

Evaluation of microtensile and tensile bond strength tests determining effects of erbium, chromium: yttrium-scandium-gallium-garnet laser pulse frequency on resin-enamel bonding

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Abstract

Objectives: The aim of the present study was to compare two different bond strength test methods (tensile and microtensile) in investing the influence of erbium, chromium: yttrium-scandium-gallium-garnet (Er, Cr: YSGG) laser pulse frequency on resin-enamel bonding.

Materials and Methods: One-hundred and twenty-five bovine incisors were used in the present study. Two test methods were used: Tensile bond strength (TBS; $n = 20$) and micro-TBS (μ TBS; $n = 5$). Those two groups were further split into three subgroups according to Er, Cr: YSGG laser frequency (20, 35, and 50 Hz). Following adhesive procedures, microhybrid composite was placed in a custom-made bonding jig for TBS testing and incrementally for μ TBS testing. TBS and μ TBS tests were carried out using a universal testing machine and a microtensile tester, respectively.

Results: Analysis of TBS results showed that means were not significantly different. For μ TBS, the Laser-50 Hz group showed the highest bond strength ($P < 0.05$), and increasing frequency significantly increased bond strength ($P < 0.05$). Comparing the two tests, the μ TBS results showed higher means and lower standard deviations.

Conclusion: It was demonstrated that increasing μ TBS pulse frequency significantly improved immediate bond strength while TBS showed no significant effect. It can, therefore, be concluded that test method may play a significant role in determining optimum laser parameters for resin bonding.

Key words: Bond strength, chromium: yttrium-scandium-gallium-garnet laser, erbium, micro-tensile, pulse frequency, tensile

Date of Acceptance: 03-Nov-2015

Introduction

Various laser technologies, including ruby (694 nm), CO₂ (9600; 10,600 nm), and neodymium-doped: Yttrium

aluminum garnet (1064 nm), have been used to remove dental caries tissue and prepare dental cavities.^[1] However, high-energy densities are required to vaporize dental hard tissues. As a result, serious thermal side effects such as melted areas and microcracks can occur within the adjacent dental tissues, with a rise in pulpal temperature due to the low tissue interactions of these lasers and

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Access this article online

Quick Response Code:



Website: www.njcponline.com

DOI: 10.4103/1119-3077.188702

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How to cite this article: Yildirim T, Ayar MK, Yesilyurt C, Kilic S. Evaluation of microtensile and tensile bond strength tests determining effects of erbium, chromium: yttrium-scandium-gallium-garnet laser pulse frequency on resin-enamel bonding. Niger J Clin Pract 2016;19:585-90.

the lack of adequate water cooling. However, thermal damage of adjacent hard tissues was reduced with the introduction of the erbium laser family, consisting of erbium-doped yttrium aluminum garnet (2940 nm) and erbium, chromium: Yttrium-scandium-gallium-garnet (Er, Cr: YSGG) (2780 nm) lasers.^[1-3] Consequently, erbium lasers are commonly used for dental hard tissue preparations that include cavity preparation, caries removal, and laser conditioning.^[1]

By adjusting hard tissue laser parameters such as laser irradiation distance,^[4] pulse duration,^[5] output power,^[6] pulse frequency,^[7] and water flow rate,^[8] the effects of laser irradiation on the interaction of resin adhesive systems with target tissues change. It is, therefore, of particular importance to exactly determine laser parameters in order to improve the bonding effectiveness of adhesive resin systems to laser-irradiated dental hard tissues, so reducing potential peripheral thermal-mechanical damage to a minimum.

The dental hard tissue ablation capacity of erbium lasers is directly associated with energy per pulse and pulse frequency, and these are the most important parameters to consider during cavity preparation.^[9] Although some studies have tried to standardize the basic parameters for ideal and safe ablation of dental hard tissues,^[10,11] available knowledge regarding the interaction of pulse frequency with bonding effectiveness of adhesive resins for dental hard tissues remains limited.

Ethical requirements stipulate laboratory bond strength testing to obtain valid *in vitro* data before initiation of clinical studies.^[12] Relevant test methods to obtain *in vitro* data for dental materials and other conditions predictive of clinical performance include bond strength tests, microleakage and nanoleakage assessments, and chemical analysis of resin-tooth interfaces and materials. Different bond strength tests such as shear (SBS), tensile (TBS), micro-SBS (μ SBS), and micro-TBS (μ TBS) may be used in determining properties of dental materials such as strength of bonding to dental hard tissues.^[13,14] However, some concerns exist about the lack of reproducibility among findings from different tests.^[12,15] Current evidence suggests that determination of treatment effects and product ranking seems to depend on which test is used.^[16] It was found that comparisons of dentin bonding systems based on the results of different bond strength tests may be misleading.^[16-18] Clearly, then, selection of an appropriate bond strength test is important.

