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Shear bond strength and SEM morphology evaluation of different dental adhesives to enamel prepared with ER:YAG laser

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Abstract

Context: Early observations of enamel surfaces prepared by erbium lasers motivated clinicians to use laser as an alternative to chemical etching. **Aims:** Evaluate shear bond strength (SBS) values of different dental adhesives on Erbium:Yttrium Aluminum Garnet (Er:YAG) laser prepared enamel and to evaluate possible etching patterns correlations between dental adhesives and SBS values. **Subjects and Methods:** One hundred bovine incisors were randomly assigned to SBS tests on enamel ($n = 15$) and to enamel morphology analysis ($n = 5$) after Er:YAG laser preparation as follows: Group I – 37% phosphoric acid (PA)+ ExciTE®; Group II – ExciTE®; Group III – AdheSE® self-etching; Group IV – FuturaBond® no-rinse. NR; Group V – Xeno® V. Teeth were treated with the adhesive systems and subjected to thermal cycling. SBS were performed in a universal testing machine at 5 mm/min. **Statistical Analysis Used:** One-way ANOVA and *post-hoc* tests ($p < 0.05$). For the morphology evaluation, specimens were immersed in Ethylenediamine tetraacetic acid (EDTA) and the etching pattern analyzed under Scanning Electron Microscope (SEM). **Results:** Mean bond strengths were Group I – 47.17 ± 1.61 MPa (type I etching pattern); Group II – 32.56 ± 1.64 MPa, Group III – 29.10 ± 1.34 MPa, Group IV – 23.32 ± 1.53 MPa (type III etching pattern); Group V – 24.43 MPa ± 1.55 (type II etching pattern). **Conclusions:** Different adhesive systems yielded significantly different SBSs. Acid etching significantly increased the adhesion in laser treated enamel. No differences in SBS values were obtained between AdheSE® and ExciTE® without condition with PA. FuturaBond® NR and Xeno® V showed similar SBS, which was lower in comparison to the others adhesives. No correlation between enamel surface morphology and SBS values was observed, except when PA was used.

Keywords: Adhesion, dental enamel, enamel-surface preparation, Er-YAG laser, laser conditioning, self-etch, SEM morphology, shear bond strength

Introduction

Adhesion to the enamel surface is based on the infiltration of resin monomers into etched enamel. An optimum etching pattern is observed when using an etch-and-rinse (ER) technique.[1] Self-etching adhesive (SE) systems have been advocated for this type of procedures as a suitable replacement for ER systems.[2] However, there is some concern about the ability of these SE systems to etch enamel, as many studies find that bond strengths of composite to enamel provided with ultra-mild, mild or intermediary-strong SE systems are lower when compared to ER systems.[2-5] Notwithstanding, SE systems seem to perform well in clinical studies.[6] The selective etching of enamel with phosphoric acid (PA) has been demonstrated as a potential technique to use with SE systems to improve their performance on enamel.[7-9] A disadvantage attributed to acid etching is the demineralization of tooth tissues, which makes them more permeable and prone to acid attacks, especially, if the demineralized substrates are not completely filled by adhesive resins.[10] Other disadvantages are the removal of the superficial enamel layer, the variation in etching depth, the subsequent contamination of the etched surface with water or oil, and the inadequate washing or drying, which can adversely affect the bond strength.[11] Effective ablation of dental hard-tissues by Erbium: Yttrium Aluminum Garnet Er:YAG laser has been reported. The 2.94 mm wavelength of the Er:YAG laser falls in an area of the spectrum where both enamel and dentin have absorption peaks (high absorbability in water and hydroxyapatite). Early observations of enamel surfaces prepared by erbium lasers demonstrated a similar etching pattern to those of PA. These findings motivated clinicians to use laser as an alternative to chemical etching.[12]

Er:YAG laser energy is absorbed by the water portion of hard-tissues, resulting in a temperature increase at the site

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of application, which leads to a rapid volume expansion on vaporization of the water. As a result, micro explosions are produced, causing disintegration of the hard-tissue.[13] However, there are controversial results regarding the effectiveness of Er:YAG laser pre-treatment of enamel and dentin[14-16] prior to bonding procedures. Most of the studies report lower shear bond strength (SBS) in enamel[17,18] while others show no differences in the results.[19] This contradictory results are probably due to the diversity of the parameters, methodologies, and adhesive systems utilized.[20] There is also some controversy concerning the need of additional acid etching application to tooth tissues irradiated by Er:YAG laser.[10,14]

The aim of this study was to evaluate the SBS and qualitative etching patterns of laser prepared enamel with different dental adhesives. The null hypothesis is that there are no differences between SBS values of different dental adhesives on Er:YAG laser prepared enamel. It was also hypothesized that there is no correlation between qualitative etching patterns of enamel and SBS values.

