ORIGINAL ARTICLE

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

Efeito da irradiação com laser de diodo sobre sistemas adesivos antes da cura versus protocolo de adesão padrão na resistência ao cisalhamento entre o cimento resinoso e a dentina

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ABSTRACT

Objective: This study aimed to evaluate the effect of diode laser irradiation (970 nm) of a one-step self-etch adhesive (Clearfil S[3] Bond/CS3B) and of the bonding agent of a two-step self-etch adhesive (Clearfil Liner Bond F/CLBF) placed on dentin before polymerization on the shear bond strength. **Material and methods:** Forty sound premolars were sectioned buccally to obtain flat dentin surfaces. The specimens were divided into 4 groups (n = 10): Group (OS) – CS3B + polymerization. Group (OS-L) – CS3B + laser + polymerization. Group (TS) – CLBF (bonding agent only) + polymerization. Group (TS-L) – CLBF (bonding agent only) + laser + polymerization. The diode laser was irradiated through an 8 mm bleaching tip for 10 seconds, (0.4 W, 10 Hz, 4 J). All samples were cemented to composite blocks and submitted to 4000 thermal cycles. The samples were tested for shear bond strength in a universal testing machine. Data obtained was analyzed using Two-way (ANOVA) ($p < 0.05$) and the Bonferroni post-hoc test. Representative samples from each group were analyzed using scanning electron microscopy (SEM). **Results:** Group TS-L – (7.43 MPa) displayed statistically significant higher shear bond strength in comparison to that of group TS – (5.13 MPa). No statistically significant difference was found between group OS-L – (6.49 MPa) and group OS – (7.28 MPa). Group TS-L exhibited the highest resin penetration beyond the hybrid layer under SEM. **Conclusions:** Diode laser irradiation of a bonding agent placed on dentin without prior priming increased the bond strength to dentin and is promising as a new dentin adhesion protocol.

RESUMO

Objetivo: Este estudo teve como objetivo avaliar o efeito da irradiação com laser de diodo (970 nm) sobre um adesivo autocondicionante de um passo (Clearfil S3 Bond / CS3B) e de um outro adesivo autocondicionante de dois passos (Clearfil Liner Bond F / CLBF) aplicado na dentina, antes de sua polimerização, na resistência ao cisalhamento. **Material e métodos:** Quarenta pré-molares hígidos foram seccionados vestibularmente para obtenção de superfícies dentinárias planas. Os espécimes foram divididos em 4 grupos (n = 10): Grupo (OS) - polimerização de CS3B +. Grupo (OS-L) - laser + CS3 + polimerização. Grupo (TS) - CLBF (apenas agente de adesão) + polimerização. Grupo (TS-L) - CLBF (somente agente de adesão) + laser + polimerização. O laser de diodo foi irradiado através de uma ponta de branqueamento de 8 mm durante 10 segundos (0,4 W, 10 Hz, 4 J). Todas as amostras foram cimentadas a blocos de compósitos, submetidos a 4.000 ciclos térmicos. As amostras foram testadas quanto à resistência ao cisalhamento em uma máquina universal de ensaios. Os dados obtidos foram analisados utilizando-se Two-way (ANOVA) ($p < 0,05$) e o teste post-hoc de Bonferroni. Amostras representativas de cada grupo foram analisadas por microscopia eletrônica de varredura (MEV). **Resultados:** O grupo TS-L - (7,43 MPa) apresentou resistência ao cisalhamento estatisticamente significativa maior em relação ao grupo TS (5,13 MPa). Não houve diferença estatisticamente significativa entre o grupo OS-L - (6,49 MPa) e grupo OS - (7,28 MPa). O grupo TS-L exibiu a maior penetração de resina além da camada híbrida em MEV. **Conclusões:** A irradiação com laser de diodo de um agente adesivo colocado sobre a dentina sem o uso prévio de primers aumentou a força de adesão à dentina e é promissora como um novo protocolo de adesão dentinária.

KEYWORDS

Dentin; Dentin-bonding agents; Lasers.

PALAVRAS-CHAVE

Dentina; Sistemas adesivos; Lasers.

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

INTRODUCTION

It has been more than half a decade since Buonocore [1] and later Nakabayashi [2] developed the concepts behind the most popular branch in restorative dentistry, dental adhesion. Owing to its ability to create intimate contact between the tooth and the restoration, adhesion theoretically eliminates marginal gaps, reduces micro-leakage, and prevents secondary caries [3]. Due to the nature of adhesive bonding, it has been proven that the quality of adhesion is proportional to the performance of the bonding agent.

Unfortunately, the performance of contemporary bonding agents is still flawed when it comes to dentin. Owing to dentin's complex heterogenic nature, hydrolysis of resin and collagen fibrils within adhesive interfaces eventually occurs [4]. According to the related literature, self-etch adhesive systems (6th, 7th, and 8th generation adhesives) seems to outperform etch and rinse adhesive systems in dentinal bonding [5–8].

