Brazilian Dental Science
Ciência Odontológica Brasileira
unesp
UNIVERSIDADE ESTADUAL PAULISTA "JÚLIO DE MESQUITA FILHO"
Instituto de Ciência e Tecnologia
Campus de São José dos Campos

ORIGINAL ARTICLE
doi: 10.14295/bds.2015.v18i4.1172

Mechanical properties of the bond interface associated with ND:YAG laser

Propriedades mecanicas da interface adesiva associada ao laser de ND:YAG

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ABSTRACT

Objective: This study evaluated the hardness and modulus of elasticity of the dentin bond interface using total-etch (Single Bond /SB) and self-etch (Clearfil SE Bond/CSEB) adhesives associated with Nd:YAG Laser irradiation through the unpolymerized adhesives. **Material and Methods:** The occlusal surfaces of 12 human third molars were ground until superficial dentin was exposed. A standardized circular cavity was performed on the occlusal surface. Specimens were sectioned in the mesio-distal direction, and the 24 hemi-crowns were divided into four groups: Group SB/Control - SB + polymerization; Group SB/Laser - SB + Nd:YAG laser (174.16J/cm^2/60s/non-contact) + polymerization; Group CSEB/Control – CSEB + polymerization; Group CSEB/Laser - CSEB + Nd:YAG laser (174.16J/cm^2/cm^2/60s/non-contact) + polymerization. Composite were placed in the cavities and polymerized. The specimens were immersed in distilled water and stored in an oven at 37ºC for 24h and then submitted to nanoindentation in a Nano Indenter® XP appliance. **Results:** The results were submitted to ANOVA, Tukey's test and Student's-t test ($p < 0.05$). **Conclusion:** It was concluded that the application of the Nd:YAG laser in both adhesive systems did not changed the hybrid layer hardness; however, it increases the modulus of elasticity in the hybrid layer for both adhesives tested and it maybe preserves the integrity of the adhesive interface and its durability. **Clinical relevance:** The application of Nd:YAG laser prior to photopolymerization of adhesive systems can increase the modulus of elasticity in the hybrid layer and may contribute to stress distribution in the adhesive interface during the polymerization preserving the integrity of the adhesive interface and its durability.

KEYWORDS

Dentin; Hardness; Dentin-bonding agents; Elastic modulus.

RESUMO

Objetivo: este estudo avaliou a dureza e o módulo de elasticidade da interface adesiva a dentina usando adesivo convencional (Sinngle Bond /SB) e adesivo autocondicionante (Clearfil SE Bond/CSEB) associados com irradiação do Laser Nd:YAH sobre os adesivos não polimerizados. **Material e Métodos:** As superfícies oclusais de 12 terceiros molares humanos foram desgastadas ate exposição de dentina sueprficial plana. Cavidades circulares padronizadas foram realizadas na superficie oclusal. Os espécimes foram seccionados no sentido mesio-distal, e as 24 hemi-coroas foram divididas em quatro grupos: Grupo SB/Controle - SB + fotopolimerização; Grupo SB/Laser - SB + Laser Nd:YAG (174,16J/cm^2/60s/não-contato) + fotopolimerização; Grupo CSEB/Controle – CSEB + fotopolimerização; Grupo CSEB/Laser - CSEB + Laser Nd:YAG (174.16J/cm^2/cm^2/60s/ não-contato) + fotopolimerização. Restaurações de resina composta foram realizadas nas cavidades e fotopolimerizadas. Os espécimes foram armazenados em água destilada a 37 ºC por 24 h e submetidas a nanoindentação no aparelho Nano Indenter® XP. **Resultados:** Os resultados foram submetidos ao ANOVA, seguidos dos testes de Tukey e T-Student ($p < 0,05$). **Conclusão:** Foi concluído que a aplicação do Laser de Nd:YAG nos adesivos não alterou a dureza da camada híbrida; entretanto, aumentou o módulo de elasticidade de ambos os adesivos testados e talvez preserve a integridade da interface adesiva e sua durabilidade.

PALAVRAS-CHAVE

Dentina; Dureza; Sistemas adesivos; Módulo de elasticidade.

INTRODUCTION

Since the introduction of the acid etching technique by Buonocore et al. [1] in 1955, dental materials and restorative techniques have being modified to improve longevity and durability of restorations. The durability of the bond interface is related to several factors, one of them is the quality of the hybrid layer. Ideally, the resin monomers of adhesive systems infiltrate the collagen fibers of dentin, which are exposed by acid etching [2]. However, recent studies have shown that this is rarely achieved [3-5]. Therefore, alternatives have been studied to improve the pattern of bonding. Among these alternatives is the use of various forms of laser energy[6-8].

In 1999, Gonçalves et al. [8] evaluated whether the application of Nd:YLF laser on dentin in the presence of an uncured adhesive system could cause fusion of the chemically modified dentin by the presence of the adhesive, and form a more resistant hybrid layer. The results obtained in that study showed a significant increase in bond strength and proved the recrystallization of dentin[8]. This methodology was later studied by other authors, who found a positive influence on bond strength [9-12] and reduction in nanoleakage [13] with the use of this dentin treatment. However, despite these positive results, the previously mentioned studies were not conclusive about the reasons that led to the increase in bond strength and reduction in nanoleakage.

