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Effect of Er, Cr: YSGG laser irradiation in the physics properties of different dentin bonding systems

Efeito da irradiação do laser de Er, Cr: YSGG nas propriedades físicas de diferentes adesivos dentinários

Efecto de la irradiación con láser Er, Cr: YSGG em las propriedades físicas de diferentes recubrimientos dentinarios

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Leticia Ferreira de Freitas Brianezzi
ORCID: https://orcid.org/0000-0002-9488-9140
University of São Paulo, Brazil
E-mail: leticia.brianezzi@hotmail.com
Giovanna Speranza Zabeu
ORCID: https://orcid.org/0000-0001-5044-6690
University of São Paulo, Brazil
E-mail: giovanna.zabeu@gmail.com
Regina Guenka Palma Dibb
ORCID: https://orcid.org/0000-0002-3247-0248
University of São Paulo, Brazil
E-mail: rgpalma@gmail.com
Patricia Aparecida Ana
ORCID: https://orcid.org/0000-0003-2857-7517
University Federal do ABC, Brazil
E-mail: patricia.ana@ufabc.edu.br
Linda Wang
ORCID: https://orcid.org/0000-0001-6308-2769
University of São Paulo, Brazil
E-mail: wang.linda@usp.br
Sérgio Kiyoshi Ishikiriama
ORCID: https://orcid.org/0000-0003-0064-1783
University of São Paulo, Brazil
E-mail: serginho@usp.br

Abstract
The aim of this study was to evaluate the effect of Er, Cr: YSGG laser irradiation in the physics properties of the different dentin bonding systems (DBS) applied in dentin substrate.This study presented two variation factors: laser, divided into two levels (Er, Cr: YSGG [L] and Control - not irradiated [C]) and DBS divided into four levels (Adper™ Scotchbond Multipurpose [MP], Adper™ Single Bond 2 [SB], Clearfil™ SE Bond [CSE], Scotchbond™ Universal Adhesive [SU]). The response variables were water sorption/solubility (μg/mm^3) [WS/WSB], degree of conversion (%) [DC], microtensile bond strength (MPa) [MS] and scanning electron microscopy [SEM] for descriptive analysis. After application of DBS over dentin substrate and before curing, in the L groups the irradiation was performed over the primer on MP and CSE and after primer/adhesive on SB and SU. After 7 days, the specimens were cut to obtain squared-shape blocks for WS/WSB, slices for one-year DC/MEV and sticks for one-year MS. The data was analyzed by two-way ANOVA, followed by Tukey test for multiple comparisons ($p<0.05$).All irradiated groups presented lower WS/WSB than respective control groups. For DC, it was noted a significant difference only for DBS factor. However, laser irradiation promoted a significant drop in MS values, except for SU-L group. In SEM, greater penetration depth of resin tags were found for total-etch DBS, especially when laser irradiated.
Keywords: Dentin bonding agents; Lasers; Dentin.

Resumo
O objetivo deste estudo foi avaliar o efeito da irradiação do laser Er, Cr:YSGG nas propriedades físicas de diferentes sistemas adesivos dentinários (DBS) aplicados em dentina.Este estudo apresentou dois fatores de variação: laser, dividido em dois níveis (Er, Cr: YSGG [L] e Controle - não irradiado [C]) e DBS dividido em quatro níveis (Adper™ Scotchbond Multipurpose [MP], AdperTM Single Bond 2 [SB], Clearfil™ SE Bond [CSE], Scotchbond™ Universal Adhesive [SU]). As variáveis de resposta foram: sorção/solubilidade de água (μg/mm^3) [WS/WSB], grau de conversão (%) [DC], resistência de adesiva à microtração (MPa) [MS] e microscopia eletrônica de varredura [SEM]

para análise descritiva. Após a aplicação do DBS sobre o substrato dentinário e antes da polimerização, a irradiação foi realizada, nos grupos L, sobre o primer em MP e CSE e após primer/adesivo em SB e SU. Após 7 dias, os espécimes foram cortados para obter blocos quadrados para WS/WSB, fatias para DC/MEV de um ano e palitos para MS de um ano. Os dados foram analisados por ANOVA two-way, seguido do teste de Tukey para comparações múltiplas ($p<0,05$). Todos os grupos irradiados apresentaram menor WS/WSB do que os respectivos grupos de controle. Para DC, notou-se diferença significativa apenas para o fator DBS. No entanto, a irradiação com laser promoveu uma queda significativa nos valores de MS, exceto para o grupo SU-L. Em MEV, maior profundidade de penetração de tags de resina foi encontrada para DBS de condicionamento total, especialmente quando irradiado com laser.
Palavras-chave: Adesivos dentinários; Lasers; Dentina.