The absence of a separate etching step evades the dilemma of over-wetting or over-drying the dentin after washing the acid [9,5,10]. The addition of resin monomers with the etchant also buffers its acidity, overcoming complications associated with strong acid use [11]. Enzymatic and hydrolytic breakdown of collagen fibers [12,13], nano-leakage [14], and hypersensitivity [5] have all been attributed to adhesive systems that incorporate strong acids (pH ≤ 1).

Yet even with milder self-etch adhesives (pH ≈ 2), numerous studies still reported water-related bond deterioration after ageing [14,15]. The presence of HEMA, water, and ethanol [16–18], as well as the differences in molecular weights between acidic and cross-linking monomers [14] have been blamed for the breakdown of adhesive interfaces created by simplified bonding systems [19–22].

Dental lasers have been commercially available for several decades and their rising popularity is vast among both practitioners and patients. Their use has been incorporated into many phases of tooth removal and preparation, including dental adhesion. SE Gonçalves et al (1999)[23] suggested a potential adhesive protocol that incorporates a soft tissue laser; they claimed that Nd:YLF (1047 nm) irradiated on an uncured total-etch adhesive before polymerization resulted in a statistically significant increase in the shear bond strength (SBS) to bovine dentin. The authors theorized that the heat energy from the laser formed a new substrate in which the hydroxyapatite has been melted and fused in the presence of bond monomers.[23]

The use of laser energy on the bonding agent before its curing has since been evaluated in further studies and has been further hypothesized to increase bond penetration [24], help evaporate the solvent [25], increase its degree of conversion (DC) [26], and increase the modulus of elasticity of the hybrid layer [27]. All supposedly through the photothermal effect of the laser on the adhesive. Heating of resin based restorative materials have been proven to increase their mechanical properties and bonding capabilities [28,29], which rationalizes why the laser may be effective in doing the same.

A decrease in nano-leakage was also observed [30–32], supposedly due to dentin recrystallization, enabling intimate contact with the bonding agent [23,31]. The majority of studies reported an increase in adhesive penetration and resin tag length under microscopic analysis [33–38]. Some authors, however, obtained negative results [39,40],finding the laser to have no effect whatsoever on the bond strength. Others found an increase in immediate bond strength that dropped dramatically after ageing [41].

Castro FLA et al. [42] found that laser irradiation of unetched dentin resulted in higher bond strengths compared to etched/lased dentin. The authors claimed that etched dentin, lacking some hydroxyapatite, suffered more physical alteration and solidification globules from the laser, hindering the adhesive penetration.

Dentinal bonding is yet to possess predictability, and warrants the need to develop materials and techniques to enhance its durability and strength. Therefore, in an attempt to further asses the validity of laser use in enhancing dentinal bonding, the aim of this study is to evaluate, in vitro, the effect

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

of diode laser irradiation on a one step self-etch adhesive and on the bonding agent of a two-step adhesive system before their polymerization on the shear bond strength between resin cement and dentin after thermocycling. This study tested the null hypothesis that the diode laser will have no effect on the shear bond strength to dentin with either adhesive system.

MATERIAL AND METHODS

Sample size calculation

Based upon the results of Marimoto AK et al. [33], using alpha (α) level of 0.05 (5%) and Beta (β) level of 0.20 (20%) i.e. power = 80%, the study included 40 samples with 10 samples per group. Sample size calculation was performed using IBM® SamplePower® Release 3.0.1. (SPSS Inc., Chicago, IL)

Sample preparation

Forty sound human premolars, extracted for orthodontic reasons, were obtained from the outpatient clinic at Misr International University after approval of the local research ethics committee (MIU-IRB-1718-048). The roots were sectioned off at the level of the cemento-enamel junction and the buccal surfaces were grinded till dentin exposure. Standardized 2 mm buccal dentin sections were obtained using a microtome sectioning device (IsoMet precision cutting microsaw, Buehler, USA) with the blade adjusted accordingly, as recommended by previous authors for hybrid layer analysis.[23,33,43] The obtained sections were then manually verified for thickness using a dental gauge caliper. Each dentin section, which measured approximately 6-7 mm in width and 7-8 mm in height, was embedded in self-curing acrylic resin using a custom-made 1.5 × 1.5 cm cylindrical Teflon mold with the dentin surface exposed at the surface. The dentinal surfaces were finished using a fine-grit diamond stone under water coolant to simulate clinical smear-layer formation [44].

Adhesive procedure

The samples were numbered from 1 – 40, then, using Microsoft Excel's random sampling tool (Microsoft® Excel for Mac 2019, Version 16.23), the 40 prepared samples were randomized and allocated into 4 groups (n = 10) according to the dentin surface treatment. The adhesive systems used and their manufacturing information are listed in Table I.

