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ORIGINAL ARTICLE

Influence of Er:YAG and Ti:sapphire laser irradiation on the microtensile bond strength of several adhesives to dentin

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Abstract The aim of the present study was to evaluate the influence of erbium:yttrium–aluminum–garnet (Er:YAG) and Ti:sapphire laser irradiation on the microtensile bond strength (MTBS) of three different adhesive systems to dentin. Flat dentin surfaces from 27 molars were divided into three groups according to laser irradiation: control, Er:YAG (2,940 nm, 100 μs, 2.7 W, 9 Hz) and Ti:sapphire laser (795 nm, 120 fs, 1 W, 1 kHz). Each group was divided into three subgroups according to the adhesive system used: two-step total-etching adhesive (Adper Scotchbond 1 XT, from now on XT), two-step self-etching adhesive (Clearfil SE Bond, from now on CSE), and all-in-one self-etching adhesive (Optibond All-in-One, from now on OAO). After 24 h of water storage, beams of section at 1 mm^2 were longitudinally cut from the samples. Each beam underwent traction test in an Instron machine. Fifteen polished dentin specimens were used for the surface morphology analysis by scanning electron microscopy (SEM). Failure modes of representative debonded microbars were SEM-assessed. Data were analyzed by ANOVA, chi-square test, and multiple linear regression ($p<0.05$). In the control group, XT obtained higher MTBS than that of laser groups that performed equally. CSE showed higher MTBS without laser than that with laser groups, where Er:YAG attained higher MTBS than ultrashort laser. When OAO was used, MTBS values were equal in the three treatments. CSE obtained the highest MTBS regardless of the surface treatment applied. The Er:YAG and ultrashort laser irradiation reduce the bonding effectiveness when a two-step total-etching adhesive or a two-step self-etching adhesive are used and do not affect their effectiveness when an all-in-one self-etching adhesive is applied.

Keywords Femtosecond · Laser · Dentin · Bond strength · Adhesion

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Introduction

Nowadays, new techniques have been developed in an attempt to avoid thermal damage, improve adhesion, and simplify clinical bonding procedures. Two main technological research fields drive this evolution: (1) new adhesive systems and (2) the use of different lasers.

Adhesive systems have evolved in the course of several generations, and manufacturers have also invested in simplified adhesives, aiming for fast and easy use and to improve bond strength [1]. Thus, the traditional application of the conditioner, primer, and adhesive in three stages has been replaced by the following three different product categories: (1) adhesive systems in which the primer and the adhesive are applied simultaneously after acid etching; (2) the self-etching primers, in which the acid-etching stage and the primer take place at the same time, followed by the application of the adhesive; and (3) the self-etching adhesives that are characterized by the acid etching, primer, and adhesives stages occurring at the same time [2, 3]. With new self-etching adhesive systems, the dentinal smear layer is no longer completely eliminated but treated like a substrate [2, 3].

Erbium lasers were introduced into dentistry specifically as an alternative to traditional mechanical instrumentation for tooth structure preparation [4–8]. The erbium:yttrium–aluminum–garnet (Er:YAG) laser emits energy at a wavelength of 2,940 nm [4]. This wavelength coincides with the major absorption peak of water, an important component of dental hard tissues, and is very close to the absorption peak of hydroxyapatite [4]. Despite its efficiency, reported bond strengths of composite resin to tooth substrate prepared by erbium laser are often confusing and contradictory. Some studies have reported higher bond strengths to laser-prepared dentin [8–10]. Others have reported significantly lower bond strengths [6, 11–17], and others have reported no significant differences [18]. These negative effects are related to the presence of subsurface microcracks and the chemical alterations that occurred in the dentin composition, especially its organic matrix [19].

The titanium:sapphire laser was introduced by Moulton in 1986 [20]. The usefulness of ultrashort pulsed lasers is the result of the combination of pulse duration and intensity. Namely, they allow access to nonlinear physical events that happen on subpicosecond time scale [21]. Recently, several experiments on ultrashort laser ablation of dentin have been described [22–31]. All of them indicated that the high beam quality and short pulse duration of the ultrafast laser used should allow the accurate preparation of cavities, with negligible damage of the underlying material.

There is only one study available in the literature that describes the influence of ultrashort laser treatment on the shear bond strength of composite resin to dentin [26]. In that work, submicrometer-sized patterns on the surface of human dentin were produced to enhance adhesion. However, the researchers did not process the whole surface of the dentin specimen, restricted the study to a two-step self-etching adhesive, and did not compare the results with Er:YAG laser-processed surfaces.

Accordingly, the aim of the present study was to evaluate the influence of Er:YAG and Ti:sapphire laser irradiation on the microtensile bond strength (MTBS) of three different adhesive systems to dentin.