In assessing the effects of laser irradiation on the bonding effectiveness of adhesive resin materials to dental hard tissue, it seems that differing bond strength tests have been used.^[19] However, any study on which bond strength test is more proper seems does not exist in the literature. In the absence of such data, the aim of the present study was to evaluate the effect of Er, Cr: YSGG laser pulse frequency on

bond strength of self-etch adhesive resin to enamel using two different bond tests: μ TBS and TBS.

Materials and Methods

One hundred and twenty-five bovine teeth were used in the present study. These were kept in dry conditions before sample preparation and then immersed in water for 2 weeks before bonding tests.^[20] Twenty-five teeth were used for μ TBS, and 100 teeth were employed for TBS testing. Roots of teeth were severed using a low-speed diamond saw under water cooling. Labial surfaces of teeth were ground using 320-grit abrasive paper under water cooling.

Laser treatments

For specimens of the laser test groups, an Er, Cr: YSGG laser device (Waterlase MD; Biolase Technology, San Clemente, CA, USA) was used with the following fixed parameters: 2.78 μ m wavelength, 140 μ s pulse duration, 3.0 W output power, 90% air pressure, 75% water pressure, 45 s irradiation time, and pulse frequency 20, 25, 50 Hz.

An area of 0.64 cm² on the flattened surface of the tooth was demarcated by means of a marker pen for precise irradiation. The demarcated area was then irradiated in noncontact mode by hand^[21] for 45 s. A bur with marker was adapted to the handpiece, using a custom-made acrylic device to fix the working distance at 1 mm. All laser groups within the present study were treated in this manner. In the bur-treated control group, a coarse diamond bur with high-speed air turbine was used by hand with almost no pressure for 15 s across the marked area.

Microtensile bond strength testing

For μ TBS testing, crowns were embedded into self-cure acrylic resin blocks. Surfaces were further ground using 600-grit abrasive paper for 30 s to obtain standardized smear layers under water cooling. Specimens were then divided randomly into five groups ($n = 5$) as follows: SiC-paper-abraded group (control); bur-treated group (control); Laser-20 Hz; Laser-35 Hz; and Laser-50 Hz. Specimen preparation for μ TBS testing is shown in Figure 1. In the SiC-paper-abraded group, surfaces were left untreated; in the laser and bur groups, surfaces were treated in the manner described above.

Following surface treatments, a two-step self-etch adhesive (Clearfil SE Bond, Kuraray, Osaka, Japan) was applied according to manufacturer instructions. Using a disposable brush tip, primer was applied to surfaces and left in place for 20 s. Volatile ingredients were then evaporated using a mild, oil-free air stream, and bond was applied to the entire surface using a disposable brush tip. After application, the bond was made as uniform as possible by use of a gentle, oil-free air stream. The bond was then light

cured for 10 s using an LED curing unit (3M Elipar S10, 3M ESPE, St. Paul, MN, USA). After adhesive application procedures, resin composite (Valux Plus, 3M ESPE) was built up in three layers to a height of 4 mm on the surfaces. Each incremental layer was cured for 20 s using an LED curing unit (3M Elipar S10, ESPE).

Bonded specimens were immersed in tap water at 37°C for 24 h before μ TBS testing. After water storage, specimens were sectioned into x and y directions using a low-speed cutting machine with diamond saw running (Micracut 125, Metkon, Bursa, Turkey) at 300 rpm under water cooling. Sectioning yielded resin-dentin sticks with dimensions of 0.9 mm \times 0.9 mm for μ TBS testing. Resin-dentin sticks from each tooth were pooled and kept separately from those of other teeth. Each resin-dentin stick was fixed to a jig with cyanoacrylate glue and forced in tension at a crosshead speed of 1 mm/min using a Bisco microtensile testing machine (Bisco, IL, USA). The μ TBS value was derived by dividing the enforced force at time of fracture by the bond area (mm²). Any occurrence of failure prior to actual testing was noted for each group, and premature failures were recorded as zero. The mode of failure was determined by stereomicroscope and recorded as “adhesive” or “cohesive” for either enamel or resin, and as “mix” failures in cases involving more than one of the dentin and resin parts. The μ TBS values of resin-dentin sticks exhibiting adhesive, mix, and premature failures were averaged for each tooth.^[22] Cohesive failures were excluded from the data sets.^[16]

