10.5005/jp-journals-10015-1046

RESEARCH ARTICLE

Influence of Er:Yag Laser on Shear Bond Strength of Self-etching Adhesives to Bovine Enamel: *In vitro* Study

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ABSTRACT

Purpose: The aim of this study was to evaluate the influence of the enamel treatment with Er:YAG laser on the bond strength of self-etching adhesives.

Materials and methods: One-hundred bovine incisors were ground to obtain flat enamel surfaces. The bond area was delimited with 3 mm diameter adhesive tape. Specimens were divided in two groups: Group L: Enamel surface received Er:YAG laser application (Kavo KEY 3) at 300 mJ/6 Hz in sweeping mode for 30 seconds; Group N: Enamel surface received no additional treatment. Each group was divided into five subgroups according to self-etching adhesive: S&E (Self & Etch), FB (Futurabond NR), XE (Xeno III), OU (One-Up bond F), CL (Clearfil SE Bond). Cylinders of Z250 composite were fabricated on the bonding area using a teflon matrix. The teeth were stored in water at 37ºC/24 hours and submitted to shear testing at a speed of 1 mm/min.

Results: The results were analyzed by two-way ANOVA and Tukey's test, obtaining p-value = 0.001. The mean values (±SD) in MPa for each group/subgroup were: N/FB—19.65(±3.87)a; N/CL—19.55(±3.78)a; N/OU—15.57(±2.85)ab; N/XE—15.32(±4.00)b; L/CL—13.91(±2.54)b; L/OU—13.16(±2.17)bc; L/XE—9.74(±1.65)cd; N/S&E—8.83(±2.63)d; L/FB—8.30(±2.27)d; L/S&E—6.56(±1.55)d.

Conclusion: It was concluded that the enamel surface treatment with Er:YAG laser reduced the bond strength significantly for most of the self-etching adhesives tested.

Keywords: Bond strength, Enamel, Laser, Self-etching adhesives.

INTRODUCTION

At present, laser technology is being widely applied in operative dentistry procedures in an attempt to replace the use of conventional handpiece instruments. However, there has been a concern about studying the principle of laser applications, effects on and consequences for dental tissue, in order to improve the application techniques on enamel and dentin.[1] Several studies have evaluated the use of Er:YAG laser for cavity preparation and decayed tissue removal, and its effects on dentin and enamel conditioning with the goal of improving the bond strength of adhesive systems.[2-4]

Researchers have started investigating the ability of erbium:YAG (Er:YAG) laser to produce an etched surface.[3,4] Er:YAG laser (2.92 mm) has a huge potential for hard tissue removal due to its ablation process, in which most of the energy is absorbed by the water present in the dental tissue, collagen and hydroxyapatite.[4,5] Thus, the thermal side effects on the dental tissue are reduced. Nevertheless, the Er:YAG laser ablation process can affect dental structure, increasing its resistance to acid and demineralization.[5]

Adhesive systems became the most experimented and studied materials in dentistry, since micromechanical adhesion to the acid-etched enamel surface was shown to be very effective.[6] However, following the modern trend of simplifying the clinical steps and saving operating time, new bonding strategies were developed. Watanabe et al in 1994[7] proposed the use of high concentrations of acidic resinous monomers, which in an aqueous solution are capable of releasing H^+ ions and etching the dental structure, at the same time as they penetrate into the substrate. These materials, called self-etching, prevent the formation of conditioned tissue without adhesive filling as these two events occur simultaneously.

Although they have presented good performance in dentin,[8,9] some researchers suggest that due to low acidity their performance in enamel is lower and capable of leading to low bond strength values in this substrate.[10,11] Er:YAG laser etching may be useful in preparing dental hard tissues for adhesion.[12,13]

Some authors recommend this treatment as an alternative to enamel etching before composite bonding.[14] However, the possible advantages and disadvantages of Er:YAG laser etching remain unclear.

Thus, prior laser treatment on the enamel surface with the use of the self-etching adhesive systems and a diminished demineralization capacity could raise some concerns. Therefore, the proposal of this study was to evaluate the effects of Er:YAG laser treatment on the shear bond strength of enamel treated with self-etching adhesives. The null hypothesis tested was that the use of Er:YAG laser does not interfere with the bond strength of the self-etching adhesives.

MATERIALS AND METHODS

One hundred bovine incisors were sectioned in the apical direction 3 mm away from the cement-enamel junction using a carborundum disk at high speed; the roots were discarded and the pulp tissue was removed with endodontic files. The teeth were embedded in colorless self-polymerizable acrylic resin blocks with the aid of a mold made of silicone. The enamel surfaces were grounded with a plaster cutting appliance (Kohlbach Motores Ltd, Jaraguá do Sul, SC, Brazil) under water-cooling, until an area of 3 mm in diameter was exposed. The superficial smear layer was standardized using 600 grit water abrasive papers coupled to a circular polishing machine (DP-10, Panambra, São Paulo, SP, Brazil). Teflon adhesive tape with a hole 3 mm in diameter was fixed over the region of the exposed enamel in order to delimit the area of adhesive system action.

