Microshear Bond Strength of a Self-adhesive Composite to Erbium Laser-Treated Primary Enamel

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Abstract
Introduction: Advances have been made in the composition of flowable composites in recent years and self-adhering composites, which do not require a bonding agent, have recently been introduced to the market. This study aimed to assess the microshear bond strength (µSBS) of a self-adhering flowable composite (Vertise) to primary enamel treated with a graphite disc with silicon carbide particles (SIC) and laser irradiation, the result of which was compared to that of a conventional flowable composite (Premise).

Methods: In this in vitro, experimental study, 72 samples of sound primary enamel were evaluated. A smooth enamel surface was obtained using a graphite disc. Next, the erbium chromium yttrium scandium gallium garnet (Er,Cr:YSGG) laser was used for enamel surface treatment in half of the samples (n = 36). All the samples were then randomly divided into 4 groups of (i) Premise flowable composite (PF) without laser (n = 18), (ii) Vertise flowable composite (VF) without laser (n = 18), (iii) PF with laser (n = 18), and (iv) VF with laser (n = 18). The teeth were then incubated at 37°C for 24 hours and were then subjected to thermocycling. The µSBS of samples was measured using a universal testing machine and reported in megapascal (MPa). Data were analyzed using SPSS via the two-way ANOVA and independent-samples t test at P<0.05.

Results: The mean µSBS of VF was significantly higher to the laser-treated samples (13.60 ± 5.47) compared with the non-treated samples (5.89 ± 2.42) (P<0.001). However, no significant difference was noted in the µSBS of PF to the laser-treated (13.18 ± 3.45) and non-treated samples (16.06 ± 3.52) (P=0.058).

Conclusion: The µSBS of the conventional flowable composite is higher than that of the self-adhering flowable composite to the enamel of primary teeth. Enamel surface treatment with laser irradiation increases the µSBS of self-adhering flowable composites.

Keywords: Microshear bond strength; Self-adhering flowable composite; Laser; Primary enamel.

Introduction
The preservation of primary teeth in the oral cavity until the time of their exfoliation is important in the growth and development of dentition as well as children's nutrition. Primary tooth restorations should have adequate durability in order to have favorable clinical service. Moreover, they should have a favorably esthetic appearance to preserve children's psychological health.

The use of composite resins, glass ionomers and a combination of both is gradually increasing in pediatric dentistry, while the application of amalgam is decreasing due to its disadvantages such as the absence of chemical adhesion to tooth structure, requiring cavity preparation with a particular design and the removal of a large portion of tooth structure and not being well accepted by the parents due to its unesthetic appearance.¹

An efficient bonding between the restorative material and tooth structure is an important factor determining the success of composite restorations.² Adhesive systems were introduced, aiming to enhance the quality of bonding of composite resins to the tooth structure. The adhesive systems can be classified into 2 groups of etching and rinse and self-etch adhesives. In the self-etch bonding systems, the demineralization of the dental substrate and resin infiltration occur at the same time; thus, these systems often cause less postoperative tooth hypersensitivity. Moreover, they have easier application and are more suitable for use in pediatric dentistry since they do not require a separate etching step.³

Flowable composite resins were first introduced in 1990 with favorable advantages such as easy clinical application and optimal adaptation to the cavity walls.⁴


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Flowable composites have several applications in pediatric dentistry and are used as fissure sealants and also for preventive resin restorations. Recently, a new generation of flowable composites, namely self-adhering flowable composites, which do not require separate etching, rinsing and bonding steps, was introduced to the market. These adhesives have been introduced as the eighth generation bonding agents. Vertise Flow (Kerr, Orange, CA, USA) is an eighth generation self-adhering composite currently available in the market. The use of VF shortens the chair time and decreases the technical sensitivity and procedural errors, which are all highly important in pediatric dentistry, especially when working on young, uncooperative patients.