Based on the alterations provided by the application of laser energy, it is believed that there may be a correlation between the mechanical properties of the bond interface and an increase in bond strength obtained with this treatment. The nanohardness and modulus of elasticity mechanical properties can be analyzed using nanoindentation. Differently from the conventional microhardness tests, a great deal of quantitative information is obtained from the load-displacement data, as opposed to only visual observations of the indentation in the sample [14-16].

Thus, this *in vitro* study uses the nanoindentation technique to evaluate the mechanical properties (nanohardness and modulus of elasticity) of the bond interface between human dentin and a conventional (Single Bond) or self-etching (Clearfil SE Bond) adhesive system, with or without Nd:YAG laser treatment before light polymerization.

The hypotheses tested were: a) Nd:YAG laser does not interfere in the nanohardness of the hybrid layer; b) Nd:YAG laser does not interfere in the modulus of elasticity of the hybrid layer; c) The hybrid layer does not show differences in the hardness and modulus of elasticity when compared to the adhesive layer, with or without laser irradiation.

MATERIAL AND METHODS

Specimen preparation

Twelve healthy, erupted, and recently extracted human third molars were used. The Research Ethics Committee of the São Jose dos Campos School of Dentistry – UNESP approved this study (protocol number 080/2009-PH/CEP). The teeth were cleaned and stored in physiological solution with 1% Thymol at a temperature of -18°C for a period from 45 to 60 days, up to the time they were used [8].

The roots were embedded in self-polymerizing acrylic resin (Classic, São Paulo, Brazil) and the occlusal surfaces were ground WITH 400-grit silicon carbide paper, in a polishing machine (Politriz, Struers A/S, Copenhagen, Denmark) under water cooling, until superficial dentin was exposed. A cavity preparation in dentin using a 3053 diamond tip (KG Sorensen, Rio de Janeiro, Brazil) at high speed under water cooling measuring a minimum of 5 X 5 mm was made on the occlusal surface, which was measured using a caliper (Golgran, São Caetano do Sul, Brazil). It was standardized 2 mm depth of cavity preparation.

The crowns were adapted to a cutting machine (Labcut 1010 – Extec Corp., Enfield, CT, USA) to perform a vertical section of the crown in the occlusal-cervical direction, dividing it into two hemi-crowns.

The 24 hemi-crowns were divided into four groups of six hemi-crowns each, according to the adhesive system and dentin treatment:

Group SB/Control - The cavities were etched for 15 s with 37% phosphoric acid gel, rinsed and the excess moisture was removed with absorbent paper. Two layers of Single Bond/SB total-etch adhesive (3M ESPE, St. Paul, MN, USA) were applied on the surface actively for 15 s and gently air dried for 10 s. The adhesive was light activated for 10 s with a Curing Light XL 3000 (3M Dental Products, St.Paul,USA) with power density of 600 mW/cm2. The tip was at a 90º angle, perpendicular and to the dentin surface, and at a distance of 1 mm from it.

Group SB/Laser - The cavities received the application of SB total-etch adhesive (3M ESPE), following the same protocol utilized for Group SB/Control. Before light polymerization, the cavities were irradiated with Nd:YAG laser based on protocol used by Marimoto et al. [9] and Ribeiro et al.[10]. The Nd:YAG laser equipment used in this study was the Laser Pulse Master 600 iQ (American Dental Technologies Inc, TX, Corpus Christi, USA) at a wavelength of 1.064 μm. The output energy of this laser device was 140 mJ per pulse; with a pulse repetition rate of 10 pulses per second (10 Hz), and was applied freehand by one calibrated operator, in non-contact mode (tip 320 μm in diameter) scanning mode for 60 s on a 5 mm X 5 mm cavity preparation. The energy density was 174.16 J/cm2. During laser application the laser tip was at a 90º angle, perpendicular and to the surface, and at a distance of 1 mm from it. Then, the adhesive was light activated for 10 s following the same protocol utilized for Group SB/Control.

Group CSEB/Control - The cavities received the application of Clearfil SE Bond/CSEB self-etch adhesive (Kuraray Medical Inc, Tokyo, Japan). One layer of primer agent was applied actively for 20 s and gently air dried for 10 s. One layer of bonding agent was applied actively for 20 s and gently air dried for 10 s. The adhesive was light activated for 10 s following the same protocol utilized for Group SB/Control.

Group CSEB/Laser - The cavities received the application of CSEB self-etch adhesive (Kuraray), following the same protocol utilized for Group CSEB/Control. However, before light polymerization, the cavities were irradiated using a Nd:YAG laser following the same protocol utilized for Group SB/Laser. Then, the adhesive was light activated for 10 s following the same protocol utilized for Group SB/Control.

Restoration placement

Two increments of resin composite, *Filtek* Z350 (3M ESPE/ St. Paul, MN, USA), were placed in the cavities and polymerized for 40 s each.