The specimens were randomly divided into two groups (G1 and G2) with 50 specimens each, according to whether or not Er:YAG laser was used:

- Group L: Enamel surfaces were irradiated by Er:YAG laser, model Kavo Key Laser 3 (Kavo Co. Biberach, Germany); wavelength 2940 nm; mean power 1.8W; pulse frequency 6 Hz; pulse duration 100 ìs; and pulse energy 300 mJ in sweeping mode for 30 seconds.
- Group N: Enamel surface received no additional treatment.

  In group L, the enamel surface was first wetted to avoid cracking and fusion and was cooled with the water spray during irradiation.[15] All the settings used in this study were selected in accordance with laser manufacturer's instructions. The irradiations were performed with the beam aligned perpendicular to the surface and moved in a sweeping fashion by hand during the exposure period, without the use of a fixed support to simulate clinical conditions as closely as possible.

  In group N, the enamel surface of specimens was washed with an air/water spray for 40 seconds and dried with a blower air jet.

  Each group was divided into five subgroups according to the self-etching adhesive used:

- Subgroup S&E: Self & Etch (Vigodent, Rio de Janeiro, RJ, Brazil);
- Subgroup FB: Futurabond NR (Voco, GMBH, Germany);
- Subgroup XE: Xeno III (Dentsply De Trey GmbH, Konstanz, Germany);
- Subgroup OU: One-Up Bond F (Tokuyama, Tokyo, Japan);
- Subgroup CL: Clearfil SE Bond (Kuraray CO., Tokyo, Japan).

The commercial brand name, chemical composition, manufacturer and pH of the materials used are presented in Table 1.

The adhesive systems were used according to the manufacturers' recommendations. Light curing was performed for 10 seconds using a Curing Light XL 3000 appliance with a power density of 600 mW/cm^2 (3M ESPE/ St. Paul, MN, USA).

The specimens were placed in a two-piece teflon matrix 4 mm thick with a 3 mm diameter hole, superimposed on the area delimited by the teflon adhesive tape. The resin composite Filtek Z250 (3M ESPE/St. Paul, MN, EUA) was inserted in two increments and light cured for 40 seconds. After removing the matrix, the resin composite cylinder received another two complementary light activations of 40 seconds each. The specimens were stored in distilled water at 37ºC/24 hours and submitted to the shear bond strength test in a universal test machine (DL-1000, EMIC, São José dos Pinhais, PR, Brazil) with a 50 Kg load cell at a speed of 1 mm/min, in accordance with the standards described in ISO TR 11405. The data obtained were submitted to the parametric two-way analysis of variance (ANOVA) to determine which of the adhesives and techniques were most efficient. To locate the differences, Tukey's test was used ($\alpha = 0.05$).

RESULTS

The ANOVA results for the interaction between factors (surface treatment × self-etching adhesives) showed a p-value = 0.001 (F = 8.25), with 4° of freedom, which resulted in significant differences among groups.

Table 2 shows the results of Tukey's test for all groups/ subgroups. The group with laser-etched surface enamel presented significantly lower bond strength means than the group that received no surface enamel treatment for the self-etching adhesive systems Xeno III, Futurabond NR and Clearfil SE Bond. The Self & Etch and One-Up Bond F self-etching adhesive systems presented similar results for the variation of surface treatments.

DISCUSSION

The development of Er:YAG laser has brought about some promising opportunities in dentistry. Several researches for studies have been conducted with the aim of improving the carious tissue removal technique; assuring biological safety, and patient and professional well-being as well as representing an alternative for performing cavity preparations, making it