Subjects and Methods

SBS test

A total of 75 sound bovine incisors were extracted for no longer than a month and kept in distilled water at 4°C. After this period of time, teeth were kept in a 0.5% chloramine solution for a week and bisected with a microtomer (Accuton-Struers, Ballerup, Denmark) to separate the crown from the root. They were then polished with a 240-grit sandpaper to create a flat surface (Carbimet Buehler-met, Buehler, and Lake Bluff, Illinois. Enamel surface preparations were performed using an Er: YAG laser system (OpusDent™, Lumenis company, London, United Kingdom, model SA5601000, series number 005-13201), with a power of 500 mJ and a 12 Hz frequency. A working distance of one mm was used in a defocused mode. The specimens were divided in five groups ($n = 15$) according to the dental adhesive system used (Group I – PA + ExciTE®; Group II – ExciTE® (without PA); Group III – AdheSE; Group IV – FuturaBond® NR; Group V – Xeno® V). All the specimens were laser treated immediately before the application of bonding, according to Table 1.

After the application of the adhesive according to the manufacturer's instructions, composite resin cylinders were bonded to the tested surfaces (Synergy D6, A2/D2, Coltène Whaledent GmbH, Langenau, Germany), and specimens were kept in distilled water for 24 h at 37°C with 100% humidity (Hemmet, Schwabach, Germany) in order to obtain the maximum resin polymerization, before being thermocycled (Aralab, mod 200E, Cascais, Portugal) for 500 cycles at 5°C and 55°C for 20 s[21] in each bath and submitted to shear testing at a crosshead speed of 0.5 mm/min (Instron, model 4502, series H3307, Instron Ltd, Bucks, England). A one-way ANOVA was run to determine, if there were differences among groups and Student knewman keuls *post-hoc* analysis to visualize between which groups these differences were detected and the confidence level was set to 95%.

Failure pattern analyses

The fracture sites of the de-bonded surfaces were examined using a binocular stereomicroscope (Stereo Microscope Zoom-10, Nikon, Melville, New York, USA) at ×15. Representative samples were chosen for examination under

Table 1: Composition of tested adhesives

Material/batch	Main component	Manufacturer	Manufacturer's instructions
Total etch (N23886)	37% phosphoric acid	IvoclarVivadent Schaan, Liechtenstein	Applied for 30 s, rinsed for 15 s, and gently dried for 5 s
Excite (P50689)	HEMA, dimethacrylate, phosphonic acid acrylate, highly dispersed silicon dioxide, initiators and stabilizers, alcohol	IvoclarVivadent Schaan, Liechtenstein	Applied for 10 s, air dried for 15 s and light-cured for 10 s light-curing
AdheSE (Primer-P30007 Bond-P23053)	Primer (dimethacrylate, phosphonic acid acrylate, initiators and stabilizers in an aqueous solution) Bond (HEMA, dimethacrylate, silicon dioxide, initiators and stabilizers)	IvoclarVivadent Schaan, Liechtenstein	Primer applied with a brush for 15 s (total reaction time should not be shorter than 30 s) dispersed with a strong stream of air until the mobile liquid was no longer visible Bond is applied and dispersed with a weak stream of air and light cured for 10 s
FuturaBond NR (0950214)	Organic acids, Bis-GMA, HEMA, TMPTMA, camphorquinone, amines (DABE), BHT, fluorides and ethanol	VOCO, Cuxhaven, Germany	Applied in a thin layer and brushed for 20 s. Air dried for at least 5 s and polymerized for 10 s
Xeno V (1002000450)	Bifunctional acrylate, acid acrylate, functionalized phosphoric acid ester, water, tertiary-butanol, initiator and stabilizer	Dentsply, DeTrey GmbH, Germany	Applied into the whole cavity uniformly, agitated gently for 20 s and the solvent evaporated with air until there was no more movement of the adhesive

