UNIVERSIDADE DE SÃO PAULO
FACULDADE DE ODONTOLOGIA DE BAURU

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Bond strength of adhesive systems irradiated with ER, CR: YSGG laser

Resistência de união de sistemas adesivos irradiados com laser ER, CR: YSGG

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Orientador: Prof. Dr. Sérgio Kiyoshi Ishikiriama

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“Por vezes sentimos que aquilo que fazemos não é senão uma gota de água no mar. Mas o mar seria menor se lhe faltasse uma gota”.

(Madre Teresa de Calcutá)

ABSTRACT

ABSTRACT

Bond strength of adhesive systems irradiated with ER, CR: YSGG laser

The use of high-power lasers has been studied to improve the mechanical and biological properties of the interface of enamel and dentin restorations when irradiated over adhesive systems prior to light-curing. The Er, Cr: YSGG laser has a wavelength of 2780nm, length where the water and hydroxyapatite absorption peak occurs, and it can present a favorable performance when irradiated in adhesive systems applied on dentin. To provide this evidence, this study had the objective of analyzing the adhesion on dentin of different adhesive systems irradiated with the Er, Cr: YSGG laser before light-curing. The experimental design of this study presented two variation factors: laser, divided into two levels (Er, Cr: YSGG [E] and Control - no irradiation [C]) and dentin-bonding system (DBS) divided into four levels (Adper™ Scotchbond Multipurpose [MP], Adper™ Single Bond 2 [SB], Clearfil™ SE Bond [CSE], Single Bond™ Universal [SBU]). The quantitative response variable was the bond strength, obtained through the microtensile test. Eighty human molars (n=10) were obtained and they were cut to expose occlusal dentin area. The specimens were restored according to their respective groups. In the irradiated groups, irradiation of the Er, Cr: YSGG laser was performed after primer or after primer/adhesive on the simplified adhesives. After seven days, the specimens were cut to obtain sticks (0.64 mm^2) and submitted to immediate microtensile test (Instron 3342) at 0.5mm/min. The fracture mode was classified according to the interface analysis in a portable digital microscope 40x. Data presented normal distribution and was analyzed by two-way ANOVA, followed by Tukey test for multiple comparisons ($p<0.05$). Only the adhesive factor presented statistical difference, where in the control group, the SBU adhesive was similar to SB and superior to CSE and MP and, in the laser group, the SBU was similar to SB and superior to CSE and MP which presented the lowest bond strength value. Based on these results, it was concluded that the bond strength variation was dependent on the adhesive systems and the laser irradiation did not affect the performance of dentin bonding systems in the immediate period.

Key words: Dentin-bonding agents. Laser. Tensile strength.

Resumo

RESUMO

Resistência de união de sistemas adesivos irradiados com o laser ER, CR: YSGG

O uso de lasers de alta potência tem sido estudado com o objetivo de melhorar as propriedades mecânicas e biológicas da interface das restaurações em esmalte e dentina, quando irradiado sobre sistemas adesivos previamente à fotopolimerização. O laser Er, Cr: YSGG apresenta um comprimento de onda de 2780nm, comprimento onde ocorre o pico de absorção da água e hidroxiapatita, podendo apresentar uma performance favorável quando irradiado em sistemas adesivos aplicados sobre dentina. Para fornecer esta evidência, este trabalho teve o objetivo de analisar a união à dentina de diferentes sistemas adesivos irradiados com o laser Er, Cr: YSGG antes de sua polimerização. O delineamento experimental desse estudo apresentou dois fatores de variação: laser, dividido em dois níveis (Er, Cr: YSGG [E] e Controle - sem irradiação [C]) e sistema adesivo, dividido em quatro níveis (Adper™ Scotchbond Multipurpose [MP], Adper™ Single Bond 2 [SB2], Clearfil™ SE Bond [CSE], Single Bond™ Universal [SBU]). A variável de resposta quantitativa foi a resistência de união, obtida por meio do teste de microtração. Oitenta molares humanos (n=10) foram obtidos e cortados para expor área de dentina oclusal. Os espécimes foram restaurados de acordo com os seus respectivos grupos e seguindo a orientação do fabricante dos materiais. Nos grupos irradiados, a irradiação do laser Er, Cr: YSGG foi feita após a aplicação do primer ou primer/adesivo nos simplificados. Após sete dias, os espécimes foram cortados para obtenção de palitos (0,64 mm^2) e submetidos ao teste de microtração imediato (Instron 3342) a 0,5mm/min. O modo de fratura foi classificado de acordo com a análise da interface em microscópio digital portátil 40x. Os dados apresentaram distribuição normal e foram analisados pelo teste de análise de variância (ANOVA) a dois critérios, seguido de teste Tukey para comparações múltiplas ($p<0,05$). Apenas o fator adesivo apresentou diferença estatística, sendo no grupo controle o adesivo SBU similar ao SB e superior ao CSE e MP. Nos grupos irradiados, o SBU apresentou valores similares ao SB e superior ao CSE e MP, que apresentou o menor valor de resistência de união. Baseado nesses resultados, conclui-se que a

variação da resistência de união foi dependente dos sistemas adesivos e a irradiação do laser não afetou o desempenho dos sistemas adesivos em dentina no período imediato.

Palavras-chave: Adesivos dentinários. Lasers. Resistência à tração.

LIST OF ILLUSTRATIONS

LIST OF TABLES

TABLE OF CONTENTS

1 INTRODUCTION

1 INTRODUCTION

Over the past century, there has been an increase in demand for aesthetic restorations among patients, growing the use of resin composite. Dental adhesives are designed to bond resin composite to enamel and dentin and are still the target of many researches (VAN LANDUYT et al., 2007). Although the unquestionable evolution of dental adhesives, the vulnerable longevity of composite restorations is still the major clinical challenge, mainly regarding their performance to dentin (MERTZ-FAIRHURST et al., 1998; DE MUNCK et al., 2005; SPENCER et al., 2010).