Aside from the new restorative materials, some novel techniques were also recently introduced for cavity preparation. Erbium lasers are used for cavity preparation in tooth and have several advantages such as the absence of vibration, pressure and noise (which are among the patient complaints in cavity preparation with moderate- and low-speed hand-pieces) and less need for local anesthesia administration; all these factors are important to encourage the pediatric patients to better cooperate during dental procedures.

Considering the more conservative nature of restorations nowadays and the availability of mi-co-tests for the assessment of bond strength, the tendency to measure the bond strength in different parts of the teeth is increasing. Very small samples of tooth structure (0.5 to 1 mm²) are used for the measurement of microshear and microtensile bond strengths, and it is believed that fewer defects occur at the resin-dentin interface under such circumstances. According to Armstrong et al compared with macro-tests, the micro-tests (microshear and microtensile) measure the bond strength of restorative materials to dentin more accurately.

Many studies have measured the bond strength of composite resins and bonding systems to permanent teeth. However, some differences exist in the structure and composition of primary and permanent teeth. Thus, the results of permanent teeth cannot be generalized to primary teeth.

This study aimed to assess the microshear bond strength (µSBS) of a self-adhering flowable composite (Vertise) to primary enamel treated with the erbium laser in comparison with a conventional flowable composite (Premise).

**Materials and Methods**

**Sample Selection and Sample Size Calculation**

This in vitro experimental study evaluated 72 primary anterior teeth. The teeth were used in the study within 3 months of extraction or exfoliation. The minimum sample size was calculated to be 16 in each of the 4 groups according to a study by Duddu et al., assuming alpha = 0.05, beta = 0.2, the standard deviation of 0.5 and the effect size of 0.45 using SPSS. We included 18 teeth in each group. The enamel surface of the teeth had to be free from caries, cracks, enamel hypoplasia or restorations. The teeth were cleaned with gauze, pumice paste, and a rubber cup and inspected under a stereomicroscope at ×40 magnification (Blue Light, USA) to ensure the absence of cracks. The teeth were then stored in a 0.5% chloramine T solution for one week; thereafter, the teeth were stored in distilled water. They were then mounted in auto-polymerizing acrylic resin (Acropars) in such a way that their buccal surface was exposed for the interventions.

**Materials**

Two flowable composites and 1 adhesive were used in this study (Table 1).

**Treatment**

A smooth enamel surface was created using a 600-grit graphite disc with silicon carbide particles (SIC) (Phoenix Beta, Buehler, Germany). Next, half of the teeth (n = 36) were subjected to enamel surface treatment by erbium chromium yttrium scandium gallium garnet (Er,Cr:YSGG) laser irradiation with a wavelength of 2.78 μm (iPlus Waterlase; Biolase, USA). The Gold MZ6 hand-piece tip was used for this purpose and the laser was irradiated with 20 Hz frequency and 1.5 W output power under water (60%) and air (60%) coolant. The laser was irradiated from 1 mm distance to the enamel surface with a sweeping motion and 60 μs pulse duration (10 seconds for each tooth surface).

Next, laser-irradiated and non-irradiated teeth were randomly divided into 2 subgroups of 18 samples each for the application of the Vertise self-adhering flowable (VF) composite (Kerr, Orange, CA, USA) and the Premise conventional flowable (PF) composite (Kerr, Italia S. r. l.).

The following groups were evaluated: (1) SIC graphite disc and PF composite, (2) SIC graphite disc and VF composite, (3) Er,Cr:YSGG laser and PF composite, and (4) Er,Cr:YSGG laser and VF composite.

