## RESEARCH ARTICLE

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# Bonding performance of universal adhesives to er,cr:YSGG laser-irradiated enamel

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#### **Abstract**

Universal adhesives have been recently introduced for use as self-etch or etch-and-rinse adhesives depending on the dental substrate and clinical condition. However, their bonding effectiveness to laser-irradiated enamel is still not well-known. Thus, the aim of this study was to compare the shear bond strength (SBS) of universal adhesives (Single Bond Universal; Nova Compo-B Plus) applied to Er,Cr:YSGG laser-irradiated enamel with SBS of the same adhesives applied in self-etch and acid-etching modes, respectively. Crown segments of sixty bovine incisors were embedded into standardized acrylic blocks. Flattened enamel surfaces were prepared. Specimens were divided into six groups according to universal adhesives and application modes randomly (n = 10), as follows: Single Bond Universal/acid-etching mode; Nova Compo-B Plus/acid-etching mode; Single Bond Universal/self-etching mode; Nova Compo-B Plus/self-etching mode; and Single Bond Universal/Er,Cr:YSGG Laser-etching mode; Nova Compo-B Plus/Er,Cr:YSGG Laser-etching mode. After surface treatments, universal adhesives were applied onto surfaces. SBS was determined after storage in water for 24 h using a universal testing machine with a crosshead speed of 0.5 mm min<sup>-1</sup>. Failure modes were evaluated using a stereomicroscope. Data was analyzed using two-way of analyses of variances (ANOVA) (p = 0.05). Two-way ANOVA revealed that adhesive had no effect on SBS (p = 0.88), but application mode significantly influenced SBS (p = 0.00). Acidetching significantly increased SBS, whereas there are no significant differences between self-etch mode and laser-etching for both adhesives. The bond strength of universal adhesives may depend on application mode. Acid etching may significantly increase bond strength, while laser etching may provide similar bond strength when compared to self-etch mode.

#### KEYWORDS

adhesion, acid-etch, bonding, enamel, laser irradiation, self-etch, universal adhesives

### **RESEARCH HIGHLIGHTS**

This study investigated the shear bond strengths (SBS) of two universal adhesives applied to enamel with different application modes. Er,Cr: YSGG laser-etching of enamel provided similar SBS when compared to self-etching mode for both adhesives.

#### 1 | INTRODUCTION

Resin adhesive systems change commercial names frequently. This makes it really challenging for dentists to catch current innovations in

Review Editor: Dr. Mingying Yang

material science and to choose which resin adhesive system to use in their clinics. Traditional resin adhesive systems have followed self-etching or acid-etching strategies. More recently, a new resin adhesive system class, which is called universal adhesives, has been marketed (Chen et al., 2015; Perdigão and Swift, 2015). Unlike to preceding adhesives, these adhesives let dentists put to use an adhesive strategy personalized to an exact clinical condition as universal adhesives may be used in self-etching mode, acid-etching mode, or in self-etching mode on dentin and in acid-etching mode on enamel (Chen et al., 2015; Perdigão and Swift, 2015). The last technique is commonly referred to as selective enamel etching (Frankenberger et al., 2008).

Applications of infrared (IR) lasers in dentistry using CO<sub>2</sub>, Nd:YAG, Ho:YAG, Er:YAG, and Er,Cr:YSGG lasers have been investigated since

Microsc Res Tech 2016; 1–7 wileyonlinelibrary.com/journal/jemt © 2016 Wiley Periodicals, Inc. 1

the 1960s. Compared to conventional mechanical drills, non-contact laser surgery eliminates vibrations and permits more precise and comfortable removal of tissue during cavity preparation procedures (Kang et al., 2007). Erbium lasers also reduce the need for local anesthesia (Keller et al., 1998; Poli and Parker, 2015). Dental hard tissues can be effectively removed with the ablation process that involves microexplosions. Lasers allow for minimally invasive caries removal and tooth preparation (de Almeida Neves et al., 2011; Matsumoto et al., 2002; Yazici et al., 2010). With the wide range of bonded materials, these smaller preparations can be restored effectively. However, bond strengths to laser-irradiated dental hard tissues in the literature are often contradictory (Lopes et al., 2015). For the success of a restoration, durable bonding should be achieved. The types of adhesive systems, restorative materials, and the method of cavity preparation affect the bond strength of resins to tooth structure (Cardoso et al., 2008; Esteves-Oliveira et al., 2007; Pashley et al., 1995).