HEMA: 2-hydroxyetil methacrylate; NR: is part of the brand mark: FuturaBond NR; Bis-GMA: Bisphenol-alpha-glycidyl methacrylate; TMPTMA: Trimethylolpropane trimethacrylate; DABE: 1,2-diaminobenzene; VOCO: is the manufacturer

Scanning Electron Microscope JEOL Ltd, JSM 6301F/Oxford Instruments Analytical INCA Energy 350/Gatan Alto 2500, Tokyo, Japan). Samples were mounted on SEM stubs and sputter-coated with gold. Examination was done a t 30 kV of accelerating voltage at different magnifications and characteristic photomicrographs were obtained at ×2000.

Morphology evaluation

Twenty five bovine incisors were divided in five groups ($n = 5$), according to the adhesive used. (Group a – 37% PA + ExciTE®; Group b – ExciTE®; Group c – AdheSE; Group d – FuturaBond® NR; Group e – Xeno® V). Samples were prepared exactly as they were for shear bond tests and after being in distilled water for 24 h at 37° C with 100% humidity (Hemmet, Schwabach, Germany), were immersed in Ethylenediamine tetraacetic acid EDTA (Queija Ferreira pharmaceutics, Porto, Portugal) 1M, pH = 5.5 for 3 weeks in a stirrer to demineralize enamel and fully expose the adhesive, to obtain a replica of the pattern of demineralization.[22] Samples were then prepared and examined on SEM as described above at ×2000. The enamel surface was classified by an experienced investigator, according to Cehreli and Altay[23] in:

Type I – Preferential dissolution of the prism cores resulting in a honey-comb-like appearance;

Type II – Preferential dissolution of the prism peripheries creating a cobblestone-like appearance;

Type III – A mixture of type I and type II patterns;

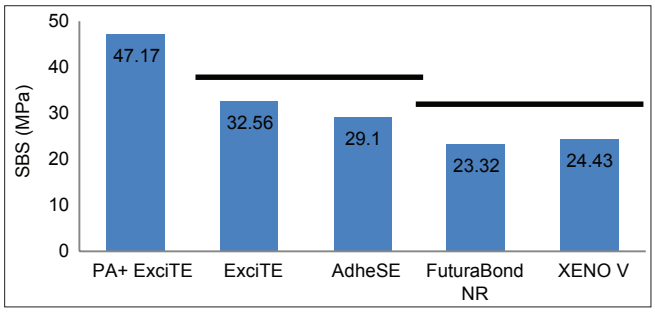
Type IV – Pitted enamel surfaces as well as structures that look like unfinished puzzles, maps or networks;

Type V – Flat, smooth surfaces.

Results

SBSs

According to the statistical analysis, SBS was affected by the type of bonding system used. SBS values were shown in Graph 1. The mean bond strengths were Group I – 47.17 ± 1.61 MPa; Group II – 32.56 ± 1.64 MPa, Group III – 29.10 ± 1.34 MPa; Group IV – 23.32 ± 1.53 MPa; Group V – 24.43 ± 1.55 MPa. The values between Group II and Group III and Group IV and Group V were not significantly different ($P > 0.05$). Group I was significantly higher than the rest of the groups ($P < 0.05$).



Graph 1: Mean shear bond strength (SBS) values (MPa). Groups under the same line show no significant differences

SEM morphology evaluation

Figures 1-5 show the enamel surface after laser irradiation and application of the different adhesive systems: 37% PA + ExciTE® (Group I); ExciTE® (Group II); AdheSE® (Group III); FuturaBond® (Group IV) and Xeno® V (Group V), respectively. Groups' II, III, and IV had similar patterns among them. The latter's enamel surface topography showed predominant dissolution of enamel peripheries with areas of prism core dissolution (a type III etching pattern). Although, Group V produced the enamel crystallite dissolution pattern similar to that produced with the other self-etch systems, the demineralization produced is less defined, shallower with a non-homogeneous type II etch pattern. With the use of PA (Group I) topography revealed a milder, homogeneous Type I etching pattern. The enamel prisms were hollowed out to deep pits or craters placed side by side separated by thick inter-prismatic enamel persisting in the form of rings.