Materials and methods

A total of 27 caries-free human third molars, freshly extracted within a 6-month period and stored in distilled water at 4 °C, were selected and cleaned with an ultrasonic system (Suprasson P5 Booster, Acteon Satelec, Merignac, France). After this, a polishing paste (Detartrine, Septodont, Saint Maur, France) was used to remove the adherent tissues from the tooth surface.

Dentin specimen preparation

The specimens were sectioned transversely at a distance of 4 mm from the occlusal surface using a precision cutting machine (IsoMet 5000; Buehler, Lake Bluff, Illinois, USA) and grinding diamond disks (IsoMet Wafering Blade Series 15LC; Buehler, Lake Bluff, Illinois, USA) with abundant water coolant in order to remove the enamel and expose a large surface of dentin. Then, the exposed dentin surfaces of each sample were grounded perpendicular to the long axis of the tooth with sandpaper granulated at 600 grit in a polishing machine (Phoenix Beta; Buehler, Lake Bluff, Illinois, USA) under running water to provide the formation of a standardized smear layer.

Dentin surfaces were controlled for the absence of enamel and pulp tissue using an Axio M1 (Carl Zeiss, Jena, Germany) light microscope. We used Epiplan ×20 and ×50 HD objectives (Carl Zeiss Vision, Aalen, Germany), attached to a 1,300×1,030-pixel digital camera (AxioCam HR, Carl Zeiss Vision, Aalen, Germany).

The roots were removed using a diamond bur, and the pulp tissue was removed from its coronal parts. This cavity was filled with Filtek Z 250 composite (3M ESPE, St. Paul, MN, USA) and adhesive technique (Clearfil SE Bond, Kuraray Medical Inc., Tokyo, Japan).

Experimental groups

Once the samples were prepared, they were randomly divided into three experimental groups according to the dentin treatment: control, Er:YAG laser, and Ti:sapphire laser. Each group was divided into three subgroups according to the adhesive system to be used: a two-step total-etching adhesive (Adper Scotchbond 1 XT, 3M ESPE, St. Paul, MN, USA) (XT); a two-step self-etching adhesive (Clearfil SE Bond, Kuraray Medical Inc., Tokyo, Japan) (CSE); and an all-in-one self-etching adhesive (Optibond All-in-One, Kerr, Orange, CA, USA) (OAO), as shown in Table 1.

Laser irradiation

Before the application of the adhesive, polished dentin surfaces were irradiated with a different laser system according to the group they belonged to.

Erbium laser

The Er:YAG laser used in this study was a Fidelis Plus III (Fotona, Slovenia), which emits at λ=2940 nm. The irradiation was performed under the following conditions: 300 mJ/pulse; pulse duration: 100 μs, 9 Hz; a focal distance of 15 mm; and a beam spot size of 0.9 mm with a noncontact hand piece (R14), using a water spray until the surface was completely irradiated.

Table 1 Manufacturers, compositions and mode of application of tested adhesives

Materials	Manufacturers	Composition	Mode/steps of application
Adper Scotchbond 1 XT-XT	3M ESPE, St. Paul, MN, USA	bisGMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, stem spherical silica particles	Each enamel for 30 s and dentine for10 s, rinse with air and water spray for 10 s, and blot excess water using a cotton pellet
		Methacrylate, copolymer of polyacrylic and polyitaconic acids	Immediately after blotting, apply 2–3 consecutive coats of adhesive to etched enamel and dentine for 15 s, with gentle agitation using a fully saturated applicator. Gently air thin for 5 s to evaporate solvent. Light cure for 10 s.
Clearfil SE, Bond CSE	Kuraray Medical Osaka Japan	1. PRIMER (self-etching primer) MDP, HEMA, hydrophilic dimethacrylate DL-camphorquinone *N-N*-diethanol-*p*-toluidine water	Primer 1. Dispense the necessary amount of PRIMER into a well of the mixing dish immediately before application. 2. Apply PRIMER to the entire cavity wall with sponge or a disposable brush tip. Leave it in place for 20 s. Use caution not to allow saliva or exudate to contact the treated surfaces for at least 20 s. 3. After conditioning the tooth surface for 20 s evaporate the volatile ingredients with a mild oil-free stream.
		2. BOND (bonding agent) MDP, bisGMA HEMA hydrophobic dimethacrylate DL-camphorquinone *N-N* diethanol-*p*-toluidine silanated colloidal silica	Bond 1. Dispense the necessary amount of BOND into a well of the mixing dish. 2. Apply BOND to the entire surface of the cavity with a sponge or disposable brush tip. 3. After application, make the bond film as uniform as possible using gentle oil-free air stream. 4. Light cure the BOND for 10 s with a visible light-curing activator.
Optibond All-in-one OAO	Kerr Orange, CA, USA	GPDM, HEMA, GDMA, bisGMA water acetone, ethanol CQ silica filler sodium, hexafluorosilicate	(1) Shake, (2) dispense, (3) close immediately, (4) dip brush, (5) apply first application with scrubbing motion (20 s), (6) dip brush, (7) apply second application with scrubbing motion, (8) air dry gently then air dry with medium force for at least 5 s, and (9) light cure for 10 s
Composite Z 250	3M ESPE, St. Paul, MN, USA	Inorganic filler (zirconium silica) loading is 60 % by volume with a particle size range of 0.01 to 3.5 microns. bisGMA, UDMA, and bisEMA, encore-GMA, UDMA, encore-EMU, zirconium/silicon 60 % (0.01 to 3.5 μm)	

