The durability of adhesion to Er,Cr:YSGG laser-irradiated enamel

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Background and aims: Adhesion durability of resin adhesives with Er,Cr:YSGG laser irradiated enamel has been rarely investigated in the literature. Thus, the present study evaluated the influence of long-term water storage (12-month) on resin bond strength with the enamel irradiated with Er,Cr:YSGG laser irradiation deployed different settings.

Materials and methods: The flattened enamel samples of 35 bovine teeth, which were embedded into acrylic blocks, were randomly divided into 7 groups (n = 5), according to surface treatments using Er,Cr:YSGG laser with different parameters 6 W/20 Hz, 6 W/35 Hz, 6 W/50 Hz, 3 W/20 Hz, 3 W/35 Hz, 3 W/50 Hz or no laser treatment (Bur-treating as a control). Adper Single Bond 2 was applied to the prepared enamel and the composites were placed and cured. Resin-enamel sticks with an approximate cross-sectional area of 0.8 mm² were obtained, and microtensile bond strength (µTBS) tests were performed at 24-hour and 12-month of water storage after bonding. The µTBS data were analyzed with two-way ANOVA and Tukey tests (p < 0.05).

Results: 24-hour water storage after bonding, the µTBS to laser-irradiated enamel in the 6W/20 Hz group was significantly lower than those of bur-treated. However, 3 W/50 Hz showed significantly higher µTBS than those of bur-treated. Two-way ANOVA revealed that 12-month water storage did not influenced µTBS.

Conclusion: It may be concluded that, initial bond strength to Er,Cr:YSGG laser irradiated enamel might be significantly influenced by power and pulse frequency settings. However, resin bonding to laser-irradiated enamel was stable over 12-month water storage regardless of tested laser parameters.

Key words: Er,Cr:YSGG laser • enamel • bond strength • bond durability • water storage

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sives and dental hard tissue surfaces is a crucial issue\textsuperscript{15, 14}, as it certainly affects the durability of restoratives. While the oral environment is the definitive test medium to assess the effectiveness of adhesive resin materials, its intricacy has resulted in the development of in-vitro test methods i.e. the use of thermocycling regimens, pH cycling, and water storage for long-term periods, that mimic the process that happens in-vivo, making available significant knowledge about the essential mechanisms of degradation of resin bonding to dental hard tissues\textsuperscript{19}. Consequently, it became likely to expect the intricate processes in the oral medium and their relations with reliability of resin bonding by the time.

Moreover, the resin bonds with enamel action was a significant factor in order to achieve successful and reliable bonding of resin adhesive systems, because it was shown that the presence of enamel yields to a durability of resin-dentin bond. The enhanced durability could be ascribed to the defensive character of the adjacent resin-enamel bond counter to degradation of interfaces\textsuperscript{16, 17}).

Er,Cr:YSGG laser is a favorable technique for preparation of dental hard tissues and to present time, any data described the bonding effectiveness of adhesive resin materials to Er,Cr:YSGG laser irradiated enamel after the long-term degradation process has not existed. Therefore, the present study aimed to evaluate the effect of 12-month of water storage on the bonds of adhesive resin with Er,Cr:YSGG laser-irradiated enamel by means of the microtensile bond strength (µTBS) test.

Materials and methods

Specimen preparation

Thirty-five permanent bovine incisors were used in this study. The teeth were cleaned to remove any tissue remnants. All teeth were embedded in self-curing acrylic resin with labial surfaces parallel to the floor using double-faced adhesive bands. Labial surfaces of teeth were ground using 320-grit abrasive paper. Then, surfaces were further ground with 600-grit abrasive paper for 60 seconds to obtain standardized smear layers. Then, teeth were divided to seven groups (n = 5) randomly as follows:

- **Group 1**: Enamel irradiated using Er,Cr:YSGG laser with 6 W-20 Hz power - pulse frequency parameters (300 mJ per pulse, 90 J/cm\textsuperscript{2} of energy density).
- **Group 2**: Enamel irradiated using Er,Cr:YSGG laser with 6 W-35 Hz power - pulse frequency parameters (171 mJ per pulse, 90 J/cm\textsuperscript{2} of energy density).
- **Group 3**: Enamel irradiated using Er,Cr:YSGG laser with 6 W-50 Hz power - pulse frequency parameters (120 mJ per pulse, 90 J/cm\textsuperscript{2} of energy density).
- **Group 4**: Enamel irradiated using Er,Cr:YSGG laser with 3 W-20 Hz power - pulse frequency parameters (150 mJ per pulse, 45 J/cm\textsuperscript{2} of energy density).
- **Group 5**: Enamel irradiated using Er,Cr:YSGG laser with 3 W-35 Hz power - pulse frequency parameters (86 mJ per pulse, 45 J/cm\textsuperscript{2} of energy density).
- **Group 6**: Enamel irradiated using Er,Cr:YSGG laser with 3 W-50 Hz power - pulse frequency parameters (60 mJ per pulse, 45 J/cm\textsuperscript{2} of energy density).
- **Group 7** (Control group): Enamel treated with coarse diamond bur using high-speed air turbine (W&H Synea TA98, Bürmoos, Austria).