In a review, Breschi et al. (2008) observed that most of the dentin bonding systems showed favorable immediate results in terms of retention and sealing of bonded interface, which was impaired after a period of six months and one year. These early failures are attributed to events as insufficient resin impregnation of dentin, high permeability of the bonded interface, sub-optimal polymerization, phase separation, activation of endogenous collagenolytic enzymes and hydrolytic degradation. Häfer et al. (2013) showed that despite of the dentin bonding system, esthetics and marginal integrity are compromised over time because of this degradation.

Hydrolytic degradation is mainly caused by remnant water in the hybrid layer, which compromises the satisfactory polymerization of the dental adhesives, leaving open channels for monomer solubilization, known as water trees (TAY; PASHLEY, 2003). On the other hand, the presence of water is essential for dental adhesives, as it provides the expansion of the collagen fibrils allowing dental adhesives to penetrate into dentin (CARVALHO et al., 1996; PASHLEY et al., 2011). In addition, water provides an ionization medium for acidic monomers, which is necessary to allow a satisfactory dentin demineralization and adhesive penetration on self-etch adhesives (TAY; PASHLEY, 2001; GIANINNI et al., 2015).

To reduce the amount of remaining water in the hybrid layer, water solvent of dental adhesives has been partially or totally replaced by ethanol and acetone. These solvents have an increased vapor pressure, which would evaporate faster than water, however, as the amount of solvent decreases through evaporation, increasing the ratio between monomer and water, it decreases the vapor pressure, and remaining water plus solvents are always trapped in the hybrid layer (CARVALHO et al., 2003). The total removal of substrate water is extremely difficult, requiring vacuum and high temperatures (CAMERON et al., 2007).

The incorporation of the ethanol solvent may favor the adhesion to dentin by removing the remaining water. This happened because when placed in a specific amount of water with the solvent they form a mixture of the same composition both in liquid and in gaseous state, called azeotrope. Thus, when the ethanol evaporated, water will evaporate together (YIU et al., 2005; LIDE; HAYNES, 2009).

Among the strategies used to improve adhesion stability in dentin, laser technology has shown some promising results. Initially, laser was proposed to replace the phosphoric-acid-etching of the substrate by determining a micro-retentive surface, without *smear layer* (VISURI et al., 1996). However, some studies demonstrated that laser irradiation did not increase, and even reduced the bond strength when dentin was prepared or conditioned with laser, since that laser created a substrate resistant to adhesive infiltration (DE MUNCK et al., 2002; MANHAES et al., 2005; MALTA et al., 2008; TACHIBANA et al., 2008).

In 1999 Gonçalves et al. introduced a different technique irradiating the laser after adhesive application before light-curing, and obtained promising results, with increased shear bond strength to dentin. Other studies using Nd: YAG laser evaluated the bond strength of two steps etch-and-rinse systems and self-etch systems irradiated with the same technique, also obtaining increased in the bond strength values when adhesives were irradiated with laser (FRANKE et al., 2006; MARIMOTO et al., 2013). Recently, Maenosono et al. (2015) proposed the use of diode laser (970nm), which has the advantage of its portability and low cost. In their study, they evaluated the bond strength of simplified dental adhesive, obtaining increased the bond strength on laser irradiated groups.

Despite the favorable results, the mechanisms that explain why lasers promoted increased bond strength are not well established yet. One of the hypotheses is that the localized heating generated by irradiation could increase water evaporation, which can improve the bond strength values (REIS et al., 2013).

Zabeu et al. (2014) evaluated the diode laser irradiation in non-simplified systems on two situations: after the application of primer and after application of the adhesive, based on the hypothesis that when irradiation is performed after the primer, solvent evaporation would be higher and, there was an increase in bond strength. Their results showed no difference in the bond strength in different irradiation strategies. In the qualitative evaluations made by scanning electronic microscopy (SEM), it was observed that the diode laser has the capacity to increase the size and quantity of the tags. However, Perdigão and Swift Jr. (1994) showed that the size of the tags did not influence the bond strength values because the tags didn't attach at the tubule walls. The factor that has the greatest influence is the thickness of the hybrid layer, which cannot be evaluated by SEM.

A comparative study between the Nd: YAG and diode lasers showed that the Nd: YAG presented the greatest potential to cause pulp damage, however as heating is localized, the risk of pulp damage could be lower (ISHIKIRIAMA et al., 2015). Brianezzi et al. (2016 *in press*) observed a significant increase in the degree of conversion of adhesives when diode laser was irradiated directly on the adhesive system, which could explain the increased bond strength.

Due to this discrepancy in the results observed in the literature, where each laser has a distinct effect on the adhesive systems and different irradiation strategies couldn't improve the bonding of non-simplified adhesive systems, other lasers were tested with this approach.

Because the interaction of the laser with any surface basically depends on the laser wavelength and the absorption peak of the substances involved, this study proposed the use of a laser, which hasn't been tested yet, to improve the adhesion, the Er, Cr: YSGG laser. This laser wavelength is longer than other lasers studied to date (2780 nm). In this wavelength, there are a peak of maximum absorption of water and hydroxyapatite, which could increase energy absorption in the surface, leading to an increased water evaporation (HOKE et al., 1990; VISURI et al., 1996).

Within this context, the aim of this study was to evaluate the bond strength of different adhesive systems irradiated with Er, Cr: YSGG laser on dentin before their light-curing. The tested null hypotheses were: 1- There is no difference on bond strength regarding the adhesives systems to dentin and; 2- There is no difference of bond strength regarding the use of laser irradiation to dentin.

2 Article

2 ARTICLE

The article presented in this Dissertation was written according to the Dental Materials instructions and guidelines for article submission.

Bond strength of adhesive systems irradiated with ER, CR: YSGG laser

Abstract

Objective. This work had the objective of analyzing the adhesion on dentin of different adhesive systems irradiated with the Er, Cr: YSGG laser before the light-curing.

