

Original Article

Bonding Strength of Universal Adhesives To Er,Cr:YSGG Laser-Irradiated Dentin

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ABSTRACT

Objectives: 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 dentin is still not well known. Therefore, the aim of this study was to compare the shear bond strength (SBS) of two universal adhesives (Single Bond Universal, Nova Compo-B Plus) applied following laser-etching with SBS of the same adhesives applied in self-etch and acid-etch modes, respectively. **Materials and Methods:** Sixty bovine incisors were used to obtain the flattened dentin surfaces. Specimens were divided into two groups according to universal adhesives. Each universal adhesive was applied with one of the following modes, self-etch, acid-etch, or laser-etch ($n = 10$). Er,Cr:YSGG laser was used for laser-etching with 1.5 W–20 Hz parameters. After adhesive applications and composite buildups, SBS was determined after storage in water for 24 h using a universal testing machine with a crosshead speed of 0.5 mm/min. Failure modes were evaluated using a stereomicroscope. Data were analyzed using two-way of analyses of variances (ANOVA) ($P = 0.05$). **Results:** Two-way ANOVA revealed that adhesive had no effect on SBS ($P > 0.05$), but application mode significantly influenced SBS ($P < 0.001$). Laser-etch significantly increased SBS for NCP when compared to self-etch mode, whereas laser-etch provided similar SBS with self-etch mode for SBU. **Conclusions:** The influence of different application modes on dentin bond strength of universal adhesives was dependent on the adhesive material. **Clinical Significance:** For universal adhesives, laser etching may provide some benefits on bonds strength but this would depend on product.

KEYWORDS: Adhesion, bonding, dentin, laser, universal adhesives

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INTRODUCTION

The popularity of the composite resin restorations among dentists and patients have significantly increased, owing to their greater aesthetic properties and the potential of minimal invasive tooth preparations.^[1] The success of the composite resin restoration at long term depends on mainly on the bond strength and durability of adhesive resin system that is a class of the resin material being used to bond composite resin to enamel and dentine.^[2] Surface pretreatments for both enamel and dentin are essential for obtaining a strong bonding between resin composite and dental hard tissues. Acid-etching of dentine surfaces has been used to remove

smear layer from the surface of the dentine, exposing dentinal collagen fibrils, and dentin tubules to be infiltrated by resin monomers. Upon polymerization of the infiltrated resin monomers, dentin hybrid layer and resin tags are formed, providing micromechanical bonding for composite resin via the use of adhesive system.^[3] Self-etching is another approach for bonding to dentin using the resin adhesive systems. Self-

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etch adhesives create dentinal hybrid layers and resin tags by dissolution of smear layer partly.^[4] As their aggressiveness is lesser than acid-etching in general, resulting in the different hybrid layer and resin tag morphology than produced by acid-etch approach.^[4] However, the clinical performance of the adhesive systems depends on adhesive brand rather than these bonding approaches.^[5]

Over recent times, an increasing consideration about the laser usage for different application in dentistry, with cavity preparation and carious removal has been exists.^[6] Er:YAG (erbium:yttrium-aluminium-garnet) and Er,Cr:YSGG (erbium, chromium:yttrium-scandium-gallium-garnet) lasers, together called as erbium lasers, are able to ablate dentin and enamel effectively due to their high absorption peaks by water and hydroxyapatite.^[7] Consequently, these lasers are most commonly used lasers in dental hard tissues. When erbium lasers are used to irradiate dentine surfaces, this result in a distinctive rough surface, open dentinal tubules without smear layers, and micro-irregularities produced by the selective ablation of the intertubular dentin, proposing that the resulting dentin surface is open to resin bonding.^[8-10] Thus, it has been suggested that erbium lasers best fulfill the prerequisites for pretreatment of the dental hard tissues prior to resin bonding.^[6] Although, the recent review of the literature has revealed a reduction in the bond strengths of laser-irradiated dentine compared with dentine prepared by conventional means.^[11]

Aside from the investment of erbium laser application for the dental hard tissue surgery, resin adhesive systems have been improved significantly toward to being more user-friendly and less technique-sensitive during the past several years.^[2] More recently, a new dental adhesive class, called universal adhesives, has been introduced. Unlike to preceding adhesive systems, these adhesives were designed to bond to enamel and dentin, with acid-etch approach or self-etch approach using the same bottle of adhesive. Manufacturer's claims that there is no compromise on bonding effectiveness when either bonding strategy is used.^[12] Surfaces irradiated by laser differ from prepared by conventional means. As all of the resin adhesive systems were designed to perform on enamel and dentin prepared by conventional means, further research is necessary for the bonding effectiveness of universal adhesives on laser-etched dental surfaces.

