

Shear bond strength of composite bonded to erbium:yttrium-aluminum-garnet laser-prepared dentin

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Abstract The purpose of this study was to evaluate the dentin bond strength to resin composite following erbium:yttrium-aluminum-garnet (Er:YAG) laser preparation using different adhesive systems. Seventy dentin specimens prepared from human molar teeth were randomly assigned to seven groups of ten. The first five groups were prepared with an Er:YAG laser 2940 nm at the manufacturer's recommended settings and (1) acid etched, and etch-and-rinse adhesive Excite was applied; (2) Excite was applied; (3) two-step self-etching adhesive AdheSE was applied; (4) laser etched (120 mJ/10 Hz), and Excite was applied; (5) laser etched, and AdheSE was applied. The last two groups were added as controls (prepared with a diamond bur): (6) acid etched, and Excite was applied; (7) AdheSE was applied. Nanohybrid composite cylinders 4 mm×2 mm were bonded to the dentin surfaces. After the specimens had been stored in distilled water and had undergone thermocycling, the shear bond strength was tested and the data were analyzed statistically. The Duncan multiple comparison test showed that specimens prepared with a diamond bur and with acid and Excite applied showed the highest mean bond strength (13.01 ± 2.09 MPa), followed by those prepared with Er:YAG and with AdheSE applied (11.5 ± 3.59 MPa) and those prepared with a diamond bur and with AdheSE applied (10.75 ± 1.95 MPa), but there were no significant

differences among them ($P > 0.05$). Er:YAG-prepared specimens, with acid, Excite (3.28 ± 0.95 MPa) and specimens that were laser etched and with AdheSE applied (3.37 ± 0.63 MPa) showed the lowest mean values for bond strength ($P < 0.05$). The results suggested that dentin surfaces prepared with Er:YAG laser may provide comparable composite resin bond strengths depending on the adhesives used.

Keywords Shear bond strength · Er:YAG laser · Dentin · Adhesive · Nanohybrid composite

Introduction

The treatment of dental tissues prior to adhesive restorative procedures is an extremely important step in the bonding protocol and accounts for the clinical success of restorations [1, 2]. Although all adhesive materials and procedures were originally developed to act on tooth substrate prepared by conventional techniques, new investigations search for alternative techniques that could produce better effects than acids produce. Among the innovations for substrate treatment, the role of the erbium:yttrium-aluminum-garnet (Er:YAG) laser has been highlighted [3].

The Er:YAG laser is one of the most recommended types of lasers to be used on dental hard tissues, because its wavelength (2.94 μm) coincides with the main absorption band of water (3.0 μm), and it is also well absorbed in hydroxyapatite [4]. The Er:YAG laser acts on dental substrate by thermo-mechanical ablation, vaporizing its water content, which causes expansion, followed by micro-explosions that produce the ejection of both organic and inorganic tissue particles, providing a surface with open dentinal tubules and no smear layer. The Er:YAG laser can

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effectively remove dental hard tissue, due to its high absorbability in both water and hydroxyapatite [4–6]. Upon laser irradiation, water within the mineral substrate is vaporized, giving volume expansion and disruption. Owing to this thermo-mechanical process, dental hard tissue is ablated, with minimal thermal damage to the surrounding tissues. Therefore, the Er:YAG laser is considered to be the device of choice for the preparation of dental hard tissue and alteration of the tooth surface [4, 6, 7].

The strength of the bond to Er:YAG-lased tooth substrate reported in the literature is often confusing and even contradictory [8–12]. Some studies report higher strengths of bonds to laser-conditioned dentin than to acid-etched dentin [13]. Others report significantly lower bond strengths, whereas, also, no significant differences were found [10, 14]. Nevertheless, the literature available on the Er:YAG laser presents varying parameters and results. Our study aimed to determine dentin shear bond strength to a nanohybrid resin composite following Er:YAG laser preparation with an etch-and-rinse and two-step self-etch adhesive system.