Group (OS) – the dentin surfaces were treated with the Clearfil S³ Bond Plus (CS3B). The bond was applied to the exposed dentin surface and agitated for 20 seconds, as per manufacturer's instructions, then air-dried for at least 5 seconds until the bond no longer moved on the surface. The bonding agent was then light cured for 10 seconds using a 650 mW/cm² LED light cure unit (LED-D, Woodpecker, Guilin, China), with the tip perpendicular and as close as possible to the surface.

Group (OS-L) – the dentin surfaces were treated with the CS3B following the same protocol used with group OS, but before polymerization, the bonding agent was irradiated with a Diode laser device (SIROLaser Advance, Sirona, Bensheim, Germany). The 8 mm bleaching tip accompanying the device was used. The bleaching tip was secured into the hand-piece and the fiber optic cable was set so that its end was located 1 mm away from the tip edge. The laser was applied for 10 seconds with the bleaching tip was placed directly on the sample's surface. The diode laser parameters used are listed in Table II. After irradiation, the samples were light cured for 10 seconds following the same protocol utilized with group (OS).

Group (TS) – the dentin surfaces were treated with the bonding resin of the Clearfil Liner Bond F (CLBF) without prior application of the primer, based on the bonding protocol used by Castro FLA et al. [42]. The bond was applied and agitated on the surface for 10 seconds, gently air-dried, and light cured for 10 seconds following the same light curing protocol utilized with group (OS).

Group (TS-L) – the dentin surfaces were treated with the bonding resin of the CLBF without prior application of the self-etching primer as done with group (TS). However, before light curing, the testing areas were irradiated with diode laser following the same irradiation protocol used with group (OS-L). The samples were then light cured for 10 seconds following the same light curing protocol utilized with group (OS).

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

Table I - The adhesive systems used and their manufacturing information

| Group	Adhesive system	Manufac-turer	Method
OS OS-L	Clearfil S³ Bond Plus/CS3B	Kurary Nori-take Dental Inc, Tokyo, Japan	Adhesive monomer (MDP), hydrophilic monomer (HEMA), cross-linking monomer (Bis-GMA), catalyst, nano-filler (silanated colloidal silica), Cam-phorquinone, water, and ethanol.
TS TS-L	Clearfil Liner Bond F/CLBF *(Bonding Agent only)*	Kurary Nori-take Dental Inc, Tokyo, Japan	*Primer:* 10-MDP, HEMA, hydrophilic dimetha-crylate, dl-Camphor-quinone, accelerators, water *Bond:* 10-MDP, HEMA, Bis-GMA, dimethacryla-te, dl-Camphorquinone, silanated colloidal silica, surface treated Sodium fluoride (NaF).

(MDP): 10-methacryloyloxydecyl dihydrogen phosphate, (HEMA): Hydroxyethylmethacrylate, (Bis-GMA): Bisphenol A-glycidyl methacrylate

Table II - Diode laser parameters used to irradiate the samples.

Parameter	Value
Peak Power	0.8 W
Time	10 seconds
Duty cycle	50%
Frequency	10 Hz
Energy	4 J
Average power	0.4 W

W: Watts, Hz: Hertz, J: Joules

Forty composite resin blocks (Nexcomp shade A3.5, Meta Biomed, Korea) were fabricated using a custom-made 6×6 mm cylindrical Teflon mold. [45] The composite resin was placed in 2 mm increments and each increment light cured for 20 seconds. For cementation, a custom-made Teflon cementation device with a 4 Kg weight was fabricated [46]. (Figure 1)
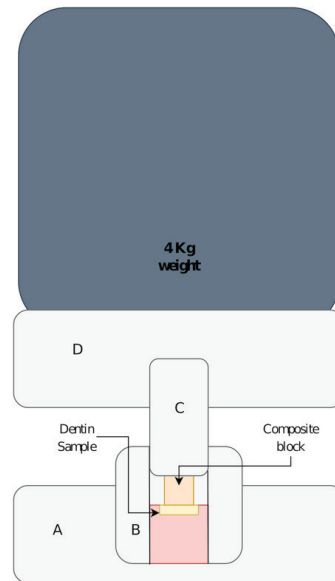
Figure 1 - Schematic illustration of the custom-made Teflon cementation device.

The cementation device consists of a 2.5×8.5 cm round Teflon base with a round 3×1.5 cm cavity in the center (unit A). A 3 cm Teflon cylinder with an internal cavity of 1.5 cm (unit B) is inserted into the base and an acrylic resin sample (1.5×1.5 cm) is placed inside. Resin cement is dispensed on the surface of the dentin and a composite block is placed on top. A Teflon 1.5×3 cm cylinder (unit C) is placed on the composite block so that around 1.5 cm of its body is protruding from unit B after placement. Another 2.5×8.5 cm round Teflon component with a central 1.5 cm cavity on the bottom and a 3 mm $\times$ 8 cm recess on the top (unit D), is placed over the protruding unit C. A 9.5 cm wide cylindrical 4 Kg weight is placed on the recess of the top component to complete the cementation device assembly.