Laser treatments

Er,Cr:YSGG laser device (Waterlase MD, Biolase Technology, San Clemente, CA, USA) was used for enamel specimens to be irradiated according to the following parameters: wavelength of 2.78 μm; pulse duration of 140 μs; spot size of 600 μm; air pressure setting of 65%; and water pressure setting of 65%; Irradiation time is 15 s; irradiation area is 1 cm\textsuperscript{2}; power of 3 - 6 W; pulse frequency of 20, 35, 50 Hz.

Area of 1 cm\textsuperscript{2} on the flattened surface was demarcated by means of a marker pen to determine an exact area to be irradiated. Then demarcated areas on teeth were irradiated at a 45° angle to the flattened surface in non-contact mode by hand. A bur with marker was adapted to the hand piece using a custom-made acrylic device to fix the working distance at 1 mm. All laser groups were treated in this manner. In the bur-treated control group, coarse diamond bur (Microdont ISO 806.314.001.524.012, Sao Paulo, Brazil) using a high-speed air turbine (W&H Synea TA98, Bürmoos, Austria) was used by hand with almost no pressure for 15 seconds across marked area.

Adhesive procedures

After following acid etching of all enamel surfaces in each group using 37% phosphoric acid gel for 20 s (All Etch, Bisco, Schaumburg, IL, USA), 2 consecutive coats of resin adhesive (Single Bond 2, 3M ESPE, St Paul, MN, USA) was applied to dried enamel surfaces with a cotton applicator and dried gently for 2-5 seconds. Light was activated for 20 seconds using a halogen lamp with an output intensity of 400 mW/cm\textsuperscript{2} (Ivoclar, Astralis 3 Ivoclar Vivadent AG, Schaan, Liechtenstein). After adhesive procedures, resin composite (Valux Plus, 3M ESPE, St Paul, MN, USA) build-ups in three layers up to a height of 4 mm, were done on the surfaces. Each increment layer was cured for 40 seconds using a halogen lamp (Ivoclar, Astralis 3).

Microtensile bond test

Summary diagram of the µTBS testing was shown in Figure 1. All bonded teeth were stored in distilled water at 37°C for 24 hours before µTBS test. Resin-enamel sticks with approximately 0.9 x 0.9 mm\textsuperscript{2} dimensions were ob-
tained using diamond saw under copious amounts of water (Micracut 125, Metkon, Bursa, Turkey) running at 300 rpm. The sectioning of each tooth into sticks took almost 45 minutes. Resin-enamel sticks from each tooth was pooled and immersed in glass vial filled with distilled water. Four of the obtained resin-enamel sticks were selected from the center of the tooth to test randomly, yielding 20 sticks for each group. Microtensile bond strength tests were performed after 24-hour and 12-month water immersion periods, respectively. The specimens were fixed to jig with cyanoacrylate glue (Pattex, Henkel, Duesseldorf, Germany) and forced in tension at a crosshead speed of 1 mm/min using a Bisco microtensile testing machine (Bisco Inc., Schaumburg, IL, USA). Exact dimensions of the interface area was measured with a digital calliper (Mitutoyo, Tokyo, Japan). The μTBS was derived by dividing the enforced force at the time of fracture by the bond area (mm²). Occurrence of failure prior to the actual testing was not included in the analysis, but a number of such pre-testing failures was noted. The mode of failure was determined by stereomicroscope under 40 x magnification (Meade Bresser Biolux, Meade Bresser, Rhede, Germany), and recorded as “adhesive” or “cohesive.” Neither enamel nor resin and “mix” failures included more than one of the enamel and resin parts.

**Statistical analysis**

Effects of the independent variables (water storage periods and surface treatments) on the dependent variable (μTBS) were analysed using two-way analysis of variance (ANOVA). Post-hoc comparisons were performed by using the Tukey test. Statistical analysis was performed with the Statistical Package for the Social Sciences (SPSS), version 13 software for Windows (SPSS Inc., Chicago, Ill, USA). All tests were done at the 0.05 level of significance.

*Figure 1: Specimen preparation for and operation of microtensile bond strength test.*
Results

The mean μTBS values, standard deviations and failure modes are shown in Table 1. Two-way ANOVA revealed that the surface treatment (p = 0.0001) significantly affected the μTBS, where water storage did not (p = 0.802). There was no significant interaction between the surface treatment and storage period (p=0.802).