Table 1: Compositions and manufacturers of materials used

Name	Composition	Manufacturer	pH
One-up Bond F	**Agent A:** MAC-10 (malonic acid 10-methacryl oxide camethylene); methacryloxyalkyl acid phosphate; Bis-phenol A diglycidyl methacrylate (Bis-GMA); triethyleneglycol dimethacrylate (TEGDMA); photoinitiators **Agent B:** 2-Hydroxyethyl methacrylate (HEMA); water; aluminum glass powder; Silica; Photoinitiators	Tokuyama Tokyo, Japan	1.2
Clearfil SE Bond	**Primer:** 10-Methacryloyloxydecyl dihydrogen phosphate (MDP); HEMA: Hydrophilic dimethacrylate; di-camphorquinone; N,N-diethanol-p-toluidine; water. **Bond**: MDP; Bis-GMA; HEMA; hydrophobic dimethacrylate; N, N-diethanol-p-toluidine; di-camphorquinone; silanated colloidal silica	Kuraray CO, Osaka, Japan	2.32
Self & Etch	**Primer:** HEMA copolymer; dimethacrylate; water; alcohol; MEP (methacryloyloxyethyl dihydrogen phosphate); photoinitiators stabilizers. **Bond:** MEP; HEMA; dimethacrylates; alcohol; Bis-GMA; microfiller; photoinitiators stabilizers	Vigodent Rio de Janeiro, RJ, Brazil	2
Futurabond NR	**Agent A:** MAC-10; methacryloxyalkyl acid phosphate; Bis-GMA; TEG-DMA; photoinitiators **Agent B:** HEMA, water, aluminum glass powder; silica; photoinitiators	Voco, Cuxhave, Germany	1.4
Xeno III	**Agent A:** HEMA, ethanol, amorphous silica, purified water, and THB (toluene hydroxybutylate). **Agent B:** Methacrylate functionalized with phosphoric acid (Piro-EMA); mono fluoro phosphazene modified (PEM-F) of urethane dimethacrylate; THB; camphoroquinone and ethyl-4-dimethylaminobenzoate.		1.4

Table 2: Results of Tukey's test for all groups/subgroups

Technique/Adhesive	Mean in MPa (± Standard deviation)	Homogeneous groups*			
Without laser/Futurabond NR	19.6 (± 3.9)	A			
Without laser/Clearfil SE Bond	19.5 (± 3.8)	A			
Without laser/One-up Bond F	15.6 (± 3.2)	A	B		
Without laser/Xeno III	15.3 (±4.0)		B		
With laser/Clearfil SE Bond	13.9 (± 2.5)		B		
With laser/One-up Bond F	13.1 (± 2.1)		B	C	
With laser/Xeno III	9.7 (± 1.6)			C	D
Without laser/Self & Etch	8.8 (± 2.6)				D
With laser/Futurabond NR	8.3 (± 2.3)				D
With laser/Self & Etch	6.6 (± 1.5)				D

*The groups accompanied by the same letters presented no significant differences.

suitable for different restorative materials in the diverse range of clinical procedures.[1,16]

The tissue removal promoted by Er:YAG lasers is based on the absorption of radiation energy by water molecules present in the dental hard tissues, which is rapidly heated to boiling high temperature producing vapor. The result of this process is increased pressure in the irradiated area leading to local micro explosions and ejection of dental hard tissue in the form of microparticles, with minimal or no thermal side effects.[17]

During the Er:YAG laser ablation process, enamel tissue undergoes structural changes, such as surface irregularity, absence of smear layer and exposure of enamel prisms. This heterogeneous microretentive pattern generated on enamel, suggesting a bee-hive like appearance on the irradiated surface, was supposed to increase the composite resin material bond strength.[18] In practice, the microporosities resulting from laser application do not have the same ideal pattern obtained with phosphoric acid. The result is a rather restructured enamel prism surface; probably due to the enormous capacity of laser to remove this dental substrate. As a result, the micro-retention promoted by the ablation process is notoriously different from the one resulting from acid etching. Consequently, this uneven enamel structure hampers the bonding effect of the composite resin material,[19] compromises cavity wall sealing and makes the adhesive vulnerable to marginal microleakage.[20] According to previous studies, the enamel surface irradiated by Er:YAG laser is less susceptible to demineralization, because the morphological alterations increase its resistance to acid etching.[18,21,22] These changes in enamel are characterized by alterations in melting and crystallinity, leading to less permeable enamel, whitening of the surface and modifications in the shape and size of hydroxyapatite crystals. Further morphological changes occur in enamel, such as an increase in calcium concentration and substantial loss of water and organic substrate (carbonate and phosphate), which are extremely soluble components of hydroxyapatite. This reorganization of ions in the crystalline structure decreases the enamel solubility in acid substances.[19,20,22]

In this study, statistical analysis of the data revealed significant difference between the control groups and the experimental groups for each adhesive system evaluated; confirming the weaker bond strength in the lased groups. Er:YAG laser increases the enamel surface resistance to etching, therefore, the acids present in the primers of the self-etching adhesive systems tested in this study were unable to promote proper demineralization on the lased surfaces.

The demineralization potential of the acidic monomers on enamel depends on the low pH of its solution. A solution considered acidic has a higher concentration of hydrogen ions than that of hydroxide ions, and releases hydrogen ions when immersed in an aqueous environment, causing unbalance in this milieu.[23] The most common acid used in dentistry is phosphoric acid, loaded with ionic radicals.