Methods. The experimental design of this study presented two variation factors: laser, divided into two levels (Er, Cr: YSGG [E] and Control - no irradiation [C]) and dentin-bonding system (DBS) divided into four levels (Adper™ Scotchbond Multipurpose [MP], Adper™ Single Bond 2 [SB], Clearfil™ SE Bond [CSE], Scotchbond™ Universal Adhesive [SBU]). The quantitative response variable was the bond strength, obtained through the microtensile test. The specimens were restored according to their respective groups and following the guidance of the materials manufacturer. In the irradiated groups, irradiation of the Er, Cr: YSGG laser was performed after primer application on the non-simplified adhesives and after primer/adhesive on the simplified adhesives. After seven days, the specimens were cut to obtain sticks with an area of approximately $0.64mm^2$ and submitted to immediate microtensile test. The data was analyzed by two-way ANOVA, followed by Tukey test for multiple comparisons ($p<0.05$).

Results. Only the adhesive factor presented statistical difference, where in the control group, the SBU adhesive was similar to SB and superior to CSE and MP and, in the laser group, the SBU was similar to SB and superior to CSE and MP and MP was the lowest value.

Significance. The use of the Er, Cr: YSGG laser to improve the solvent evaporation at higher temperature did not affected the performance of dentin bonding systems in the immediate period.

Key words: Dentin-bonding agents. Laser. Tensile strength.

1. Introduction

Although the unquestionable evolution of dental adhesives, the vulnerable longevity of composite restorations is still the major clinical challenge, mainly regarding their performance to dentin [1-3]

One of the hypothesis that explains this lower longevity is related to hydrolytic degradation of the interface between composite and dentin [4-5]. Hydrolytic degradation is mainly caused by remnant water in the hybrid layer, which prevents the satisfactory polymerization of the dental adhesives, leaving open channels for monomer solubilization, observed by Tay and Pashley [6] as water trees. On the other hand, the presence of water is essential for dental adhesives, as it provides the expansion of the collagen fibrils allowing dental adhesives to penetrate into dentin [4, 7], and, in addition to this, water provides an ionization medium for acidic monomers, which is necessary to allow a satisfactory dentin demineralization and adhesive penetration on self-etch adhesives [8-9].

Among the strategies used to improve adhesion stability in dentin, the laser technology has shown some promising results. Initially, laser was proposed to replace the phosphoric-acid-etching of the substrate by determining a micro-retentive the surface, without *smear layer* [10]. However, many studies demonstrated that laser irradiation did not increase, and even reduced the bond strength to dentin [11-14].

In 1999 some authors introduced a different technique irradiating the laser after adhesive application before the light-curing, and they obtained favorable results, with increased shear bond strength to dentin [15]. Other studies using Nd: YAG laser evaluated the bond strength of two steps etch-and-rinse systems and self-etch systems irradiated with the same technique, also obtaining increased in the bond strength values when adhesives were irradiated with laser [16-17] Recently, Maenosono et al. [18] proposed the use of diode laser (970nm) which has the advantage of its portability and lower cost. In their study, they evaluated the bond strength of simplified dental adhesive, obtaining increased microtensile bond strength on laser irradiated groups.

Despite of the favorable results, the mechanisms that explain why lasers promoted increased bond strength have not been established yet. One of the hypothesis is that the localized heating generated by irradiation can result in water evaporation, increasing the bond strength values [19]. The interaction of the laser with any surface basically depends on the laser wavelength and the absorption peak of the substances involved. Therefore, this study proposed the use of Er, Cr: YSGG laser, which has not been tested yet, to improve the adhesion. This laser wavelength is longer than other lasers studied to date (2780 nm). Hoke et al. [20] and Visuri et al. [21] have shown that in this wavelength there are a peak of maximum absorption of water and hydroxyapatite, which can increase energy absorption in the surface, leading to an increased water evaporation.

Within this context, the aim of this study was to evaluate the bond strength of different adhesive systems irradiated with Er, Cr: YSGG laser on dentin before their light-curing.

2. Materials and Methods

This study was previously approved by the Ethics Committee of Research on Human Beings at Bauru School of Dentistry, University of São Paulo n. 49812415. 1.0000.5417 (Attachment 1).

2.1. Experimental design

This *in vitro* study involved two factors: laser in two levels (Er, Cr: YSGG laser irradiation [E] and no irradiation-Control [C]) and dentin bonding system (DBS) in four levels (Adper^{TM} Scotchbond Multipurpose [MP], Adper^{TM} Single Bond 2 [SB], Clearfil™ SE Bond [CSE], Scotchbond^{TM} Universal [SBU]). The quantitative response variable was the bond strength by means of microtensile test. The failure mode was also analyzed with a portable digital microscopy at 40x magnification.

The tested null hypotheses were: 1- There is no difference on bond strength regarding the tested adhesives systems to dentin and; 2- There is no difference of bonding strength regarding the use of laser irradiation to dentin compared with no laser.

The information of the tested materials is presented in Table 1 and 2.

Table 1 - Commercially available systems, their classification and compositions.

Dentin Bonding	Manufacturer	Classification	Composition
Adper™ Scotchbond Multipurpose	3M ESPE, St Paul, MN, USA	Three-step etch-and-rinse system	***Primer:*** HEMA**, water, copolymer of polyalcenoic acid. ***Bond:*** HEMA**, Big-GMA*** and camphorquinone
Adper™ Single Bond 2	3M ESPE, Sumaré, SP, Brazil.	Two-step Etch-and-rinse system	HEMA**; Bis-GMA***, ethanol; silane treated silica filler; glycerol 1,3 dimethacrylates; diuretanedimethacrylate and copolymer of polyacrylic and polyitaconic acids.
Clearfil SE™ Bond	Kuraray, Okayama, Japan.	Two-step self-etch system	***Primer:*** MDP*; HEMA**; hydrophilic aliphatic dimethacrylate; dl-Camphorquinone; N,N-Diethanol-p-toluidine; Water. ***Bond:*** MDP*; HEMA**; Big-GMA***; hydrophobic aliphatic dimethacrylate; dl-Camphorquinone; N,N-Diethanol-p-toluidine; colloidal silica.
Scotchbond™ Universal	3M ESPE, Seefeld, Germany.	Universal system (used as one-step self-etch system with wet-technique)	MDP*; HEMA**; Bis-GMA***; silica treated silane; ethanol; decamethylenedimethacrylate; water; 1,10-decanediol dimethacrylate; copolymer of polyacrylic and polyitaconic acids; Camphorquinone; N,N-dimethylbenzocaine; methacrylate 2-dimethylmonoethyl; methyl etyl ketone.

