser applications give similar results to acid-etching techniques. Ozer et al. (2008) investigated the SBS of brackets that they applied on enamel prepared with 0.75 W Er, Cr: YSGG, 1.5 W Er, Cr: YSGG, 37% Orthophosphoric acid or self-etching primer. They found that the 0.75 W laser-applied group was significantly less in regard to SBS than all other groups, although there was no statistically significant difference among the other groups. Lee et al. (2007) etched enamel surfaces with acid, a laser, and acid and a laser together and investigated the SBS of the orthodontic brackets. They did not find statistically significant differences in the laser- and acid-etched surfaces. Further, they reported that the application of both could enhance the bond strength. However, acid etching after laser irritation is not able to eliminate the laser-modified layer completely.

In the present study, Total-etch adhesive produced bond strength higher than self-etch adhesive in non-lased enamel and lased enamel groups. The results of present study showed that acid etching pretreatment alone is more effective than Er,Cr;YSGG laser etching followed by acid etching. This is in agreement with Ceballos et al (2001) that proposed acid-etching alone yields shear bond strength values that are significantly higher than those achieved with laser ablation alone, or in combination with acid-etching. The self-etching primer adhesive system yielded the lowest bond strength in the Er,Cr:YSGG-lased group and confirmed the outcomes of previously reported studies (Eguro et al. 2002, De Munck et al. 2002). Contreas-Bulnes et al. (2013) found that Er, YAG laser irradiation could not de an option for enamel conditioning.

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 enamel surface. The total-etch systems showed a limited number of cohesive failures in enamel (8%), which were not observed for the self-etch system. 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 enamel. For the self-etching system, a significant number of cohesive failures in enamel was observed (28%).
Conclusion
Within the limitations of this laboratory study, the following conclusions were drawn:
1. No significant differences were noted between non-lased and Er,Cr:YSGG lased enamel for each adhesive tested.
2. Er,Cr:YSGG laser pretreatment followed by acid etching or self-etch adhesives did not enhance the adhesion of composite resin to enamel surface compared with acid etching.

References
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