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ORIGINAL ARTICLE

Evaluation of bond strength of resin cement to Er:YAG laser-etched enamel and dentin after cementation of ceramic discs

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Abstract

The purpose of this study was to investigate the shear bond strength (SBS) of ceramic discs luted to differently etched enamel and dentin surfaces. Occlusal surfaces of 64 carious-free human molars and vestibule surfaces of 64 first maxillary incisors were ground to get flat superficial dentin and flattened enamel respectively. After generating 4 groups according to the surface etching method (37% orthophosphoric acid, Er:YAG laser–contact handpiece/scanning handpiece (1 or 2 times of scanning)), ceramic discs were luted to the surfaces with adhesive resin cement (Variolink N, Vivadent Ets., Schaan/Liechtenstein). After etching and cementation, thermocycling of 5000 cycles (Sd Mechatronık Gmbh, Feldkirchen-Westerham, Germany) and SBS test (Servopulser EHFFD1; Shimadzu, Kyoto, Japan) were performed respectively. The surface morphologies of 2 specimens, etched enamel and dentin, prepared for each group were examined with SEM analysis. Failure modes were determined under a USB digital microscope. Data were analyzed with one-way analysis of variance (ANOVA) and Tukey HSD test ($\alpha = 0.05$). SBS values in dentin surfaces showed statistically significant differences ($p < 0.05$) among tested groups. The highest SBS among dentin groups was determined in the group which had 2 times etching by Er:YAG laser (11.42 MPa) by a scanning handpiece. No statistical differences were observed in the other dentin or enamel groups. Laser etching seems to be a viable alternative to acid etching on both enamel and dentin surfaces while double etching of dentin with a scanning handpiece can improve the adhesion.

Keywords Er:YAG laser · Shear bond strength · IPS e-max Press ceramic · Surface treatment · Enamel · Dentin

Introduction

The search for optimal techniques and materials in dental restorations for recovering esthetic and functional properties, with the least discomfort for the patient, is constant in dentistry. Indirect porcelain restorations used with adhesive resin systems seem to be capable of obtaining acceptable results in restorative dental treatment. Furthermore, the combination of high biocompatibility and good optical and mechanical properties [1] in all ceramic systems appears to meet both patient expectations and clinical requirements, offering unequaled esthetics and resemblance to tooth structure [2].

One of the main aspects of success in dental restorative treatment is adhesion which represents the process of physical and chemical attachment of two different substances of different physical properties. Based on this, different surface alterations are usually needed in order to achieve better adhesion; therefore, there is always a search for the best surface treatment technique in dental practice [3]. The concept of phosphoric acid etching was born with the study of Buonocore in 1955 [4]. His idea of increasing adhesion area by creating irregularities on dental tissues served as a mainstay on the improvement of bonding chemistry and led to generation of many bonding systems, such as etch-and-rinse and self-etch systems that are available today [5]. Since the acid etching technique remains as one of the most commonly used surface conditioning methods in dental practice [6], it has been suggested as a control method in this study.

In addition to advancements in acid-bonded systems, alternative methods to increase bond strength of resin materials as crystal growth [7], micro-etching with pressurized pumice [8],

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and maleic acid application [9] were suggested. However, integration of laser technologies into dental practice attracted the huge attention of authors.

A long time has passed since the first application of laser in dentistry practice [10]. This technology that promised valuable perspectives and great improvement in dental treatment became a center of interest, especially its effect on dental hard and soft tissue lesions. Dentin desensitization [11, 12], inhibition of caries by increasing the resistance of enamel to demineralization [13], ability of evaporation, and crater formation on dental hard tissues by ablation [14, 15] became some of the main options of laser technology.