Tensile bond strength testing

For TBS testing, crowns were embedded into individual plexiglass molds with self-cure acrylic resin and divided

randomly into five groups ($n = 20$) as for μ TBS testing [Figure 2]. Surfaces were further ground using 600-grit abrasive paper for 30 s to obtain standardized smear layers under water cooling. Surface treatments were applied in the same way as described for μ TBS testing. Following surface treatment, a plexiglass mold was fixed on the specimen, using adhesive bands and self-etch adhesive resin applied according to manufacturer instructions. The diameter of the hole in the mold was 3 mm. Resin composite (Valux Plus, 3M ESPE) was then placed in the mold in two-three layers to a height of 4 mm. Each incremental layer was cured for 20 s using an LED curing unit (3M Elipar S10, ESPE).

Bonded specimens were stored in tap water at 37°C for 24 h before TBS testing. Specimens were then placed in the apparatus with retention sides [Figure 2]. This configuration was loaded in tension, using a universal testing machine (Instron 3382A, Norwood, MA, USA) at a crosshead speed of 1 mm/min and a 10 kg load cell until fracture. The mode of failure was determined by stereomicroscope and recorded as “adhesive” or “cohesive” for either enamel or resin, and as “mix” failures for cases involving more than one of the dentin and resin parts.

Statistical analyses

Normal distribution and homogeneity of data were checked using the Kolmogorov–Smirnov and Levene tests, respectively. After ensuring that the data provided parametric test conditions, one-way analysis of variance and least squares difference tests were applied. All statistical analyses were performed using SPSS 13 (SPSS Inc., Chicago, Ill, USA).

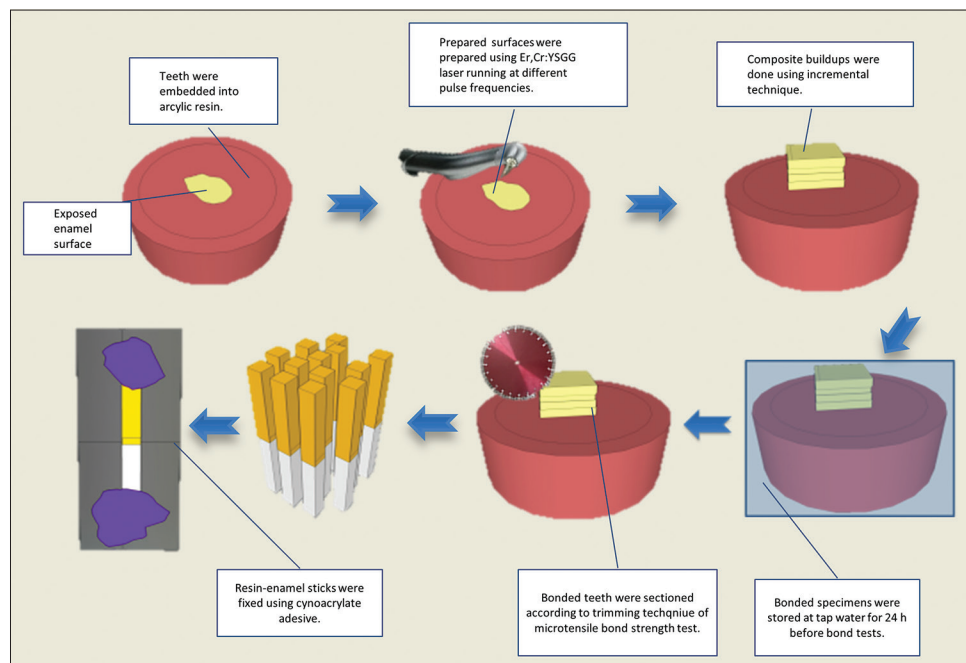


Figure 1: The illustrations show specimen preparation for and operation of microtensile bond strength test