Resumen
El objetivo de este estudio fue evaluar el efecto de la irradiación con láser Er, Cr: YSGG en las propiedades físicas de los diferentes sistemas de unión a dentina (DBS) aplicados en el sustrato de dentina. Este estudio presentó dos factores de variación: láser, dividido en dos niveles (Er, Cr: YSGG [L] y Control - no irradiado [C]) y DBS dividido en cuatro niveles (Adper™ Scotchbond Multipurpose [MP], Adper™ Single Bond 2 [SB], Clearfil™ SE Bond [CSE], Scotchbond™ Universal Adhesive [SU]). Las variables de respuesta fueron: sorción de agua/solubilidad (μg/mm^3) [WS/WSB], grado de conversión (%) [DC], fuerza de unión a la microtracción (MPa) [MS] y microscopía electrónica de barrido [SEM] para el análisis descriptivo. Después de la aplicación de DBS sobre el sustrato de dentina y antes del curado, en los grupos L la irradiación se realizó sobre el primer en MP y CSE y después del primer/adhesivo en SB y SU. Después de 7 días, los especímenes se cortaron para obtener bloques de forma cuadrada para WS/WSB, rebanadas para DC/MEV de un año y barras para MS de un año. Los datos se analizaron mediante ANOVA de dos vías, seguido de la prueba de Tukey para comparaciones múltiples ($p<0,05$). Todos los grupos irradiados presentaron menor WS/WSB que los respectivos grupos de control. Para DC, se observó una diferencia significativa solo para el factor DBS. Sin embargo, la irradiación con láser promovió una caída significativa en los valores de MS, excepto para el grupo SU-L. En SEM, se encontró una mayor profundidad de penetración de las etiquetas de resina para DBS de grabado total, especialmente cuando se irradia con láser.
Palabras clave: Recubrimientos dentinarios; Rayo láser; Dentina.

1. Introduction

The heterogeneity of dentin limited the adhesive bonding procedures; the same water required to keep the collagen fibers network open for resin penetration (Pashley et al., 2011; Van Meerbeek et al., 2011) limited the degree of conversion. Furthermore, this water promoted hydrolytic degradation of collagen matrix and dentin bonding system (DBS), compromising the durability of the bond strength over the time. (Tjäderhane et al., 2013; Pashley et al., 2007; Tjäderhane L, 2015). Biodegradation of the adhesive interfaces also increases bacterial microleakage (Kermanshahi et al., 2010) leading to secondary caries.

To improve the performance of the different DBS, some methods are being investigated to try to eliminate this water/solvent present in the hybrid layer after the application of the DBS, such as heating the DBS with heated air, (Bail et al., 2012) increasing the evaporation time and irradiation of different types of lasers as a form of heating (Batista et al., 2015). Some studies (Bail et al., 2012; Vale et al., 2014; Castro et al., 2013] have reported that the increase of temperature aid in the solvent evaporation of the DBS and improve the bonding strength.

Lasers have been used to improve the bond strength of DBS, specially one bottle DBS. The method described by Gonçalves et al. 1999 have been showed promising results for diode and Nd: YAG lasers. In these methods the DBS is first applied on dentin and irradiation occur previously light polymerization. (Goncalves et al.,1999; Franke et al., 2006; Marimoto et al., 2013; Maenosono et al.,2015). Some studies have mentioned that the laser could recrystallize the hydroxyapatite, (Goncalves et al.,1999) help evaporation process of the solvent due to heating and improve the microtensile bond strength using this method (Goncalves et al.,1999; Franke et al., 2006; Marimoto et al., 2013). Other studies related that this solvent evaporation by laser can promote increase in degree of conversion that could justify many favorable results from reports. (Franke etl al., 2006; Batista et al.,2015; Brianezzi et al., 2017)

As the heat promoted by laser depends of the interaction between laser wavelength and substrate, among other factors, *Erbium*, *Chromium* doped Yttrium Scandium Gallium Garnet (Er, Cr: YSGG) laser could be the best assist to remove this water/solvent off the collagen network after penetration of DBS due the greater interaction with water. Its wavelength of 2780nm is very close to the absorption peak of water and hydroxyapatite, that are abundant components present in dentin substrate (Meng et al., 2022). Thus, the resulting higher temperature increase may be a better aid in the evaporation process of water/solvent from hybrid layer.