The mean μTBS values ranged from 12.99 MPa (6 W/20 Hz) to 36.22 MPa (3 W/50 Hz) for 24 hours. Comparisons among the materials using one-way ANOVA and Tukey’s Post hoc test at baseline (24 hours), 3W/50 Hz showed the significantly highest μTBS value among all groups, whereas, 6 W/20 Hz exhibited the lowest μTBS value at 24 hours. No significant differences exist among bond strengths of other groups and those of the control group. After 12 months of water storage there was no statistically significant decrease in enamel μTBS when compared with the 24 hour samples observed in all groups.

The distribution of failure modes is presented in Table 1. In the all groups, with exceptions of 3 W/50 Hz and Bur-treated, the predominant mode of failure was mix at 24 hours. In contrast, in 3 W/50 Hz and Bur-treated the most frequent failure mode was of cohesive at 24 hours. The 12 month water storage did not alter failure modes of test groups.

SEM evaluation of resin-enamel interfaces showed that interfaces were intact in general and resin tag formations were confirmed. However, it was revealed that laser irradiation would result in subsurface damage within enamel substrate, depending on laser parameters (Figure 2). In parallel to bond strength data, the intact interfaces were achieved in the bur-treated group, and 3 W/ 50 Hz laser group. In other groups, extensive micro cracks induced by laser irradiation, preventing formation of regular resin tag formation and resulting in weakening substrate, hence obtaining strong bond between resin adhesive and enamel.

Discussion

The evaluation of bonding strength of adhesive resins to dental hard tissue is one of the main laboratory tests in order to rank and compare the performance of adhesive materials, because it offers an opportunity to obtain an understanding into the bonding effectiveness of adhesive resin agents to clinically related bonding locations and substrates [10]. Bond durability is another important feature that has a critical effect on the acceptable clinic effectiveness of adhesive resin restorative fillings, as it is highly linked to the degradation of resin bonding joints that could impair the mechanical properties of adhesive systems and components of hybrid layers [19].

The degradation of resin bonds with dental hard tissues and morphological alterations within bonding interfaces were reported after long term intra oral function by in vivo studies [20, 21]. In the present study, resin-enamel interfaces were aged by an in vitro method, long-term water storage that truly imitates what could happen under in vivo conditions. Storage in water is the most conventional in vitro aging technique to expect the effectiveness of adhesive resin materials [22], since the water is critical for the degradation of interface of resin materials with enamel and dentin [23].

At present, various wavelengths are offered for diverse dental aims [24]. Amongst them, the Er,Er:YSGG laser has been presented to offer numerous benefits above the traditional turbines and dental burs for preparation of enamel and dentin. Er,Er:YSGG laser also offers lessened noise and vibration, and reduced or no pain [2, 3, 25]. Furthermore, Er,Er:YSGG laser device has been particularly encouraged for the cavity preparation based on the principles of minimal invasive dentistry without producing thermal injury to the dental pulp [20, 29].

The proper choice of laser settings to deploy while Er,Er:YSGG laser irradiation should be considered. It is clear that important irradiation parameters i.e. frequency,

| Groups         | 24-hour μTBS   | Failure modes | 12-month μTBS    | Failure modes |
|----------------|----------------|---------------|------------------|---------------|
| 6 W/20 Hz      | 12.99 ± 9.2 a  | M > C > A     | 15.42 ± 3.8 a    | M > C > A     |
| 6 W/35 Hz      | 21.17 ± 5.4 b  | M > C = A     | 22.49 ± 6.9 b    | M > A > C     |
| 6 W/50 Hz      | 27.16 ± 8.6 c  | M > C = A     | 26.44 ± 6.9 b    | M > A > C     |
| 3 W/20 Hz      | 25.52 ± 9.2 b  | A > M > C     | 26.79 ± 7.0 b    | A > M > C     |
| 3 W/35 Hz      | 22.29 ± 8.0 b  | M > A > C     | 22.39 ± 6.6 b    | M > A > C     |
| 3 W/50 Hz      | 36.22 ± 5.8 d  | C > A > M     | 33.84 ± 8.1 d    | C > A > M     |
| Bur-treated    | 26.60 ± 8.5 b  | C > A > M     | 26.11 ± 5.7 b    | C > A > M     |

The same lower superscript indicates no significant differences p < 0.05.
A: adhesive failure; C: cohesive failure in enamel or composite; M: mix failure.
power, pulse duration, and water/air cooling ratios are significant to have significant effects in order to obtain optimum bonding strengths. However, there is a noteworthy discrepancy between these settings in Er,Cr:YSGG laser studies. This makes the assessment of results, challenging. Therefore, in the present study, Er,Cr:YSGG laser irradiation was deployed with different power and pulse frequency settings, including high (6.0 W) and low power (3.0 W) combinations with three pulse frequencies (20 Hz, 35 Hz and 50 Hz). 6.0 W of Er,Cr:YSGG laser irradiation as a cavity preparation setting. Previously, Cardoso et al. reported that 6.0 W - 20 Hz Er,Cr:YSGG laser parameters resulted in significantly lower enamel bond strength when compared with bur treating as also supported by findings of the present study 5). However, increasing pulse frequency from 20 Hz to 35 Hz provided similar enamel bond strength when compared with bur treating. Increasing pulse frequency with fixed output power lowers pulse energy. This might reduce potential thermo-mechanical damage of erbium laser irradiation to bonding substrate 11).