Failure pattern analyses

Graph 2 shows the bonding failure pattern analysis after SBS test.

Discussion

Laser etching of dental enamel is a painless procedure, which does not impart vibration or heat, nonetheless providing an etching pattern that seems ideal for resin penetration.[24] Moreover, the surface produced by laser irradiation seems to be more resistant to secondary caries.[25] The mechanism of tissue removal by laser, unlike acid etching, is not demineralization. Instead a micro-ablative process causes vaporization of water and dental organic components, promoting micro explosions, which on their turn, cause the destruction of inorganic substances[13] resulting in

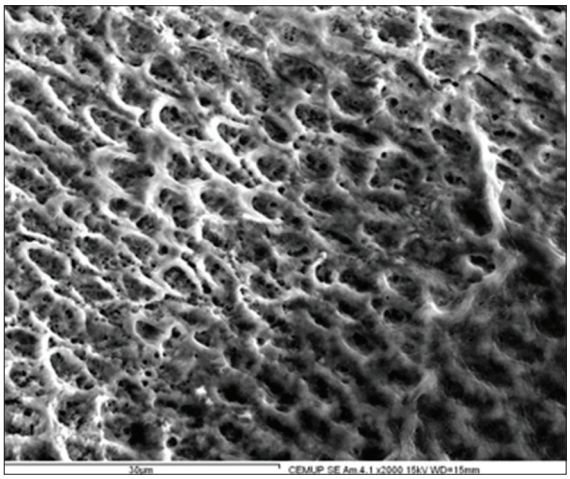
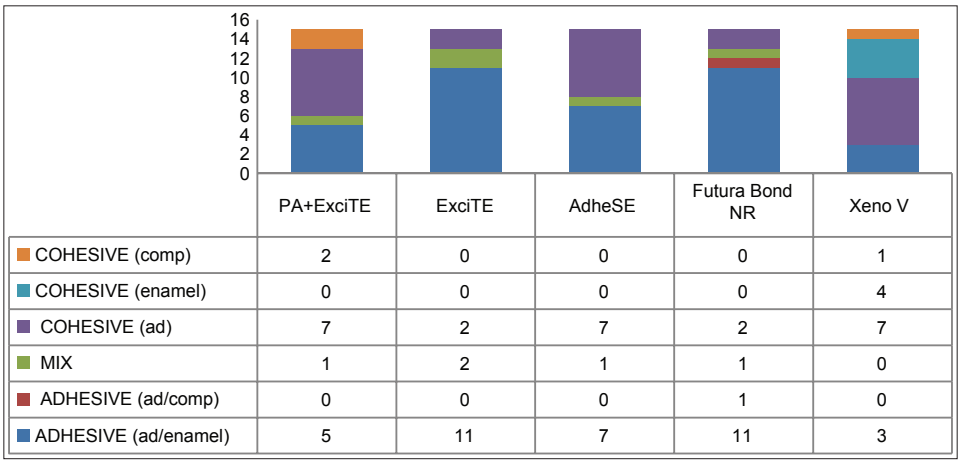


Figure 1: Scanning Electron Microscope SEM micrograph of Group I (PA + ExciTE®). This image shows the enamel surface after Er:YAG laser and phosphoric-acid treatment (Group I). This morphology has a honeycomb appearance and more flattened surface. Type I (preferential prism center etching) etching pattern



	PA+ExciTE	ExciTE	AdheSE	Futura Bond NR	Xeno V
COHESIVE (comp)	2	0	0	0	1
COHESIVE (enamel)	0	0	0	0	4
COHESIVE (ad)	7	2	7	2	7
MIX	1	2	1	1	0
ADHESIVE (ad/comp)	0	0	0	1	0
ADHESIVE (ad/enamel)	5	11	7	11	3