**Table 1. Materials Used in the Study**

<table>
<thead>
<tr>
<th>Material</th>
<th>Composition</th>
<th>Manufacture</th>
<th>Lot no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vertise Flow</td>
<td>GPDM, HEMA, prepolymerized filler, nano-sized ytterbium fluoride, barium glass filler, nano-sized colloidal silica (65 wt%)</td>
<td>Kerr, Orange, CA, USA</td>
<td>5285208</td>
</tr>
<tr>
<td>Premise Flow</td>
<td>Uncured methacrylate ester monomers, titanium dioxide, benzoyl peroxide, propylene carbonate, antimonite salts, dibutyl phthalate</td>
<td>Kerr Italia S.r.l.</td>
<td>5867585</td>
</tr>
<tr>
<td>Adper Single Bond 2</td>
<td>Bis-GMA, HEMA, UDMA, dimethacrylates, ethanol, camphorquinon, photoinitiators, the copolymer of polialcenoic acid, silica (5 nm)</td>
<td>3M ESPE St Paul, USA</td>
<td>N662648</td>
</tr>
</tbody>
</table>

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Tygon tube cylinders with an internal diameter of 0.7 mm and 3 mm height were used for the application of composites. In groups 1 and 3, the PF composite was applied after the application of acid etchant and Single Bond 2 (3M ESPE Adper st Paul, USA) according to the manufacturer’s instructions. Etching was performed using 37% phosphoric acid for 20 seconds followed by rinsing for 15 seconds. The surface was then dried to obtain a chalky white appearance. The single Bond 2 fifth generation bonding agent was applied to the surface in 2 coats and light-cured for 20 seconds using a light-curing unit (Bluephase C8; Ivoclar Vivadent, Lichtenstein) according to the manufacturer’s instructions. Next, the PF composite was injected into the cylinders and cured for 40 seconds. In groups 2 and 4, the VF composite was used according to the manufacturer’s instructions. It did not require separate etching or adhesive application. The treated tooth surface was then rinsed for 5 seconds and dried with air spray. VF was then injected into the cylinders, and after a few seconds, it was cured for 40 seconds.

Thermocycling
Next, the Tygon tubes were cut with a scalpel and the samples were immersed in distilled water and incubated at 37°C for 24 hours. The samples then underwent thermocycling (Nemo, Iran) for 500 thermal cycles between 5-55°C with a dwell time of 30 seconds and transfer time of 15 seconds. After thermocycling, the samples were immersed in distilled water and incubated at 37°C.

Shear Bond Strength
Each sample was then transferred to a universal testing machine (STM20, Santam, Bongshin, Korea) with a crosshead speed of 0.5 mm/min. The load cell of UTM was: capacity- 50 kg, R.O-3Mv/v, model-DBBP-50, and S/N-M16972. The load was applied by a wedge-shaped blade with a 0.2 mm² surface area. The amount of load required for debonding was determined and the µSBS was calculated in megapascal (MPa).

Statistical Analysis
The collected data were analysed using SPSS version 16.0. The mean µSBS of the groups was calculated, and the groups were compared using two-way ANOVA and independent-samples t test.

Results
The µSBS of primary teeth with and without laser treatment to the Premise conventional flowable composite and the Vertise self-adhering flowable composite was measured (n = 18 in each group).

The maximum µSBS was noted in the PF group without laser irradiation (16.06 ± 3.52 MPa), while the minimum µSBS was noted in the VF group without laser irradiation (5.89 ± 2.42 MPa). Two-way ANOVA revealed that the interaction effect of material and group on µSBS was significant (P<0.001). An independent-samples t-test was used to assess the independent effect of each parameter on the µSBS. The results are presented in Tables 2 and 3. The mean µSBS of VF in laser-irradiated samples was significantly higher than that of non-laser-irradiated samples (P<0.001). There was no significant difference in scores for the µSBS strength of the PF composite to both laser-irradiated and non-laser irradiated samples (P = 0.058). Figure 1 shows the mean µSBS of the 4 groups.