Preceding researches have mostly concentrated on the bond strength of the preceding adhesive systems to laser-etched dentin surfaces.^[11,13-15] However, to the best knowledge of the authors, there is scant information

regarding the shear bond strength (SBS) of universal adhesive to laser-etched dentin in the literature. Consequently, the aim of the present study was to determine the SBS of two universal adhesives that were applied to dentin with three application modes including (i) self-etch mode, (ii) acid-etch mode, (iii) laser-etch mode. The null hypotheses tested were (1) there is no difference between the two universal adhesives when they are used for bonding to dentin in acid-etch mode, or self-etch mode, or laser-etch mode; (2) the adhesive application mode has no effect on SBS of universal adhesives to dentin.

MATERIALS AND METHODS

Experimental design

A factorial design 2×3 was used to evaluate the variables "adhesives" in two levels (Single Bond Universal, Nova Compo-B Plus) and "application modes" in three levels (self-etch, acid-etch, laser-etch). The dependent variable evaluated was SBS values.

Er,Cr:YSGG laser device

Laser irradiations were performed using an Er,Cr:YSGG laser (Waterlase MD, Biolase Technology; San Clemente, California, USA) with following parameters, wavelength 2.780 nm, power of 1.5 W, frequency of 20 Hz, pulse duration of 140 μ s; spot size of 600 μ m; tip MGG6; air pressure setting of 65%; and water pressure setting of 55%. 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.

Specimen preparation

Sixty bovine incisors with no visible defects 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.^[16] Roots were severed by low speed diamond saw under water-cooling. Enamel surfaces were removed by using 320-grit silicon carbide (SiC) abrasive papers by hand under water-cooling, exposing dentin surfaces. 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. Dentin surfaces were finished using 600-grit SiC abrasive papers under water-cooling for 1 min.

Bonding procedures

Prepared teeth samples were randomly divided into two groups of 30 teeth each and assigned to one of the two universal adhesives tested in the present study: Single Bond Universal, Nova Compo-B Plus. The compositions of the adhesives are shown in [Table 1].

Each adhesive was applied to the prepared dentin surfaces in the acid-etch mode, or the self-etch mode, or the laser-etch mode ($n = 10$). For the acid-etch mode, a 37% phosphoric acid gel (Etch Royale, Pulpdent) was used to etching dentine surfaces for 15 s. After this, etched dentine surfaces were rinsed for 10 s to remove etching gel completely. For self-etching mode, no acid-etching gel was used. For laser-etching mode, Er,Cr:YSGG laser was deployed in the way mentioned above. The adhesives were used according to manufacturer's instruction for use [Table 1].

Following adhesive applications, 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 using LED curing unit with power of 1100 mW/cm².

Shear bond strength test

The bonded teeth were stored in the water for 24 h at 37°C before bond strength testing. Specimens were loaded in shear mode until fracture happened with the use of universal testing machine (Instron 3220; Instron Corporation, Canton, Massachusetts, USA) at crosshead speed of 1.0 mm/min using 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. Following the SBS tests, all of the failure specimens were observed with stereomicroscope at 10 × to determine the failure modes. Failure modes were divided into adhesive, cohesive, and mixed failure.

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).