Materials and methods

Table 1 shows the materials used in this study. Seventy extracted, caries-free, unrestored, human molars were used. Any remaining soft tissues were thoroughly hand-scaled and cleaned from the tooth surfaces and disinfected in 0.5% chloramine solution and placed in distilled water for up to 1 month at -20°C . The teeth were rinsed in running dis-

tilled water for 30 min and then embedded in auto-polymerizing acrylic resin (Simplex Rapid, Kemdent, Associated Dental Products Ltd, Wiltshire, UK), with the buccal surfaces positioned for surface treatment and composite bonding. After polymerization of the embedding resin, the buccal surfaces were abraded and then sequentially polished in a polishing machine (Mecapol P230, Presi Tavernoles 38, Briect Angonnes, France) using 400 grit and 600 grit silicon carbide paper until a uniform layer of peripheral dentin was observed. After the dentin surfaces had been controlled for the absence of enamel and/or pulp tissue with a stereo-microscope (Nikon SE, Tokyo, Japan), they were randomly divided into seven groups. The first five groups were prepared with an Er:YAG laser (Fidelis, Fotona Medical Lasers, Ljubljana, Slovenia), and last two groups were prepared with a high-speed diamond bur and served as control groups:

Group 1 The dentin surfaces were irradiated with an Er:YAG laser with $2.94\ \mu\text{m}$ wavelength and a contact tip with a repetition rate of 20 Hz/200 mJ and pulse duration of 100 μs , under water spray. The laser beam spot size was 0.6 mm and was moved in a sweeping fashion by hand over an area 4 mm in diameter. The specimens were then chemically etched with 37% phosphoric acid (Vivadent, Ivoclar, Schaan, Liechtenstein) for 15 s and rinsed with distilled water for 15 s. To prevent desiccation of the treated surfaces, the excess water was carefully removed with a damp cotton pellet instead of being dried with com-

Table 1 Materials used in the study

Brand name	Manufacturer	Material type	Composition	Application procedures	Batch number
Excite	Ivoclar, Vivadent, Schaan, Liechtenstein	Etch-and-rinse adhesive system	HEMA (hydroxymethyl methacrylate), dimethacrylates, phosphonic acid, acrylate, silicon dioxide, ethanol photo-initiators	Apply 1 coat, gently agitate for 10 s, thoroughly air dry, light cure for 20 s.	J01968
AdheSE	Ivoclar, Vivadent Schaan, Liechtenstein	Self-etching adhesive system	Primer: phosphonic acid, acrylate, bis-acrylamide, water, initiators, stabilizers	Apply primer and when thoroughly coated, brush for 15 s. Disperse excess amounts with a strong stream of air	H36141
			Adhesive: dimethacrylate, hydroxyethyl methacrylate, highly dispersed silicon dioxide, initiators, stabilizers	Apply bond, disperse with a very weak stream of air, light cure for 10 s	H34680
TetricEvo Ceram	Ivoclar, Vivadent Schaan, Liechtenstein	Nanohybrid composite resin	Dimethacrylates, barium glass filler, ytterbium tri-fluoride, mixed oxides, prepolymers, additives, stabilizers, catalysts, pigments	Light cure for 40 s	H24275

pressed air. Excite, an etch-and-rinse dentin bonding agent, was used as adhesive, according to the manufacturer's instructions.

- Group 2 Dentin specimens were treated with Er:YAG laser as in group 1, and Excite was applied in the same manner but without acid etching
- Group 3 Dentin specimens were irradiated with Er:YAG laser, and AdheSE, a two-step self-etching adhesive, was used as bonding agent, according to the manufacturer's instructions.
- Group 4 Dentin specimens were irradiated with Er:YAG laser and conditioned by Er:YAG laser with a contact tip, a repetition rate of 10 Hz/120 mJ and pulse duration 100 μ s. Excite was applied as above.
- Group 5 Dentin specimens were irradiated with Er:YAG laser, laser etched as in group 4, and AdheSE was applied as before.
- Group 6 Dentin specimens in this group were prepared with high-speed diamond bur (Standard 837 R, Diatech, Switzerland) at approximately 200,000 r.p.m. with air/water coolant. The specimens were acid etched, and Excite was applied as in group 1.
- Group 7 Dentin specimens were prepared with high-speed diamond bur as in group 6, and AdheSE was applied as mentioned above.