Discussion
This study assessed the µSBS of 2 flowable composites to primary enamel. One limitation of this study was that we only had 2 groups with 2 different surface treatments and 2 types of composites because the collection of primary teeth with sound enamel surface was difficult. If we had a higher number of groups, for example, a self-

Table 2. Comparison of the Mean Microshear Bond Strength (MPa) of Laser-Irradiated and Non-laser Irradiated Samples to the 2 Types of Composites

<table>
<thead>
<tr>
<th>Material</th>
<th>Group</th>
<th>Mean</th>
<th>SD</th>
<th>P Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vertise</td>
<td>No laser</td>
<td>5.89</td>
<td>2.42</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td></td>
<td>Laser</td>
<td>13.60</td>
<td>5.47</td>
<td></td>
</tr>
<tr>
<td>Premise</td>
<td>No laser</td>
<td>16.06</td>
<td>3.52</td>
<td>0.058</td>
</tr>
<tr>
<td></td>
<td>Laser</td>
<td>13.18</td>
<td>3.45</td>
<td></td>
</tr>
</tbody>
</table>

Table 3. Comparison of the Mean Microshear Bond Strength (MPa) of Vertise Flow and Premise to Laser-Irradiated and Non-laser Irradiated Samples

<table>
<thead>
<tr>
<th>Material</th>
<th>Group</th>
<th>Mean</th>
<th>SD</th>
<th>P Value</th>
</tr>
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<tr>
<td></td>
<td>No laser</td>
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<td></td>
<td></td>
<td>13.18</td>
<td>3.45</td>
</tr>
</tbody>
</table>

Figure 1. Mean Microshear Bond Strength of the 4 Groups. 0: Without laser; 1: With laser.
adhering flowable composite group with acid etching, the generalization of the results to the clinical setting would be more accurate.

The results showed that the mean μSBS of the Vertis Flow self-adhering composite to laser-treated samples was significantly higher than that to non-laser treated samples. On the other hand, there was no significant difference in the mean scores for the μSBS of Premise, which is another product from the same manufacturer (Kerr), to both laser-irradiated and non-laser irradiated samples. Also, the μSBS of Premise in both groups was higher than that of Vertise. The bonding mechanism of self-adhering composites is based on the presence of glycerol phosphate dimethacrylate monomer in their composition, which is responsible for etching of the tooth surface and the chemical bond to calcium ions in tooth structure.\(^{15-17}\) Similar to our study, Moslemi et al\(^{11}\) used the Er,Cr:YSGG laser for tooth surface treatment. Minimum μSBS of the self-adhesive flowable composite to the non-laser irradiated samples was observed. Maximum bond strength was noted between the conventional flowable composite and the laser-treated samples. However, it should be noted that they used primary dentin. In our study, minimum bond strength was noted between the self-adhesive flowable composite and the non-laser irradiated samples. The bond strength of this composite was higher to the laser-irradiated samples. However, the conventional flowable composite used in our study showed no significant difference in bond strength to laser-treated and non-laser treated samples. This difference can be due to the type of tooth used. The self-adhesive flowable composite contains hydroxyethyl methacrylate, which is responsible for increased wetting and the penetration of resin into dentin. This may explain the enhanced bond strength of the composite to dentin in their study. However, it had no effect on bond strength to primary enamel, which was used in our study. In this manner, Mine et al\(^{11}\) showed that the self-adhesive composite had limited interaction with smear-covered substrates and prismatic enamel, which explains its inferior diminished bonding capacity to enamel in comparison with current adhesive.

On the other hand, Alamaz et al\(^{16}\) measured the μSBS of self-adhering flowable composites to dentin samples, which was similar to the methodology adopted by Moslemi et al.\(^{11}\) The results showed that self-adhering flowable composites yielded the lowest μSBS, which was in agreement with our findings. Sachdeva et al,\(^{19}\) Pacific et al,\(^{20}\) and Poorzandpoush et al\(^{11}\) used primary dentin samples for the assessment of shear bond strength. Their results were in line with ours, although we evaluated primary enamel. They showed that the conventional flowable composite yielded the highest and the self-adhering composite yielded lower bond strength. Considering the results of previous studies on this topic as well as the findings of the current study, it may be concluded that in contrast to the claims of the manufacturers of self-adhering composites, introduced as the eighth generation adhesives\(^{21-27}\) that do not require separate etching, rinsing and bonding agent application, these composites do not provide an optimal bond to the enamel and dentin of primary or permanent teeth. It seems that the used monomers are not strong enough to etch the tooth surface and enable the bonding of composite resin to tooth structure at the same time. Thus, it seems that the tooth surface should be prepared with other methods. Similarly, Memarpour et al\(^{22}\) showed that the use of all-in-one OptiBond enhanced the shear bond strength of the self-adhering composite to primary enamel and dentin. Shafiei and Saadat\(^{23}\) showed that phosphoric acid etching for 15 seconds significantly increased the bond strength to the self-adhering composite.