In addition to pH, the acid-dissociation constant, defined as the negative logarithm of the acid dissociation constant Ka equation (pKa = –logKa), also determines the strength of an acid.[24] The acid-dissociation constant describes the tendency of the molecules to dissociate. The higher the acid dissociation, the stronger is the acid and lower the pKa.[25] Muecke et al (2003)[25] evaluated the pKa, reported that the monomers have a heavier molecular weight than the phosphoric acid, lowering their effectiveness for the dissolution of hydroxyapatite.

Adhesive systems rely on different pHs to etch enamel and dentin.[26] Consequently, the microporosities created by the self-etching adhesive systems tested on the irradiated enamel may differ from one another according to the acidic monomer present in the formula. Strong primers are more acidic ($pH \leq 1$), and they generate more porosity on the enamel surface due to their demineralization potential close to that of phosphoric acid.[27]

Although Clearfill SE Bond adhesive system is classified as a self-etching adhesive with a moderate demineralization potential[27](pH = 2.32), in this investigation, the group treated with this product obtained the highest mean value of adhesive bond strength (16.73 MPa); in comparison with Xeno III adhesive system, pH = 1.4 (12.53 MPa); Futurabond NR adhesive system, pH = 1.4 (13.97 MPa); and Self & Etch adhesive system, pH = 2 (7.70 MPa). The One-Up Bond F adhesive system, which had the lowest pH (1.2) did not perform better in the adhesive bond strength evaluation than the Clearfil SE Bond adhesive system; obtaining a mean bond strength value of 14.36 MPa. This can be explained by the high concentration of acidic monomer MDP (30%) in the Clearfil SE primer, giving the solution a superior etching potential, pKa = 2.2.[25]

From the results, it can be inferred that although the primers used in this research have acid components with low pH and all the self-etching adhesive systems used are classified as strong or moderate (Table 2), which should warrant the etching on all mineralized structures; it was not enough to create an efficient resin imbrication in enamel.[28] The shallow etching pattern generated on the dental surface and subsequent reduced primer penetration into the surface microporosities, caused by the high concentration of calcium and phosphate ions precipitated in the inner solution, are responsible for this weak interaction.[6,26,29]

From this study, it can also be observed that the mean bond strength value for all adhesive systems tested was significantly lower in the groups irradiated by Er:YAG laser, when compared with the unlased groups. Similar findings were observed in an earlier investigation, in which the primer did not penetrate sufficiently into the lased enamel, causing unsatisfactory adhesion of the self-etching primer to the enamel surface.[30]

A well-accepted explanation for the low bond strength values obtained when self-etching adhesives were applied on enamel irradiated by Er:YAG laser concerns the presence of calcium and phosphate in the microstructures created by the laser on the dental surface. Once enamel is irradiated, it releases calcium present in a high concentration in dental substrate, which reacts with the phosphate contained in the primer molecules. This chemical reaction is very likely to occur due to the high affinity between these two ions.[22] Thus, the pH of the primer molecules is briefly neutralized, blocking the etching action of this compound and restraining the demineralization pattern.[28]

The Self & Etch adhesive system had the lowest value recorded for bond strength both in the lased and unlased groups; being even lower in the former. This may be the result of the chemical nature of the acidic monomer itself, which lacks efficiency in promoting etching.

The present study found unfavorable results as regards obtaining an acceptable pattern on the enamel surface treated by Er:YAG laser prior to applying the adhesive system coating, a finding similar to earlier investigations.[30-32] The laser treatment alone was insufficient to achieve an effective adhesive bond strength,[32] therefore additional methods are indicated to improve the bond to enamel treated by laser, such as the association with phosphoric acid.

Previous studies have reported that the Er:YAG laser treatment associated with supplementary etching with phosphoric acid can actually improve the composite bond strength to enamel.[33,34] There are no conclusive data in the literature about the ultimate action and the effectiveness of

Er:YAG irradiation treatment on the enamel substrate. In addition, the pH, pKa and type of acidic monomer present in the self-etching adhesive systems seem to play a major role in the bond of adhesives to enamel; particularly after the laser treatment, which increases the resistance to etching of the enamel surface.

The null hypothesis was rejected. It was noted that the laser may adversely affect bond strength of the self-etching adhesive systems. Therefore, it is mandatory that new researches for studies be conducted to clarify the efficiency of the Er:YAG laser on enamel tissue and to define the optimal range of Er:YAG irradiation for conditioning the enamel surface previous to application of the adhesive system.

CONCLUSIONS

Within the limitations imposed by the *in vitro* study and based on the test results, it may be concluded that:

- There was an undeniably adverse effect on the bond strength of self-etching adhesives applied on an enamel surface treated with Er:YAG laser
- The bond strength performance differed notably among the adhesive systems.

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