Following the respective pretreatment sequences, a Teflon tube with an inner diameter of 4 mm and a height of 2 mm was attached to the prepared dentin surfaces. A nanohybrid composite resin (TetricEvo Ceram, Vivadent) was applied and polymerized for 40 s (Elipar Free Light, 3M Espe, St Paul, MN, USA). After curing had been completed, the Teflon tube surrounding the composite was carefully removed. The specimens were stored in distilled water at 37°C for 24 h and thermocycled for 500 cycles between 5°C and 55°C, with a dwell time of 30 s each. The specimens were then tested in shear mode with a knife-edge testing apparatus in a universal testing machine (LR50K, Lloyd Instruments Ltd., Fareham, Hants, UK) at a crosshead speed of 1 mm/min. Shear bond strength was calculated as the ratio of fracture load and bonding area expressed in megapascals (MPa). Data were subjected to analysis of variance (ANOVA) and Duncan multiple comparison tests.

Results

Table 2 and Fig. 1 show the means and standard deviations of shear bond strength for the tested treatment groups. Averages and standard deviations were calculated, and the data were submitted to ANOVA. As the results showed differences among the measurements, they were subjected to the Duncan multiple comparison test. Dentin specimens

Table 2 Shear bond strengths (in megapascals) of resin composite bonded to dentin. There are no significant differences in groups with the same superscript letter

Groups	Number	Mean	Standard deviation	Minimum	Maximum
1 ^a	10	3.28	0.95	2.10	5.30
2 ^b	10	7.31	2.13	4.30	10.10
3 ^c	10	11.5	3.59	7.40	18.30
4 ^b	10	5.75	0.95	4.60	7.40
5 ⁿ	10	3.37	0.63	2.50	4.30
6 ^c	10	13.01	2.09	10.35	17.43
7 ^c	10	10.75	1.95	7.64	13.93

that had been prepared with a diamond bur, were acid etched and had had Excite applied, showed the highest mean bond strength (13.01 \pm 2.09 MPa), followed by Er:YAG-prepared specimens (using the power setting of 20 Hz/200 mJ) and with AdheSE applied (11.5 \pm 3.59 MPa), and diamond bur-prepared specimens with AdheSE applied (10.75 \pm 1.95 MPa), but there were no differences among them ($P>0.05$). The specimens prepared with Er:YAG (20 Hz/200 mJ), acid etched and with Excite applied (3.28 \pm 0.95 MPa), and the specimens prepared with Er:YAG (20 Hz/200 mJ), laser etched (10 Hz/120 mJ) and with AdheSE applied (3.37 \pm 0.63 MPa), showed the lowest mean bond strengths ($P<0.05$).

Discussion

Our study compared the in vitro shear bond strength of a nanohybrid composite resin to human dentin that was prepared with an Er:YAG laser and treated with two different adhesive systems (a conventional etch-and-rinse method that required prior conditioning with phosphoric acid, and self-etching). For comparison with the laser system, dentin surfaces prepared with a regular grid diamond bur commonly used clinically to prepare cavities for adhesives were also added to the study. The extracted teeth were stored in distilled water at -20°C, the preferred method for testing the bond strength of resin composites to dentin, as suggested by Titley and others [15]. Dentin adhesives tend to function well in bond strength tests when tested shortly after application [16, 17]. Clinically, the oral environment represents a challenge to durability of bond strength because of temperature changes, masticatory load cycling, water sorption and pH fluctuations. Although thermal cycling represents only one of these challenges, the specimens were subjected to thermal cycling and shear bond strength tests within 2 days.

It is known that the hydrophobic nature of restorative composite renders bonding to hard dental tissues difficult.