Graph 2: Bonding failure pattern analysis after shear bond strength test

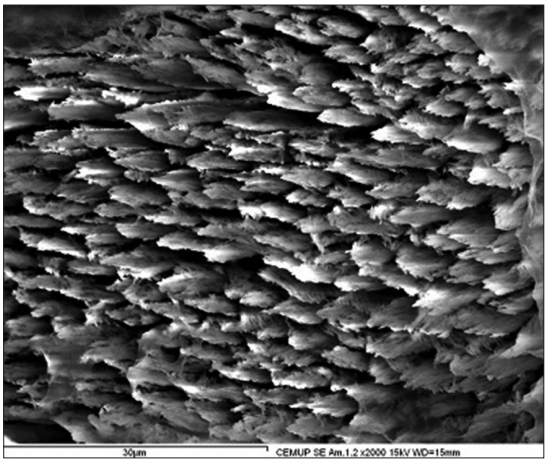


Figure 2: Scanning Electron Microscope SEM micrograph of Group II (ExciTE® without PA). SEM shows sharp, jagged projections of enamel prisms after laser application. This rich and complex etching pattern was solely produced by laser etching as the adhesive was applied without any prior conditioning

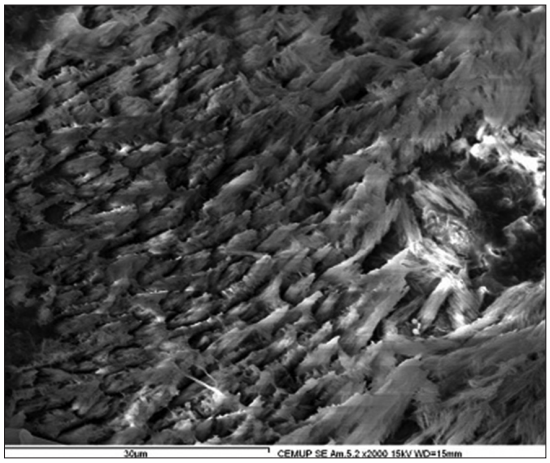


Figure 3: Scanning Electron Microscope SEM micrograph of Group III (AdheSE). A selective demineralization on the periphery of the enamel prisms can be observed. The resin was able to penetrate into these spaces providing a good resin-enamel bonding

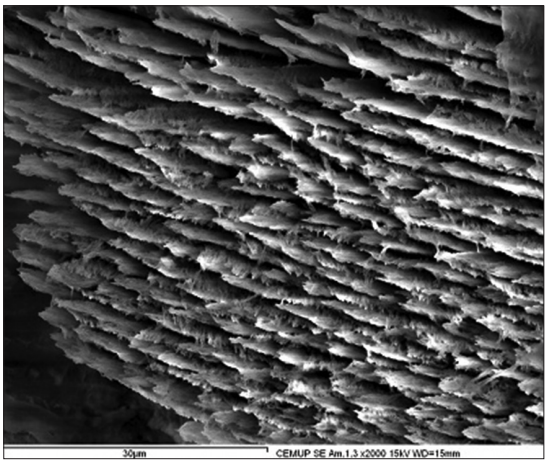


Figure 4: Scanning Electron Microscope SEM micrograph of Group IV (FuturaBond® NR). A selective demineralization on the periphery of the enamel prisms can be observed. Prism cores were also superficially demineralized increasing the bonding area

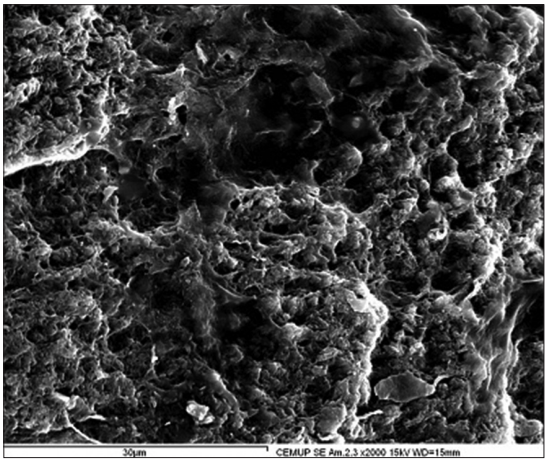


Figure 5: Scanning Electron Microscope SEM micrograph of Group V (Xeno® V) shows rough irradiated surface and irregular ablated pattern compared with Group I. Demineralization resembles type II pattern, prism peripheries were demineralized more deeply than prism cores. Shallower demineralization compared with the other adhesives

microscopic surface irregularities in which the adhesive system can penetrate, fostering retention, and thus, good SBS values.[26]