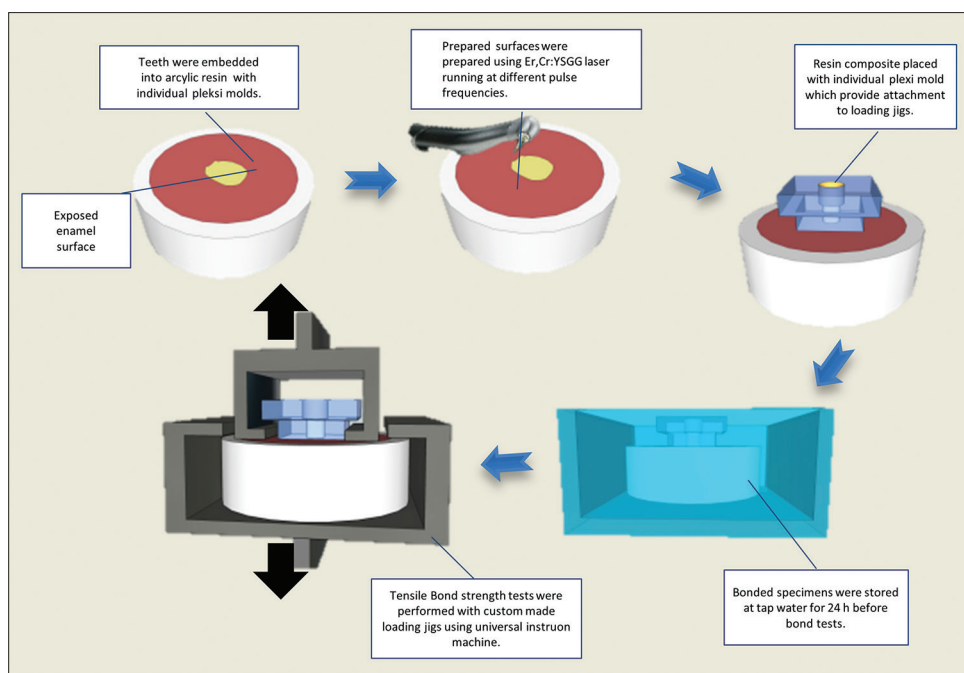


Figure 2: Schematics show specimen preparation for and operation of tensile bond strength test

Table 1: Microtensile bond strength and tensile bond strength means (μ TBS, TBS; MPa), standard deviations, failure patterns

Test groups	μ TBS	μ TBS-failure patterns	TBS	TBS-failure patterns
Control (SiC paper)	16.06 ± 3.57 ^a	A > M > C	2.38 ± 1.89 ^a	A > M > C
Control (Bur)	16.81 ± 2.84 ^a	A > M > C	2.84 ± 1.64 ^a	A > M = C
Laser-20-Hz	14.64 ± 2.11 ^a	A > M > C	2.99 ± 3.63 ^a	A > C > M
Laser-35-Hz	19.47 ± 3.71 ^b	A > M = C	2.82 ± 2.03 ^a	A > M > C
Laser-50-Hz	24.32 ± 3.19 ^c	A > C > M	2.74 ± 2.43 ^a	A > M = C

Different superscripts indicate statistically differences in the same column ($P < 0.05$). M=Mix failure; A=Adhesive failure; C=Cohesive failure in neither enamel nor composite

Results

The bond strength means of groups are shown in Table 1. The μ TBS tests revealed significant differences among test groups ($P < 0.05$). Among the μ TBS test groups, the lowest bond strength was obtained from the Laser-20 Hz group (14.64 ± 2.11 MPa, $P < 0.05$), while there are no significant differences among the Laser-20 Hz group and both control groups ($P > 0.05$). Laser-50 Hz delivered the highest bond strength, with significant differences from other groups (24.32 ± 3.19 MPa, $P < 0.05$). Increasing pulse frequency resulted in a significant increase in bond strength. Adhesive failure patterns were dominant within μ TBS test groups. For TBS test, no significant differences were found among groups. Adhesive failure patterns were again dominant within TBS test groups.

Discussion

Erbium laser irradiation on dental hard tissues has been widely studied in restorative dentistry since the first experimental research by Hibst and Keller.^[23] At present, erbium lasers are frequently used for dental hard tissue operations such as cavity preparation, caries removal, and laser conditioning.^[1]

The effects of erbium lasers on the effectiveness of bonding of resin adhesives to enamel and dentin remain controversial. One reason for these contested results may be the complicated interactions of resin adhesive systems with laser-irradiated dental substrate, which depend on multiple laser parameters including laser irradiation distance,^[4] pulse duration,^[5] output power,^[6] pulse frequency,^[7] and water flow rate,^[8] all of which affect the bond strength of adhesives to laser-irradiated dental hard tissues.

Pulse frequency and pulse energy are important parameters because the ablation ability of erbium lasers depends on them.^[8,10,11] However, the effects of these parameters on resin bonding to Er, Cr: YSGG laser-irradiated dentin and enamel have rarely been investigated. One previous study suggested that increasing pulse frequency might result in more favorable outcomes when etch-and-rinse adhesive was used on enamel.^[24] However, the effects of different pulse frequencies on bond strength of self-etch adhesive to enamel have not to date been studied. To rectify this deficit, the present study investigated the effects of pulse

frequency (20, 35, and 50 Hz) at output power of 3 W on the enamel bond strength of self-etch adhesive.