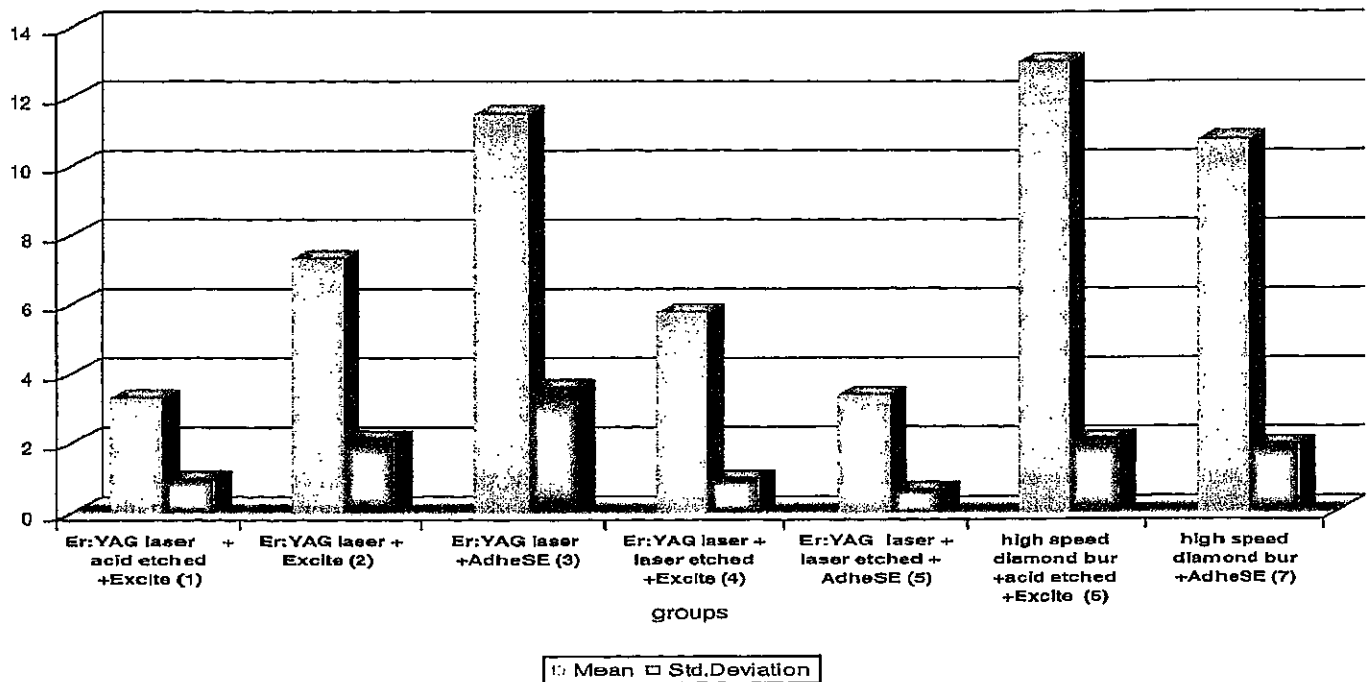


Fig. 1 Shear bond strengths (in megapascals) of resin composite bonded to dentin

Therefore, for bonding of these resins to be achieved, it is necessary for one to alter the topography of the tooth surface and use hydrophilic resins [1, 2, 9]. In this study, we modified the surface morphology by cutting the tooth surface with an Er:YAG laser or a bur and conditioning the surface with phosphoric acid or with self-etching primer and with laser in order to alter the chemistry of the dentin surfaces, so that they were able to bond with the composite.

Preparation of dentin with rotary instruments leaves a smear layer on the surface. The smear layer consists mainly of pulverized enamel and dentin and caries debris and bacteria, which are created by the bur. The low surface energy of this layer hinders impregnation of the enamel and dentin with the adhesive agent and, thus, prevents adequate adhesion [18]. The standard approach to this problem has been acid etching, first proposed by Buonocore [18] and later applied to dentin by Fusayama [19].

On the other hand, it was postulated that the laser-dentin surface possessed an advantage because of an apparent enlarged surface area for adhesion, based on the scaly and flaky surface appearance following laser irradiation [20]. This is caused by the micro-explosions during the laser procedure, due to its thermo-mechanical ablation [21]. The erbium laser initially vaporizes water and other hydrated organic components of the tissue. On vaporization, internal pressure increases in the tissue until explosive destruction of inorganic substance occurs. Since intertubular dentin contains more water and has a lower mineral content than peritubular dentin, it is selectively ablated more than the

peritubular dentin, leaving protruding dentinal tubules with a cuff-like appearance [21, 22]. This may also contribute to an increase in the adhesive area. Patent tubules and the absence of smear layer are additional factors that may enhance bonding to laser-treated dentin. Adhesion to laser-treated dentin could be explained by the mechanical retention provided by resin tag formation and the infiltration of adhesive resin into the micro-irregularities in laser-demineralized dentin.

In this study, although there were no significant differences among them, the highest bond strength was achieved by diamond bur preparation, acid-etching and application of Excite, followed by Er:YAG preparation and application of AdheSE and by diamond bur preparation and application of AdheSE. Excite is a two-step, etch-and-rinse system, where the primer and adhesive resin are combined into one solution. Generally, an etch-and-rinse procedure involves the use of phosphoric acid. The main objectives of acid conditioning are to remove the smear layer (and smear plugs) and render enamel and dentin surfaces more receptive to bonding. With the introduction of self-etching adhesives, the use of separate acid etching step was eliminated. AdheSE is a two-step, self-etching, adhesive system that uses an etching-priming solution followed by a separate adhesive. Self-etching priming-adhesive systems dissolve the smear layer and partially demineralize the underlying dentin surface. The dissolved smear layer is incorporated into the bonding process. We used the adhesive systems with the manufacturer's composite (TetricEvo

Ceram) to avoid incompatibilities with the bonding agent. Special attention was given to correct application procedures, in particular to the application of both adhesives to tooth substrates that were prepared in both ways.