The parameters for laser enamel conditioning are generally of low power. Several researches that have deployed low power parameters have stated that enamel conditioning with the Er,Cr:YSGG laser is less amenable to resin adhesive bonding than with acid etching 6, 7). Therefore, it was claimed that traditional phosphoric acid etching after laser irradiation is required when the Er,Cr:YSGG laser is deployed in order to achieve similar findings with acid-etched enamel. In the present study, it was revealed that adjusting pulse frequency at the low power setting has a significant effect on enamel bonding. While bonding to enamel surfaces irradiated by the Er,Cr:YSGG laser with 3.0 W - 20 Hz and 3.0 W - 25 Hz parameters yielded similar bond strength when compared to bur treating, increasing pulse frequency further, to 50 Hz, resulted in significant improvement in the enamel bonding. Laser irradiation as a form of low pulse energy with high pulse frequency might increase surface roughness, and combination with conventional acid etching might induce a significant improvement in the initial enamel bond strength.

**Figure 2:** (a) SEM image of an acid-etched cross section of resin-enamel interface, using acid-etching. Interfaces were intact. Resin tag formations which provide microretention for resin adhesive were seen through to the interface. (b) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 6 W-20 Hz parameters. There was a gap at the interface. Extensive micro cracks (MC) occurred throughout subsurface enamel, possibly weakening the structure. Resin tags were not evident. (c) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 6 W-35 Hz parameters. Resin-enamel interface was intact. Resin tag formations (RT) were rarely seen at the interface where micro cracks (MC) were absent. (d) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 6 W-50 Hz parameters. The resin-enamel interface was intact. Although subsurface micro cracks (MC) were evident, formation of regular resin tags (RT) were more common to observe. (e) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 3 W-20 Hz parameters. Subsurface micro cracks were evident (MC). (f) SEM image of acid-etched cross section of resin-enamel interface, using laser irradiation with 3 W-35 Hz parameters. Although thinner micro cracks (MC) were evident, the interface was very similar to the interface of bur-treated group in the manner of resin tag formation and integrity of interface. (R: resin composite, A: adhesive layer, E: enamel, D: dentin, RT: resin tag, MC: micro crack induced by laser irradiation)
Durability of resin bonding to enamel, which is prepared by traditional cavity preparation methods is generally accepted as more durable than the durability of bonding to dentin. The collagenous structure of dentin itself promotes the biodegradation of resin-dentin interfaces due to activation of endogenous proteolytic enzymes upon acid etching. These proteolytic enzymes degrade exposed collagen fibrils that provide mechanical retention to resin composite, thus jeopardizing the stability of adhesive bonded restoration over time. Unlike dentin, non-collagenous structure of enamel is quite durable in aqueous medium, thus long-term water storage might not reduce mechanical properties of enamel. However, bonding to enamel with hydrophilic adhesives is amenable to degradation after long-term water storage as mechanical properties of hydrophilic adhesives is inverse to water storage.

Enamel bond durability of the adhesive system used in the present study (Single Bond 2) was found quite stable after a 12-month water storage, as reported previously by several studies. However, the bond durability of adhesive resin to Er:YSGG irradiated enamel after a long-term water storage period has been described in the present study. Data revealed that the 12-month water storage did not affect adhesion in the Er:Cr:YSGG laser-irradiated groups, negatively. Considering the influence of different laser parameters on enamel bond durability of an etch-and-rinse adhesive, it can be concluded that tested laser parameters did not cause any alterations on enamel, which might induce favorable or unfavorable changes in degradation of resin-enamel interfaces during a 12-month water storage.

Even when the limitations of the present study are taken into account, the findings of the present study provide viewpoints for further investigations that assess the durability of resin bonding to the Er,Cr:YSGG laser-irradiated dental hard tissues. It is probable that other resin adhesive systems and bonding strategies might lead to different results, and further studies should discover their relations with the Er,Cr:YSGG laser irradiated enamel and dentin in order to support the ideal and safety practicability of the Er,Cr:YSGG laser technology to dentistry.

Conclusions

It has been shown that bonding to the Er,Cr:YSGG laser irradiated enamel was quite reliable after a 12-month water storage in the present study. However, enamel bond strength significantly depends on selected laser parameters. Therefore, the selection of proper parameters i.e. power and pulse frequency is critical for bonding to laser-irradiated enamel.

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