In recent years, lasers with different wavelengths and parameters have developed and already become an integral part of dental practice. And this rapid development of lasers may continue to have a major impact on the scope and practice of dentistry [16, 17]. Er:YAG laser is one of the most preferred laser types in dental practice and it has an affinity for the wavelength to be highly absorbed by the hydroxyl groups in hydroxyapatite, collagen, and especially water [18–20]. During Er:YAG laser irradiation, a vaporization after a sudden heating that occurs in water content of enamel and dentin leads to the production of micro-explosions: consequently, the elimination of both organic and inorganic components from dental hard tissues and cavitation of the irradiated area [18–20].

Surfaces irradiated by this laser showed a characteristic rough surface, clean and without debris, open dentinal tubules, and micro-irregularities caused by the preferential removal of the intertubular dentin, suggesting that the resultant dentin surface is receptive to adhesive procedures [21]. Furthermore, cavitation caused by laser etching on enamel and dentin presented an anfractuous surface (fractured and uneven), which is in agreement with theoretical suggestion of Vissuri et al., stating that a rougher surface may contribute to better adhesion [22]. However, there are studies reporting that the SBS values of superficial parts of surfaces irradiated using laser handpieces have decreased when compared with surfaces prepared by conventional instruments [23, 24].

In many publications, researching Er:YAG laser can be found in academic dental journals. The considerable number of study reports is focused on comparison of conventional surface etching of dental tissues with surface etching using manual handpieces of erbium lasers [25–28]. Today, we have an opportunity to compare these techniques with a novel Er:YAG scanning handpiece etching method that offers the benefits of homogenous surface ablation, better focal spot overlapping, and ability to adjust the shape and size of adhesion surface. It should also be stressed that there is no study in literature comparing the effect of automatic laser etching method on adhesion of ceramics on both enamel and dentin.

Therefore, the purpose of this study was to compare the shear bond strength (SBS) of all ceramic material (IPS e-max Press, Ivoclar, Schaan/Liechtenstein) specimens bonded to enamel and dentin after three different surface etching techniques (Er:YAG laser manual handpiece (H14), Er:YAG scanning handpiece (X-Runner), and 37% phosphoric acid etching (control)). The enamel and dentin morphologic structures were also investigated with scanning electron microscopy (SEM) after different etching procedures. The hypothesis tested was that the SBS obtained after etching of enamel and dentin surfaces using the Er:YAG scanning handpiece is similar to that obtained after acid etching.

Materials and methods

The total number of caries-free human teeth, stored in distilled water at 10 °C immediately after extraction, used in this study was 128 (64 molars and 64 first maxillary incisors). The distilled water was changed weekly. Roots were severed by a low-speed diamond saw under water-cooling. The occlusal surfaces of molars and vestibular surfaces of incisors were embedded into self-cure acrylic resin in 1.5 × 2.5 × 1.5 cm silicone pattern, parallel to the shear force direction. The enamel layer on the occlusal surfaces of molars was ground to get flat superficial dentin and vestibule surfaces of incisors were ground to flatten enamel surfaces for standardization. All surfaces were polished using 240–600-grit silicone carbide abrasive papers under water-cooling (Minitech 233 Polishing-Machines; France). The center of each specimen was marked using 0.5-mm graphite pencil to form a circle of 3-mm diameter for adhesive area standardization, and each etching procedure was limited inside circle borders.

The study was carried out in 8 groups with 15 different samples in each group, using 2 different dental tissues (enamel and dentin) and 4 different surface etching techniques. One enamel and dentin specimen representing each group was stored for SEM analysis after surface treatment procedure. Ceramic discs with a diameter of 3 mm and a thickness of 3 mm were fabricated according to the manufacturer's recommendations. The initial shapes of ceramic materials were manufactured by press technique with cylinder rod length of 12 mm and 3 mm diameter using waxed (Elastiwax; Ivoclar Vivadent AG, Schaan, Liechtenstein) models of the same shapes. Ceramic rods were separated using a low-speed diamond saw sectioning machine (Buehler Ltd., USA) to obtain the final dimensions.