RESULTS

SBS values were presented in both [Figure 1] and [Table 2]. Two-way ANOVA showed that SBS was significantly influenced by application mode ($P = 0.00$) but not significantly influenced by adhesive ($P = 0.676$). The interactions of these two factors were significant ($P = 0.014$), indicating that the differences that existed among application modes

depended on the adhesive system. Therefore, additional one-way ANOVA with post-hoc Tukey tests were performed to reveal the effects of application mode on bond strength of each adhesive. For Single Bond Universal, the self-etch mode showed similar bond strength with the acid-etch mode ($P = 0.102$) and the laser-etch mode ($P = 0.643$). However, the acid-etch mode provided significantly higher bond strength than the laser-etch mode ($P = 0.014$) for Single Bond Universal. For Nova Compo-B Plus, the self-etch mode showed significantly lower bond strength when compared with the acid-etch mode ($P = 0.000$) and the laser-etch mode ($P = 0.031$). The laser-etch mode increased bond strength, but the acid-etch mode provided significantly highest bond strength among all application modes for Nova Compo-B Plus.

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. Self-etch specimens showed higher incidences of adhesive failure for both adhesives [Table 2].

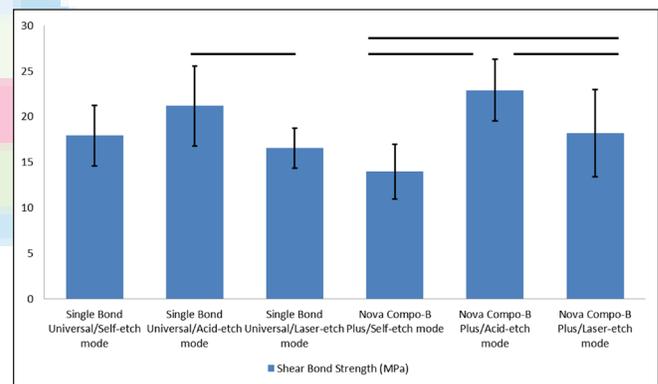


Figure 1: An error-bar graph of dentin bond strength of Single Bond Universal and Nova Compo-B Plus by using different application modes. Data connected by a horizontal line are significantly different ($P < 0.05$).

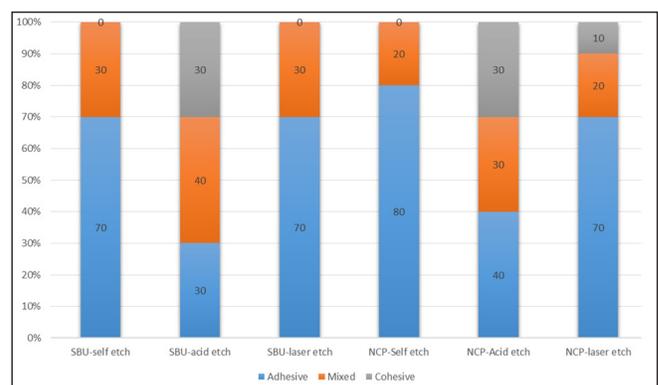


Figure 2: The failure mode distribution of the shear bond strength test. SBU = Single Bond Universal; NCP = Nova Compo-B Plus.

Table 1: Compositions, manufacturer instructions for use the universal adhesives tested in the present study

Adhesive	Manufacturer	Composition	Manufacturer instructions
Single Bond Universal pH 2.7	3M ESPE, St. Paul, Minnesota, 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 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 dihydrogenphosphate; HEMA = 2-hydroxyethyl methacrylate; Bis-GMA = bisphenol A diglycidyl methacrylate; 4-META = 4-methacryloyloxyethyl trimellitate anhydride.

Table 2: Means of shear bond strength (MPa), standard deviation (SD), and failure modes

Application modes	Universal Adhesives							
	Single Bond Universal				Nova Compo-B Plus			
	Bond strength	Failure modes			Bond strength	Failure modes		
		C	M	A		C	M	A
Self-etch	17.90 ± 3.3	0	30	70	13.96 ± 3.0	0	20	80
Acid-etch	21.15 ± 4.4	30	40	30	22.89 ± 3.4	30	30	40
Laser-etch	16.52 ± 2.2	0	30	70	18.17 ± 4.8	10	20	70

Data connected by a vertical line are significantly different ($P < 0.05$) in each column. Groups designated with different superscript letters are significantly different ($P < 0.05$). C = cohesive failure; M = mixed failure; A = adhesive failure.

DISCUSSION

The findings of the present study suggested that first null hypothesis that “there is no difference between the two universal adhesives when they are used for bonding to dentin in either application mode” could not be rejected as, both of tested universal adhesives exhibited similar dentin bond strength, regardless of the application mode. However, the second null hypothesis that “adhesive application mode has no effect on shear bond strength of universal adhesives to dentin” has to be rejected, as it was found that application mode affected significantly bond strength of universal adhesives.