For cementation, each dentin sample was placed in the analogous hole in the cementation device, dual-cure resin cement (Panavia SA Cement Plus, Kurary Medical Inc., Osaka, Japan) was dispensed evenly on its whole surface and the composite block placed over the dentin surface. Before cementation, the surface of each composite block to come in contact with the cement was air abraded with 50 μm aluminum oxide particles for 10 seconds from a 1.5 cm distance [47].

Excess cement was removed with a disposable microbrush, then the top component with 4 kg weight were placed. Light curing of

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

the specimen from outside the mold is done for a total of 40 seconds (20 seconds from 2 opposite angles). The cement was then left to self-cure for 5 min at 37 °C to ensure total auto-polymerization. The finished specimens were stored in distilled water until testing.

Ageing process

To simulate cyclic thermal fluctuations during clinical service [48], the specimens were submitted to thermocycling for 4000 cycles between temperatures 5 °C and 55 °C in a thermo cycling device (Julabo GmbH, FT 200, Seelbach, Germany).

Shear bond strength

A universal testing machine (Instron, Norwood, MA) was used to test the macro SBS of the adhesive bond. The shear force rod was set to travel at a speed of 0.5 mm/min with a load cell of 500 N. The bond strength was determined from the highest point on the stress-strain curve (maximum stress) measured by the load cell of the testing machine. Results obtained were expressed in Megapascals (MPa).

Scanning electron microscope analysis

An additional 2 samples were made in each group for analysis of the hybrid layer using scanning electron microscopy. The specially prepared samples for SEM analysis were thermocycled but were not submitted to shear bond strength testing to preserve the integrity of the adhesive interface.

Each sample was sectioned vertically in half using an automated microsaw (IsoMet precision cutting microsaw, Buehler, USA). The sections were then decalcified using 32% phosphoric acid for 30 seconds, deproteinized using 2% sodium hypochlorite for 2 minutes etched, rinsed with water, and submitted to 70%, 80%, 90%, and 99% alcohol concentrations for total elimination of the water content [49].

The sections were then sputter coated with gold using a gold sputter (Emitech/Quorum sputter coater K500X, England). The gold-sputtered samples were examined under a scanning electron microscope (Quanta 250 FEG, FEI Co., Netherlands) with an accelerating voltage of 30 K.V under a magnification of 2000X. The SEM images were analyzed using the software ImageJ (ImageJ 1.52k, Wayne Rasband, NIH, USA).

Statistical analysis

For the statistical analysis, the numerical data was first explored for normality by checking the distribution of data using the tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Data showed normal (parametric) distribution. Data were presented as mean, standard deviation (SD) and 95% Confidence Interval (95% CI) for the mean values.

For the effect of type of adhesive system (variable 1), the presence or absence of laser irradiation (variable 2) and their interactions on mean shear bond strength, Two-way Analysis of Variance (ANOVA) was used. Bonferroni's post-hoc test was then used for pair-wise comparisons when the ANOVA test proved the data to be significant. The significance level was set at (P ≤ 0.05). Statistical analysis was performed with the software IBM SPSS Statistics Version 20 for Windows (IBM Corporation, NY, USA).

RESULTS

Shear bond strength

Two-way (ANOVA) proved the interaction between the variables had a statistically significant effect on the mean SBS (P-value < 0.001). According to the Bonferroni post-hoc test, the bonding agent of the two-step adhesive system (CLBF) showed statistically significant higher mean SBS in the irradiated group (group TS-L) in comparison to the non-irradiated group (group TS), (P-value < 0.001, Effect size = 0.332).

Regarding the one-step adhesive (CS3B), there was no statistically significant difference between the mean SBS of the irradiated group (group OS-L) and the non-irradiated group (group OS), (P-value = 0.156, Effect size = 0.055).

Group TS-L showed no significant difference in mean shear bond strength when compared to groups OS and OS-L (P ≤ 0.05).

Group TS showed significantly lower mean shear bond strength compared to the rest of the groups. The mean, standard deviation, and confidence interval values as well as the results of Two-Way ANOVA test are presented in Table III. Mean SBS values of the groups are graphically represented in Figure 2.

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

Table III - The mean, standard deviation (SD), 95% Confidence Interval (95% CI) values and results of two-way ANOVA test