Different bond strength tests have resulted in wide variations among the findings reported in the literature, indicating that the kind of test used for assessing treatment effects can be a determining factor in bond strength testing. It has previously been claimed that ranking of bond strength values was more meaningful than any direct comparison of those values.^[25] More recently, however, a literature review showed that ranking of bond strength values also depends on the test used.^[16] For that reason, the effects of Er, Cr: YSGG laser pulse frequency on the bonding effectiveness of self-etch adhesive to enamel were assessed by means of two different bond strength tests: μ TBS and conventional TBS tests.

Although the bonding effectiveness of composite resins to Er, Cr: YSGG laser-irradiated enamel in conjunction with different adhesive resin systems has been assessed by numerous studies, evidence is scarce as to the influence of Er, Cr: YSGG laser pulse frequency on bond strength of resin to enamel bonding. The μ TBS tests revealed that increasing pulse frequency promotes significant improvement in adhesion, but conventional TBS testing showed no such effect in the present study. This means that the effects of Er, Cr: YSGG laser parameters on bond strength may depend on the test used, confirming the advantages of μ TBS testing over conventional TBS testing.

Several bond strength test methods, including shear, tensile, and microtensile testing, have been used to investigate adhesion forces between adhesive resin and laser-irradiated dental hard tissues. However, each of these test methods has both advantages and limitations,^[13,14] and results from the different methods do not always correlate.^[16,26] It follows that the selection and use of the most feasible test method appear to be an important parameter when conducting a laboratory evaluation.

The μ TBS testing uses specimens with smaller interface dimensions than those used in conventional TBS testing, which results in lower defect concentration within the bonded interface. This explains why μ TBS testing is advantageous, in that it tends to provide much higher bond strengths with lower standard deviations than conventional TBS testing,^[14,16] making μ TBS testing more sensitive to the effects of test parameters on the dependent variable.^[14] The present study supports this argument, as only μ TBS testing was able to reveal a significant improvement in bond strength as a result of pulse frequency variation.

Another advantage of the μ TBS test is that it permits testing of irregular surfaces, beyond the flat surfaces used in conventional tensile and SBS tests.^[14] In the present study, enamel surfaces were irradiated for time frame in

which surface were irradiated across whole demarked surface as possible as without resulting in any cavitation. It was observed that laser irradiation for 45 s was best fitted for this purpose. Inevitably, laser-irradiated surfaces show increased irregularity, which may require the use of μ TBS.

TBS test mean values, in the present study, were lower than some of those reported regarding the effects of laser irradiation on TBS in literature,^[27,28] while some others reported TBS means similar to those of the present study.^[29,30] The range of variables in test protocols (tooth substrate, bonding agent, bonding technique and methods employed) might be expected to yield wide variations in bond strength obtained by conventional tensile testing.^[15,25,26] According to Sudsangiam and van Noort, different methods (or even small modifications of the same method) can produce great differences in bond strength values for a given product,^[15] making direct comparison of results with those of previous studies difficult.

However, two main factors may play a role in the lower mean TBS values recorded in the present study: Constraining the adhesive application area to avoid occurrence of adhesive flash for better stress distribution at the resin-enamel interfaces^[31] and the use of self-etch adhesive for bonding to enamel.^[32] According to Van Noort *et al.*, delimitation of the bonding area before application of adhesive resin and placement of resin composite could give lower (by a factor of two to four) but more realistic bond strength means. They found that constraining the adhesive application area lowered bond strength mean from 6.90 MPa to 3.10 MPa.^[31] In addition, it is known that the bonding effectiveness of self-etch adhesives to enamel may be lower when compared to etch-and-rinse adhesives and can be increased by additional phosphoric acid.^[32,33]

Conclusion

Based on the findings of the present study, the following conclusions can be drawn.

- The effects of Er, Cr: YSGG laser irradiation on bond strength of self-etch adhesive to enamel depend on laser pulse frequency and the test being used
- μ TBS revealed the effect of pulse frequency on bond strength
- Increasing pulse frequency may improve the bonding effectiveness of self-etch adhesive to enamel
- Further clinical studies are warranted to assess the findings of the present *in vitro* study in terms of their importance for clinical performance of composite resin restoration placed on laser-irradiated enamel and dentin surfaces.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

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