Enamel and dentin surfaces were irradiated with Er:YAG laser (Lightwalker, Fotona, Slovenia) emitting photons at a wavelength of 2.94 μm and pulse duration of 100 μs in all laser groups. The output power and repetition rate of this equipment were adjusted to be the same for all laser groups to 120 mJ and 10 Hz in enamel [29] and dentin surfaces, respectively. The energy densities in group Er:YAG-H14 were 9.05 J/cm^2, while in XR and XR2 groups it was calculated as 18.87 J/cm^2. The specimen surfaces were bathed with an adjustable air/water spray using air level of 20% and water level of 40%.

Group acid The 37% phosphoric acid was applied with a disposable micro-brush for 30 s to enamel and for 15 s to dentin, then all surfaces were rinsed for 15 s and gently air-dried.

Group XR The surfaces of specimens were irradiated with the Er:YAG scanning handpiece (X-Runner, Fotona, Slovenia) adjusted to etch a circle-shaped area of 3 mm in diameter.

Group XR2 The procedure applied on the specimen surfaces was similar to that in group 2, but the surfaces of the specimens were scanned 2 times. The distance to the surface was 10 mm, which was accomplished using a designed system.

Group Er:YAG-H14 Specimens were irradiated with the Er:YAG laser delivering the energy via the H14-N handpiece by a 1.3-mm-diameter, 8-mm-length sapphire tip. The distance between the tip and the surface was kept at 1 mm and the laser beam was applied to the entire surface for 15 s manually.

After ultrasonic cleaning for 5 min in distilled water, ceramic discs (IPS Ceramic Etching Gel; Ivoclar Vivadent, Schaan, Lichtenstein) etched by hydrofluoric acid for 20 s and treated with silane coupling agent (Monobond N, Vivadent Ets., Schaan/Liechtenstein) were cemented to the specimen surfaces by using adhesive resin cement (Variolink N, Vivadent Ets., Schaan/Liechtenstein) according to the manufacturer's instructions. Excess cement was cleaned with a micro-brush before polymerization and then light-cured (Valo Ultradent, South Jordan, UT, USA; light intensity 1.400 mW/cm^2, wavelength 385–515 nm). After storage in distilled water at 37 °C for 24 h, all bonded specimens were subjected to thermal cycling between 5 and 55 °C for 5000 cycles of 30 s cold and 30 s hot storage in distilled water with a transfer time of 2 s [28].

The SBS test was performed using a universal test machine (Servopulser EHFFD1; Shimadzu, Kyoto, Japan) at a crosshead speed of 0.5 mm/min, and values were recorded.

The fractured surfaces were analyzed with a digital microscope (U500X Digital Microscope, CoolingTech OEM/ODM, Guangdong, China) at × 20 magnification to determine the predominant failure mode classification (cohesive, adhesive, or mixed failure at the ceramic or the composite surface). The percentages of failure modes were determined.

After data collection, means for each group were calculated in megapascal for relevant statistical analysis. The values obtained from the SBS test were analyzed with one-way analysis of variance (ANOVA) (SPSS v16.0; SPSS Inc., Chicago, IL, USA) as dentin and enamel groups, respectively. As significant differences were observed, comparisons were made between the groups by post hoc Tukey's HSD test. The level of statistical significance was set at $p < 0.05$.

SEM images of each enamel and dentin specimen from each representative group were evaluated at × 2000 magnification to observe changes on the enamel and dentin surfaces after surface treatment procedures (Evo LS10, Carl Zeiss, Cambridge, England).

The number and mean diameters of dentin tubules per squared millimeter were determined at the center of each dentin specimen from each representative group on SEM images of × 500 magnification and mean diameters were measured on the SEM images of × 2000 for each sample in accordance with the study of Schilke et al. in 2000 [30]. The counting and measurements were performed using an image editing software (GIMP 2.10; The GIMP Development Team; Berkeley, CA, USA).

Results

Mean SBS values and standard deviations for different surface treatment methods on enamel and dentin in each group are shown in Table 1.

