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Longitudinal Evaluation of Bond Strength to Enamel of Dental Adhesive Systems Associated with Nd:YAG Laser

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Clinical Relevance

The photothermal mechanism of action of the Nd:YAG laser for the parameters used in this study likely promoted alterations at the bond interfaces of enamel, negatively influencing the bond strength and, consequently, the durability of resin composite restorations.

SUMMARY

Objectives: This study evaluated the durability of bond strength to enamel using total-etch (Single Bond/SB) and self-etch (Clearfil SE Bond/CSEB) adhesives associated with neodymium:yttrium-aluminu- garnet (Nd:YAG) laser irradiation through the uncured adhesives.

Methods: Bovine incisors were worn to expose an area of enamel and were divided into four groups: group 1 (control) SB + polymerization; group 2 (control) CSEB + polymerization; group 3 (laser) – SB + Nd:YAG laser (174.16 J/

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cm^2) + polymerization; and group 4 (laser) CSEB + Nd:YAG (174.16 J/cm^2) + polymerization. Blocks of composite were fabricated and stored for 24 hours or 12 months, sectioned into beams, and submitted to microtensile tests. Results were analyzed by three-way analysis of variance (ANOVA) (adhesive, technique, and storage time) and Tukey tests.

Results: ANOVA revealed significant differences for adhesive × technique and technique × storage time ($p<0.05$). The mean values (MPa) for interaction adhesive × technique (standard deviation) were as follows: SB/control = 35.78 (6.04)a; SB/laser = 26.40 (7.25)b, CSEB/control = 26.32 (5.71)b, CSEB/laser = 23.90 (7.49)b. For interaction technique × storage time the mean values were as follows: control/24 hours = 32.58 (6.49)a; control/12 months = 29.52 (8.38)a; laser/24 hours = 29.37 (5.71)a; laser/12 months = 20.92 (6.5)b. Groups with the same letters showed no statistically significant differences.

Conclusion: Scanning electron microscope analysis showed evident areas of micromorphological alterations in lased samples after 12 months of water storage. Nd:YAG laser irradiation of enamel through unpolymerized total-etch adhesive significantly reduced bond strength compared with the control. Bond strength decreased when enamel samples irradiated with Nd:YAG laser through unpolymerized adhesives were stored in water for 12 months.

INTRODUCTION

The post-Buonocore,[1] 1955 era, known as the "adhesive era," has shown that the total acid etch adhesive systems present excellent performance in this substrate and maintain the longitudinal stability of the bond interface. The enamel acid-etching technique is based on selective demineralization of the hydroxyapatite crystals present in tooth enamel, resulting in an extremely roughened surface with high energy. These characteristics provide high wetting capacity of the resinous monomers, which, on polymerizing, results in the formation of prolongations called tags that "anchor" the resin to the tooth.[2]

On the other hand, following the modern trend of simplifying the clinical steps and saving operating time, new bonding strategies were developed. In 1994, Watanabe and others[3] introduced the self-etching adhesives to the market with the proposal of optimizing the bond process, reducing the clinical steps, eliminating the acid-etching and washing steps, and providing better interaction with the dentinal substrate (because these adhesives are less aggressive). However, their etching capacity has been shown to be more restricted, as they present low reactivity with the mineral component, lower availability of H^+ ions, and high molecular weight, compared with phosphoric acid, promoting etching that is not as deep and retentive on dental enamel.[4,5] The highly hydrophilic nature of their composition may also contribute to the reduction in longevity of restorations in areas of extensive availability of enamel,[6,7] since this characteristic favors the sorption of liquids, nanoleakage, and consequent degradation of the bond. Therefore, total acid etch adhesive systems are considered the "gold standard" when compared with the self-etching adhesives existent in the market.

New alternatives for perfecting the bonding pattern have been exhaustively studied. Gonçalves and others[8] developed an irradiation technique with Nd:yttrium-lithium-fluoride (YLF) laser on dentin impregnated with unpolymerized adhesive. They obtained significantly higher shear bond strength values compared with unradiated dentin, leading them to believe that irradiation with laser could lead to the formation of a more resistant substrate with more chemical affinity for the bonding/adhesive process. After this, other authors[9,10] observed that this technique now used on enamel also improved the bond strength and could consequently optimize the longevity of the restoration.

