

Evaluation of Microshear Bond Strength of Resin Composites to Enamel of Dental Adhesive Systems Associated with Er,Cr:YSGG Laser

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ABSTRACT

The aim of this in vitro study was to evaluate the microshear bond strength (μ SBS) of resin composite to enamel etching by Er,Cr:YSGG laser with the use of two different adhesives systems. Fifty freshly extracted human molars halves were embedded in acrylic resin before preparation for the study, making a total of up to 100 available samples. The specimens were randomly assigned into six groups (n=10) according to substrate pre-treatment and adhesive system on the enamel. A two-step self-etching primer system (Clearfil SE Bond) and a universal adhesive used as an etch-and-rinse adhesive (Adper Single Bond Universal) were applied to the nonirradiated enamel surface according to manufacturer's instructions, as control groups (Control CF and Control SB, respectively). For the other groups, enamel surfaces were previously irradiated with the Er,Cr:YSGG laser with 0.5 W, 75 mJ and 66 J/cm² (CF 5 Hz and SB 5 Hz) and 1.25 W, 50 mJ and 44 J/cm² (CF 15 Hz and SB 15 Hz). Irradiation was performed under air (50%) and water (50%) cooling. An independent *t*-test was performed to compare the adhesive systems. Mean μ SBS \pm sd (MPa) for each group was 16.857 \pm 2.61, 17.87 \pm 5.83, 12.23 \pm 2.02, 9.88 \pm 2.26, 15.94 \pm 1.98, 17.62 \pm 2.10, respectively. The control groups and the 50 mJ laser groups showed no statistically significant differences, regardless of the adhesive system used. The results obtained lead us to affirm that the bonding interaction of adhesives to enamel depends not only on the morphological aspects of the dental surface, but also on the characteristics of the adhesive employed and the parameters of the laser.

Keywords: Microshear bond strength. Er,Cr:YSGG laser. Adhesive system.

1. INTRODUCTION

Since the introduction of the acid etching technique by Buonocore in 1955, a standard protocol to remove the smear layer for successful bonding, different etching methods have been proposed to foment adhesion of composite resins to the dental structure. The self-etching primer systems, which need not necessarily previous smear layer removal, were introduced to simplifying adhesive procedures [1]. Self-etch systems are in turn divided in two-step self-etch and one-step self-etch (all-in-one) adhesives. In the two-step self-etch adhesives, the conditioning agent and the primer are placed in one bottle and the adhesive resin in another, whereas onestep adhesives combine self-etch primer and hydrophobic resin into one application, which results in simplification of the bonding procedure [2]. These systems simultaneously promote demineralization and resin infiltration through the demineralized dental surfaces.

Both types of self-etching systems have been reported to produce predictable bond strengths to dentin. However, controversial and unpredictable bonding values to enamel has been reported, with limited evidence suitability for replacing conventional phosphoric acid etching [3]. It was observed a reduction of enamel bonding effectiveness when universal adhesives were applied on enamel as self-etching adhesives [4]. Nevertheless, with the use of selective phosphoric acid etching, the performance of a one-step self-etch adhesives was significantly improved [5].

High power lasers have been introduced as an alternative to promote chemical/morphological changes on the tooth surface. Laser irradiation produces enamel and dentin surfaces that are free of smear layer, imbricate pattern, and crack formation. Erbium lasers (Er:YAG and Er,Cr:YSGG) have been considered the most promising lasers to be used on mineralized tissues because both wavelengths show high absorption by water and hydroxyapatite. Erbium lasers are

able to cause thermomechanical ablation process on hard tissues, like the etching process, forming a rough structure in dental tissue [6]. The use of both laser and acid together has been reported to enhance the strength of bonding to hard tooth surfaces relative to those exposed to acid alone [7]. Meanwhile, there are not well-designed studies comparing acid etching followed by laser ablation or laser ablation followed by acid etching.

The aim of this in vitro study was to evaluate the microshear bond strength (μ SBS) of resin composite to enamel etching by Er,Cr:YSGG laser with the use of two different adhesives systems.