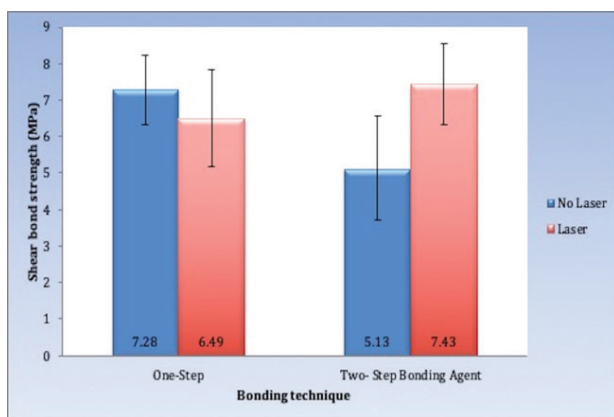| Laser irradiation	One Step (OS)		Two Steps (TS)		P-value (Between techniques)	Effect size (Partial Eta Squared)
	Mean (SD)	95% CI	Mean (SD)	95% CI		
No Laser	7.28 (0.95)	6.5 – 8.06	5.13 (1.42)	4.35 – 5.91	< 0.001*	0.301
Laser (L)	6.49 (1.33)	5.71 – 7.27	7.43 (1.11)	6.65 – 8.21	0.091	0.077
P-value (Between Laser and No Laser)	0.156		< 0.001*			
Effect size (Partial Eta Squared)	0.055		0.332			

*: Significant at P ≤ 0.05

Figure 2 - Bar chart representing the mean and standard deviation values of the shear bond strengths obtained in each group.

DISCUSSION

Due to the nature of the acquired data, the null hypothesis that the diode laser will have no effect on the SBS to dentin with either adhesive system was partly rejected. No statistically significant difference was found between the lased and non-lased groups that received the Clearfil S3 Bond Plus (CS3B); group OS-L (6.49 ± 1.33 MPa) and group OS (7.28 ± 0.95 MPa).

The lack of increase in bond strength in group OS-L contradicts with the findings of Matos et al.,[38] JN Rolla et al.,[37] Marimoto et al., [33] and Maenosono RM et al., [45] all of which found an increase in dentin bond strength after irradiating a one-step self-etch

adhesive before curing. The difference between the results of the current study and that of the previously mentioned studies, aside from the differences in laser parameters, may be due to the thermocycling process that was done in this study. All the previously mentioned studies subjected the irradiated samples to mechanical testing without prior aging, which could have resulted in less favorable outcomes.

Thermocycling is a laboratory method used to simulate the clinical condition with the accompanying deteriorating effects [48]. Thermal stresses, water sorption, leakage, and other destructive water and time related effects appear well after a period of use, and one cannot ensure their complete or even partial absence without proper aging.

According to Gale M.S. and Darvell B.W. [48], 10,000 thermal cycles arbitrarily corresponds to a year of clinical service, implying that the 4000 cycles chosen in this study roughly represents 4.5 months of thermal fluctuations inside the oral cavity. However, according to a review by Morresi et al. [50] on the use of thermal cycling in invitro studies, there is still no established consensus on a proposed number of cycles to correspond to a specific time in the oral cavity.

After the aging process, the samples were tested for the macro shear bond strength (SBS). The shear bond strength test measures

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

the strength of adhesive interfaces exposed to shearing forces associated with cemented indirect restorations [51]. Due to the nature of the study, the macro SBS was chosen as it has been demonstrated under finite element analysis that it more accurately represents the bond strength values than μ SBS, especially for cemented samples [52] as well as sustain sectioning procedures, as done for the SEM samples, without microcrack formation [53].

The diode laser parameters as well as the laser tip chosen to irradiate the samples are considered pilot factors incorporated in this adhesive protocol. To the current authors' knowledge, in all the related studies, the laser was applied freehand by a calibrated operator who positioned the optic fiber tip perpendicular to the surface and scanned the test area for 30 - 60 seconds in contact or non-contact mode.

Since the laser's optic fiber tip typically measures 200 - 400 μm in diameter, running such a small diameter tip on the entire bonding surface can be highly unpredictable when rendered to the clinical situation. In the present study, an 8 mm bleaching tip (cup) was used to irradiate the samples to ensure complete irradiation of all the test surfaces with the least human error possible.

As for the laser parameters, no standardization of any sort was found between the studies, however success was found with power within the range of 0.75 – 1 W.[26,30,37,45] The parameters used in the current study have been chosen to complement the chosen laser tip and its application manner. The bleaching tip was placed directly on each sample without movement.

Therefore, to avoid the risk of overheating the surface, causing cracks or solidification globules [42], the authors decided to irradiate each sample for exactly 10 seconds in 50% duty cycle (pulsed mode), for thermal relaxation, with a 0.4 W power and 4 J total energy. SEM analysis of the samples showed no structural abnormalities in any of the groups. However, such defects may have occurred in the nanometric

scale and would not have been detected by the scanning electron microscopy employed here.

The authors believe that the inherent nature of the one-step self-etch adhesive used in this study is responsible for the bond deterioration seen in groups OS and OS-L. The bond used is a 'mild' light-cured one step self-etching adhesive that contains acidic and functional monomers as well as water and ethanol [5]. The water, which is mandatory in self-etch adhesives,[20] acts as an ionizing medium to allow 'self-etching', while the ethanol ensures the water's evaporation with drying.[16] However, it has been reported that during drying, the hydrophobic monomers separate and the water 'bubbles [5, 19]. These water bubbles are more difficult to blow away and get retained on the substrate's surface [5].