* *MDP: 10-methacrylo*yloxydecyl dihydrogen phosphate
** HEMA: 2-hydroxyethyl methacrylate
*** Bis-GMA: Bisphenol A diglycidyl methacrylate

Table 2 - Laser's manufacture and classification

Laser	Manufacturer	Classification
Er, Cr: YSGG laser	Water Lase iPlus, Biolase, Irvine, CA, USA	High power

2.2. Specimen preparation

Eighty molars were used in this study (Attachment 2), which were randomized according to the dimension of exposed dentin area into eight groups (n=10). The crowns were sectioned transversely on the occlusal third to expose dentin using a sectioning machine (Isomet™ Low Speed Saw®, Buehler, Lake Bluff, IL, USA) with water-cooled diamond disc (Extec Corporation, Enfield, CA, USA). Remaining enamel was removed through #320 grit silicon carbide paper (Carbimet Paper Discs, Buehler, Lake Bluff, IL, USA) on a polishing machine (Arotec, Cotia, SP, Brazil). Then, they were polished for 30 seconds with a #600 grit silicon carbide paper to simulate smear layer formation (Figure 1).

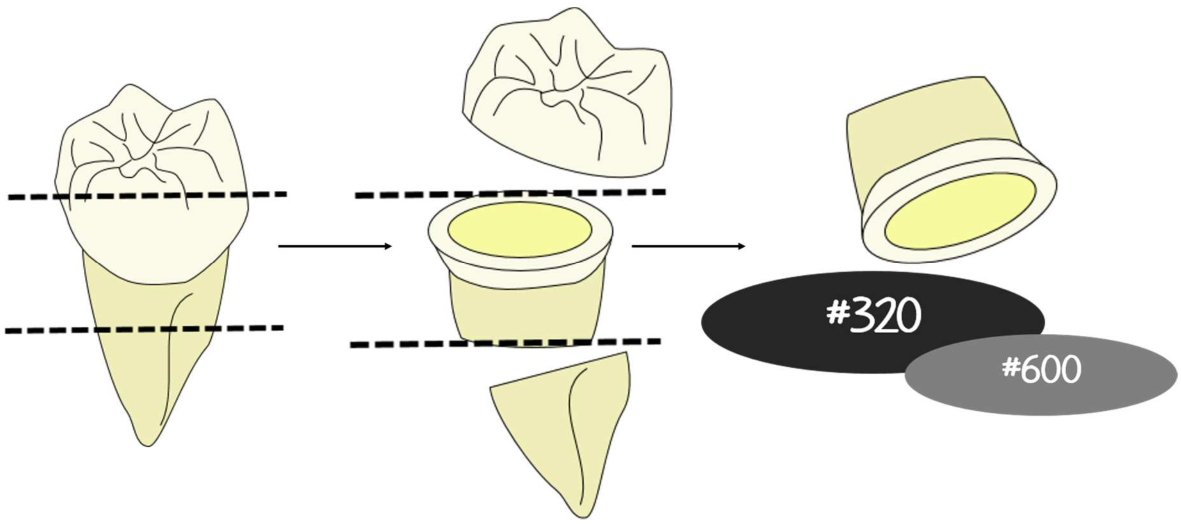

Figure 1 - Specimen preparation – cut and polished specimen.

2.3. Lasers treatments

After the DBS application (Table 3), the Er, Cr: YSGG laser was activated in a distance of 3 mm from the surface substrate (non-contact mode), 90° inclination and by automatic scanning of the area, standardized by XY table (BioPDI, São Carlos, SP, Brazil). The table does an automatic zigzag scan, the displacement of the X axis being determined by the extent of the test area and the displacement of the Y axis based on the thickness of the fiber optical tip (Figure 2). The time was standardized according to the largest specimen, so that the entire dentin area of all specimens received irradiation with the same energy density. The parameters used for laser irradiation are listed in Table 4.

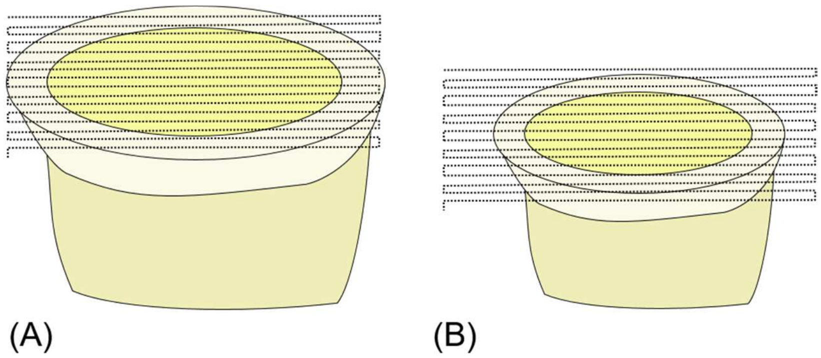

Figure 2 - Standardization design of the trajectory and irradiation time based on the largest area specimen (A). Therefore, all the specimens could receive the same energy density throughout the dentin area (B).

Table 3 - Application technique for each DBS according to its manufacturer's instructions.