The objective of this in vitro study was to investigate the effect of Er, Cr: YSGG laser irradiation over some DBS physics properties: water sorption (WS) and water solubility (WSB), degree of conversion (DC) and microtensile bond strength (MS).

2. Methodology

Experimental Design

This article is a laboratory experiment of a quantitative and qualitative e nature.This study involved two factors: treatment in two levels (irradiated[L] or not irradiated [C] with Er, Cr: YSGG laser- WaterLase IPlus, Biolase, Irvine, CA, USA) and dentin bonding system in four levels (Adper™Scotchbond™ Multi-Purpose [MP] - 3M ESPE, St Paul, MN, EUA; Adper™ SingleBond 2 [SB] -3M ESPE, St Paul, MN, EUA; Clearfil™ SE Bond [CSE] - Kuraray Co. Ltda., Osaka, Japan; Adper™ Single Bond Universal [SU] -3M ESPE, St Paul, MN, EUA). The quantitative response variables were water sorption/solubility (WS/WSB) (μg/mm^3), degree of conversion (DC) (%) and microtensile bond strength (MS) (MPa). A qualitative response variable was also described by scanning electron microscopy analysis (SEM).

The null hypotheses were: 1- there was no difference between the DBSs irradiated or not with laser in the water sorption/ water solubility properties, 2- there was no difference between the DBSs irradiated or not with laser in the degree of conversion, 3- there was no difference between the DBSs irradiated or not with laser in microtensile bond strength values, 4- there was no difference between the DBSs irradiated or not with laser in scanning electron microscopy.

Composition of materials used are described in the Table 1.

Table 1 - Commercially available dentin bond systems (DBS), their classification, compositions and application technique according to manufacturer.

DBS	Classification	Composition	Application technique
Adper™ Scotchbond Multipurpose (MP)	Three-step etch-and-rinse system	***Primer:*** HEMA**, water, copolymer of polyalcenoic acid. ***Bond:*** HEMA**, Big-GMA*** and camphorquinone	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
Adper™ Single Bond 2 (SB)	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.	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
Clearfil SE™ Bond (CSE)	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.	Apply primer with a microbrush for 20s Gently dry-air for 5s Apply bond with a microbrush and remove excess
Scotchbond™ Universal (SU)	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.	Apply bond actively for 20s Gently air-dry for 5s

Source: Authors.

***Water sorption and water solubility (*WS/WSB)**

Sample preparation

This study was previously approved by the Ethics Committee of Research on Human Beings at Bauru School of Dentistry, University of São Paulo (n. 49813315.5.0000.5417).

The WS/WSB tests was performed according to ISO 4049 as reference with some modifications (dimensions, sample type and substrate because of the interaction of laser with water and hydroxyapatite). Square-shaped blocks of dentin (6,0x 6,0x 1,0mm) were obtained, weighed, and randomized before the application of DBS that was performed according to manufacturer instructions (Table1). For treatment with laser, after the DBSs application over the dentin blocks, they were irradiated with Er, Cr: YSGG laser with parameters described in Table 2. The laser tip was positioned perpendicularly at 3 mm from specimen for automatic zigzag scanning (BioPDI, XY Table, São Carlos, SP, Brazil). For MP and CSE groups the laser irradiation was performed after primer and for SB e SU after primer/bond. After the treatments and before DBS curing, all the specimens were air-drying for 20s from 10 cm to help the evaporation of solvents. After all the specimens were light cured with LED curing unit (Blue Star 2 light, Microdont, São Paulo, SP, Brazil) covering the entire specimen surface at a power density of 1000mW/cm^2 for 20s.