According to the one-way ANOVA test results, differences between the two dentin groups were statistically significant ($p < 0.05$). Post hoc Tukey's HSD test was applied in order to determine the groups. Significance among dentin groups is shown in Table 1 using lowercase letters. Statistical significance was observed between group XR2 and other dentin groups ($p < 0.05$), where 2 times Er:YAG laser-scanned dentin specimens showed highest SBS values. No statistical significance was observed among other dentin groups ($p > 0.05$). According to the results of ANOVA, no statistical significance was observed among the enamel groups.

As a result of the surface examination, no cohesive-type failure was observed on any tooth or ceramic material surfaces. Mixed failures within resin cement (65%) and adhesive

Table 1 Mean SBS values and SDs for different etching methods (MPa). Superscript letters a and b in the table indicate statistical significance between dentin groups. Identical lowercase letters denote no significant differences among groups ($p > 0.05$). Superscript letters C and D in the table indicate statistical significance between enamel groups. Identical uppercase letters denote no significant differences among groups ($p > 0.05$). *MPa*, megapascal; *Acid*, acid-etched surfaces; *XR*, laser-etched surfaces using scanning handpiece; XR2, surfaces etched 2 times using scanning handpiece; *Er:YAG-H14*, surfaces etched manually using laser

Group	Dentin	Enamel
Acid	^{a} 9.18 ± 1.52	^{C} 15.43 ± 3.88
XR	^{a} 8.52 ± 3.08	^{C} 14.68 ± 3.70
XR2	^{b} 11.42 ± 2.37	^{C} 15.17 ± 3.39
Er:YAG-H14	^{a} 8.58 ± 1.94	^{C} 15.55 ± 4.26

failures between teeth surfaces and resin cement (35%) were mostly observed.

SEM images of each dentin group after surface treatment are shown in Fig. 1. SEM image of group acid shows clearly observable dentin tubules without smear layer (Fig. 1a). The mean diameters of dentin tubules per squared millimeter were determined as 2.28 μm for group acid, 1.66 μm for group XR, 1.2 μm for group Er:YAG-H14, and 1 μm for group XR2, where the acid and the laser groups were similar to those in the study of de Los Angeles Moyaho-Bernal et al. [26]. The diameters of exposed dentin tubules in laser groups (groups XR, XR2, Er:YAG-H14) were comparatively smaller than those of the acid group specimens and funnel shapes and peritubular dentin demineralization could not have been observed (Fig. 1b–d).

SEM images of each enamel group after surface treatment are shown in Fig. 2. SEM image examinations of enamel group specimens were made according to the surface treatment model firstly published by Silverstone et al. in 1975. In the group acid, generalized roughening of the enamel surface was observed. Demineralized enamel surface appeared to combine hollowing centers of the enamel prisms with relatively intact peripheral regions as a type 1 etching pattern (Fig. 2a). Different surface etching patterns are observed in groups XR and XR2, as the formation of geometrical motive with a more uniform and homogenous pattern (Fig. 2b and c), and irregular hollowing surface formation with non-homogenous depth of ablated regions in group Er:YAG-H14 (Fig. 2d). Enamel surfaces of laser-treated groups showed the mixed etching pattern that differed from known classification.

Discussion

In the present study, standardized enamel and dentin surfaces were conditioned using four different surface treatment methods and bonded with all ceramic discs by using adhesive resin cement as an adhesive luting material. After the thermocycling of all bonded specimens, bonding efficacy was determined by SBS test. It was hypothesized that there will be no statistical differences among all groups; however, significant differences were observed among dentin groups, where the dentin group that was etched 2 times using the Er:YAG laser scanning handpiece showed the highest SBS values. Therefore, the null hypothesis was rejected relying on this statistical result.