MDP 10-methacryloxydecyl dihydrogen phosphate; *HEMA* 2-hydroxyethyl methacrylate; *PI* photoinitiator; *bisGMA* 2,2-bis(4-(2-hydroxy-3-methacryloxypropoxy)phenyl)propane; *bisEMA(6)* bisphenol A polyetheylene glycol diether dimethacrylate; *UDMA* urethane dimethacrylatel; *TEGDMA* tri[ethylene glycol] dimethacrylatel; *A174* gamma-methacryloxypropyltrimethoxysilane; *BHT* 2,6-di-(*tert*-butyl)-4-methylphenol; *BSA* benzene sulfinic acid sodium salt; *CQ* 1,7,7-trimethylbicyclo-[2, 2, 1]-hepta-2,3-dione; *EtOH* ethanol; *GDM* glycerol dimethacrylate; *GPDM* glycerol phosphate dimethacrylate; *HFGA-GDM* hexafluoroglutaric anhydride-glyceroldimethacrylate adduct; *EHQ* 4-methoxyphenol; *Na2 Si6 F* disodium hexafluorosilicate; *ODMAB* 2-(ethylhexyl)-4-(dimetylamino)benzoate; *OX-50* fumed silicon dioxide; *PAMA* phthalic acid monomethacrylate; *SP345* barium aluminoborosilicate; *TS530* fumed silicon dioxide

Ti:sapphire laser

The laser system consists of a Ti:sapphire oscillator (Tsunami; Spectra Physics, USA), which provides pulses in the near infrared (λ=795 nm) and a duration of approximately 120 fs and energies of the order of 10 nJ. These energies are too low to produce massive ablation of the materials, so, in order to provide them with sufficient energy, they will be amplified with a system called regenerative (Spectra Physics, Spitfire) based on the chirped pulse amplification technique. Finally, the pulses were 120 fs long with a repetition rate of 1 kHz and the maximum pulse energy was 1 mJ. The pulse energy was

finely controlled by a half-wave plate and a linear polarizer. Neutral density filters were used when further energy reduction was required. The average power of the beam was measured with a thermopile detector (407A, Spectra Physics, USA). The transversal mode is nearly a Gaussian TEM00 with a 9-mm beam diameter (at $1/e^2$). The laser pulses were focused by means of an achromatic doublet lens (f=100 mm). The spot size with this focusing system has a diameter of approximately 12 μm.

The specimens were fixed on a computer-controlled XYZ motorized stage (Micos ES100, Nanotec, Germany). The laser pulses impinged vertically on the dentin surfaces. Horizontal movements XY allowed to scan the area to be microstructured. The laser beam was defocused by elevating the samples to 1 mm, in order to obtain a more uniform pattern across the surface, thus minimizing the depth of the grooves generated by laser ablation. The pulse energy was 0.045 mJ, the scanning velocity was 0.5 mm/s, and the scanning step was 0.03 mm. We processed the specimens in a saturated vapor atmosphere to preserve the tissues from drying.

Adhesive system and sample restoration

The adhesives of each subgroup were bonded to the dentin surfaces according to the manufacturer's instructions. The mode of application, components, and manufacturers of these adhesives are shown in Table 1.

After photopolymerization of the adhesive, a resin-based composite crown was constructed with 1.5-mm layers of Filtek Z 250 composite (3M ESPE, St Paul, MN, USA) to reach a height of approximately 4–5 mm. Each layer was photocured for 10 s with a LED light-curing unit (Bluephase G2; Ivoclar Vivadent, Liechtenstein). Light intensity output was monitored with a curing radiometer (Bluephase Meter, Ivoclar Vivadent, Liechtenstein) to be at least 1,000 mW/cm^2.