2. MATERIALS AND METHODS

2.1 Specimen preparation

A total of 50 extracted human molars were visually examined to confirm the absence of physical damage such as deep grooves or cracks. The specimens were cleaned with distilled water and brushes, and then disinfected in 0.5% chloramine for 2 weeks. The roots were sectioned 2 mm from the enamel–cementum junction, and the crowns were divided into halves with a slow-speed diamond saw in a sectioning machine. To facilitate handling of the specimens throughout the experiment, each half was embedded in acrylic resin. The outer surface of the enamel specimens was then ground flat with water-cooled sandpaper of decreasing grit (400, 600) in order to produce a clinical relevant and standardize smear layer.

2.2 Experimental Design

The enamel specimens (n=10) were randomly assigned into six groups according to etching treatment and adhesive system, as summarized in Table 1. The control groups were submitted to polishing only and the enamel surfaces were etching with phosphoric acid and treated with: Control CF - a two-step self-etching primer system (Clearfil SE Bond, Kuraray Medical Inc., Kurashiki, Japan) and Control SB - a universal adhesive used as a two-step etch-and-rinse adhesive (Adper Single Bond Universal, 3M ESPE, St. Paul, MN).

For the other groups, enamel surfaces were irradiated with the Er,Cr:YSGG laser (Waterlase Millennium, Biolase, San Clemente, CA) that works at 2780 nm. The tip was positioned at 1mm (90°) from the enamel surface. In Groups CF 5 Hz and SB 5 Hz the output power was 0.5 W with 75 mJ and 66 J/cm². For Groups CF 15 Hz and SB 15 Hz the output Power was 1,25 W with 50 mJ and 44 J/cm². Irradiation was performed under air (50%) and water (50%) cooling for all groups. The same adhesives were applied on Er,Cr:YSGG laser-irradiated enamels.

Table 1 – Groups and etching protocols.

Group	Adhesive system	Etching protocol
Control CF	Clearfil SE Bond	Phosphoric acid (according to the manufacturer's instructions)
Control SB	Adper Single Bond Universal	Phosphoric acid (according to the manufacturer's instructions)
CF 5 Hz	Clearfil SE Bond	Er, Cr:YSGG - 5 Hz, 0.5 W, 66 J/cm ²
SB 5 Hz	Adper Single Bond Universal	Er, Cr:YSGG - 5Hz, 0.5 W, 66 J/cm ²
CF 15 Hz	Clearfil SE Bond	Er, Cr:YSGG - 15 Hz, 1.25 W, 44 j/cm ²
SB 15 Hz	Adper Single Bond Universal	Er, Cr:YSGG - 15 Hz, 1.25 W, 44 j/cm ²

2.3 Restorative Procedure

The tested adhesive systems were applied to the enamel surfaces according to manufacturer's instructions. Transparent polyethylene tubes with 0.9 mm for the internal diameter and 1 mm in height were positioned over a double-faced perforated tapes and then resin composite (Tetric N-Ceram,) was carefully packed inside the tubes. The resin composite was light-cured for 20 s using a LED light-curing with 1200 mW/cm² (Radii-cal, SDI, Bayswater, Victoria, Australia).

The specimens were kept in distilled water for one hour at 37 °C and then the tubes and the double-faced tapes were removed with a blade, exposing the cylinders of resin composite. Right after, the specimens were stored in distilled water for a week at 37 °C. All specimens were examined under a stereomicroscope at x10 magnification to observe possible defects.

2.4 Microshear Bond Strength Test

The specimens were tested in a universal testing machine (Kratos, Kratos Equipamentos Industriais Ltda, Cotia, SP, Brazil). A thin orthodontic wire (0.2 mm) was looped around the cylinder aligned with the setup to ensure the correct orientation of the forces. The crosshead speed was set at 0.5 mm/min until failure.

2.5 Statistical analysis

Data were organized into an Excel spreadsheet (Microsoft Office 2007) and analyzed using SPSS 13.0 (Statistical Package for the Social Sciences, Chicago, IL, USA) for Windows. Statistical measures were obtained and the Komogorov-Smirnov test was used to assess the normality of the data. An independent *t*-test was performed to compare the adhesive systems. All tests were applied with 95% confidence.

3. RESULTS

Figure 1 shows the box plots of microshear bond strength, displaying the interquartile range and the standard deviation. The small square inside the boxes represent the median, the parallel line inside the boxes represents the mean. Table 2 shows the mean shear bond strengths values (in MPa) of two-step self-etch (Clearfil SE Bond) and one-step self-etch (all-in-one) (Adper Single Bond Universal) adhesive bonded to enamel. Microshear bond strength values obtained for control groups on enamel were in average of 16.857 ± 2.61 MPa for Control CF and 11.875 ± 5.83 MPa for Control SB. The maximum bond strength was recorded for the SB 15 Hz group and the minimum bond strength was recorded for the SB 5 Hz group.