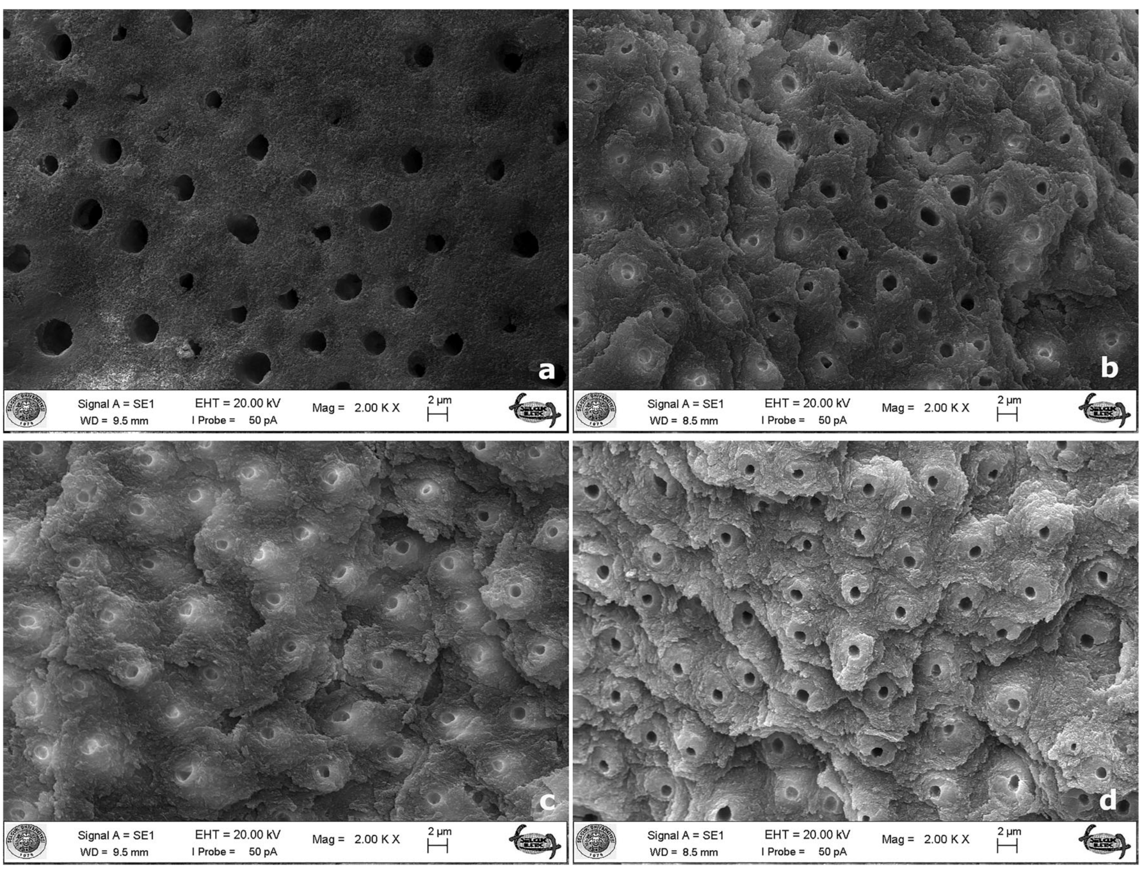

Fig. 1 SEM images of dentin groups (× 2000). **a** Group acid: acid-etched dentin surfaces. **b** Group XR: laser-etched dentin surfaces using scanning handpiece. **c** Group XR2: dentin surfaces etched 2 times using scanning handpiece. **d** Group Er:YAG-H14: dentin surfaces etched manually using laser