Dentin Bonding Systems	**Application technique**
MP	Etch dentin surface with phosphoric acid 37% for 15s Rinse for 15s Gently dry with absorbent paper Apply primer with a microbrush Gently air-dry for 5s Apply adhesive with a microbrush and remove excess
SB	Etch dentin surface with phosphoric acid 37% for 15s Rinse for 15s Gently dry with absorbent paper Apply adhesive with a microbrush and remove excess Gently air-dry for 5s
CSE	Apply primer with a microbrush for 20s Gently dry-air for 5s Apply bond with a microbrush and remove excess
SBU	Apply bond actively for 20s Gently air-dry for 5s

Table 4 - Parameters used for irradiation

Parameter	Value
Energy per pulse (output)	25 mJ
Frequency	10 Hz
Power	0.25 W
Energy density	20.83 J/cm^2
Thickness tip	800 µm

After the irradiation, the specimens were light-cured for 20 seconds using a LED Blue Star 2 (Microdont, São Paulo, SP, Brazil) with 1.000 mW/cm^2 and restored with Filtek Z250 (3M ESPE, St Paul, MN, USA) in three consecutive 1.5 mm increments. Specimens were stored in deionized water at 37°C for 7 days. Then, the specimens were sectioned longitudinally to obtain sticks with approximately 0.64 mm^2 area (Figure 3).

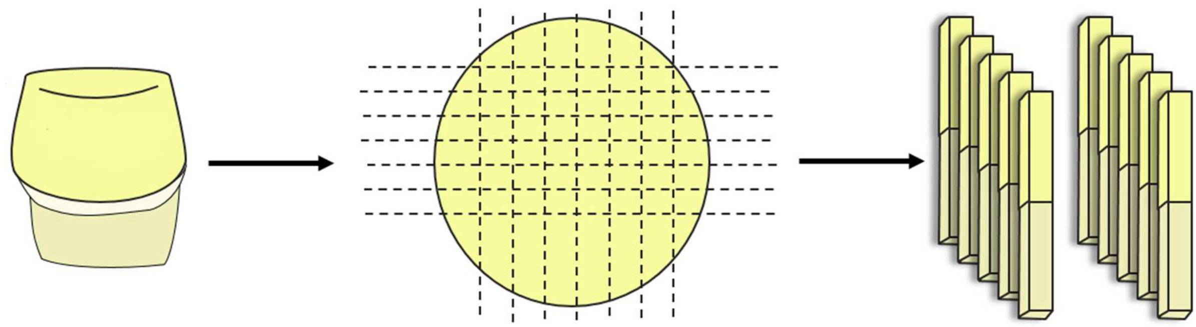

Figure 3 - Restoration using Filtek Z250. After 7 days, dentin/resin sticks of 0.64 mm^2 were obtained and subjected to the microtensile test.

2.4. Microtensile test

The specimens were tested with the universal testing machine Instron 3342 (Illinois Tool Works, Norwood, IL, USA). The cross-sectional area of each specimen was measured with a digital caliper (Digimatic Caliper Absolute, Mitutoyo Corp, Kawasaki, Japan) and the values were inserted into the onboard BlueHill software (BlueHill® Materials Testing Software, Norwood, IL, USA). Then, the sticks were fixed individually with a cyanoacrylate-based adhesive (Loctite Super Bonder Gel Control, Henkel Ltda, São Paulo, SP, Brazil) into the machine's dispositive (JIG 1 Microtensile, Odeme, Santa Catarina, Brazil). The adhesive interface was positioned perpendicular to the tensile forces generated by the testing machine. The tension was applied at a constant speed of 0.5 mm/min, with maximum load of 500 N that measured in Newtons (N) the force required to break the stick.

2.5. Failure mode analysis

Both segments of the fractured specimens were evaluated to define the type of the failure with a portable digital microscope (Dino Lite Microscope Plus, AnMo Electronics Corp, New Taipei City, Taiwan) at 40x magnification and classified by failure modes: Adhesive (A); Cohesive in Dentin (CD); Cohesive in Resin (CR); and Mixed (M). The percentage of each type of failure was obtained.

3. Statistical analysis

Data was calculated and analyzed statistically with Statistica software (Statsoft®, Tulsa, OK, USA). The assumptions of normal distribution and of equality of variances were checked for all the variables using Kolmogorov-Smirnov and Levene test, respectively. As the assumptions were satisfied, data was subjected to two-way ANOVA ($p<0.05$) followed by Tukey's test ($p<0.05$) for individual comparisons.

4. Results

Bond strength means and standard deviations are summarized in Table 5. Data revealed significance only for DBS factor. There were no significant differences related to laser factor.

Regarding the DBS factor, in the control group the SBU presented the highest bond strength value, being statistically different to CSE and MP and similar to SB. In the laser group, the SBU also presented the highest bond strength value, being like SB and statistically different to CSE and MP. SB was similar to CSE, and MP was different to all DBS, presenting the lowest value.

The most predominant failure mode for all groups was adhesive and mixed. Description of the distribution is presented in table 6.

Table 5 - Microtensile bond strength values (MPa)

DBS	Control	Er, Cr: YSGG
MP	34.23 ± 2.22 Ab	30.43 ± 2.67 Ac
SB	39.43 ± 2.74 Aab	42.59 ± 6.04 Aab
CSE	36.13 ± 4.27 Ab	38.93 ± 2.27 Ab
SBU	42.45 ± 4.87 Aa	46.62 ± 4.49 Aa

Upper case letters indicate differences between column in the same row.
Lower case letters indicate differences between raw in the same column.

Table 6 - Failure modes distribution (%)

Groups	A	M	CD	CR
MP-C	81%	13%	4%	2%
SB-C	79%	9%	3%	9%
CSE-C	74%	16%	4%	6%
SBU-C	78%	14%	4%	4%
MP-E	71%	16%	6%	7%
SB-E	75%	12%	8%	5%
CSE-E	80%	10%	5%	5%
SBU-E	81%	8%	6%	5%

5. Discussion

The present study investigated a high-power laser treatment for the dentin substrate, to improve the adhesive interface hybridization in different DBS. The results reject the first null hypothesis, since there were statistical differences between the adhesives, independently of the treatment. The highest bond strength value was obtained by the SBU adhesive. In a systematic review and meta-analysis by Rosa et al. [22], the studies that used the same trademark and similar aspects of mode and time of storage presented values of bond strength similar to the findings of this study, where the bond strength in dentin varied between 32.30 MPa [23] and 44.00 MPa [24] in the self-etch mode.