Microtensile bond strength test

After a 24-h water storage at 37 °C, to permit adequate water absorption and equilibration, the specimens were then serially sectioned in a longitudinal manner into 1-mm-thick slabs using a low-speed diamond saw under water cooling (Isomet 5000; Buehler, USA). Each slab was sectioned into beams with a cross-sectional area of approximately 1 mm^2 using a low-speed diamond saw, following the method described by Shono et al. [32]. Approximately 50 beams resulted from each subgroup, and these were submitted to MTBS evaluation, using a universal testing machine (Instron 3345; Instron Corp., USA), running at a crosshead speed of 0.5 mm/min until fracture.

Failure mode analysis

Fracture specimens were examined with an optical microscope Axio M1 (Carl Zeiss, Germany) at ×40 magnification to determine the mode of failure. Failure modes were classified as adhesive (no signs of dentin fracture or remnants of resin on the tooth, failure in adhesion), cohesive (complete fracture of dentin or resin, failure of the tooth substrate, or failure of the resin composite), or mixed (samples showing both adhesive and cohesive failures).

Scanning electron microscope analysis

Fifteen dentin specimens, cut and polished as they were for MTBS test, were treated according to the three different procedures (control, Er:YAG laser, and Ti:sapphire laser) for surface morphology analysis with a variable pressure scanning electron microscope (Zeiss EVO MA25; Carl Zeiss, Germany). Specific regions across the surface were explored to obtain a paramount view of the effect of laser processing.

In addition, representative fractured specimens from each subgroup were dehydrated for 48 h in a desiccator (Sample Dry Keeper Simulate Corp., Japan) and then mounted on aluminum stubs with carbon cement. They were sputter coated with pure gold by means of a sputter-coating Unit E500 (Polaron Equipment Ltd., Watford, England) and then observed with the same scanning electron microscope in order to examine the morphology of the debonded interfaces.

Statistical analysis

The bond strength values were measured in megapascal. The data were analyzed with SPSS v16 (Statistical Package for the Social sciences, Chicago, IL), using a p value below 0.05 as threshold for the statistical significance. ANOVA test was applied for comparing the MTBS values between dentin treatment and type of adhesive subgroups, respectively. When ANOVA test detected significant differences, Bonferroni post hoc comparisons were performed to quantify the differences between two subgroups. Then, a multiple linear regression analysis was performed using MTBS as the dependent variable, and the laser treatment and adhesive treatment were introduced as predictors being transformed each one into two dummy variables. For the adhesive predictor, the two dichotomous variables created for either CSE or OAO used the XT subgroup as reference. For the laser treatment, both the Er:YAG and the Ti:sapphire laser used the control subgroup as reference. The chi-square test was used to detect differences in the type of failures among the adhesive subgroups.

Results

Microtensile bond strength test

Means and standard deviations of MTBS are summarized per experimental subgroup in Table 2. The results of the Bonferroni post hoc comparisons showed that when laser was not applied as well as when the surface was irradiated with one of the two lasers used in this study (Er:YAG and Ti:sapphire), CSE showed higher MTBS values than XT and OAO, which showed no statistically significant difference. When XT was used, the MTBS values were statistically higher in the control group than in the laser groups (Er:YAG and Ti:sapphire), which were not statistically different. When CSE was applied, the MTBS values were statistically higher in the control group than in the laser groups (Er:YAG and Ti:sapphire), where the Er:YAG obtained values statistically higher than the Ti:sapphire laser. Finally, when OAO was used, the ANOVA revealed no statistical difference on MTBS values within the experimental groups.

The linear regression model (R^2=0.38; F=66.19; p< 0.001) revealed a significant decrease of 7.6 and 4.6 MPa in MTBS when Ti:sapphire laser and Er:YAG laser were used, respectively, instead of the control group (p<0.001, data not shown). When CSE was applied, the MTBS mean values increased significantly—11.1 MPa— as compared to XT. However, there was no statistically significant difference between the application of XT and OAO.

Failure mode analysis

The main failure mode in specimens showing low bond strengths was mixed failure, while cohesive failures were observed with higher bond strengths (F=13.205; p<0.001, data not shown). Adhesive failure was predominantly observed in the control and Ti:sapphire laser groups. Conversely, mixed failures took place predominantly in the Er:YAG group. The cohesive failures were predominantly observed in the CSE group. Among the control group, XT produced significantly higher rates of mixed failures. Among the Er:YAG group, OAO produced significantly higher rates of mixed failures than its counterparts (Table 3).