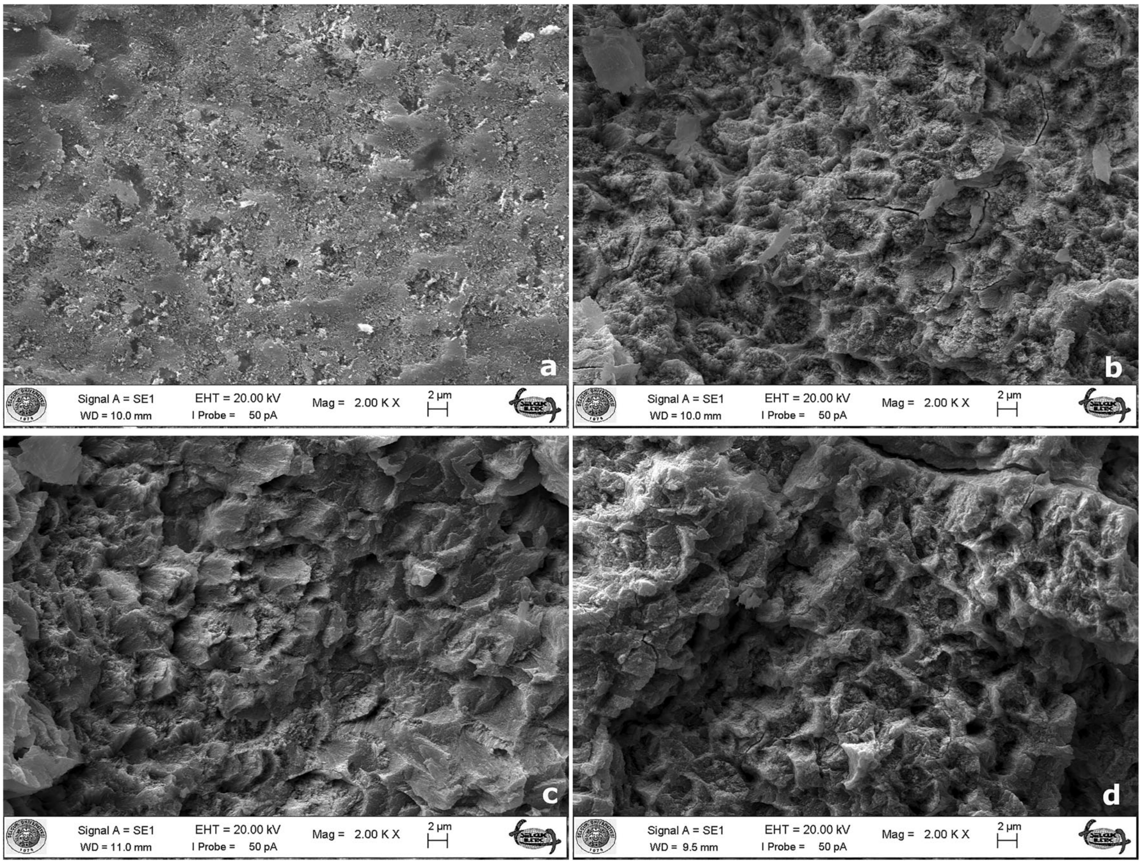

Fig. 2 SEM images of enamel groups (× 2000). **a** Group acid: acid-etched enamel surfaces. **b** Group XR: laser-etched enamel surfaces using scanning handpiece. **c** Group XR2: enamel surfaces etched 2 times using scanning handpiece. **d** Group Er:YAG-H14: enamel surfaces etched manually using laser

The specimens stored in distilled water rely on the study of Mobarak et al., which mentioned controversial opinions regarding optimum storage conditions and reported that pretest storage conditions of extracted teeth have no effect on the bond strength of composite resin materials as well as rehydration of dry stored teeth in distilled water [31]. The temperature variations in oral cavity have detrimental effects on the bond strength between tooth structures and all ceramic restorative materials. Therefore, the method of simulating the oral environment in testing bond strength between biomaterials and tooth substance is of great importance. In the present study, thermocycling, the most commonly used artificial aging method, was used in terms of obtaining physical conditions closest to the oral environment [32].