Many laboratory studies have examined surface characteristics and bond strength to enamel following laser irradiation (Lopes et al., 2015). However, the bond strength to laser-cut enamel cited in the literature is often contradictory. While some researchers have reported that an Er,Cr:YSGG laser provided favorable enamel surfaces without smear layers and thermal damage (Hossain et al., 1999; Lin et al., 1999), and they showed similar and even better enamel bond strength when compared to high-speed bur (Lin et al., 1999), others have shown that an Er,Cr:YSGG laser could present a substrate less receptive to adhesion of the current resin adhesives than bur-cut enamel (Cardoso et al., 2008; Esteves-Oliveira et al., 2007; Usumez et al., 2002). Cardoso et al. suggested that bond strength to laser-irradiated enamel might depend on class and composition of the resin adhesive system (Cardoso et al., 2008). Several studies reported that acid-etching mode for enamel provides a higher bond strength of universal adhesives than self-etching mode. These results have been obtained by applying universal adhesives on bur-cut enamel; however, comparison of acidetching and self-etching modes on laser-cut enamel has not been reported. Therefore, the aim of the present study was to test the null hypothesis that different application modes, including self-etching, acid-etching, and laser-etching modes, do not affect the shear bond strength (SBS) of universal adhesive systems to enamel surfaces.

## 2 | MATERIALS AND METHODS

#### 2.1 | Experimental design

A factorial  $2\times 3$  design was used to evaluate the variables "adhesives" in two levels and "application modes" in three levels. The dependent variable evaluated was SBS value. The compositions, manufacturer instructions for use the universal adhesives tested in the present study were shown in Table 1.

#### 2.2 | Er,cr:YSGG laser device

Laser irradiations were performed using an Er,Cr:YSGG laser (Waterlase MD, Biolase Technology; San Clemente, CA) with following param-

eters: wavelength 2.780 nm; power of 1.5 W; frequency of 20 Hz; pulse duration of 140  $\mu$ s; spot size of 600  $\mu$ m; tip MGG6; air pressure setting of 90%; and water pressure setting of 75%. The irradiation was performed in the noncontact and focused mode, with a cylinder fiber tip positioned perpendicular to the enamel surface at a distance of 1–1.5 mm from the target tissue. Laser irradiation of enamel surfaces was accomplished by hand, using a sweeping motion. Consequently, irradiation distance ranges from 1 to 1.5 mm.

### 2.3 | Specimen preparation

Sixty bovine incisors with no visible defects in enamel were used in the present study. Teeth were stored in dry condition until needed and immersed into distilled water for 2 weeks before being used (Mobarak et al., 2010). Roots were severed by low speed diamond saw under water-cooling. Enamel surfaces were primarily flattened by using 320-grit silicon carbide (SiC) abrasive papers by hand under water-cooling. Then, all crowns were embedded into self-cure acrylic resin in plexiglass cylinders individually in order to allow for standardized and secure placement during the SBS test. Enamel surfaces were finished using 600-grit SiC abrasive papers under water-cooling for 1 min. Enamel surfaces were randomly divided into six groups according to the universal adhesives and application modes (n=10) as follows:

Group 1. (SBU-SE): Single Bond Universal adhesive was applied on enamel surfaces in self-etching mode. Adhesive was applied to enamel surfaces and agitated for 20 s, then gently air-dried for 5 s, and light cured for 10 s.

Group 2. (SBU-AE): Single Bond Universal adhesive was applied on enamel surfaces in acid-etching mode. Enamel surfaces were acid-etched by using 37% phosphoric acid for 20 s, rinsed for 10 s, and airdried for 2 s, and then adhesive was applied as for the self-etch mode.

*Group 3.* (*SBU-LE*): Single Bond Universal adhesive was applied on enamel surfaces after laser irradiation with setting given below. Then, adhesive was applied as for self-etch mode.

*Group 4.* (*NCP-SE*): Nova Compo-B Plus universal adhesive was applied on enamel surfaces in self-etching mode. Adhesive was applied to enamel surfaces and agitated for 20 s, then gently air-dried for 5 s, and light cured for 10 s.

Group 5. (NCP-AE): Nova Compo-B Plus universal adhesive was applied on enamel surfaces in acid-etching mode. Enamel surfaces were acid-etched by using 37% phosphoric acid for 20 s, rinsed for 10 s, and air-dried for 2 s, and then adhesive was applied as for the self-etch mode.

*Group 6.* (*NCP-LE*): Nova Compo-B Plus universal adhesive was applied on enamel surfaces after laser irradiation with setting given below. Then, adhesive was applied as for self-etch mode.