Nonetheless, according to some authors, the treatment of the enamel surface exclusively by Er:YAG laser seems to result in a higher degree of micro-leakage[27,28] and lower SBS values.[16] These results are not in agreement with other studies that obtained identical[29,30] SBS values when enamel surface was prepared with Er: YAG laser only. This discrepancy may be possibly explained by different laser outputs used or the different characteristics of the adhesive systems employed[31] after the use of laser irradiation, being SE or ER systems.[29]

SBS test

Regarding SE systems, the overall bond strength of these to enamel is also controversially discussed in the literature. Some studies have reported similar results in comparison to ER systems[32,33] while other studies consider these systems to be less reliable to enamel bonding.[2,5,34] These differences may be due to the use of different bonding procedures or different methods for enamel preparation, as these may affect SBS results. SE systems seems to be more dependent on the type of enamel preparation than ER systems[35] and the use of laser as an enamel pre-conditioner for SE adhesives could be highly beneficial. This is because the laser treated surface may provide a deeper etching pattern, which elicits better mechanical adhesion[36] and compensates for the weaker acidity of the SE systems in comparison to ER systems. Moreover, laser treated surfaces are absent of smear layer, which may be important, especially, for intermediary-strong SE systems, which are generally buffered by thick smear layers.[37]

The evaluation of bond strength through SBS tests is recognized as a reliable mean to assess the adhesive resistance to failure. According to the results of this study, the SBS of ExciTE (Group II, laser without PA), was 32.56 MPa. However, when PA etching was used after the laser application (Group I, laser and PA), the SBS values increased significantly to 47.17 MPa ($P < 0.05$). These findings seem to show that when Er:YAG laser is used to etch enamel, the additional use of PA does influence the performance of a ER system, as the mean SBS values obtained were significantly higher. However, the SBS value obtained for Group II (laser without PA) seem to be acceptable for enamel adhesion, according to Bowen who stated that SBS values between 15 MPa and 30 MPa are acceptable for enamel adhesion.[38] In addition, the SBS values obtained for Group II (laser without PA) was higher than the mean SBS values obtained in our previous study for ExciTE with PA (27.95 MPa), when enamel was prepared with diamond burs and without laser. Thus, there was an increase in the bonding performance of ExciTE (Group I) on enamel prepared with laser only when compared to enamel that was prepared with a bur and acid etched. Based on our results, we believe that laser can be a reliable alternative as a replacement for PA etching as SBS values seems to be clinically acceptable. Incidentally, previous studies show that laser prepared enamel may provide a better surface for adhesion.[37,39] In spite of imparting a significantly lower SBS compared to G I, G II performed well in terms of SBS. Additionally, there are further benefits for using laser on enamel. It is important to consider that laser is associated with enamel re-mineralization and reinforcement, and it is less likely to impart adverse effects, which have been associated with ER systems, such as enamel decalcification, which leaves the enamel susceptible to caries, especially, if subsequent resin impregnation is incomplete or defective.[18]

Regarding groups III, IV and V; the three SE Adhesives used in our study, AdheSE (GIII) yielded significantly higher SBS than FuturaBond® NR (GIV) and Xeno® V (GV), both of which presented statistically insignificantly differences in terms of SBS values. The SBS of AdheSE (GIII) was similar to ExciTE without PA (GII). These values were 29.10 MPa and 32.56 MPa, respectively ($P > 0.05$). According to these results, the two-step SE AdheSE performed relatively well in enamel treated with laser. This is in accordance to other studies that state that two-step SE performs better in terms of SBS than one-step SE systems.[40] In addition, AdheSE contains phosphonic acid acrylate in its primer. In general, phosphorus-containing monomers, e.g., phosphonic acids or acidic phosphates, are capable of etching enamel well. In addition, these monomers promote monomer diffusion into the acid-conditioned enamel.[41,42]

The SBS values for the two one-step SE adhesives on laser treated enamel, regardless of being one (Xeno® V) or two-component (FuturaBond® NR), were similar: 24.43 MPa and 23.32 MPa ($P > 0.05$), respectively; and significantly lower than all the other groups. Xeno® V and FuturaBond® NR are intermediary strong self-etch systems (pH 1.5 and 1.4, respectively) and despite the factor of imparting relatively lower SBS values, their performance is found to be clinical acceptable[38] and thus, its use is recommended for bonding to enamel prepared with laser, however, these findings are not in accordance with others that state that strong and intermediary strong self-etch systems may reach bond strength values to enamel as high as those obtained with PA, provided that the enamel is roughened.[32,43,44]