Its best performance may be related to its composition, mainly 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), a phosphate acidified functional monomer that allows a stable chemical bond with the hydroxyapatite [25]. The nano-layer interface between hydroxyapatite and MDP presents multifunctional properties, such as the durability of nano-layering, protection of the hybrid layer by its hydrophobic nature, protection of collagen against degradation and hydroxyapatite strength to acid dissolution [26].

In addition, the SBU presents ethanol in this composition. Ethanol was included as a solvent to facilitate the penetration of resin monomers on the dentin surface [27] and allows azeotrope phenomenon, a combination of substances which, when under a certain pressure, have the same composition in the liquid and gaseous state, resembling a pure substance. Thus, when ethanol evaporates, water will evaporate together [28-29].

Despite these advances in adhesive materials, water remains retained in the dentin and the hydrolytic degradation of the hybrid layer continues to occur over time [3]. Several studies have tested different strategies for evaporation of solvents present in DBS. Argolo et al. [30] evaluated the prolonged solvent evaporation time in the micro-shear bond strength and degree of conversion to ethanol based adhesive. According to the results, 60 seconds drying between the application of the adhesive and the light curing significantly increased the bond strength and degree of conversion. Another study evaluated the use of hot and cold air for the evaporation of solvents from the Adper Single Bond 2 and Prime & Bond 2.1 (Dentsply) adhesives based on ethanol/water and acetone, respectively. Based on the results, they concluded that the hot air flow can improve the bond strength and decrease the nanofiltration, since it reduced the number of pores inside the hybrid layer [31]. Matuda et al. [32] also observed that the increase in temperature allied to a pre-treatment of dentin with proanthocyanidins improved solvent volatilization and mechanical properties after six months.

Considering the results observed in the mentioned studies, it is noted that strategies for solvent evaporation seem to improve the performance of the bond interface. This study used Er, Cr: YSGG laser irradiated on dentin previously impregnated with adhesive system. The hypothesis was that the laser, which has the same wavelength (2780nm) than water and the hydroxyapatite absorption peak, could be able to eliminate the solvents and water present in the substrate with increasing surface temperature. Based on the results, it was not possible to observe a difference in the values of bond strength between the control and laser groups, affirming the second hypothesis of this study.

The findings of this study are contrary to previous studies that have suggested that the laser can improve bond strength. Maenosono et al. [18] used the diode laser and observed an increase in bond strength of the simplified adhesive systems. Similar results were previously observed with the Nd: YAG laser, which presents a wavelength close to diode laser [16-17]. Despite these favorable results, any study could prove the effect of laser irradiation on dentin treated with the adhesive system.

A hypothesis raised in the studies would be that the increase in surface temperature could evaporate the solvents and water present in the substrate [18]. Recently, Batista et al. [33] evaluated the effect of Nd: YAG laser on solvent evaporation in total-etch and self-etch adhesives and concluded that the irradiation with the Nd: YAG laser increased the evaporation of the solvent in both adhesives tested.

The fact that the Er, Cr: YSGG laser does not decrease the values of bond strength, shows that it didn't interfere negatively on the bond interface. However, only by the immediate bond strength test we cannot say that the laser was not able to evaporate the solvent and create an adhesive interface with better performance, since durability is more important than higher bond strength values.

Based on the knowledge acquired in this study, it is possible to state that the Er, Cr: YSGG laser did not influence the performance of adhesive systems tested in the immediate bond strength.

6. Conclusion

Based on the results, it was concluded that the irradiation with the Er, Cr: YSGG laser in adhesive systems before light curing applied on the dentin didn't influence the bond strength, independent of the adhesive system tested.

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3 Discussion

3 DISCUSSION

The main objective of Restorative Dentistry is to guarantee an adhesive interface with durability and maximum sealing, reducing the risks of microleakage. However, besides of the evolution of adhesive materials, dentin restoration continues to present the highest failure index due to its complex composition (SPENCER et al., 2010). Therefore, the present study investigated a high-power laser treatment for the dentin substrate to improve the adhesive interface hybridization in different DBS.

The results of this laboratory test reject the first null hypothesis, since there were statistical differences between the adhesives, independently of the treatment. The highest bond strength value was obtained by the SBU adhesive. In a systematic review and meta-analysis by Rosa et al. (2015), the studies that used the same trademark and similar aspects of mode and time of storage presented values of bond strength similar to the findings of this study, where the bond strength in dentin varied between 32.30 MPa (LUQUE-MARTINEZ et al., 2014) and 44.00 MPa (WARGNER et al., 2014) in the self-etch mode.

Its best performance may be related to its composition, mainly 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), a phosphate acidified functional monomer that allows a stable chemical bond with the hydroxyapatite (YOSHIHARA et al., 2011). The nano-layer interface between hydroxyapatite and MDP presents multifunctional properties, such as the durability of nano-layering, protection of the hybrid layer by its hydrophobic nature, protection of collagen against degradation and strength to the hydroxyapatite against to acid dissolution (YOSHIDA et al., 2012).

In addition, the SBU presents ethanol in its composition. As mentioned in the literature review, ethanol was included as a solvent to facilitate the penetration of resin monomers on the dentin surface (CARVALHO et al., 2003) and allows azeotrope phenomenon, a combination of substances which, when under a certain pressure, have the same composition in the liquid and gaseous state, resembling a pure substance, favoring the evaporation of water that is attached to ethanol (YIU et al., 2005; LIDE; HAYNES, 2009).