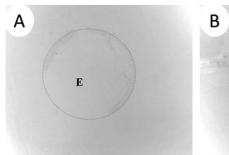
## 2.4 | Shear bond strength test

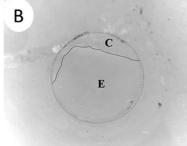
Following adhesive applications, a two-piece removal plexiglass mold was fixed on the surface, giving a cylindrical cavity 4 mm in height and 3 mm in diameter. Valux Plus microhybrid resin composite was placed into the cavity incrementally. Each increment was polymerized for 20 s.

TABLE 1 Compositions, manufacturer instructions for use, and the universal adhesives tested in the present study

Adhesive	Manufacturer	Composition	Manufacturer instructions
Single bond universal Lot # 611430;pH = 2.7	3M ESPE, St. Paul, MN, USA	10-MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane.	Apply with agitation for 20 s. Gently air-dry for 5 s. Light cure for 10 s.
Nova Compo-B Plus; Lot # 16256;pH = 2.5-2.7	Imicryl, Konya, Turkey	Bis-GMA, HEMA, ethanol, 10-MDP, 4-META, silanated nano silica, initiators, water.	Apply with agitation for 20 s. Gently air-dry for 5 s. Light cure for 10 s.

10-MDP = 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA = bisphenol glycidyl methacrylate; HEMA = 2-hydroxyethyl methacrylate; 4-META = 4-methacryloxyethyl trimellitateanhydride.





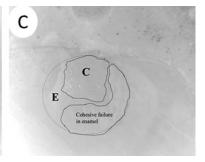


FIGURE 1 Representative images from stereomicroscope of failure modes of shear bond strength test. E: enamel, C: composite. (A) Adhesive failure, (B) Mixed failure, (C) cohesive failure

The bonded teeth were stored in water for 24 h at 37  $^{\circ}$ C before bond strength testing. Specimens were loaded in shear mode until fracture happened with the use of a universal testing machine (Instron 3220, Instron Corporation, Canton, MA) at crosshead speed of 1.0 mm min $^{-1}$  using a knife-edged chisel. The direction of the applied load was from the cervical to the incisal of the tooth. The SBS (in MPa) was calculated by dividing the maximum load by the cross-sectional area of the bonded surface.

## 2.5 | Failure mode analysis

Following the SBS tests, all of the failure specimens were observed with a stereomicroscope at  $10\times$  to determine the failure modes. Failure modes were divided into adhesive, cohesive, and mixed failure (Figure 1). Additionally, representative samples were examined using a scanning electron microscope (SEM, Zeiss EVLO LS10, Bruker, Bremen, Germany).

#### 2.6 | Statistical analysis

Data were statistically analyzed with a two-way analysis of variance (ANOVA) with bond strength data as a dependent variable and adhesives and application modes as factors. One-way ANOVAs with Tukey post hoc tests were then used to determine groups with significant differences. All tests were performed at a significance level of 0.05. The analyses were done by SPSS software (SPSS 13.0 for Windows).

#### 3 | RESULTS

SBS values are presented in Table 2. Two-way ANOVA showed that SBS was significantly influenced by application mode (p = 0.00) but not

significantly influenced by adhesive (p = 0.88). The interaction of these two factors was not significant (p = 0.488), indicating that the differences that existed among application modes were not dependent on the types of adhesive system. One-way ANOVAs deployed to evaluate the application modes on each adhesive revealed that statistically significant differences existed among different application modes of the universal adhesives.

Post hoc tests showed that while no significant differences for either material existed in the groups of self-etch mode and laser-etch mode, mean SBS of total-etch modes were significantly higher than those of other modes for both adhesives.

The distribution of failure modes is presented in Figure 2. Stereomicroscope evaluation determined a higher incidence of mixed and cohesive failures for acid-etched specimens of both adhesives. Selfetch specimens showed higher incidences of adhesive failure for both adhesives

**TABLE 2** Shear bond strength data (Mean ± SD, MPa)

Adhesive	Application mode	Enamel
Single bond universal	Self-etching mode	$14.99 \pm 2.7^{a}$
	Acid-etching mode	$23.53\pm7.0^b$
	Er,Cr:YSGG laser-etch	$17.15 \pm 4.4^{a}$
Nova Compo-B Plus	Self-etching mode	$18.77\pm4.3^{\text{a}}$
	Acid-etching mode	$23.70 \pm 4.9^b$
	Er,Cr:YSGG laser-etch	$19.65 \pm 4.6^{a}$

The same letter indicates no significant differences for each adhesive (p > 0.05), n = 10.