In a control group, enamel and dentin specimens were conditioned using 37% orthophosphoric acid, which is accepted as the most popular surface etching method. The formation of the resin tags on the enamel surface after acid etching is a desired result in attaining good micromechanical adhesion [33]. On the other hand, dentin adhesion has a more sophisticated mechanism, where acid etching leads to demineralization of superficial dentin and total dissolving of the smear layer. Furthermore, by penetration into the opened dentin tubules and setting among the exposed collagen fibrils on the decalcified layer, the adhesive material can attain intimate contact with conditioned area and bonding to the dentin surface [34]. Despite the positive results that have been reached in the surface conditioning of the tooth structures by acid etching, some disadvantages of this technique still exist. Possible decalcification of dental tissues may reduce the defense of demineralized regions against oral environment and end with caries [35]. On the other hand, laser therapy offers the advantages of sterilization, enforcement of dental surfaces, and preferable adhesive structure achieved by formation of a microscopically rough substrate surface without demineralization and open dentinal tubules without smear layer production [22, 23]. Although, according to Usumez et al., hand-controlled laser application on the substrate surfaces may reduce adhesion to enamel, it was commented that the manual etching process is prone to errors which could be caused by irregular and non-standardized hand-controlled laser application, where unexposed substrate surfaces will show etching pattern with failed continuity [36]. The Er:YAG scanning handpiece has an advantage of obviating a hand-controlled irregular movement. Surfaces are etched in particular shape, region, and without any movement. Therefore, a more uniform etching pattern can be obtained. Relying on this information, the effect of Er:YAG scanning handpiece was analyzed on both enamel and dentin surfaces.

In the study of Sağır et al., Er:YAG laser etching of the enamel surface in different pulse durations and modes (QSP or MSP modes) was considered a successful alternative to acid etching [29]. A similar conclusion was made by Sobha et al., where increased SBS values surpassing those of acid etched were obtained in Er:YAG laser in 100-μs pulse duration in MSP mode [37]. The etching parameters (MSP), SBS procedures, and bonding techniques in the studies mentioned above were similar to the enamel groups of our study, although the differences as the acid etching time of 20 s, larger bonding area of 10.55 mm^2 for brackets, lack of etching surface, and area standardization could alter the SBS results. Furthermore, the SBS values higher than those in acid groups were found to be statistically significant after surface etching in MSP mode in these two studies, while there were no significant differences among acid groups in our study.

Some authors reported comparable SBS values on bonding to enamel and dentin when Er:YAG handpiece settings were adjusted to 120 mJ, 10 Hz, and 1.2 W [29, 38–40]. Therefore, Er:YAG laser etching in MSP mode with settings of 120 mJ, 10 Hz, and 1.2 W was applied in this study. Additionally, laser treatment distances of 1 mm in group Er:YAG-H14 and 10 mm in XR and XR2 groups were suggested in accordance with the study carried out by Yilanci et al. in 2017 [28]. Since there is limited data regarding the best number of scans using the scanning handpiece, the surface conditioning in groups XR and XR2 was made 1 and 2 times respectively.

Despite the studies reporting that laser etching enhances the bond strength between resin cement and tooth structures, there are controversial opinions between authors. The conclusion of the study carried out by Martines-Insua was that the formation of extensive subsurface fissuring on enamel and dentin surfaces after Er:YAG laser conditioning exerts a detrimental influence on adhesion leading to weaker bond strength values than acid-etched surfaces [41]. Moreover, Hoshing et al. reported significantly lower SBS values in enamel after Er:YAG laser etching [42]. Apart from this, Ceballo et al. reported the lower SBS values in dentin surface prepared using Er:YAG laser compared with acid etching. It was considered that the limited resin penetration to subsurface of interfibril space caused by fusion of collagen fibrils is the main factor of reduced SBS values [23].

However, the results of this study indicate that no significant differences were found among the groups in terms of SBS values except the dentin group that was irradiated 2 times using the scanning handpiece. SBS values in group XR, group Er:YAG-H14, and enamel specimens of group XR2 are in agreement with the studies earlier reported that the Er:YAG laser pretreatment of the tooth structures is similar to acid etching. However, dentin specimens in group XR2 showed significantly higher SBS values. According to Ceballo et al., dentin surfaces irradiated using erbium lasers are deprived of the exposed collagen layer [23], which claimed to be one of the main parts of strong dentin adhesion mechanism [34]. Also, the ablation rate of dental surfaces is dictated by the water and collagen amount in the structure of etched surfaces [20].