Table 2 - Parameters used for irradiation.

Parameter	**Value**
Energy per pulse (output)	6.25 mJ
Frequency	10 Hz
Power	0.0625 W
Time	30s
Area	36mm^2
Energy density	5.20 J/cm^2
Tip diameter	800 µm

Source: Authors.

Water Sorption and Water Solubility Analysis

The samples were stored in desiccators at 37ºC [Cuber with silica gel (Mesh Blue 2-4 mm, Synth, São Paulo, SP, Brazil)]. Test had three phases for obtaining M1, M2 and M3.

M1: mass of the specimen after the first desiccation. After the treatments, the specimens were weighed daily in an analytical balance (GR-202, A & D Engineering, INC., San Jose, USA), until to obtain a constant mass (oscillation of 0.002g).

M2: mass of specimen after immersion in water. The specimens were stored in distilled water at 37ºC and were weighed daily until to obtain a constant mass (oscillation of 0.002g). Before the weighing each specimen was dried with three absorbent papers.

M3: mass of the specimen after a new desiccation. After M2, the specimens were subjected to a further desiccation process on silica and a new weighing were carried out to obtaining M3, observing the oscillation limit of 0.0002 g.

The values of WS/WSB, in micrograms per cubic millimeter (µg / mm³), were calculated using the following equations:

(Sorption) $WS = \frac{m2 - m3}{V}$ (Solubility) $WSB = \frac{m1 - m3}{V}$

The volume (V) in mm³ of each specimen was determined by the formula of V = x².h, where x² corresponds to the working area and h is the mean value of height (thickness) that was measured at three different points (Digimatic Calibrator, Mitutoyo Sul Americana, Rio de Janeiro, Brazil).

Degree of conversion

Sample preparation

For Raman spectroscopy, 24 teeth had the crown sectioned, then the area of 36mm^2 for irradiation was delimited in the exposed dentin with white nail enamel (Risque, Barueri, SP, Brazil) and the treatments were performed as described in the previous test. After application of the DBS, irradiation and light polymerization, the composite resin (Filtek™ Z250, 3M ESPE, St Paul, USA) was inserted by the incremental technique with consecutive deposition of two 1.5mm thick horizontal increments. The curing of each increment was performed with the same LED for 20s. After application of the resin, the teeth were immersed in deionized water and kept in an oven at 37 ° C.

After 1 year, the specimens were sectioned in the long axis on the serial cutting machine (Isomet™ Low Speed Saw®, Buehler, Lake Bluff, USA) to obtain slices with 1.0mm thickness. Additional wear was performed when necessary to obtain standardized slices of average thickness of 1.0 mm ± 0.1mm.

Degree of Conversion Analysis

The Raman spectra of the specimens were recorded on a Micro Raman spectrometer (CRAIC Micro Raman, model PV 20/30, San Dimas, CA, USA) with excitation at 830nm, coupled to a microscope with 40x magnification. The Micro-Raman spectrophotometer was first calibrated to zero and then to the coefficient values using a silicon sample. The spectra were recorded on the hybrid layer of each specimen with a Raman camera. Uncured DBS spectra (R) were taken as reference. The ratio of the double bond content of the monomer to the polymer was calculated according to the following formula:

$$DC = (1 - \frac{R\ cured}{R\ uncured}) \times 100$$

• R is the ratio between the aliphatic and aromatic peaks at 1638 centimeters-1 and 1610 centimeter-1 in cured and uncured adhesives.

Microtensile Bond Strength (MS)

Sample preparation

For microtensile strength test the sample was prepared in the same way of degree of conversion. However, the sample were sectioned further longitudinally to obtain square sticks with approximately 0.64mm^2 area (n=10).

Microtensile Bond Strength Analysis

This test was performed only after 1 year of water storage using 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, Article 31 Odeme, Santa Catarina, Brazil). The adhesive interface was positioned perpendicular to the tensile forces generated by the testing machine. The tension (N) was applied at a constant speed of 0.5 mm/min, with maximum load of 500 N, and the force required to break the stick was registered.

Failure mode analysis (FM)

The segments of the fractured samples 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.