Another issue is the presence of HEMA in the adhesive's composition. HEMA (2-hydroxyethyl methacrylate) – a hydrophilic monomer incorporated in bond formulations to prevent phase separation [16] – polymerizes into linear poly-HEMA chains that contain residual water. This enables it to uptake water even after curing, causing water sorption from the host dentin, and nano-leakage within the bonded surface [14,16,54–56]. Some authors even deemed one-step self-etch adhesives as permeable membranes after polymerization [57], which might explain why any aging process greatly decreases the bond strength of one-step self-etch adhesives [7,58].

The SEM analysis of groups OS (Figure 3) and OS-L (Figure 4) presented separation gaps beneath the hybrid layer, with very thin layers of the underlying dentin attached to the composite side of the separation. This indicates that fracture would have occurred under the hybrid layer rather than at the dentin-adhesive interface. A possible explanation is that the 'mild' etchant present in the adhesive was unable to penetrate well beyond the formed hybrid layer [18], creating a weak link below where the adhesive failed to reach.

Sub-hybrid layer separation was also found by Van Landuyt KL et al. [19] after

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

water storage of self-etch adhesives for 6 months. The authors claimed that this may be due to incomplete smear layer dissolution and bypassing by the 'mild' self-etch adhesive, especially if the smear layer formed is thick and compact as that formed by a diamond bur. In this case, the adhesive monomers will not have reached the underlying dentin, resulting in separation beneath the loosely adapted smear layer,[11] as seen in this study.

Tay FR et al. [14] claimed that bond degradation of self-etch adhesives occur due to differences in molecular weight between the acidic and non-acidic monomers, which causes areas of etched dentin that does not get infiltrated by the heavier non-acidic monomers, resulting in separation and nano-leakage underneath the hybrid layer.

Other authors suggested that hydrolytic breakdown of acidic monomers found in one-step self-etch adhesives can result in the formation of non-curable acids that continue to etch the underlying dentine after polymerization, causing weak areas beneath the hybrid layer devoid of hydrophobic monomers [21].

SEM analysis of group OS-L, however, showed areas of minute resin tags beneath the hybrid layer that were not seen in group OS, Figure 4(A). Thicker well-formed resin tags were also observed in some images, Figure 4(B). However, the results of the shear bond strength test suggest that diode laser irradiation did not significantly affect the bond strength of the Clearfil S^3 Bond.

A possible explanation is that the laser did increase the penetration of the bonding agent in group OS-L, probably due to evaporation of some of the water content [25]. The presence of hydrophilic monomers (HEMA) and acidic components in the formulation of the Clearfil S3 Bond, however, may have caused enough hydrolytic and enzymatic damage that was not redeemable by the increased penetration of the bond.

As for the groups that received the bonding agent of the two-step self-etch adhesive

(CLBF) without prior use of the primer, the diode laser significantly increased the SBS to dentin in group TS-L (7.43 ± 1.11 MPa) in comparison to the non-lased samples treated with the same adhesive; group TS (5.13 ± 1.42 MPa) where (P-value < 0.001). Castro FLA et al. [42], who achieved the same result in a similar study but with a control group following the manufacturer's instructions (primer + bonding agent), claimed the etchant in the primer is what compromised the effect of the laser.

The authors claimed that the primed dentin, lacking some hydroxyapatite, suffered more physical alteration and solidification globules from the laser, hindering the adhesive penetration [42]. The unprimed dentin, however, was not seemingly altered. In addition, non-etched dentin generally contains less water at the surface [5], which could have facilitated the diffusion of the adhesive further and lead to the higher bond values. This might justify the minimum to no resin tag formation observed in groups OS and OS-L.

On the un-etched dentin, however, the preservation of the smear layer allowed the laser to "seal" the dentin, reducing permeability and water seepage [42]. The absence of etching also preserved dentin's calcium content, which may have allowed its chemical bond to the MDP functional monomers found in the bonding agent [5].

The absence of acidic components and water in the bonding agent used, as well as the presence of a larger quantity of hydrophobic monomers and fillers may have also played a role. However, the bonding agent does contain HEMA. The current authors suggest that the diode laser may have enhanced HEMA's inferior polymerization efficiency by increasing its degree of conversion [26,28,29].

In addition, the absence of water and ethanol may have subdued some of the damaging effect of HEMA. Some authors have claimed that HEMA decreases the vapor pressure of water and solvents like ethanol, preventing their complete evaporation [59], justifying the intact

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

hybrid layer seen even after thermocycling in group TS-L.

Another possible explanation for the increase in strength in group TS-L is the sodium fluoride (NaF) content present in the bonding agent. Sodium Fluoride has been proven to inhibit dentinal esterase enzymes even in very low concentrations [60]. Fluoride ions are also believed to be gradually released into the underlying dentin, increasing the mineral content and protecting against micro-leakage [61]. Indeed, fluoride containing adhesives have also been found to be more durable than fluoride free adhesives after months of water storage [61,62].