Despite these advances in adhesive materials, water remains retained in the dentin and the hydrolytic degradation of the hybrid layer continues to occur over time

(SPENCER et al., 2010). Several studies have tested different strategies for evaporation of solvents present in DBS. Argolo et al. (2012) evaluated the micro-shear bond strength and degree of conversion to ethanol-based adhesive prolonging the solvent evaporation time. According to the results, 60 seconds drying between the application of the adhesive and the light-curing increased significantly the bond strength and degree of conversion. Another study evaluated the use of hot and cold air for the evaporation of solvents from the Adper Single Bond 2 and Prime & Bond 2.1 (Dentsply) adhesives based on ethanol/water and acetone, respectively. Based on the results, they concluded that the hot air flow can improve the bond strength and decrease the nanoinfiltration, since it reduced the number of pores inside the hybrid layer (KLEIN-JUNIOR et al., 2008). Matuda et al. (2016) also observed that the increase in temperature allied to a pre-treatment of dentin with proanthocyanidins improved solvent volatilization and mechanical properties after six months.

Considering the results observed in the mentioned studies, it is noted that strategies for solvent evaporation seem to improve the performance of the bond interface. This study used Er, Cr: YSGG laser irradiated on dentin impregnated with adhesive system before light-curing. The hypothesis was that the laser, which has the same wavelength (2780nm) than water and the hydroxyapatite absorption peak, could be able to eliminate the solvents and water present in the substrate with the increasing of the surface temperature. Based on the results, it was not possible to observe a difference in the values of bond strength between the control and laser groups, accepting the second null hypothesis of this study.

The Er, Cr: YSGG laser had not been studied yet in Dentistry with the aim of improving the DBS performance. Due to its ability to promote the mechanism of ablation of hard tissues, since its high energy is absorbed by the tissues, vaporizing the water and the hydrated components, its use was initiated with the intention of removing hard dental tissue (RIZOIU; DESHAZER, 1994). However, studies have shown that the Er, Cr: YSGG laser could cause morphological changes in the structure such as the creation of a rough surface without the presence of smear layer, open dentinal tubules, fissures and cracks of peritubular dentin (HOSSAIN et al., 1999; MORETTO et al., 2010). In addition, Cardoso et al. (2008) used laser for cavity preparation and observed a significant decrease in bond strength in different adhesive systems.

Since then, Er, Cr: YSGG began to be investigated in other situations, such as in the conditioning of dentin to improve the adhesion quality of glass ionomer cements (GARBUI et al., 2013), to prevent enamel and dentin demineralization (RAMALHO et al., 2015) and additional laser treatments were proposed to improve the quality of the restoration interface (CARVALHO et al., 2011).

The findings of this study are contrary to previous studies that have suggested that the laser can improve bond strength. Maenosono et al. (2015) used the diode laser and observed an increase in bond strength of the simplified adhesive systems. Similar results were observed previously with the Nd:YAG laser, which presents a wavelength close to diode laser (FRANKE et al., 2006; MARIMOTO et al., 2012). Despite of these favorable results, no study could prove the effect of laser irradiation on dentin treated with the adhesive system. One of the hypotheses is that the laser irradiation on the dentin and adhesive creates a new substrate where the hydroxyapatite and the adhesive fuse by the laser action (MARIMOTO et al., 2012). However, Zuerlein et al. (1999) stated that the melt of hydroxyapatite occurs at very high temperatures (about 800°C). Pilot studies carried out by this work team evaluated the temperature that the substrate can reach with the diode and Er, Cr: YSGG lasers through the thermography test and the results showed that the diode laser cannot reach more than 6ºC on the surface while the Er, Cr: YSGG laser can reach a maximum temperature of 130°C on the surface, not being able to reach the temperature to occur the surface melt.

Another hypothesis suggested in the studies is that the increase in surface temperature may evaporate the solvents and water present in the substrate (MAENOSONO et al., 2015). Recently, Batista et al. (2015) evaluated the effect of Nd: YAG laser on solvent evaporation in total-etch and self-etch adhesives. In the control group, the solvent was evaporated spontaneously for 5 minutes and in the laser group it was irradiated for 1 minute, followed by evaporation spontaneous for 4 minutes. The mass was measured every 10 seconds in the first 2 minutes and the rest of the time for every minute. The obtained results showed that the irradiation with the Nd: YAG laser increased the evaporation of the solvent in the two adhesives tested.

The fact that the Er, Cr: YSGG laser do not decrease the values of bond strength shows that it did not interfere negatively on the bond interface. However, only by the immediate bond strength test it not cannot be said that the laser was not able to evaporate the solvent and create an adhesive interface with better performance, since durability is more important than high bond strength values. The analysis in the immediate form presents itself in a valid way by bringing information about the resistance capacity of adhesive materials in relation to gap formation (BURROW et al., 1994). However, this analysis is not sufficient to conclude that the laser has no effect on the substrate. Further studies must be performed after a period of storage so that values can be related to the clinical performance of the restorations in the long term (DE MUNCK et al., 2012).

Based on the knowledge acquired in this study it is possible to state that the Er, Cr: YSGG laser did not influence the performance of adhesive systems tested in the immediate bond strength. Further studies which consider the longevity of the restoration and other morphological and chemical aspects must be carried out for a better understanding of the phenomenon that occurs in the adhesive interface irradiated with laser.

4 Final Considerations

4 FINAL CONSIDERATIONS

Based on the results, it was concluded that the irradiation with the Er, Cr: YSGG laser in adhesive systems before light-curing applied on the dentin did not influence the bond strength independent of the adhesive system tested.

Thus, the null hypotheses of this study must be:

1. Rejected, as the DBS presented different values of bond strength, regardless of the treatment being the SBU adhesive with highest bond strength.

2. Accepted, since there was no statistical difference between the groups with irradiation and without irradiation independent of the DBS used.