Scanning Electron Microscopy (SEM)

Sample PreparationFor SEM analysis, 24 teeth divided in 8 groups according to the DBS and laser irradiation factors (n= 3) were used. The teeth had the roots sectioned, and the crowns had the occlusal surface flattened with #320 and #600 grit. The Laser energy density was standardized for irradiation. After the application of DBS and irradiation of the laser, the DBSs

were light cured and the composite resin (Filtek™ Z250 (3M ESPE, St Paul, USA)) was inserted by incremental technique. The restored teeth were immersed in deionized water and kept in an oven at 37 °C.

After the storage period, the specimens were sectioned in longitudinal slices (1.0 mm ± 0.1mm) with a serial cut machine (Isomet ™ Low Speed Saw®, Buehler, Lake Bluff, USA) and diamond blade (Extec Dia. Wafer blade 5 "x.015x1 / 2, Extec Corp-Einfeld, USA) at low speed and under water cooling. One slice of each tooth was randomly selected for morphological analysis.

The selected slices were stored in deionized water and then immersed in 18% hydrochloric acid solution (HCl 5η) for 30s, to remove the superficial smear layer, washed for 30s with deionized water, followed by immersion in 5% sodium hypochlorite for 15 minutes to remove all collagen not infiltrated by DBS and subsequent washing for 30 seconds. The samples remained 12 hours drying at room temperature and then mounted on aluminum stubs and metallized with gold-palladium (Dentron Vacuum - Desk IV Moorestonn / NJ - USA) prior to SEM observation (JSM-T220A, JOEL,Inc, Tokyo, Japan).

In the SEM, the representative specimen of each group was qualitatively analyzed in the dentin of the adhesive interface with a standardized magnification of 1000x and 1500x.

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.

3. Results and Discussion

For water sorption (WS), no difference was observed for DBS, only treatment. Groups irradiated by laser presented lower values than non-irradiated.

Table 3 - Mean and standard deviation of the Water Sorption (ug/mm³).

Treatment	MP	SB	CSE	SU
Control	123,44 ± 15,4 Aa	124,87 ± 8,0 Aa	130,25± 28,5 Aa	136,0± 22,6 Aa
Laser	119,94 ± 23,9 Ba	101,24 ± 13,7 Ba	103,41 ± 9,5 Ba	116,0 ± 12,0 Ba

* Upper case letters show difference between lines. ** Lowercase letters show difference between columns. Source: Authors.

For water solubility (WSB), it was observed significant results for the two factors, as well as the interaction between them. In the control groups, significant differences between DBSs were found (CSE>SU=SB=MP). Laser irradiation promoted a significant decrease in WSB values for all groups. In the irradiated groups, significant differences between DBSs also occurred (CSE-L>SU-L=SB-L= MP-L).

Table 4 - Mean and standard deviation of Water Solubility (μg/mm³).

Treatment	MP	SB	CSE	SU
Control	-1,49 ± 5,28 Aab	-6,92 ± 4,31Ab	12,39 ± 13,51 Aa	-7,67 ± 5,03 Ab
Laser	-47,8 ± 17,1 Bd	-33,68 ± 5,25 Bcd	-12,13 ±5,7 Bb	-31,97 ± 5,50 Bc

* Upper case letters show difference between lines. ** Lowercase letters show difference between columns. Source: Authors.

For DC, it was noted a significant difference only for DBS factor. Laser irradiation did not promote any significant difference in DC (Table 5). SU presented higher values of degree of conversion (SU>MP>CSE=SB).

Table 5 - Mean and standard deviation of Degree of Conversion (%).

Treatment	MP	SB	CSE	SU
Control	52,0±7,6 Ab	35,8±0,8Ac	38,3±1,65Ac	72,37±0,51Aa
Laser	57,2 ±2,0Ab	35,4±0,83Ac	38,0±3,0Ac	73,2±1,52Aa

*Upper case letters show difference between lines. ** Lowercase letters show difference between columns. Source: Authors.

For MS, it was observed significant differences for the two factors and interaction between them. In the control groups, no significant differences between DBSs could be noted. It was observed that Laser irradiation promoted a significant drop in MS values for all groups, with exception of SU-L (SU-L> CSE-L= SB-L= MP-L).

Table 6 - Mean and standard deviation of Microtensile Strength (MPa).