Group TS displayed the least bond strength of all groups. Under SEM, group TS showed generalized separation within the hybrid layer itself, suggesting an adhesive breakdown, Figure 5. This phenomenon has been observed with adhesives that are unable to penetrate the full length of the smear layer, either due to their weak etching ability, the thickness of the smear layer, or both [16,63]. Unmodified smear layer remnants were observed within the formed interface, probably due to the lack of a priming agent.

In this study, the dentin samples were finished with a fine grit diamond stone (20 – 30 μm) under water coolant to simulate clinical smear layer formation [44], which is known to produce a 1.2 μm thick smear layer on the dentinal surface [64]. The thickness of the smear layer combined with the lack of etching are probably responsible for the hybrid layer separation as well as the low bond strength in group TS.

The increase in bond strength and penetration of the bonding agent of the CLBF without the primer (group TS-L) was thus directly related to the diode laser irradiation. It seems that the laser energy facilitated the bonding agent's penetration through the smear layer to the underlying dentin. The SEM analysis of group TS-L shows rounded resin tag formation reaching up to 120 μm in length. (Figure 6).
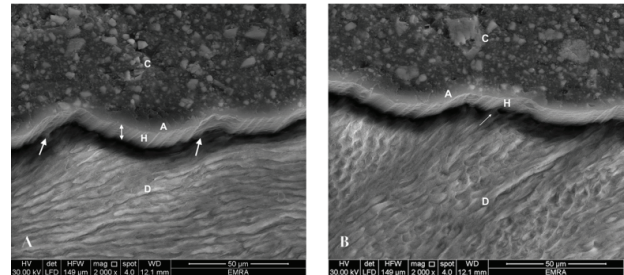

Figure 3 - SEM micrographs (2000x) of group OS, after 4000 thermal cycles. A 7 μm hybrid layer formation is evident (double-ended white arrow). Evidence of sub-hybrid layer separation is seen with parts of the dentin attached to the hybrid layer (white arrows). (H – Hybrid layer, A – Adhesive, C – Composite, D – Dentin).
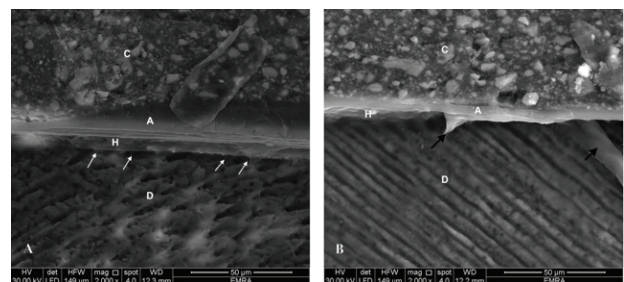

Figure 4 - SEM micrograph (2000x) of group OS-L, after 4000 thermal cycles. A) Minute resin tag formations are seen (white arrows). B) Evidence of thick resin tag formation is seen (black arrows).
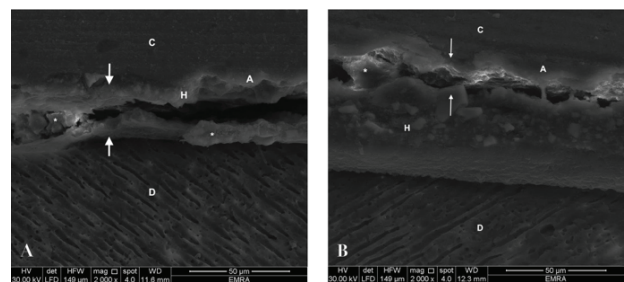

Figure 5 - SEM micrographs (2000x) of group TS, after 4000 thermal cycles. An interrupted hybrid layer is seen (in between white arrows) with a separation gap. Smear layer remnants are found in the formed interfaces (asterisks).
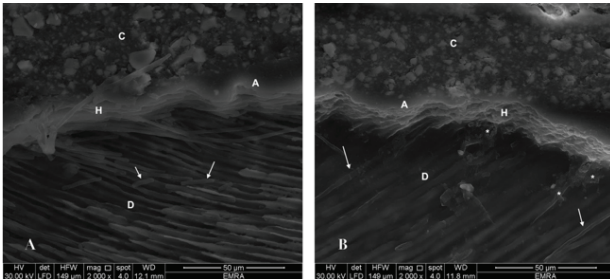
El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

Figure 6 - SEM micrographs (2000x) of group TS-L, after 4000 thermal cycles. A) A well-formed hybrid layer is seen with up to 120 μm long rounded resin tag extensions. Some resin tag sections have become separated (white arrows). B) Resin tag extensions could be seen (white arrows). Isolated irregular or pebble-shaped resin inclusions are found (asterisks). No separation gaps related the hybrid layer are seen.