Many researchers have suggested the use of different types of lasers as an alternative to dentin conditioning [7, 23–25], and several reports have compared bond strengths of erbium lasers. In general, there is variability among the dentin bond strength values reported by various studies [26, 27]. This may be attributed to different testing methods and conditions, the varying nature of dentin as a substrate, the composite–adhesive used, and also to laser energy parameters. Er:YAG laser irradiation promotes structural and morphological changes in dental hard tissues that depend on fluence, repetition rate, beam spot size and water presence [3, 7].

With regard to the settings of the Er:YAG laser, the technique utilized in this study was first to irradiate the dentin surfaces at the setting of 200 mJ/20 Hz. As the preparation was completed, the surface for bonding was then prepared with a setting of 120 mJ/10 Hz for better efficiency, the minimal number of induced changes, and favorable surface characteristics.

There are serious doubts about which energy density is the most appropriate to obtain a suitable micro-retentive pattern for adhesion procedures. Some clinicians have preferred to prepare the bonding surface by utilizing lower energy settings. de Sousa et al. [28] used the setting 80 mJ/2 Hz, whereas Eguro et al. [29] used 100 mJ/4 Hz, and De Munck et al. [30] used 80 mJ/10 Hz.

Visuri et al. [13] compared the bonding of composite resin to dentin following the preparation of the dentinal surface with either an Er:YAG laser with 350 mJ/6 Hz or a standard dental bur and with or without a subsequent acid-etching treatment (10% phosphoric acid applied for 30 s) and reported a significantly higher shear bond strength of composite to dentin prepared with an Er:YAG laser. In contrast, Dunn et al. [12] (using energy of 140 mJ/30 Hz) and Ceballos et al. [11] (180 mJ/2 Hz) reported a decrease in bond strength to laser-irradiated dentin.

Bertrand et al. [8] showed that the values of shear bond strength of bur-prepared dentin surfaces with acid applied; with Er:YAG laser applied (500 mJ/10 Hz) or Er:YAG laser and acid applied, did not differ significantly, whereas Trajtenberg et al. [9] (160 mJ/10 Hz) reported highest bond strengths when the tooth surfaces were acid-etched after preparation with either the laser or bur prior to the application of the bonding agent.

Data from our study demonstrated that higher bond strengths were achieved when the dentin surfaces were prepared with a diamond bur and were acid etched prior to the application of Excite, and prepared with an Er:YAG laser prior to the application of AdheSE, and prepared with

a diamond bur prior to the application of AdheSE. Bond strengths were significantly weaker when dentin surfaces were prepared with an Er:YAG laser and acid-etched prior to application of Excite and prepared with Er:YAG laser and Er:YAG laser-etched prior to application of AdheSE. Although Er:YAG laser has been flagged up as a promising technology, there is still a need for more research to determine the best adhesive protocol and appropriate parameters of Er:YAG laser for its application in restorative dentistry. With regard to the bond strength to dentin with Er:YAG laser and the constant development of adhesive materials, studies are always necessary to consolidate new concepts.

Conclusion

Within the limitation of this in vitro study, it may be concluded that:

1. The highest bond strengths were obtained when dentin specimens were prepared with a diamond bur, then etched with 37% phosphoric acid, and Excite (an etch and rinse adhesive) was used.
2. Application of acid and Excite after Er:YAG laser preparation using power settings 20 Hz/200 mJ with 100 μ s pulse duration decreased the bond strengths.
3. Application of AdheSE (two-step, self-etching adhesive) after Er:YAG laser preparation showed higher bond strengths, but Er:YAG laser etching (10 Hz/120 mJ) after Er:YAG laser preparation showed lower bond strengths.
4. Improvements in laser technology, and the increased interest in their potential for hard tissue application, warrant further investigations of Er:YAG laser-prepared teeth and adhesion with resin-based composites.

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