Treatment	MP	SB	CSE	SU
Control	24,3 ± 14,0 Aabce	35,6 ± 13,0 Aab	29,6 ± 9,8Aabc	29,5 ± 9,8Aabc
Laser	9,8 ± 8,8 Be	17,7±13,9 Bec	20,8 ±10,8 Bcbe	38,4 ± 9,5 Ba

*Upper case letters show difference between lines. ** Lowercase letters show difference between columns. Source: Authors.

For FM, the most predominant failure mode for all groups was adhesive degradation. Description of the distribution is presented in Table 7.

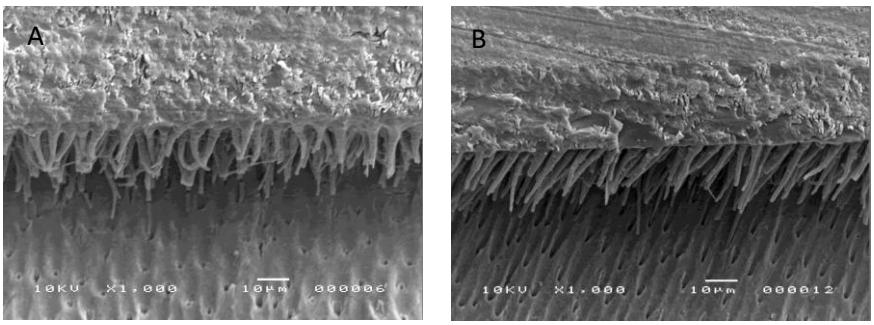
Table 7 - Failure mode analysis (%).

Groups	A	CD	CR	M
MP	63%	8%	8%	4%
SB	75%	11%	7%	3%
CSE	81%	13%	2%	2%
SU	67%	8%	8%	4%
MP-L	68%	13%	0%	0%
SB-L	81%	5%	2%	0%
CSE-L	94%	3%	0%	3%
SU-L	80%	12%	8%	0%

Source: Authors.

In SEM analysis it was not observed morphological differences between MP and MPL group, although the irradiation was performed after primer (Figure 1). For SB-L, it was possible to observe longer tags than SB (Figure 2). For CSE-L, it was observed a quantitative increase of tags regarding CSE, however with shorter tags characteristic of self-etch DBS (Figure 3). For SU no morphological difference could be evidenced by the laser irradiation (Figure 4).

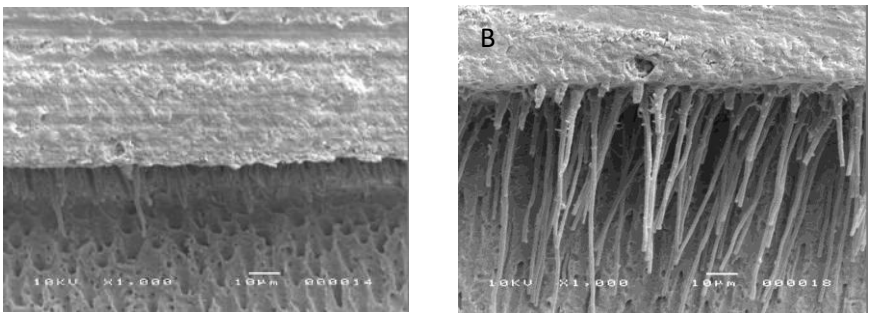
Figure 1 - SEM images of adhesive-dentin interface with Adper™Scotchbond™ Multi-Purpose: A- MP without laser irradiation; B- MPL with laser irradiation.



Source: Authors.

It was observed Figure 1B the total etch pattern with longer tags in greater quantity. It was not observed difference between MP and MP-L.

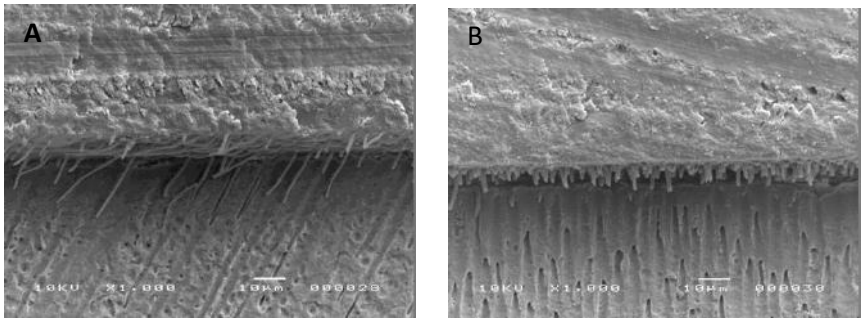
Figure 2 - SEM images of adhesive-dentin interface with Adper™ SingleBond 2 A- SB without laser irradiation; B- SBL with laser irradiation.