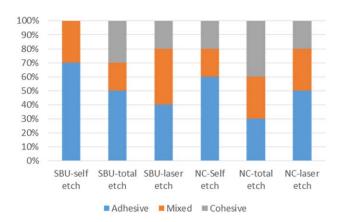


FIGURE 2 The failure mode distribution of the shear bond strength test [Color figure can be viewed at wileyonlinelibrary.com]

Representative SEM images taken from adhesive failure areas of the debonded specimens are shown in Figure 3a-c. Self-etching mode (Figure 3a) and acid-etching mode (Figure 3b) of universal adhesive produced uniform surfaces when compared to laser-etching mode (Figure 3c). Laser irradiation left un-lased, untouched enamel surfaces.

#### 4 | DISCUSSION

This in vitro study demonstrated that the etch-and-rinse or selective-etch technique is an effective approach to achieving more predictable micromechanical bonding of composite to enamel. Similar findings were reported in recent study by McLean et al. (2015). They reported that etching enamel significantly increased the SBS of two universal adhesives to enamel, respectively. As surface treatment significantly affected the SBS of composite to enamel for the universal bonding agents, the null hypothesis that there would be no difference based on surface treatment must be rejected.

From the results of the present study, the SBSs of the universal adhesives to enamel were improved when the bonding agents were applied as two-step, etch-and-rinse adhesives with acid-etching mode rather than one-step, self-etch adhesives with self-etching mode. This was attributed to an improved micromechanical bond being produced with the addition of the etch-and-rinse or selective etch surface treat-

ment. Etch-and-rinse or selective etch adhesive systems are characterized by an initial etching step, typically with 32-37% phosphoric acid, followed by a thorough rinsing procedure that is responsible for the complete removal of the smear layer and selective dissolution of the enamel rods. This creates microporosities in the enamel that are easily infiltrated by resin adhesives via capillary attraction (Gwinnett and Matsui, 1967). Following polymerization, micromechanical interlocking of tiny resin tags within the etched enamel surface provides a strong micromechanical bond to enamel. The alternative self-etch approach only dissolves the smear layer but does not remove it, as there is no rinsing step, leaving the dissolved products to become incorporated within the bonded layer (Van Meerbeek et al., 2011). Furthermore, the degree of demineralization produced by self-etch adhesives depends largely on the acidity or etching aggressiveness of the functional monomer and is material dependent. According to Sunfeld and others, the penetration of the adhesive system may be restricted to the more superficial enamel layers with creation of shorter resin tags when selfetch adhesives are used without a selective-etch step (Sundfeld et al., 2005).

The usage of laser irradiation has been suggested to improve the bonding of resin to dental hard tissues and thus, it might eliminate the acid-etching requirement due to the surface roughness created by laser irradiation, so allowing mechanical interlocking with resin materials (Kameyama et al., 2008). This phenomena is termed as laser etching (Martinez-Insua et al., 2000). Nevertheless the effectiveness of this technique is controversial; while some researchers support the preparation or etching ability of laser to enamel (Basaran et al., 2007; Kameyama et al., 2008; Memarpour et al., 2016; Ozer et al., 2008; Shafiei et al., 2014), others deny its efficacy (Martinez-Insua et al., 2000: Usumez et al., 2002).

To our knowledge, there is limited information in the literature about bond strength of universal adhesives to laser irradiated enamel. Our findings indicate that Er,Cr:YSGG laser irradiation of enamel with 1.5–20 Hz parameters did not impair bonding effectiveness of universal adhesives tested in the present study to enamel. It can be suggested that self-etching and chemical bonding potentials of universal adhesives would be enough for comparable bond strength with single step application to enamel. The different enamel surface characteristics were reported following laser irradiation depending on various

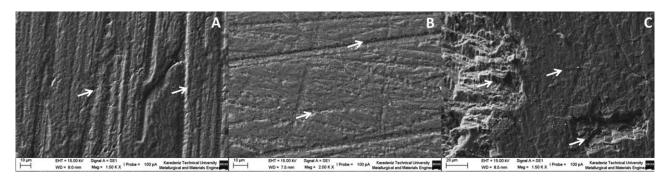


FIGURE 3 Representative SEM images taken from adhesive failure areas of the debonded specimens. Surface was uniform and starches resulted from SiC abrasion were seen (arrows) in acid-etching groups (A) and self-etching (B). However, laser irradiation yielded nonuniform surface with unlased enamel surfaces (arrows) (C)

irradiation parameters used, including extensive fissuring and subsurface cracking, fusing and blocking micropores, and compositional alterations such as an increase in quantities of calcium (Adebayo et al., 2012; Ayar, 2015; Delfino et al., 2007). A previous ultra-morphological study on the interfaces of resin/laser irradiated enamel with different output power parameters suggested that Er,Cr:YSGG laser parameters exceeding 100 mJ output energies resulted in significant micromorphological alterations within resin-enamel interfaces and subsurface enamel (Ayar, 2015). Enamel surfaces were irradiated with 1.5 W to 20 Hz parameters, yielding 75 mJ pulse energy in this study. Therefore, it can be speculated that the power setting deployed in the present study could be safe for resin bonding to enamel with universal adhesives.