Although the laser irradiation technique developed by Gonçalves and others[8] had been promising in improving the bond strength to both dentin and enamel,[9,10] only the immediate results were observed. No longitudinal studies were found. Therefore, the longitudinal evaluation of bond strength achieved when using the laser irradiation technique on the unpolymerized adhesive is shown to be relevant. The aim of this study was to evaluate *in vitro* the influence of neodymium:yttrium-aluminugarnet (Nd:YAG) laser on the microtensile bond strength to enamel of a two-step total-etch and a two-step self-etch adhesive when the laser was applied on the unpolymerized adhesives at time intervals of 24 hours and after a one-year period of storage in water at 37°C. In this study, three null hypotheses were tested: 1) The different adhesive systems do not affect the bond strength to enamel; 2) Nd:YAG laser irradiation through unpolymerized adhesives does not affect bond strength to enamel; and 3) The storage period does not affect the bonding effectiveness of etch-and-rinse and self-etch adhesives to enamel.

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METHODS AND MATERIALS

One hundred and twenty freshly extracted bovine incisor teeth were used in this study. The roots were sectioned with a steel diamond disc (KG Sorensen, Rio de Janeiro, Brazil) at the cement-enamel junction. The buccal surfaces were worn using abrasive papers (600 grit) coupled to a circular polishing machine (PA-10, Panambra, São Paulo, Brazil) under water cooling to obtain a 5-mm^2 area of flat enamel.

The teeth were divided into four groups (n=30) according to the surface treatment performed, as follows:

- Group 1 (control): The surfaces were etched for 15 seconds with 37% phosphoric acid gel, rinsed, and dried with air spray for 10 seconds. Two layers of Single Bond/SB total-etch adhesive (3M ESPE, St Paul, MN, USA) were actively applied on the surface for 15 seconds and gently air-dried for 10 seconds. The adhesive was light-activated for 10 seconds with a LED light unit (Emitter A, Schuster, Santa Maria, RS, Brazil) with a power density of 600 mW/cm^2.
- Group 2 (control): The surfaces received the application of Clearfil SE Bond/CSEB self-etch adhesive (Kuraray Medical Inc, Tokyo, Japan). One layer of primer agent was applied actively for 20 seconds and gently air-dried for 10 seconds. One layer of bonding agent was applied actively for 20 seconds and gently air-dried for 10 seconds. The adhesive was light-activated for 10 seconds.
- Group 3 (experimental/laser): The surfaces received the application of SB total-etch adhesive (3M ESPE), following the same protocol used for group 1. Before light polymerization, the surfaces were irradiated with Nd:YAG laser in noncontact mode, scanning for 60 seconds. The adhesive was light-activated for 10 seconds.
- Group 4 (experimental/laser): The surfaces received the application of CSEB self-etch adhesive (Kuraray), following the same protocol used for group 2. Before light polymerization, the surfaces were irradiated with Nd:YAG laser in noncontact mode, scanning for 60 seconds. The adhesive was light-activated for 10 seconds.

Treatment with Nd:YAG Laser

The Nd:YAG laser equipment used in this study was the Laser Pulse Master 600 iQ (American Dental Technologies Inc, Corpus Christi, TX, USA) at a wavelength of 1.064 μm. The output energy of this laser device was 140 mJ per pulse, with a pulse repetition rate of 10 pulses/s (10 Hz) for 60 seconds. The laser was fitted with a noncontact tip 320 μm in diameter and was applied freehand by one calibrated operator in noncontact mode scanning over a 5-mm × 5-mm area of flat enamel. The energy density was 174.16 J/cm^2. During laser application the laser tip was at a 90° angle, perpendicular to the surface, and at a distance of 1 mm from the surface.[9-11]

Restoration Placement

Nanocomposite resin blocks (Filtek Z350, 3M ESPE), approximately 4 mm high, were built on the treated surfaces using a two-piece split Teflon mold. Each 2-mm portion was light-activated for 40 seconds.