Considering the difference in etching patterns of acid-etched dentin and laser-etched dentin surfaces, it can be suggested that the higher bond strength obtained from dentin specimens in group XR2 relies on the increased adhesive area produced by additional irradiation. Uniform irradiation of dentin surface by the scanning handpiece produced the bonding conditions similar to acid etching, while double irradiation of dentin surface using the same handpiece with the same parameters caused the extension of adhesive area leading to higher SBS values. The mean diameters of exposed dentinal tubules per squared millimeter were determined from the highest to the lowest as 2.28 μm for group acid, 1.66 μm for group XR, 1.2 μm for group Er:YAG-H14, and 1 μm for group XR2. This difference between diameters of acid-etched and laser-etched groups was found to be associated to the demineralization of peritubular and intertubular dentin by phosphoric acid leading to increased tubule diameters, when Er:YAG lasers perform a selective ablation on intertubular dentin leaving the tubule orifices with highly mineralized peritubular dentin tissue leading to minimal change in their diameters; and it was shown that the thermal effect of laser ablation managed to alter the organic content of the treated dentin surface [43]. Considering the conclusion that reduced dentin tubule diameters have a potential adverse effect on SBS in terms of lower resin penetration, it could be expected that the test results of all laser groups would end up with much lower SBS values. However, the collagen layer loss coupled with the micromechanical environment created by surface irregularities which tend to increase with laser treatment endowed dentin surfaces with bond values comparable with those of the acid-etched group and led to even higher SBS values in group XR2.

Nahas et al. concluded that dentinal ablation increased proportionally to the increase in energy density delivered [44]. In the study of Gisler and Gutknecht in 2012, it was suggested that the ablation threshold of dentin in freshly extracted human teeth is 4 J/cm^2 and the energy densities of 30.86 J/cm^2 and 72.6 J/cm^2 could not be the minimal invasive approach due to its rapid ablative rate [45]. In this study, etching parameters slightly exceeding the ablation threshold of dentin surface exerted a beneficial influence on the adhesion between resin cement material and dentin surfaces.

Failure analysis of the specimens revealed 35% of adhesive failure between resin and tooth structures, and 65% of mixed failure in resin cement which were in agreement with SBS values. The increased number of mixed failures was observed on both enamel and dentin groups etched two times using a scanning handpiece, which might be the outcome of additional laser etching.

This in vitro study was performed under certain conditions where only one type of resin-based cement and ceramic material were used. However, many adhesive systems and ceramic materials can be included in this evaluation of a novel method of laser etching. The thermocycling may not be the certain reflection of actual oral environment and its real aging patterns. Furthermore, diameters of exposed dentin tubules were measured from only one specimen representative for each group. Therefore, our results may not be interpreted as the certain reflection of in vivo conditions. Further clinical studies should be conducted.

Conclusion

Considering the limitations of this study, the following can be concluded:

Different etching methods of dental surfaces as acid etching, manual laser etching, and etching with a scanning handpiece showed similar SBS values.

Additional etching of the dentin surfaces with a scanning handpiece before final luting of ceramic materials improved the bonding between resin cement used as adhesive material and dentin.

Laser etching seems to be a viable alternative to acid etching on both enamel and dentin surfaces.

Authors' contributions Conceptualization, methodology, formal analysis and investigation, writing–original draft preparation: Artur Ismatullaev. Writing–review and editing: Simge Taşın. Supervision: Aslıhan Üşümez.

Data availability Not applicable

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

Ethics approval Ethical approval was obtained from the Clinical Research Ethics Committee of Bezmialem University in Istanbul, Turkey (No. 71306642/050-01-04/208).

Consent to participate Not applicable

Consent for publication Not applicable

Code availability Not applicable

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