Another consideration regarding irradiation of enamel surfaces with pulsed lasers would be that laser irradiation yields heterogeneous surfaces with non-lased enamel areas (Sasaki et al., 2008). It was reported that acid etching these non-lased enamel surfaces after laser etching would improve bond strength when compared to specimens that were treated with an Er:YAG laser alone either with acid etching (Sasaki et al., 2008). Similar SEM findings were observed in this study (Figure 3c). However, the aforementioned benefit of a combination of laser irradiation and acid etching on resin bonding was not observed in this study. This may be due to less aggressiveness of self-etching adhesive when compared to phosphoric acid etching. Universal adhesives tested in this study could be considered as ultra-mild, as their pH values range from 2.5 to 2.7.

One of the important abilities of self-etching adhesives is chemical bonding abilities of their functional monomers to hydroxyapatite. The 10-methacryloyloxydecyl dihydrogenphosphate (MDP) has shown a very effective and reliable for bonding to enamel and dentin among the currently used functional monomers (Van Meerbeek et al., 2011). Yoshida et al. showed that calcium salt of MDP monomer with hydroxyapatite forms nano-layers at the adhesive interfaces (Yoshida et al., 2012). It was claimed that the low solubility and hydrophobic nature of these calcium salt depositions of MDP with hydroxyapatite would account to this success of the MDP monomer (Yoshida et al., 2004, 2012). Both universal adhesives tested in this study have MDP functional monomers in their compositions, so that they bond chemically to enamel surfaces. Previous research reported that incorporation of MDP monomer into universal adhesive would provide higher enamel bond strength when compared to universal adhesives without MDP monomer (Loguercio et al., 2015; Tsuchiya et al., 2016). Additionally, single bond universal also contains a methacrylate-modified polyalkenoic acid copolymer while Nova Compo-B Plus includes 4-META; both with potential for chemical bonding. On the other hand, micromechanical interlocking by means of phosphoric acid etching of enamel has been shown to improve enamel bond strength of universal and/or selfetch adhesives (Loguercio et al., 2015; McLean et al., 2015; Tsuchiya et al., 2016).

In terms of failure mode, it was reported that a relationship between the bond strength and fracture failure mode existed (Al-Salehi and Burke, 1997). From the findings of the present study, the higher bond strengths did correlate with greater mixed fractures or cohesive plus adhesive failure modes. The universal bonding agents produced more mixed and cohesive fractures when used in acid-etching mode and laser-etch mode than self-etch mode, which also correlated with bond strength.

One of the limitations of our study has to do with the lack of thermal cycling or long-term water storage. Thermal cycling may not be relevant because the weakness of adhesives to thermal cycling depends on the chemical configuration of adhesive system (Perdigão et al., 2011). The appropriate sample size has been a matter of dispute and has served as a criterion of the soundness of research, since it is likely that sample sizes of <10 specimens per group may not follow a normal distribution (Eliades and Brantley, 2000). However, the sample size of this study (n = 10) was determined by considering previous studies using SBS testing to assess the effect of different laser irradiations on resin-enamel/dentin bonding SBSs. According to a recent review of Lopes et al., sample sizes of these studies varied from 6 to 10 (Lopes et al., 2015). Nevertheless, some authors criticize the publication of studies reporting mean bond strength values derived from groups containing <10 specimens (Eliades and Brantley, 2000). This can be considered as a limitation of this study. Nevertheless, interpretation of the results of the present study must be made carefully. However, as data of the present study were normally distributed and variances among groups were homogenous, it is believed the results have some useful validity.

Because the present study is one of the first publications on effects of laser irradiation on bonding effectiveness of universal adhesives, further studies covering effects of different laser wavelengths (Er:YAG, Er,Cr:YSGG), power settings (high and low), application modes (laser etching and laser cavity preparation), and adhesives should be conducted in the future.

#### **5** | CONCLUSION

The present study investigated the SBSs of two universal adhesives applied to enamel with different application modes (acid-etching mode, self-etch mode, and Er,Cr:YSGG laser etch mode, respectively).

- 1. Acid-etching mode significantly increased SBSs of adhesives when compared to self-etching and laser-etching modes.
- There were significant differences among the bond strengths of application modes regardless the universal adhesives tested.
- 3. For laser-irradiated enamel, no significant differences in SBS were noted when compared to self-etching mode for both adhesives.

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