The bonded teeth were stored in distilled water (pH=7.0) at 37°C for 24 hours or 12 months.[12-16] The water was changed every week during the course of one year.[15]

The test specimens were cut into parallel sections measuring approximately 1 mm, made from the mesial to the distal and from the cervical to the occlusal surface, using a diamond disc attached to a Labcut 1010 (Extec Technologies Inc, Enfield, CT, USA) cutting machine to obtain sticks, producing a minimum of seven sticks per tooth. The sections were made at low speed under water cooling to prevent stress induction at the bond interface.

The sticks were attached to a microtensile device in a universal testing machine (DL-1000, EMIC, São José dos Pinhais, PR, Brazil) with a 10-kg load cell at a crosshead speed of 0.5 mm/min, in accordance with the ISO 11405 Standard. The bond strength data were expressed in megapascals (MPa).

Statistical Analysis

To the pretest failures (PTFs) and debonded resin blocks, the lowest measured value was assigned.[17] To the cohesive failures (enamel or composite), the specimens were discarded.[17] The mean value for the sticks originating from each tooth was calculated and used for the statistical analysis.

Data (expressed in MPa) were analyzed by three-way analysis of variance (ANOVA; adhesive, technique, and storage time) followed by Tukey test (α=5%).

Scanning Electron Microscopy (SEM) Examination

Two teeth from each group were prepared for SEM analysis. The specimens were sectioned perpendicu-

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Table 1: *Descriptive Analysis for the Different Groups*

Adhesive	Technique	Storage Time	Mean[a]	SD
SB	Control	24 h	36.44 A	6.72
SB	Control	12 mo	35.79 AB	5.50
SB	Laser	24 h	30.19 ABC	5.83
CSEB	Control	24 h	29.38 BCD	3.93
CSEB	Laser	24 h	28.56 CDE	5.67
CSEB	Control	12 mo	23.26 DEF	5.66
SB	Laser	12 mo	22.61 EF	6.63
CSEB	Laser	12 mo	19.24 F	6.13

Abbreviation: SD, standard deviation.
[a] *Means followed by the same letters do not differ statistically* ($p>0.05$).

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larly to the bond interface. The sections were polished with 2000 and 4000 mesh sheets. Phosphoric acid etchant was applied for 15 seconds and then rinsed off with water for 10 seconds. Specimens were dehydrated, sputter-coated with gold-palladium (according to Marimoto and others[10]), and examined by SEM.

RESULTS

The mean bond strength values in all groups are presented in Table 1.

ANOVA revealed that total-etch adhesive presented higher bond strength values compared with self-etch adhesive (p=0.000); the unlased surface treatment presented higher bond strength values compared with the lased surface treatment (p=0.000); and storage in water for 24 hours presented higher bond strength values compared with storage in water for 12 months (p=0.0000).

Table 2 shows the results of the Tukey test for the interaction between the independent variables of adhesive and technique (p=0.0014). The adhesive SB, associated with the control technique, presented significantly higher mean bond strength values when compared with SB associated with the laser technique or with CSEB for both techniques used.

Table 3 shows the results of the Tukey test for the interaction between the independent variables of technique and storage time (p=0.0123). The technique of irradiating the tissue with Nd:YAG laser in the longitudinal time interval (12 months of storage) presented significantly lower mean bond strength values when compared with the technique of irradiating the tissue with Nd:YAG laser in the time interval of 24 hours and the control technique, regardless of the storage time.

For the fracture type, an increase in the occurrence of adhesive fractures in the time interval of 12 months was observed, regardless of the technique used.

Figures 1 through 3 show SEM images obtained of the interfaces created in all of the groups.

DISCUSSION

In this study we used the indirect storage technique (storage of restored teeth) for evaluating the longitudinal bond strength, as in the studies conducted by De Munck and others,[12] Toledano and others,[13] Osorio and others,[14,15] and Abdalla.[16] According to Osorio and others[15] and Pashley and Tay,[18] the water sorption phenomenon in resin-enamel bond occurs over time, inducing resin swelling and weakening of the adhesive joint.