Table 2 Descriptive statistics of microtensile bond strength in megapascals of the specimens tested

	Control M (SD)	Er:YAG laser M (SD)	Ti:sapphire laser M (SD)
Adper Scotchbond 1XT	28.2 (8.0) Ba	23.9 (8.0) Bb	22.4 (8.2) Bb
Clearfil SE Bond	43.3 (10.9) Aa	34.9 (10.4) Ab	29.2 (6.7) Ac
Optibond All-in-One	24.1 (7.4) Ba	23.9 (5.9) Ba	21.0 (7.5) Ba

ANOVA test: F=37.542; p<0.001. Mean values followed by the same lowercase letter in rows and the same uppercase letter in columns were not significantly different

M mean, *SD* standard deviation

Scanning electron microscopy

Analysis of surface treatment

The scanning electron microscope (SEM) micrographs in Fig. 1a, b show the morphology of the dentin surface after ultrashort pulsed laser irradiation. The processed surfaces present an irregular and rough appearance (Fig. 1a). The smear layer was entirely removed by the laser treatment, and some dentinal tubules are exposed and open (labeled in white) (Fig. 1b). No sign of melted material or microcracks are observed in the SEM images.

Failure mode analysis

Representative SEM micrographs of fractured specimens interface after MTBS tests are presented in Fig. 2a–f.

Figure 2a shows a representative micrograph of a fractured dentin–adhesive interface from the dentin side when CSE was bonded to raw and laser-treated dentin— mixed failure. At higher magnification, a microcrack can be clearly observed (Fig. 2b).

Figure 2c shows a representative micrograph of a fractured dentin–adhesive interface at the dentin side when CSE was bonded to Er:YAG laser-irradiated dentin—mixed failure with some resin composite on the surface. At higher magnification, a rough surface with some partially obliterated dentinal tubules can be observed (labeled in white) (Fig. 2d).

Figure 2e shows a representative micrograph of a fractured dentin–adhesive interface at the dentin side when CSE was bonded to Ti:sapphire laser-irradiated dentin—adhesive failure. At higher magnification, a rougher surface than that in the Er:YAG group with some partially obliterated dentinal tubules can be observed (labeled in white) (Fig. 2f).

Discussion

In order to study the bonding effectiveness of composite resin to laser-irradiated dentin (Er:YAG and Ti:sapphire laser), we selected three commercially available adhesives that represent the most common adhesives used in dentistry: (1) a two-step total-etching (XT), (2) a two-step self-etching (CSE), and (3) an all-in-one self-etching adhesive (OAO). The overall conclusion of the study is that not only the adhesive system but also the laser irradiation influences the bond strength of composite resin to dentin.

Table 3 Cross-tabulation of the effect of laser irradiation within the laser group specimens among the adhesive groups on the type of failure (adhesive, cohesive, and mixed)

	Control, *n* (%)			Er:YAG laser, *n* (%)			Ti:sapphire laser, *n* (%)		
Failure mode	XT	CSE	OAO	XT	CSE	OAO	XT	CSE	OAO
Adhesive	23 (34.8)	33 (66.0)	30 (76.9)	11 (26.2)	10 (17.2)	1 (2.9)	26 (55.3)	28 (63.6)	32 (50.0)
Cohesive	5 (7.6)	13 (26.0)	0 (0.0)	1 (2.4)	4 (6.9)	1 (2.9)	1 (2.1)	1 (2.3)	0 (0.0)
Mixed	38 (57.6)	4 (8.0)	9 (23.1)	30 (71.4)	44 (75.9)	32 (94.1)	20 (42.6)	15 (34.1)	32 (50.0)
	Chi-square=46.439; $p<0.001$			Chi-square=8.937; $p=0.06$			Chi-square=3.818; $p=0.43$		

The linear regression model revealed a significant decrease by 7.6 and 4.6 MPa in MTBS when Ti:sapphire laser and Er:YAG laser were used. When a two-step total-etching (XT) and a two-step self-etching (CSE) were applied, the Ti:sapphire group obtained lower MTBS values than the control group; however, no statistical differences were