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ATTACHMENTS

ATTACHMENT A – Approval by the Ethics Committee of Research on Human Beings at Bauru School of Dentistry

FACULDADE DE ODONTOLOGIA DE BAURU-USP

PlataForma Brasil

PARECER CONSUBSTANCIADO DO CEP

DADOS DO PROJETO DE PESQUISA

Título da Pesquisa: Avaliação in vitro da resistência de união de sistemas adesivos com MDP irradiados com LASERs de alta potência

Pesquisador: Giovanna Speranza Zabeu

Área Temática:

Versão: 1

CAAE: 49812415.1.0000.5417

Instituição Proponente: Faculdade de Odontologia de Bauru

Patrocinador Principal: Financiamento Próprio

DADOS DO PARECER

Número do Parecer: 1.292.361

Apresentação do Projeto:

Trata-se de um projeto intitulado "Avaliação in vitro da resistência de união de sistemas adesivos com MDP irradiados com LASERs de alta potência", de autoria de Giovanna Speranza Zabeu, orientação do Prof. Dr. Sérgio Kiyoshi Ishikiriama. Segundo a pesquisadora, o mecanismo de ação do LASER e como ele pode interferir sobre os agentes do sistema adesivo ainda não estão bem estabelecidos. Para fornecer estas evidências, esse projeto de pesquisa irá avaliar e comparar o LASER de Diodo com um LASER com comprimento de onda próximo ao da água, o Er,Cr,YSGG, para verificar a interação com os solventes e evaporação da água. Para tal a pesquisadora se utilizará de 99 molares humanos que serão doados a pesquisadora por cirurgiões dentistas de clínicas particulares, cujo modelo de termo de doação/cessão se encontra anexado para análise.

Objetivo da Pesquisa:

O presente projeto de pesquisa tem como objetivo avaliar a resistência de união à dentina de sistemas adesivos que apresentam MDP (metacriloxidecil, dihidrogênio e fosfato), em sua composição irradiados com laser de Diodo e laser Er,Cr,YSGG após a aplicação do adesivo antes da fotopolimerização.

Avaliação dos Riscos e Benefícios:

Riscos:

Endereço: DOUTOR OCTAVIO PINHEIRO BRISOLLA 75 QUADRA 9
Bairro: VILA NOVA CIDADE UNIVERSITARIA **CEP:** 17.012-901
UF: SP **Município:** BAURU
Telefone: (14)3235-8356 **Fax:** (14)3235-8356 **E-mail:** cep@fob.usp.br

FACULDADE DE ODONTOLOGIA DE BAURU-USP

Continuação do Parecer: 1.292.361

Segundo a pesquisadora, não há nenhum risco, por ser uma pesquisa laboratorial, não envolvendo de forma direta os participantes da pesquisa.
Benefícios:
Espere-se haver benefícios após os resultados obtidos na pesquisa para a população.

Comentários e Considerações sobre a Pesquisa:
Trata-se de uma pesquisa bem interessante que poderá contribuir para a população em geral, quando, por ocasião de um eventual tratamento dentário, fizer uso de sistemas adesivos com MDP irradiados com LASERs de alta potência. A pesquisadora ainda não possui os dentes para a sua pesquisa, mas apresentou o modelo do termo de cessão de dentes que deverão ser assinados pelos cirurgiões dentistas que doarão os dentes para a pesquisadora. Ela justificou a contento a dispensa do TCLE e informou que pretende destruir as sobras dos dentes ao final da pesquisa. Não existe nenhum problema ético que torna a pesquisa inviável.

Considerações sobre os Termos de apresentação obrigatória:
Foram apresentados todos os documentos necessários para que a pesquisa fosse avaliada do ponto de vista ético.

Recomendações:
Não se aplica.

Conclusões ou Pendências e Lista de Inadequações:
Trata-se de uma pesquisa bem interessante que poderá contribuir para a população em geral, quando, por ocasião de um eventual tratamento dentário, fizer uso de sistemas adesivos com MDP irradiados com LASERs de alta potência. A pesquisadora ainda não possui os dentes para a sua pesquisa, mas apresentou o modelo do termo de cessão de dentes que deverão ser assinados pelos cirurgiões dentistas que doarão os dentes para a pesquisadora. Ela justificou a contento a dispensa do TCLE e informou que pretende destruir as sobras dos dentes ao final da pesquisa. Não existe nenhum problema ético que torna a pesquisa inviável, razão pela qual sou de parecer pela aprovação da pesquisa.

Considerações Finais a critério do CEP:
Esse projeto foi considerado APROVADO na reunião ordinária do CEP de 21.10.2015, com base nas normas éticas da Resolução CNS 466/12. Ao término da pesquisa o CEP-FOB/USP exige a

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ATTACHMENT B – Term of assignment of teeth by private practice

TERMO DE CESSÃO DE DENTES - Cirurgião-dentista

Eu, Marina Alves Lara, cirurgião-dentista, inscrito no CRO 112.542, com consultório situado na Rua São João nº 783, bairro Alto, cidade Piracicaba, UF SP CEP 13416-585, cedo o(s) 80 dente(s) permanente(s) para o desenvolvimento da pesquisa **"Avaliação da resistência de união de sistemas adesivos irradiados com o laser ER, CR: YSGG"**, de autoria de *Giovanna Speranza Zabeu*, sob orientação do *Prof. Dr. Sérgio Kiyoshi Ishikiriama*, declarando que este(s) dente(s) foi(ram) extraído(s) por indicação terapêutica, cujos históricos fazem parte dos prontuários dos pacientes de quem se originam, arquivados sob minha responsabilidade. Estou ciente de que o(s) dente(s) será(ão) utilizado(s) para a referida pesquisa a qual deverá ter sido previamente aprovada por um Comitê de Ética em Pesquisa, sendo preservada minha identidade na divulgação do trabalho.

DECLARO, cumprir as exigências contidas na Resolução 466/12.

Piracicaba, 01 de Junho de 2016.

Nome/Assinatura do cirurgião-dentista

Marina Alves Lara

Dra. Marina Alves Lara
Cirurgiã - Dentista
CRO-SP 112.542