The first null hypothesis was rejected, because the results of the present research showed the superiority of SB compared with CSEB. The 10-methacryl-

Table 2: *Results of Tukey Test (5%) for Interaction Between Factors Adhesive × Technique*

Adhesive	Technique	Mean (SD)	Homogeneous Groups[a]	
Single Bond	Control	35.78 (6.04)	A	
Single Bond	Laser	26.40 (7.25)		B
Clearfil SE Bond	Control	26.32 (5.71)		B
Clearfil SE Bond	Laser	23.90 (7.49)		B

Abbreviation: SD, standard deviation.
[a] *Means followed by the same letters do not differ statistically* ($p>0.05$).

Table 3: *Results of Tukey Test (5%) for Interaction Between Factors Technique × Storage Time*

Technique	Storage Time	Mean (SD)	Homogeneous Groups[a]	
Control	24 h	32.58 (6.49)	A	
Control	12 mo	29.52 (8.38)	A	
Laser	24 h	29.37 (5.71)	A	
Laser	12 mo	20.92 (6.50)		B

Abbreviation: SD, standard deviation.
[a] *Means followed by the same letters do not differ statistically* ($p>0.05$).

oyloxydecyl dihydrogen phosphate (MDP) acidic monomer results in a lower number of ionizable radicals in an aqueous solution, determining a pH ≈ 2.0. However, the phosphoric acid (35-37%) exhibits a large number of ionizable radicals in an aqueous solution, resulting in a pH ≈ 0.6. Thus, the etching capacity of self-etch CSEB to demineralize the substrate is more restricted when compared with that of total-etch SB.[19] CSEB is considered a self-etching adhesive of mild or weak aggressiveness[20] and presents low reactivity with the mineral component. As enamel has a high mineral content, and as a result of the lower availability of H^+ ions to the acidic monomers, these ions may be practically or completely neutralized by the minerals dissolved from enamel before they perform an adequate etching pattern.[21]

In addition, the higher the ionization constant (Ka) value, the greater the force of the acid. Consequently, the greater the acidity, the lower the pKa value of the acid (negative logarithm of the ionization constant). MDP has a pKa = 2.2, with a capacity to dissolve 1.6 g of hydroxyapatite for each gram of MDP acidic monomer. Phosphoric acid has a pKa = 2.0, with a capacity to dissolve 5.1 g of hydroxyapatite for each gram of phosphoric acid. Salz and others[22] explained that the acidic monomers have a high molecular weight in comparison with phosphoric acid, having a negative influence on the capacity of hydroxyapatite dissolution. There-