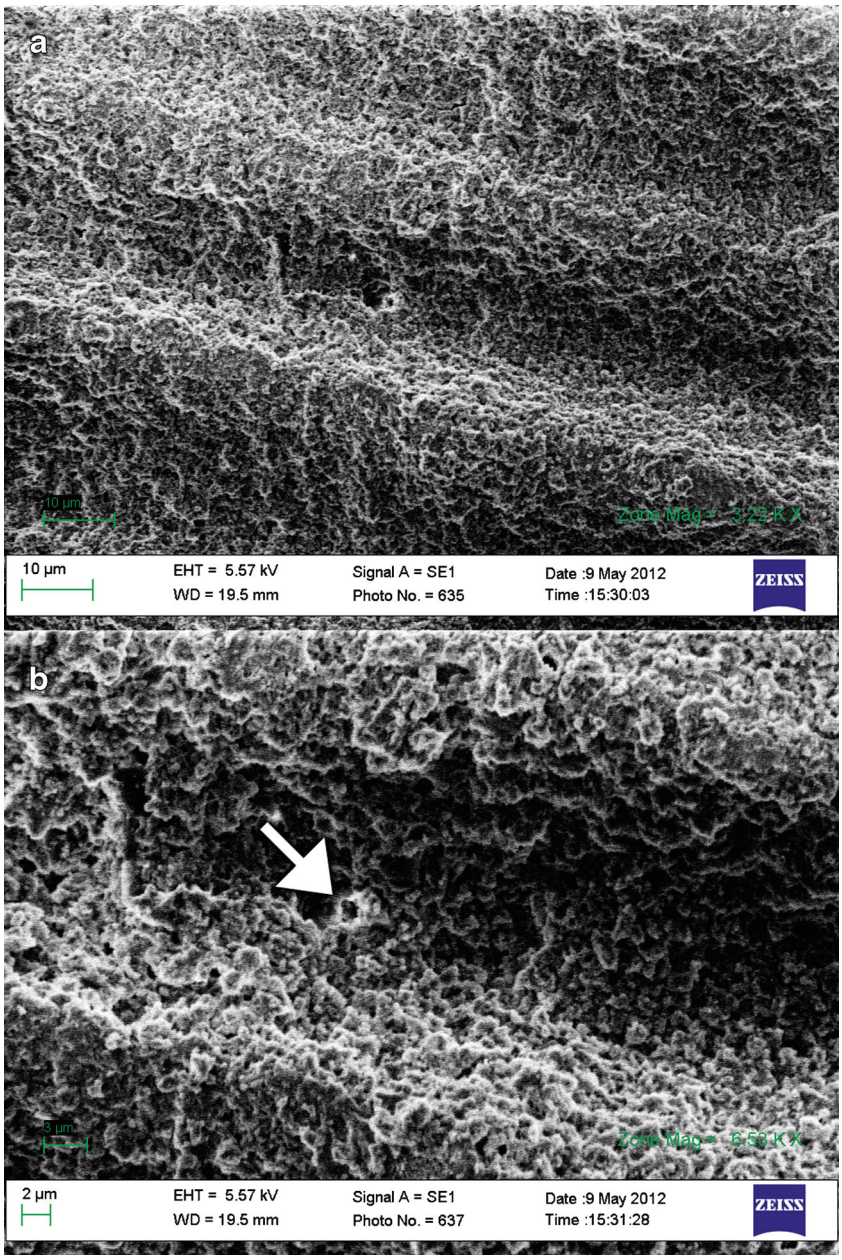

Fig. 1 SEM micrographs of dentin surface after Ti:sapphire laser irradiation at 5.57 kV. Original magnification: **a** ×3,200 and **b** ×6,530