Laser irradiation is another method currently used for tooth surface treatment. Erbium laser irradiation creates an irregular rough surface, which is ideal for bonding to composites and is comparable to etched surfaces.\(^{22}\) However, Jaberi Ansari et al\(^{24}\) suggested further etching of surfaces treated with the Er,Cr:YSGG laser with phosphoric acid to increase the μSBS to composites. Similar to our study, Memarpour et al\(^{22}\) used primary enamel samples and applied lasers for their surface treatment. They showed that the shear bond strength of the self-adhering composite to non-laser irradiated samples was lower than the bond strength of other groups.

Davari et al\(^{25}\) showed that the shear bond strength of composites to acid-etched permanent dentin and acid-etched plus laser-irradiated dentin was significantly higher than that in other groups (laser, laser + acid etching, and no treatment control group). However, no difference existed between these 2 groups. Thus, the authors stated that laser irradiation after acid etching of the surface is not necessary. In our study, the samples in the conventional flowable composite group were etched following the use of the laser, and the obtained results were in agreement with those of Davari et al.\(^{25}\) They observed no significant difference in the shear bond strength of the composite between laser-treated samples and those that were laser-treated first and then etched with acid.

Erbium lasers cause thermomechanical wear of the tooth structure and by the evaporation of water present in the hydroxyapatite matrix, they cause physical and macroscopic structural changes and create a rough surface for bonding of composite resins.\(^{24,25}\) Compared with enamel etching with acid (which creates micromechanical bonding of resin tags created in the microscopic porosities),\(^{26}\) the application of lasers alone has an insignificant effect on the bond strength of the conventional composite to the tooth structure, similar to our study. Thus, it seems that laser irradiation is more effective in increasing the bond strength of self-adhering composite resins and self-etch bonding systems.\(^{27}\)

One advantage of this study was the use of the
Waterlase Er,Cr:YSGG laser, which has superiorities to the erbium yttrium aluminum garnet (Er,YAG) laser. In the application of the Er:YAG laser, water in dentinal tubules and enamel prisms evaporates fast; thus, enamel hydroxyapatites absorb less energy and therefore, the efficacy of fast tissue ablation by the laser decreases, resulting in higher tissue damage. However, the Waterlase laser hand-piece never touches the tooth structure. Thus, vibrations decrease and microscopic cracks and tissue damage are prevented.\textsuperscript{11}

In this study, the microshear test was used to assess the bond strength of the composite to the enamel. Different methods are available for the mechanical testing of resin materials, including shear, microshear, tensile, microtensile and flexural tests. There is no industrial standard for bond strength tests. However, some of these tests have tried to earn it. The µSBS test is simple. The preparation of samples for this test is also relatively easy. It has simple instructions and optimal reliability, resulting in its common use.\textsuperscript{28}

McDonough et al\textsuperscript{28} in 2004, found that the µSBS is an efficient tool to understand the complex interactions between dental composites and enamel and dentin, especially at the interface. The macroshear test is not normally sufficient for this purpose.

**Conclusion**

The current results revealed that the bond strength of the conventional flowable composite to both laser-treated and non-laser treated samples was higher than that of the self-adhering flowable composite. The application of the laser for primary enamel surface treatment did not enhance the bond strength of the conventional flowable composite, but it increased the bond strength of the self-adhering flowable composite to the primary enamel. The self-adhering flowable composite could not provide an optimal bond to the non-laser-treated primary enamel.

**Conflict of Interest**

The authors declare no conflict of interest.

**Ethical Considerations**

The protocol was reviewed in the research committee of Tehran University of Medical Sciences.

**Acknowledgment**

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