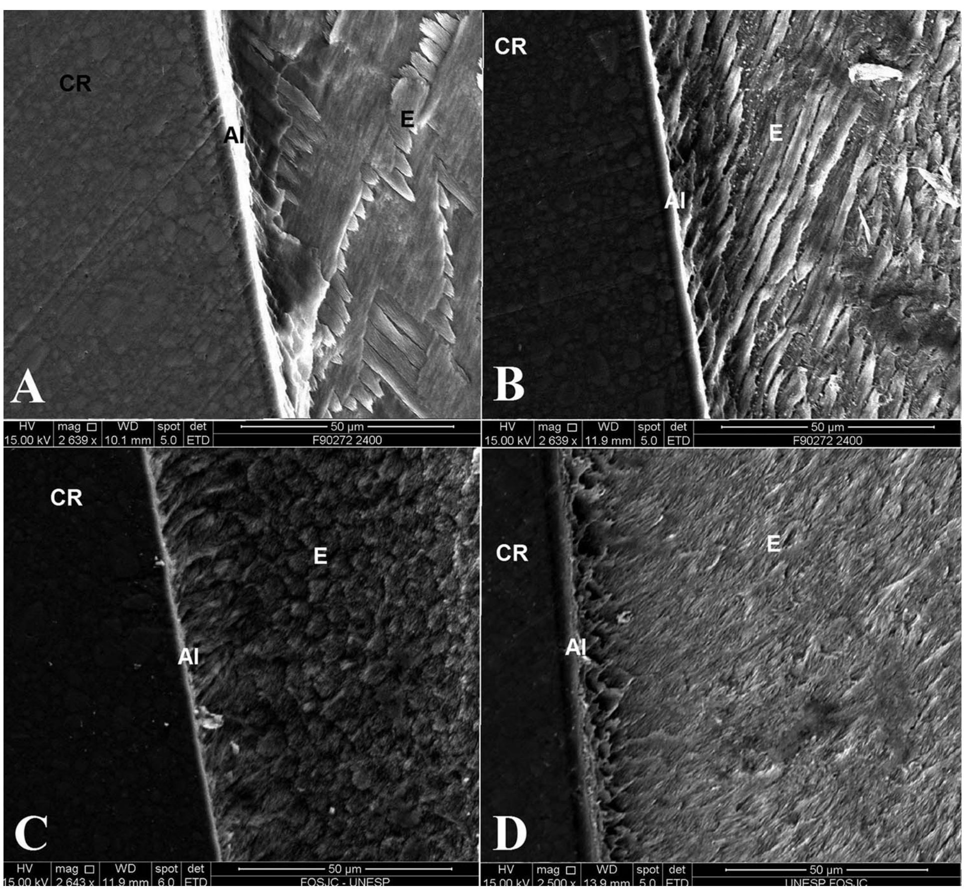

Figure 1. *(A, B) Bond interface that received, respectively, SB and CSEB by control technique and storage for 24 hours. The formation of a thicker bond interface may be observed for the group that received SB; (C, D) Bond interface that received, respectively, SB and CSEB by control technique and storage for 12 months. No areas of degradation of adhesive layer were observed over time. (Legend: CR, composite resin; AI, adhesive interface; E, enamel.)*

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fore, the higher pH and pKa values may explain the lower bond strength values for CSEB compared with SB observed in this study. Total-etch adhesives in enamel are considered the "gold standard," because phosphoric acid is capable of producing a satisfactory enamel etching pattern, resulting in excellent micromechanical entanglement by the tags formed.[23]

When acid etching is deeper and more retentive on enamel, greater penetration of the adhesive into the substrate occurs, promoting a thicker bond interface.[24] As can be observed in Figure 1A, the interface formed by SB is thicker when compared with that formed by CSEB (Figure 1B), which is thinner. This phenomenon likely occurred as a result of the etching attaining the interprismatic, peripheral, and central layers of the enamel prisms,[24] confirming the greater power of action and dissolution of hydroxyapatite by the phosphoric acid in SB.

The second null hypothesis was rejected, because Nd:YAG laser irradiation significantly decreased the bond strength in comparison with the control technique. According to Castro and others,[25] SB is not capable of absorbing wavelengths between 950 and 1100 nm, which includes the wavelength of 1064 nm emitted by the Nd:YAG laser. As the Nd:YAG laser causes a change only when absorbed, this adhesive likely did not undergo any change from the direct action of laser. Moreover, Arrais and others[26] observed that SB and CSEB present absorption spectra of a similar spectral band. It may therefore be speculated that these adhesives presented a similar behavior with regard to the nonabsorption of the wavelength emitted by the Nd:YAG laser.

The results of this study are in contrast to those of Matos and others[9] and Marimoto and others,[10] who observed a significant improvement in the bond strength of enamel impregnated with nonpolymerized adhesives and irradiated with Nd:YAG laser, using 49.76 J/cm^2 and 174.16 J/cm^2, respectively. They believe that this technique would promote the fusion and recrystallization of this tissue in the presence of the adhesive,[8,10] resulting in the formation of a new substrate formed of melted hydroxyapatite and adhesive, and this would be mechanically interlocked and have greater affinity for bonding.[8,10]