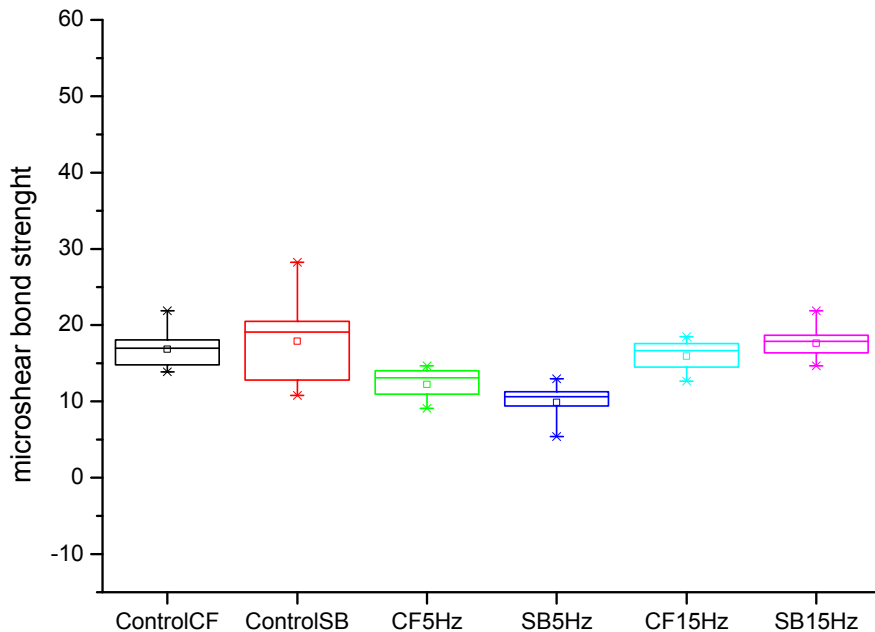


Figure 1 - Box plots of microshear bond strength display the interquartile range and the standard deviation.. The small square inside the boxes represent the median, the parallel line inside the boxes represents the mean.

Table 2 - Microshear bond strengths (MPa) and standard deviation (\pm sd) of the tested adhesives to enamel. Different superscript letters indicate statistical differences ($p < 0.05$)

	<i>Clearfil SE Bond</i>	<i>Adper Single Bond Universal</i>
Control	16.857 \pm 2.61 ^a	11.875 \pm 5.83 ^b
5 Hz	12.230 \pm 2.02 ^d	9.886 \pm 2.26 ^c
15 Hz	15.947 \pm 1.98 ^{a,c}	17.628 \pm 2.10 ^{b,c}

An independent *t*-test was performed to compare the effect of Clearfil SE Bond and Adper Single Bond Universal adhesive systems on Bond strength. The results of t-Test showed no statistical differences between the control groups ($p = 0.62095$). Comparison of mean values of μ SBS of the groups ($n = 10$) is shown in Table 2-

4. DISCUSSION

The bond strength of adhesive systems is one of the main factors to be considered in composite resin restorations. For years the standard protocol to remove the smear layer for successful bonding has been acid etching, that appears to improve retention by selectively eroding certain hydroxyapatite formations, characterized by surface irregularities and demineralization areas, facilitating penetration by the development of resin tags [8].

New bonding agents have been introduced to the market as self-etch primer and single-bottle systems to simplifying adhesive procedures. These systems simultaneously promote demineralization and resin infiltration through the demineralized dental surfaces. However, a reduction of enamel bonding effectiveness when universal adhesives were applied on enamel as self-etch adhesives has been recently reported [9]. Their etching capacity has been shown to be more restricted, as they present low reactivity with the mineral component, lower availability of H⁺ ions, and high molecular weight, compared with phosphoric acid, promoting etching that is not as deep and retentive on dental enamel [10]. Therefore, the use of selective phosphoric acid etching has been recommended. Meantime, because of the demineralization areas acid-induced, enamel becomes more susceptible to caries attack [11].