Source: Authors.

It was also observed a pattern for total-etch DBS (Figure 2A). Furthermore, it is possible to observe (Figure 2B) longer tags promoted by laser irradiation(SBL) than control (SB).

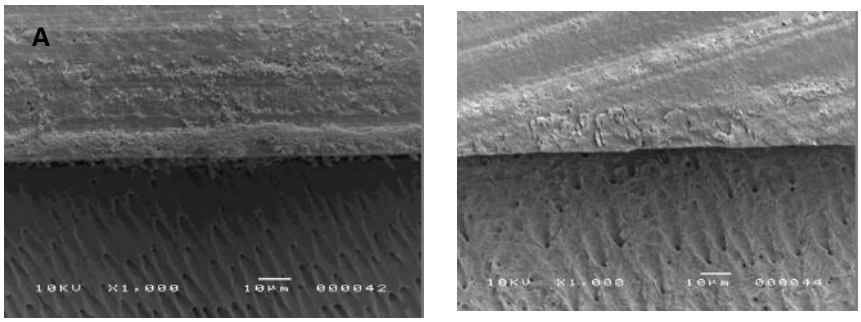
Figure 3 - SEM images of adhesive-dentin interface with Clearfil™ SE Bond. A- CSE without laser irradiation; B- CSEL with laser irradiation.



Source: Authors.

I was observed a pattern of self-etch DBS with shorter tags and smaller quantity than total-etch DBS(Figure 3A);In addition, it was observed a quantitative increase and thicker tags for CSEL (Figure 3B).

Figure 4 - SEM images of adhesive-dentin interface with Adper™ Single Bond Universal in self-etch mode. A- SBU without laser irradiation.



Source: Authors.

It was observed shorter tags and in smaller number than CSE(Figure 4A).For this group no morphological difference could be evidenced by the laser irradiation (Figure 4B).

Lasers have been tested to improve the physicochemical interactions between the moist dentin substrate and the resin-based DBS (Pashley et al., 2007). Heating promoted by lasers and dental literature (Bail et al., 2012; Vale et al., 2014; Castro et al.,2013) have been reported the increase of temperature aid in the evaporation of the solvents presented in DBS as well as improvement of mechanical properties. Furthermore, many authors (Goncalves et al., 1999; Maenosono et al., 2015) observed that neodymium and diode laser improved the bond strength of DBS irradiated prior to their curing. However, that among the available lasers, it presents wavelength with high interaction with elements present in dentin, such as water and hydroxypatite this situation has not yet been investigated.

In this study, the Er, Cr: YSGG laser promoted a significant drop in the sorption (WS) of the different DBSs. Some studies have indicated that increasing WS may negatively impact the long-term stability of the polymer (Malacarne-Zanon et al., 2009). These smaller values of WS could have occurred due to the greater evaporation of the hydrophilic solvent promoted by the heating of the laser. These results agree with other studies that related those longer periods of evaporation, heated airstream or preheating of DBS can reduce the WS/WSB (Bail et al.,2012; Vale et al.,2014).

However, laser irradiation also promoted a significant drop in the solubility values of DBSs. MP-L, SB-L and SU-L presented significant difference than respective control groups. Many factors could affect the solubility of DBS: presence of chemical groups capable of forming hydrogen bonds with water; crosslinking degree; presence of residual monomers; and crystallinity of the polymer (Nishitani et al.,2006). Sideridou et al. 2003 reported that methacrylate polymers exhibit a spatial heterogeneity in which some parts are densely cross-linked and some parts are loosely cross-linked.