However, lower parameters than 174.16 J/cm^2 of Nd:YAG laser irradiation promote chemical and morphological alterations of the enamel surface, such as formation of small bubbles, similar to craters, irregular elevations and fine cracks, and melted and solidified enamel with craters at the surface[27,28]; and generated the formation of areas of decalcification of 15 μm and the formation of craters, with the enamel surface fractured, melted/fused, and recrystallized with a glazelike aspect.[28,29] Ariyaratnam and others[30] believed that the formation of craters, fissures, and fractured enamel could occur as a result of the rapid thermal cycle on the enamel surface during irradiation with Nd:YAG laser. The formation of craters similar to bubbles would be the result of overheating of the enamel surface submitted to subsequent cooling to ambient temperature, and the higher the energy density (ED), the greater are the photothermal effects on the tissues.[30] Fowler and Kuroda[31] explained that even low EDs (between 9 and 120 J/cm^2) could promote slight melting of the enamel surface, which indicates that temperatures $>$1400°C could be attained on the enamel surface. In addition, high EDs promote chemical and structural alterations on the tooth surface, making the irradiated enamel surface more fragile[32] and significantly reducing its surface microhardness.[33]

Therefore, according to the results of this study, it is believed that the photothermal effects promoted by the high ED of the Nd:YAG laser (174.16 J/cm^2) chemically and morphologically changed the enamel surface impregnated with the adhesive systems, with the formation of microcracks, fractured areas, and fissures on the irradiated enamel surface[27-30] and reduction in the microhardness of the enamel surface,[32,33] which have a negative influence on the bonding process, when compared with the control technique.

The third hypothesis was rejected, because significant reductions in resin-enamel bond strengths were observed after 12 months of water storage. The phenomenon of water sorption occurs with the passage of time, inducing an increase in the volume of resin and rupturing the adhesive bonds at the tooth-restoration interface.[15] Both CSEB and SB have water and alcohol as solvents.[34] Poor solvent evaporation during the application of adhesive systems may determine separation of the phases and a lower rate of polymerization, resulting in the formation of weakened interfaces, with increasing susceptibility of the adhesive layer to degradation over the course of time.[15,35]

According to the results of this study, significant differences were observed for the interaction between factors adhesive × technique. Unlased SB presented significantly higher bond strength values when compared with lased SB and compared with lased and unlased CSEB. As previously explained, the higher pH and pKa values of MDP, when

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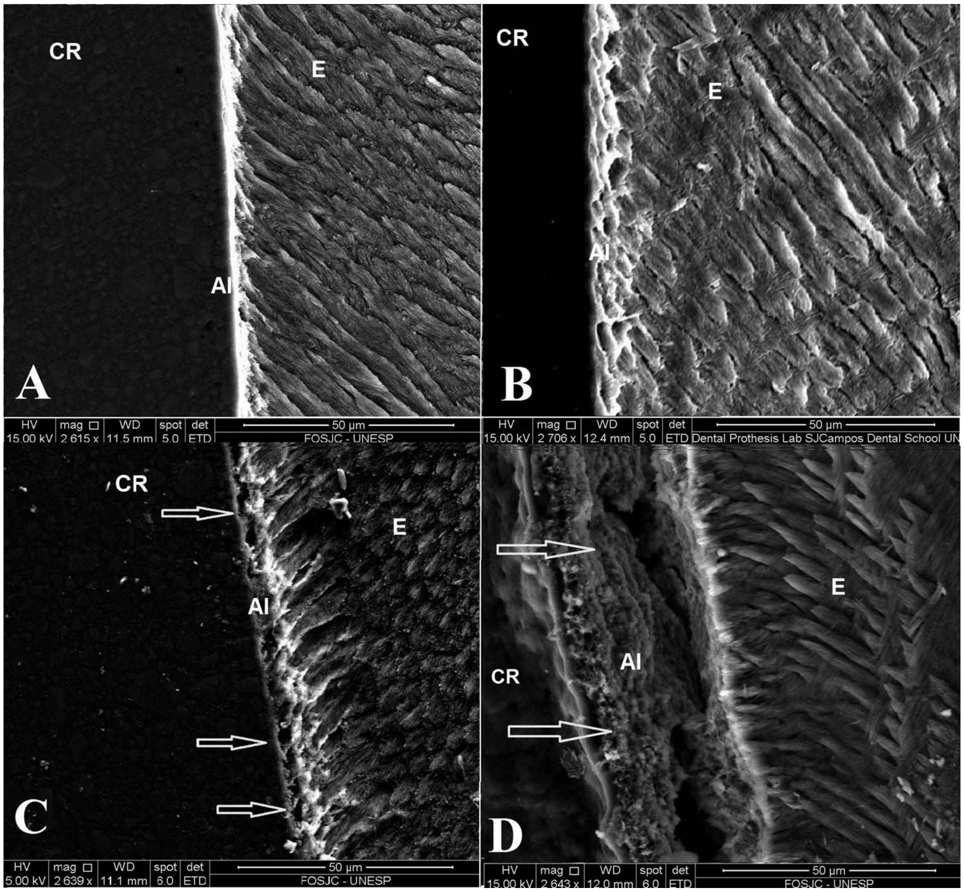