Laser etching has become an alternative to acid etching of enamel. Laser can promote physical changes such as melting and recrystallization in the enamel [12]. Laser irradiation laser ablation are able to eject hard tissue particles, resulting in a typical imbricate patterned surface with an evidently rough aspect without smearlayer [13]. These morphological features seem to be enough for adhesion, since the interaction of self-etch adhesives with dental substrates is expected to be better in the absence of smear debris [14].

Laser irradiation of the enamel modifies the calcium-phosphate ratio and leads to the formation of more stable and less acid-soluble compounds [11]. Increased resistance to acid etching of Er:YAG-irradiated enamel has been reported [15]. However, a more mineralized, acid resistant enamel surface may resist etching by the weak acid of a self-etching primer adhesive [16], which would be a limitation. Besides that, on enamel, surface roughness may influence bond strengths for self-etching primer adhesives [17]. Variability in bonding effectiveness of this group of adhesives may be attributed in part to their pH and etching aggressiveness on the enamel and dentin substrate and to the particular enamel-dentin adhesive used [18].

Conflicting reports have described the effectiveness of resin bonding following laser tooth preparation. Some studies have found significant lowering of enamel bond strengths with a two-step adhesive, a one-step self-etching primer adhesive, and a total-etch adhesive following Er,Cr:YSGG [12]. Part of the results of our study confirmed these findings, since the use of Er,Cr:YAGG laser with 5 Hz, 0.5 W, 75 mJ and 66 J/cm² reduced the bond strength to enamel, regardless of the adhesive system used. The occurrence of thermal alterations on the irradiated enamel, such as melting and chemical changes, could render the enamel less receptive for adhesion according Cardoso et al. (2008) [12]. Another reported explanation is that Er,Cr:YSGG-laser irradiation can reduce the acid dissolution of dental hard tissues that could hamper the acid conditioning of enamel, especially considering the limited etching potential of “mild” self-etch systems [19].

Differently, Basaran et al. (2007) [8] found that bond strength obtained with Er,Cr:YSGG laser (operated at 1 W or 2 W for 15 seconds) was comparable to that obtained with acid etching. In our study, when the laser parameters 15 Hz, 1.25 W, 50 mJ and 44 J/cm² were used, the results of bond strength were similar to those in the control groups.

According to Hossain et al. (2003) [20] laser-irradiated enamel shows a significant increase in calcium in its composition. The 10-MDP molecule, present in the used adhesives, can partially decalcify dental hard tissues, penetrate the demineralized substrate and chemically bond to the remaining calcium [19], which could explain these results.

5. CONCLUSIONS

The results obtained lead us to affirm that the bonding interaction of adhesives to enamel depends not only on the morphological aspects of the dental surface, but also on the characteristics of the adhesive employed and the parameters of the laser. The highest value obtained for the Microshear bond strengths was 17.628 ± 2.10 Mpa, using Adper Single Bond Universal with the laser operating at 15Hz, 1.25 W, 44 j/cm^2 .

6. ACKNOWLEDGEMENTS

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7. CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

REFERENCES

- [1] Loguercio, A.D., Munõz, M.A., Luque-Martinez, I., Hass, V., Reis, A. and Perdigão, J., "Does active application of universal adhesives to enamel in self-etch mode improve their performance?," *Journal of Dentistry* 43(9), 1060-70 (2015).
- [2] Kimyai, S., Ajami, A.A., Chaharom, M.E.E., Oskoe, J.S., "Comparison of Microleakage of Three Adhesive Systems in Class V Composite Restorations Prepared with Er,Cr:YSGG Laser," *Photomedicine and Laser Surgery* 28(4), (2010).
- [3] Perdigão, J., Gomes, G., Gondo, R., Fundingsland, J.W., "In vitro bonding performance of all-in-one adhesives. Part 1 - Microtensile bond strengths," *J Adhes Dent* 8, 367-373 (2006).
- [4] Perdigão, J. and Loguercio, A.D., "Universal or multi-mode adhesives: why and how?," *Journal of Adhesive Dentistry* 16, 193-4 (2014).
- [5] Van Landuyt, K.L., Peumans, M., De Munck, J., Lambrechts, P. and Van Meerbeek, B., "Extension of a one-step self-etch adhesive into a multi-step adhesive," *Dental Materials* 22, 533-44 (2006).
- [6] Ansari, Z.J., Fekrazad, R., Feizi, S., Younessian, F., Kalhori, K.A.M., Gutknecht, N., "The effect of an Er,Cr:YSGG laser on the micro-shear Bond strength of composite to the enamel and dentin of human permanent teeth," *Lasers Med Sci* 27, 761-765 (2012).
- [7] Lee, B.S., Hsieh, T.T., Lee, Y.L. et al., "Bond strengths of orthodontic bracket after acid-etched, Er:YAG laser-irradiated and combined treatment on enamel surface," *Angle Orthodontist* 73, 565-570 (2003).
- [8] Basaran, G., Özer, T., Berk, N. and Hamamci, O., "Etching Enamel for Orthodontics with an Erbium, Chromium:Yttrium-Scandium-Gallium-Garnet Laser System," *Angle Orthodontist* 77(1), (2007).
- [9] De Goes, M.F., Shinohara, M.S. and Freitas, M.S., "Performance of a new one-step multi-mode adhesive on etched VS non-etched enamel on Bond strength and interfacial morphology," *Journal of Adhesive Dentistry* 16, 243-50 (2014).
- [10] Rotta, M., Bresciani, P., Moura, S.K., Grande, R.H., Hilgert, L.A., Baratieri, L.N., Loguercio, A.D. and Reis, A., "Effects of phosphoric acid pretreatment and substitution of bonding resin on bonding effectiveness of self-etching systems to enamel," *Journal of Adhesive Dentistry* 9(6), 537-545 (2007).