The universal adhesives would be considered as ultra-mild/mild one-step self-etch adhesives ($\text{pH} > 2.5$) in terms of chemical components and clinical application procedures. Similar to the preceding self-etch adhesives, universal adhesives also include the acidic resin monomer to render the adhesive blend into more acidic/aggressive solution to etch the dentine and the enamel. Most of the current universal adhesives have been now incorporated with 10-MDP functional monomer.^[12] This resin monomer has been considered as a quintessential functional resin monomer for chemical bonding to apatite.^[12] However, Single Bond Universal contains also polyalkenoic acid copolymer (Vitrebond), which a known component from preceding adhesives of Single Bond Universal’ manufacturer. Therefore, it has been claimed that universal adhesives should perform no differently from previous generations of etch-and-rinse adhesives or self-etch adhesives.^[12]

When Single Bond Universal and Nova Compo-B Plus were applied with acid-etch mode, Nova Compo-B Plus produced significantly higher dentin bond strength when compared with self-etch mode, while Single Bond Universal showed similar bond strength in acid-etch or self-etch modes. Bonding properties of Single Bond Universal tested in the present study is well-known material in the literature and a recent review of universal adhesives proposed that the application mode of Single Bond Universal adhesive might have no effect on dentin bond strength.^[17] However, some recent reports suggested that acid-etch mode may provide better dentin adhesion for universal adhesives when compared with self-etch mode.^[18–20] Muñoz *et al.*,^[20] reported that in acid-etch mode. All Bond Universal and G-Bond Plus showed higher bond strength than in self-eth mode, whereas application modes did not influence on bond strength of Single Bond Universal. Therefore, it seems that the effect of application mode might depend on the brand of adhesive and not the adhesive class.

Another finding of the present study is that laser-etching of dentin surfaces prior to application of Single Bond Universal had no effect on bond strength. The similar finding has been reported by some studies, recently.^[18,19] Yazici *et al.*^[19] evaluate the bond strength of Single Bond Universal to dentin prepared with SiC paper or an Er,Cr:YSGG laser using acid-etch or self-etch mode. They stated that regardless of preparation, laser etching did not affect the bond strength of the Single Bond Universal.^[19] In other study, Buyukhatipoglu *et al.*^[18] also reported that, laser-etch of Single Bond Universal

showed similar bond strength when compared with self-etch mode.

However, we found that laser-etch mode improved bond strength of Nova Compo-B Plus significantly. The differences in the monomer chemistry of the tested adhesives may also result in differences among the laser-etch modes of universal adhesives. Considering laser-etch mode, the irradiation of dentin surfaces prior to adhesive application may remove smear layer from the dentin surface and leave clean surface as reported in the literature.^[18] In addition, laser-etching increases surface roughness thus may enhance adhesion.^[21] Therefore, we think that the benefits of laser-etching may contribute significantly better bond strength of Nova Compo-B-Plus that was found in this study.

In terms of failure mode, it was reported that a relationship between the bond strength and failure mode was existed.^[22] 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 adhesives produced more mixed and cohesive fractures when used in acid-etch mode than self-etch mode and laser-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.^[23] However, aging of resin-tooth interfaces by long-term water storage may show a better relationship with clinical performance. Indeed, Heintze and others stated that dentin bond strength of adhesive systems after 6-month water storage exhibited a good correlation with marginal discoloration in clinical Class V restorations.^[24]

Since 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 on long-term bonding effectiveness of universal adhesives should be conducted in the future.

CONCLUSIONS

The findings of this *in-vitro* study showed that influence of the application mode on the bond strength of universal adhesives might depend on adhesive brand, rather than adhesive class. No significant differences were found in self-etch and laser-etch groups for Single Bond Universal adhesive. However, laser-etching improved bond strength

for Nova Compo-B Plus when compared to self-etch mode. Additional researches are required to confirm the benefits of laser-etching on bonding effectiveness of universal adhesives in terms of long-term bond strength.

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Conflicts of interest

There are no conflicts of interest

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