Figure 2. *(A, B) Bond interface that received, respectively, SB and CSEB adhesives by experimental/laser technique and storage for 24 hours; (C, D) Bond interface that received, respectively, SB and CSEB by experimental/laser technique and storage for 12 months. For SB, the presence of gaps in the bond interface (A). For CSEB, we observed the presence of a melted mass in the irradiated substrate (B) (arrows). (Legend: CR, composite resin; AI, adhesive interface; E, enamel.)*

compared with those of phosphoric acid, may explain the lower bond strength values for unlased CSEB compared with unlased SB observed in this study.

Furthermore, regardless of the adhesive used, the lased enamel negatively influenced the bond strength. It is believed that the high ED of Nd:YAG laser on the enamel impregnated with the non-polymerized adhesives may have promoted changes in the morphology of the enamel surface, creating areas with microcracks, fractures, and fissures[27-29] and a reduction in the microhardness of the irradiated enamel surface,[32,33] harming the bond strength as a result of the formation of a more debilitated enamel-resin interface (in comparison to that associated with the control technique).

For the interaction between the variables technique and storage time, lased enamel stored for 12 months presented significantly lower bond strength when compared with lased enamel stored for 24 hours or with unlased enamel stored for 24 hours or 12 months. Oho and Morioka[28] observed that EDs between 67 and 160 J/cm^2 on enamel caused a reduction in the following components: water, carbonate, and organic substances. Theoretically, the reduction of water and organic substances should improve the bond strength longitudinally. The smaller the quantity of organic components (proteins) in the tissue, the less the degradation of the bond interface with the passage of time.[15] Moreover, the smaller the quantity of water in the tissue, the greater the possibility of preventing the action of water on the monomers when the adhesive is light-activated, making the bond interface more stable with the passage of time.[36] However, one of the hypotheses proposed was that the high ED of Nd:YAG laser on the surface of the could have promoted deleterious changes in the morphology[27-29] and reduction in microhardness of the enamel surface,[32,33] which may have contributed to accelerating the degradation, in comparison with the control technique.

As was observed in the SEM images (Figures 2A,B and 3B), there was formation of a bond interface with the characteristics of melting and fusion, with an aspect of "melted lava." These images could indicate that temperatures >1400°C were attained on the enamel surface.[31] According to Lin and