It can be observed that the WSB test presented negative results which is not a common finding in the literature. This may have happened due to modification in the test using DBS applied on the dentin due to the interaction of the laser with the substrate/DBS and not only pure DBS. For control groups, the negative results could be explained due to the presence of remaining water in the dentin. For laser groups, the hypothesis is that high heating promoted by Er,Cr:YSGG laser could have been aided the solvent evaporation decreasing the WS/WSB, but at the same time could have affected the substrate due interaction with hydroxyapatite and spatial heterogeneity of polymer resulting in a greater amount of the chemical groups capable of forming hydrogen bonds with water, allowing that part of the water obtained in M2 to be maintained at the desiccation end (M3>M1) in addition to the water retained in the dentin, was occurred in the control groups. Since laser irradiation affected the WS/WSB of most DBSs, the first null hypothesis was denied.

The irradiation with Er,Cr:YSGG laser did not affect DC after 1 year. It was observed only significant differences among DBSs, with higher values for SU. Scientific studies showed that this DBS presents a different technology which allows them to be used as etch-and-rise or self-etching. Furthermore, these results could be justified due the viscosity and composition of this DBS that presents silane, vitrebond copolymer and 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP) that could promote a higher stability of the polymer (Yoshida et al., 2012; Giacomini et al.,2020). Besides the differences in the chemical composition, clinically it is possible to observe that this DBS is less viscous than the others. According Danroch et al., 2005 lowest viscosity may confer an increase in the mobility of the radicals and reacted monomers, resulting in further curing and higher DC. In this way, the second null hypothesis was accepted.

For MS, it was observed significant differences between irradiated and not irradiated groups. Irradiated groups were affected negatively, with exception SU-L that did not present a significant drop in the MS values. These data corroborate the work of Zabeu et al.,2021 however in the period of 6 months. Drop in bond strength values over time are expected (Yoshida et al., 2012) regardless of the adhesive type, however the laser irradiation promoted a significantly greater drop. In this way, it is believed that although the heating promoted by the erbium laser has assisted in the evaporation of the solvent, it may have altered the substrate and spatial heterogeneity of the polymer as previously discussed. This phenomenon may have allowed the formed polymer to have a greater amount of the chemical groups capable of forming hydrogen bonds with water or greater amount of loosely cross-linked, that after 1-year resulted in greater degradation of the adhesive interface. It is observed that the SU-L group was not affected like the others, and this may have occurred due to the higher values of DC obtained for this DBS.

Another factor that could also interfered in the MS test is the degradation of collagens fibrils promoted by the high temperature increase. Moreto et al. 2011 reported that the Er,Cr:YSGG laser parameters used in his study resulted in a specific morphological pattern of dentin and collagen fibrils that negatively affected the bond strength, due the high interaction of this laser with the dental hard tissue. However, according to Schaller et al. 2004, the degradation of collagen fibrils begins from values up to about 146ºC (420K). According to data not yet published on this research group the temperature of this study did not reach this level, and therefore this degradation probably did not occur.

Regarding the morphological characteristics of the hybrid layer, the results of the present study are in accordance with the literature. It was observed a significant difference between total-etch and self-etch DBS when the qualitative/quantitative of resin tags was compared. Both conventional DBS presented larger and more numerous resin tags when compared with self-

etch ones. The self-etch DBS presented shorter and fewer resin tags, and the one step self-etch DBS presented almost total absence of tags (Lafuente et al.,2012; Reis et al.,2009).

The increase of temperature is important to promote the evaporation of solvents and reduce the viscosity of the DBS (Reis et al.,2009). Probably SB had a higher evaporation of the water due to the presence of ethanol in the DBS. This may have been responsible for the greater infiltration of this DBS in the substrate (longer tags). In the self-etching DBS, these differences were not evident due to the characteristics such as: lower formation of tags due to the absence of previous acid conditioning.

Since all laser irradiation presents a large variety of parameters for irradiation, other configurations should be tested since some parameters may achieve the ideal temperature to promote benefits to the physics properties of the DBSs.

4. Conclusion

Based on the results, Er, Cr: YSGG laser irradiation in an attempt to improve adhesion should be better investigated, since the irradiation parameters and strategy used in the present study compromised negatively the most physics properties of the most DBSs. More studies with different lasers and parameters are needed to be able to establish a protocol to improve adhesion.

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