Isolated 'broken' horizontal resin tags were found separated from the main tags, Figure 6(A), probably due to the aging process. [65] Irregular, pebble-shaped inclusions were also found, Figure 6(B). Transmission electron microscopy is needed to identify the nature of such inclusions. An Intact hybrid layer was found with no evidence of separation in any of the TS-L sections.

The absence of separation may be due to the complete lack of water in the bonding interface, which preserved the integrity of the bond even after thermal cycling. In addition, the presence of MDP and NaF in the Clearfil Liner Bond F may have contributed to the increased bond strength. Barcellos et al. [27] found laser irradiation to increase the modulus of elasticity of the formed hybrid layer, causing less polymerization shrinkage stresses at the interface. This may explain the intact hybrid layer seen in group TS-L.

It is unclear whether the laser morphologically altered the dentin to receive the adhesive in group TS-L, or if the laser had modified the smear layer to allow the penetration of the bond. It has been suggested that the laser energy could morphologically alter the smear layer, causing its melting and evaporation [36]. More in-depth analysis with transmission electron microscopy is needed to observe the formed bond interface on the structural level.

Despite the variations in hybrid layer formation, there was no statistically significant difference in the SBS between group TS-L and groups OS/OS-L after 4000 thermal cycles. However, SEM analysis of groups OS and OS-L suggests the initiation of deterioration of the adhesive interface, denoting future failure. More investigations with longer aging periods are therefore required.

According to Sakaguchi et al. [51], typical values of macro shear bond strength tests range from 10 to 50 MPa. The macro shear bond strength values obtained in this study, however, range from 3.8 – 8.8 MPa. The lower values are most probably due to the thermocycling process that was done, which favors proteolytic hydrolysis of bonded interfaces [14]. Especially with self-etch adhesives, water aging has been found to cause water uptake and plasticization [19], compromising the bond integrity.

Another plausible reason for the low values is the difference in the modulus of elasticity and strength between the resin cement used and dentin. According to Mondragon E. et al. [66], shear bond strength is heavily dependent on the strength of the substrate. A study by Raphaela R. et al. [67] on the shear bond strength between resin cement and bovine dentin that was treated with a primer and bonding agent resulted in values as low as 0.34 MPa after thermocycling.

A difference between the current study and other studies that evaluated the effect of laser irradiation on adhesives placed on dentin is the usage of composite blocks cemented to the dentin via resin cement as done in the current study versus the direct build-up of composite on the dentin's surface. This could explain the lower values obtained in this study, due to the higher strength of the direct composite filing as opposed to the strength of resin cement.

In this study, a self-adhesive resin cement was used. Self-adhesive resin cement possess lower mechanical properties in comparison to conventional resin cements [68,69]. Owing to their chemical composition, self-adhesive resin cements often possess inadequate neutralization

El-Hakim NM et al.

Effect of diode laser irradiation of bonding agents before curing versus standard bonding protocol on the shear bond strength between resin cement and dentin

capacity [70] and are prone to delayed or even incomplete conversion upon curing, especially if left to self-cure [71].

The surface area of the bond interface used in this study have also played a role. The diameter of the composite block cemented to the dentin samples measured 6 mm, with a surface area of 28 mm2. According to the shear bond strength calculating equation, $(\tau) = F / \pi dh$,[51] where d is the diameter of the specimen, the wider the interface, the less the shear bond strength value. It is worth noting that all previously mentioned studies that incorporated macro SBS testing bonded areas 3 – 4 mm in diameter, which explains the higher values.

This being an invitro study, limitations exist regarding its correlation to the clinical setting. One such limitation is the 2 mm dentinal sections used in this study, which do not represent the clinical situation, in which the vital dentin has its own inherent 'wetness' from the pulpal outwards flow of fluid with different pressure changes within the tooth.[5,19] Water content from the dentin can lead to different outcomes with this technique, especially since simplified water-based adhesives have been deemed permeable to dentinal fluids [15,18].

The results of this study indicate that diode laser irradiation of an uncured adhesive has the ability to increase adhesive penetration, bond strength, and durability after aging despite the lack of a priming agent. Evidence also support that the success of this technique is related to the type of adhesive system used. Further studies thus are encouraged to optimize this bonding protocol in more clinically oriented settings.

CONCLUSIONS

Within the limitations of this study, based on the results obtained it can be concluded that:

1. Diode laser irradiation of an uncured two-step self-etch adhesive bonding agent without prior priming, group TS-L, increased the shear bond strength to dentin;

2. Diode laser irradiation of an uncured one-step self-etch adhesive, group OS-L, did not influence the bond strength to dentin;

3. As was evident from SEM analysis, group TS-L exhibited the highest adhesive penetration and resin tag lengths;

4. Diode laser irradiation of an acid-free bonding agent before its curing is capable of producing a higher bond strength to diamond bur cut dentin. This technique could have the potential to replace acid etching and achieve high bond strengths without subjecting dentin to acidity.

DISCLOSURE

No conflicts of interest.

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