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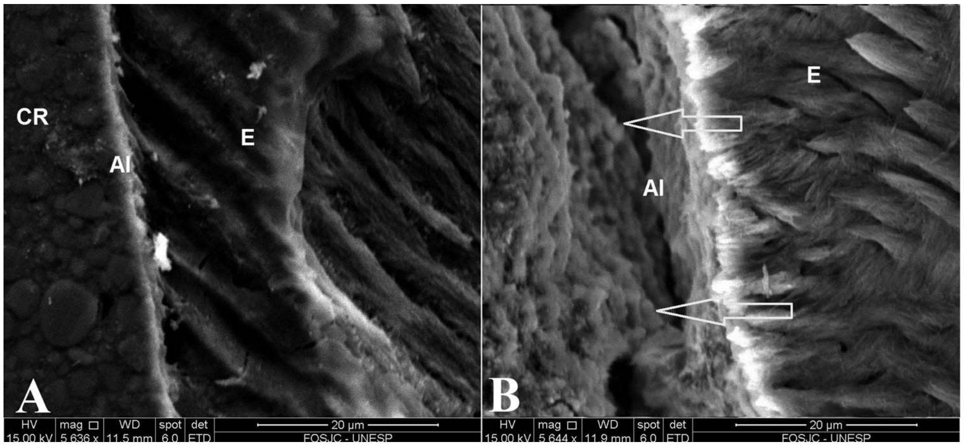

Figure 3. *(A, B) Bond interface that received CSEB by experimental/laser technique and storage for, respectively, 24 hours and 12 months (5700× magnification). For 12 months of storage, complete loss of the bond interface may be observed, as is the presence of a melted mass formed by adhesive and tissue, with formation of a large gap between the enamel and resin (arrows) (B). (Legend: CR, composite resin; AI, adhesive interface; E, enamel.)*

others,[37] temperatures above 1125°C promote the formation of a new crystalline phase, tricalcium phosphate-α [Ca3(PO4)2-α]. Furthermore, Kawasaki and others[38] observed that an ED, as from 100 J/cm^2 of Nd:YAG laser, could produce Ca3(PO4)2-α formation. This new crystalline phase has a higher degree of solubility and degradability than hydroxyapatite, which could reduce the chemical stability of the irradiated tissue[37] and consequently reduce the longitudinal bond strength.

The SEM images clearly illustrate the bond interface modified by Nd:YAG laser in the specimens stored for 12 months. When the bond interface formed by SB (Figure 2C) was exposed to the water for 12 months, we observed a bond interface with various areas of degradation (empty spaces that separated the resin from the tooth enamel) and the presence of subjacent enamel modified by the laser irradiation. At the interface formed by CSEB (Figures 2D and 3A) we observed a complete loss of characterization of the bond interface, with the presence of a melted, hydrolyzed mass and gaps, clearly demonstrating the advanced degradation of this interface.

With regard to the fracture type analysis, Leloup and others[39] explained that adhesive systems with high microtensile bond strength values presented higher rates of cohesive failure, whereas adhesive systems showing low bond strength values after the microtensile test presented higher rates of adhesive failures[39] and, consequently, high rates of pretest failure.[18] The SB/control/24 hours presented the highest bond strength values (36.44±6.72) and the highest number of cohesive failures, which was higher than the number of adhesive fractures, without the occurrence of pretest failure. However, the CSEB/laser/12 months presented the lowest bond strength values (19.24±6.13) and the highest values of adhesive failure and premature failure.

Although the results obtained in this study cannot be directly extrapolated to a clinical situation, it may be suggested that the photothermal mechanism of action of Nd:YAG laser for the high ED used in this study likely promoted alterations at the bond interfaces of the enamel, negatively influencing the bond strength and, consequently, the durability of resin composite restorations.

Further *in vitro* studies are necessary to observe the longitudinal behavior of the bond interface using the irradiation technique with Nd:YAG laser with different parameters from those evaluated in the present study. Different power, energy density, frequency, and scanning time parameters of Nd:YAG laser may modify dental tissues without promoting harmful photothermal effects on the formation of the bond interface.

CONCLUSIONS

According to the methodology used, and based on the results obtained, we may conclude that

- Self-etch adhesive showed reduced bond strength to enamel compared with total-etch adhesive;
- Nd:YAG laser irradiation through unpolymerized adhesives affected bond strength to enamel;
- The 12-month water storage period affected the bonding effectiveness of adhesives to enamel;
- Nd: YAG laser irradiation of enamel through the unpolymerized total-etch adhesive significantly reduced bond strength; and
- Bond strength decreased when enamel irradiated with Nd:YAG laser through the unpolymerized adhesives was stored in water for 12 months.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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