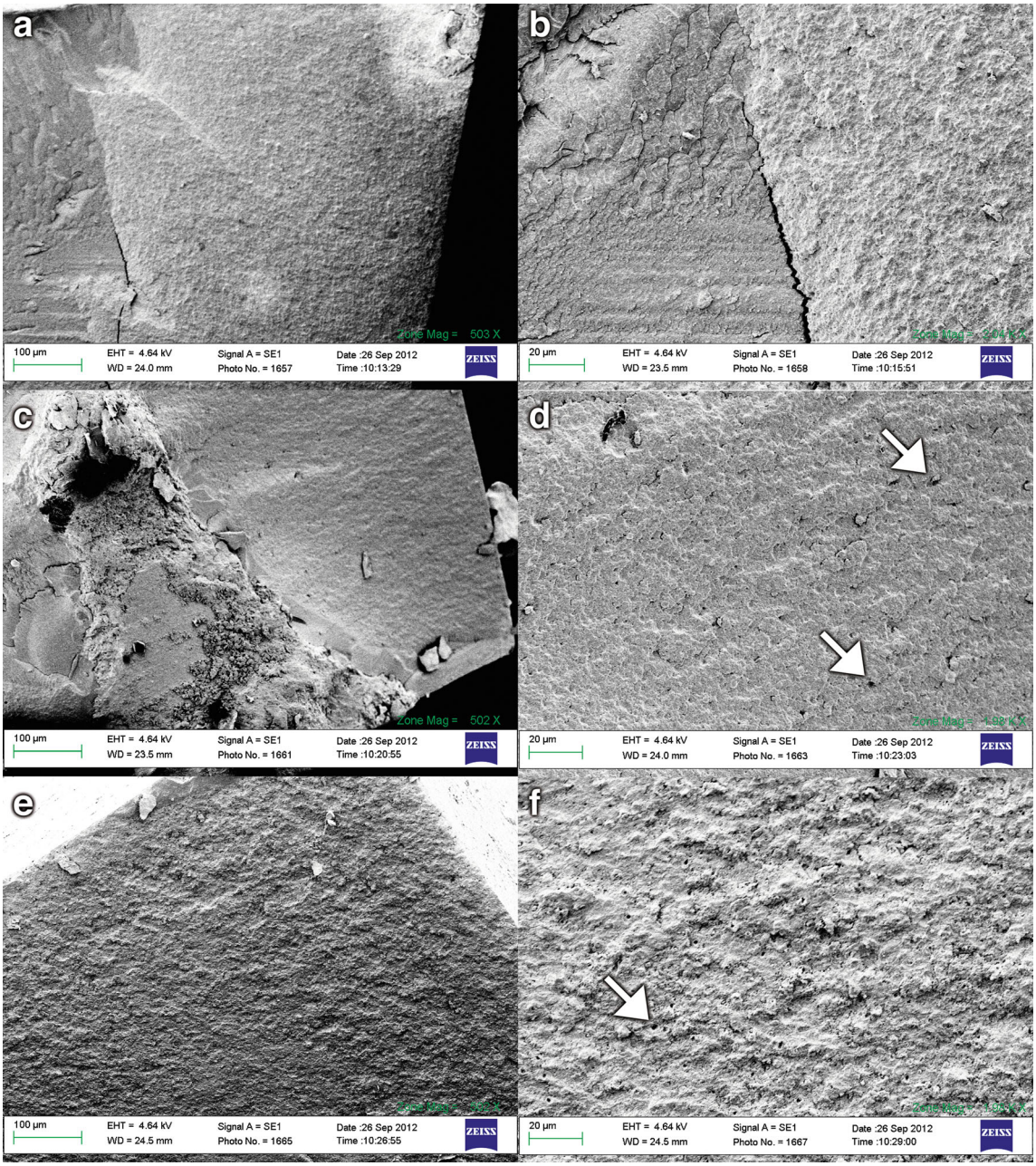

Fig. 2 SEM micrographs of the debonded dentin specimen from Clearfil SE at 4.64 kV: control (**a**, **b**), Er:YAG laser (**c**, **d**), and Ti:sapphire laser (**e**, **f**) groups. Original magnification: ×500 (*left column*); ×2,000 (*right column*)

observed when an all-in-one self-etching adhesive (OAO) was used. As far as we know, this is the first time that the influence of ultrashort laser irradiation on the microtensile bond strength of three dentin adhesives used in routine clinical practice has been studied. Gerhardt-Szep et al. reported the effect of submicrometer-sized patterns generated with ultrashort laser treatment to improve bonding effectiveness of dentin–resin adhesion interface with CSE. They introduced an artificial microstructure on the surface consisting of grooves separated 80 or 160 µm from each other with areas of intact dentin in between and concluded that dentin treatment with ultrashort laser in a 160-µm-sized cross pattern did not significantly affect shear bond strength (SBS) as compared with the control group, whereas the same cross pattern with half the pitch resulted in significantly lower SBS [26].

Laser ablation of the dentin by ultrashort lasers was previously studied by several authors [22–25, 27–29, 31]. These previous studies showed that the irradiated dentin presents an irregular and rough appearance with no signs of melting, deformation, cracking, or carbonization. Moreover, the chemical dentin composition is not significantly modified by the ultrashort laser treatment [31], but they did

not study the possible denaturation of the collagen fibrils that could take place. Alves et al. observed that the surface resulting from treatment with ultrashort lasers should be favorable to standard bonding procedures because it presents a microretentive irregular topography, free of smear layer, and with open dentinal tubules [31], as can be observed in SEM micrographs of the dentin surface after Ti:sapphire laser irradiation (Fig. 1); however, these observations are not in accordance with the results obtained in our study, which showed that these morphological characteristics do not improve the MTBS independent of the type of dentin adhesive used. The main factors that could explain these results are as follows: (1) Despite the fact that the surface roughness of laser-irradiated dentin was significantly higher than that for acid-etched dentin, these irregularities were so prominent that they may decrease the bond strength by preventing uniform stress distribution at the adhesive–dentin interface. Moreover, these irregularities produce a non-uniform hybrid layer that results in a decrease of the bonding effectiveness of adhesives to the dentin [33], as was suggested by Tay et al. who observed the absence of correlation between hybrid layer thickness and bonding efficacy as long as a uniform demineralization front is created within the underlying dentin that is fully impregnated by resin [34]. (2) Despite that the chemical dentin composition was not being significantly modified by the ultrashort laser treatment [31], the ablation of dentin could melt collagen fibrils, resulting in a lack of the interfibrillar space necessary for the diffusion of the adhesive. This lack of resin penetration could explain the lower bond strengths [6, 35], while, at the same time, this decalcified noninfiltrated zone at the base of the hybrid layer is susceptible to degradation as time goes by [2].