SEM morphology evaluation

Bonding to enamel is primarily based on the micromechanical interlocking of a low-viscosity resin, which penetrates into enamel micro-porosities. Considering this, apart from the mechanical properties of the adhesive itself, the surface morphology, that is, the extent, depth and etching pattern should influence the performance of the evaluated adhesive.[32] Therefore, we think it is important to understand how these surface alterations are performed on enamel and how it responds to different conditioning materials and methods.

Although, the etching pattern provided by the two one-step SE systems used in this study FuturaBond® NR and Xeno® V,

were not as deep and evident as the PA etching, selective enamel etching could be observed, which may have played a role in the relatively good performance of these adhesives. Previous studies reported that the etching pattern may not be a determinant factor for enamel bond strength[45] and despite the similar SBS and similar pH values the etching pattern provided by FuturaBond® was deeper than the one provided by Xeno® V, which is in accordance to Tsujimoto *et al*., as Xeno® V did not present the same etching pattern than FuturaBond® but still imparted similar SBS values. The different etching patterns produced by these adhesives may be due to their different chemical compositions. Xeno® V is a methacrylamide-based that has no 2-hydroxyetil methacrylate (HEMA), on the contrary of all the others adhesives used in this study. HEMA is a reactive diluent instable in aqueous acid solutions due to formation of hydrolysis-prone associates.[46] We hypothesize that the absence of HEMA could have prevented deeper etching patterns. Another possible explanation is the fact that SE adhesives without HEMA appear to be prone to phase separation showing voids in the adhesive layer.[47]

A similar performance was found among GII, GIII, and GIV. These groups elicited a rich and complex etching pattern with a selective demineralization on the periphery of the enamel prisms and the prism cores were also superficially demineralized increasing the bonding area. These results may be attributed to the absence of debris as laser produces no enamel smear layer or alteration in enamel morphology.[48] It is somewhat surprising that very little information is available on the morphology of enamel smear-layers.[49] Another possible explanation for the good etching patterns of these groups may be the chemical bonding of monomers to hydroxyapatite, which may contribute for the favorable etching patterns obtained. The parameters' used for laser irradiation may also have been important for the results, as different outputs seem to produce different results.[50]

The typical honeycomb etching appearance was observed when enamel was treated with the ER system after laser irradiation, which is thought to be related with good SBS values.[11]

Failure pattern analyses

As with any study that evaluates the acute and immediate SBS of dental adhesives, the surface failure pattern should be examined carefully to better understand the limitations of dental adhesives as well as possible mechanisms to develop better adhesive systems. Cohesive failures in laser irradiated surfaces are probably due to the presence of micro-cracks,[16] which are points of stress concentration and maybe responsible for lower bond strengths, however, only Xeno® V had cohesive failures on enamel. GI had the higher SBS and the most prevalent failure was cohesive on the adhesive, which is in accordance with other studies that state that cohesive failures may also be explained by a higher mechanical strength of the adhesive itself in comparison to enamel, which is brittle.[51]

For groups GII and GIV, adhesive failures were more frequent. The increase in adhesive failures observed when ExciTE was used without PA and when SE systems were also used may be explained by the reduced etching effect of the SE systems on the enamel surface, thus, reducing surface area available for adhesion. Only Xeno® V doesn't fit in the explanation, which may be due to its unique chemical composition.[45] Further investigations are necessary to understand this discrepancy.

The null hypothesis that SBS values are not affected when enamel is prepared with Er:YAG laser was rejected. It was also hypothesized that etching patterns of enamel have no correlation effects with the values of SBS. This hypothesis was confirmed. Despite similar etching patterns for some groups (GII, GIII, GIV), SBS values were different for the adhesives present in the study.

Further *in vitro* research should be performed to evaluate the long-term bond strength (water storage) of restorations bonded to substrates subjected to laser treatment and *in vivo* research should assess their clinical longevity.

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