- [11] Türkmen, C., Sazak-Öveçoglu, H., Günday, M., Güngör, G., Durkan, M. and Öksüz, M., “Shear bond strength of composite bonded with three adhesives to Er,Cr:YSGG laser-prepared enamel,” *Quintessence Int* 41, 119–124 (2010).
- [12] Cardoso, M.V., De Munck, J., Coutinho, E., Ermis, R.B., Van Landuyt, K., De Carvalho, R.C.R. and Van Meerbeek, B., “Influence of ErCr : YSGG laser treatment on microtensile bond strength of adhesives to enamel” *Operative Dentistry* 33(4), 448-455 (2008).
- [13] De Munck, J., Van Meerbeek, B., Yudhira, R., Lambrechts, P. and Vanherle, G., “Micro-tensile bond strength of two adhesives to erbium:YAG-lased vs bur-cut enamel and dentin,” *European Journal of Oral Sciences* 110(4), 322-329 (2002).
- [14] Ogata, M., Harada, N., Yamaguchi, S., Nakajima, M., Pereira, P.N.R. and Tagami, J., “Effects of different burs on dentin bond strengths of self-etching primer bonding systems,” *Operative Dentistry* 26(4), 375-382 (2001).
- [15] Kim, J.H., Kwon, O.W., Kim, H.I. and Kwon, Y.H., “Acid resistance of erbium-doped yttrium aluminum garnet laser-treated and phosphoric acid-etched enamels,” *Angle Orthodontist* 76(6), 1052-1056 (2006).
- [16] Tay, F.R., Pashley, D.H., King, N.M., Carvalho, R.M., Tsai, J., Lai, S.C.N. and Marquezini, L. Jr., “Aggressiveness of self-etch adhesives on unground enamel,” *Operative Dentistry* 29(3), 309-316 (2004).
- [17] Van Meerbeek, B., De Munck, J., Mattar, D., Van Landuyt, K. and Lambrechts, P., “Microtensile bond strengths of an etch & rinse and self-etch adhesive to enamel and dentin as a function of surface treatment,” *Operative Dentistry* 28(5), 647-660 (2003).
- [18] De Oliveira, M.T., Di Francescantonio, M., Consani, S., Giannini, M. and Ambrosano, G.M.B., “Influence of dentin smear layer created by chemo-mechanical or bur excavation methods on adhesion of self-etching primers and a conventional adhesive,” *Journal of Adhesion* 83(9), 821-835 (2007).
- [19] Perdigão, J. and Geraldeli, S., “Bonding characteristics of self-etching adhesives to intact versus prepared enamel,” *Journal of Esthetic and Restorative Dentistry* 15(1), 32-42 (2003).
- [20] Hossain, M., Nakamura, Y., Tamaki, Y., Yamada, Y., Murakami, Y. and Matsumoto, K., “Atomic analysis and Knoop Hardness measurement of the cavity floor prepared by Er,Cr:YSGG laser irradiation *in vitro*,” *Journal of Oral Rehabilitation* 30(5), 515-521 (2003).