In the present study, the Er:YAG processed specimens performed worse than the control group, regardless of the adhesive system used with the exception of OAO, and no statistical difference was observed. Contradictory results in MTBS test after Er:YAG laser treatment may be found in the literature because of the wide range of different experimental setups. Li et al. explained that Er:YAG laser ablates hard tissues, inducing microexplosions within inorganic structures in teeth. Initially, Er:YAG pulses vaporize water and other hydrated organic components until internal pressure causes the explosion of the inorganic component [36]. Tachibana et al. observed that irradiated dentin presented opened dentinal tubules with protruded peritubular dentin distributed on a scaly surface free of smear layer. These characteristics may contribute to an increase in the effective adhesion surface [16]; however, the morphology of the dentin surface is not the only factor relevant to bonding. According to Ceballos et al. the irradiated dentin showed a superficial layer that consists of a scaly surface where the collagen fibrils are completely melted and vaporized [6]. This superficial layer varied in appearance and thickness, depending on the pulse energy. The basal part contains the rest of the denatured collagen fibrils that were fused and weakly attached to the underlying dentin with reduced interfibrillary spaces [6]. The presence of this layer prevents deep infiltration of the adhesive, resulting in lower bond strength values [6]. Despite the controversy, researchers have reached some degree of agreement on the fact that Er:YAG irradiation of dentin negatively affects the bonding effectiveness of resin-based adhesives [6, 11]. The results of the present study agree with these studies in which the bond strength of different adhesive systems applied to Er:YAG-irradiated dentin was tested until failure, and the interfacial morphology was observed under SEM. De Munck et al. concluded that the total and self-etching adhesives bonded significantly less effectively to Er:YAG-processed dentin than to bur-cut dentin. The subsurface damage initiated by Er:YAG ablation is probably the main reason for the decrease in MTBS and might compromise clinical bonding in the long term [13]. Brulat et al. also showed lower adhesion values for the Er:YAG-irradiated dentin with different self-etching adhesive systems [17].

The two-step self-etching adhesive (CSE) exhibited the highest bond strengths compared with the two other adhesives, both in the control and laser groups (Er:YAG and Ti:sapphire). These results are in agreement with results of other authors [17, 37–39]. CSE is an ultra-mild self-etching adhesive (pH=2.7). Its low acidity produces a considerably reduced ability to dissolve the smear layer and demineralizes the underlying dentin [40], and it can only superficially expose collagen on dentin, creating a characteristic nanometric hybrid layer. However, an effective and stable chemical bonding to the mineral content of the partially demineralized dentin resulting in higher MTBS is made possible by the presence of 10-MDP in its composition [3, 40]. By the way, it plays an important role in resisting long-term hydrolytic degradation [9, 13, 16, 17].

When comparing the two self-etching adhesives, the OAO showed lower adhesion values than the two-step adhesive (CSE); however, the total-etching adhesive (XT) showed no statistical differences with OAO. These results are in agreement with those of previous studies reporting lower values obtained with the all-in-one adhesives as compared to the two-step self-etching adhesives [17, 37, 40]. The all-in-one adhesives are more hydrophilic than the two-step self-etching adhesives. This hydrophilicity could result in stronger water absorption by the adhesive resin, which could act as a semipermeable membrane that allows water transport and therefore could affect its mechanical properties. By the way, this could contribute to the hydrolysis of resin polymers and the subsequent degradation of the tooth–resin bond [17, 37]. In addition, the all-in-one adhesives are less viscous, which makes them difficult to fix properly

[40]. These properties may lead to a wide variety of seemingly unrelated problems that may jeopardize the effectiveness and stability of adhesion to the dentin [40].

The dentin–composite–adhesive interface consists of a complex structure with many potential fracture locations [41]. The final pattern of fracture at this interface is determined by local stress distribution during the test, crack propagation, material structure properties, and dynamics of the fracture itself. Based on these facts, fracture sites highlight differences in the methodology of the experiments or the sample shapes submitted to microtensile test [38]. In our study, the predominant failure mode in specimens showing low bond strengths was mixed failure, whereas cohesive failures were mainly observed associated to higher bond strengths.

Ultrashort laser conditioning of the dentin is influenced by different parameters: wavelength, pulse duration, pulse energy, repetition rate, scanning velocity, and step. In our study, the Ti:sapphire laser pulses were applied in the following conditions: 795 nm, 120 fs, 0.045 mJ, 1 kHz, 0.5 mm/s, and 0.03 mm; and the results obtained with different adhesive systems did not improve the MTBS values. Future investigations could focus on the parameters that could enhance the bonding effectiveness of dentin–resin adhesion interface and replace the dentin conditioners used nowadays.

Conclusions

In conclusion, the use of Er:YAG and Ti:sapphire laser for conditioning the dentin did not improve the dentin–resin adhesion interface because laser irradiation reduced the bonding effectiveness when a two-step total-etching adhesive or a two-step self-etching adhesive were used and did not affect this effectiveness when an all-in-one adhesive was applied.

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