The test specimens were then immersed in distilled water and stored in an oven at 37ºC for 24 h. After this period, the bond interfaces were finished with wet abrasive papers for 30 s each in a sequence from 600 to 4000 grit; polished in a Polishing machine using a felt disc and 1 μ diamond paste, until a mirror surface was obtained, and ultrasonically cleaned for 20 min.

Nanoindentation

The interface of each sample was measured to evaluate the nanohardness and modulus of elasticity using a nanoindenter, Nano Indenter® XP (MTS®, MN, USA). The test was computer-controlled, and indentations were made at a temperature of 26 ± 0.5 ºC.

The measurements were made at intervals starting from the hybrid layer in a direction towards the adhesive layer, with an interval of 25 μm distance from each measurement diagonally along the interface. A total of 5 indentations were performed in each layer (hybrid layer and adhesive layer). The load used in this study was 4 mN; loading and unloading time was 30 s and between each cycle, the load was maintained constant for a period of 5 s. The tests were performed using a Berkovich tip with a pyramid shape.

A curve of applied force (P) was constructed as a function of vertical displacement of the tip (h), which was recorded considering the time. The curve of force was characterized by a region of load and the other of unloading. During loading, the tip came into contact with the tooth surface and the force was gradually increased up to 4 mN. Once the stress between the tip and surface overtook the limit of flow, the load curve resulted in a combination of elastic and plastic deformation of the tooth. After attaining the maximum value of force, the tip started to move backwards, with the evaluated surface accompanying the tip until all of the elastic recovery occurred. These data characterized the unloading curve, which is the response with reference to the elastic recovery of the surface. Analysis of the results obtained was made using the method developed by Oliver and Pharr [17], in the following manner:

The basic equation for the hardness measurements is:

$$H = \frac{P}{A}$$

The projected area of contact, A is calculated evaluating a function of the area determined at a depth of contact hc; that is:

$$A = f(h_c)$$

This calculation is obtained during calibration of the tip. For an ideal Berkovich penetrator, it follows that:

$$A = 24.5h_c^2$$

In order to obtain the values of the modulus of elasticity, the effect of the indenters that are not perfectly rigid; that is to say, the deformations they undergo are taken into consideration with the introduction of the so-called reduced elastic modulus Er, defined by the equation:

$$E^* = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_p}}$$

The specimens were individually tested, and the nanohardness and modulus of elasticity (GPa) values were submitted to ANOVA and Tukey tests, at a level of significance of 5% ($p < 0.05$).

RESULTS

Hardness

Table 1 shows the results of ANOVA. When the factor layer and its interaction with the factor laser and the factor adhesive were analyzed, statistical differences were observed.

Table 1 - ANOVA for the hardness data obtained

Effect	GL	F	p
Laser	1	2.03	0.170
Adhesive	1	0.29	0.598
Laser X Adhesive	1	0.76	0.394
Layer	1	39.67	0.000*
Laser X Layer	1	5.66	0.027*
Adhesive X Layer	1	19.14	0.000*
Laser X Adhesive X Layer	1	0.07	0.800

*p<0.05.

Table 2 presents the results of the Tukey test (5%) for the factor Adhesive Layer. The Group CSEB/Control presented significantly lower nanohardness values when compared with the other groups.

Table 2 - Results of the Tukey Test with reference to hardness for the factor Adhesive Layer

Layer	Adhesive	Laser	Mean (GPa)	Homogeneous Groups	
Adhesive	CSEB	Without Laser	0.253	A	
Adhesive	SB	Without Laser	0.349		B
Adhesive	CSEB	With Laser	0.352		B
Adhesive	SB	With Laser	0.390		B

* The groups accompanied by the same letters presented no significant differences.

Modulus of Elasticity

Table 3 presents the results of ANOVA for the experimental conditions. The interaction of the three study variables was significant. Thus, the relationship between the adhesive system - with regards to the application of laser - and the adhesive layer was not the same as it was in the hybrid layer.

Table 3 - ANOVA for the modulus of elasticity data

Effect	GL	F	*p
Laser	1	5.94	0.024*
Adhesive	1	0.02	0.903
Laser X Adhesive	1	4.85	0.039*
Residue I	**20**		
Layer	1	23.94	0.001*
Laser X Layer	1	0.50	0.485
Residue II	**20**		
Adhesive X Layer	1	0.06	0.805
Laser X Adhesive X Layer	1	7.52	0.012*

*p<0.05.

The results of the Tukey test for the adhesive layer are shown in Table 4. The Group CSEB/Laser presented significantly lower modulus of elasticity values when compared with SB/Laser. The SB/Control presented significantly lower modulus of elasticity values when compared with SB/Laser and CSEB/Control.

DISCUSSION

This current study evaluated the alterations in the nanohardness and modulus of elasticity promoted by Nd:YAG laser irradiation using the nanoindentation technique on the bond interface. Physical and mechanical tests such as hardness and modulus of elasticity are important to observe the stress distribution in the adhesive interface, as this region is subject to tensile and shear forces during chewing. The elasticity of the adhesive interface is important to determine the stress generated in this region during the polymerization shrinkage[18]. Depending on the value of the modulus of elasticity of the adhesive system and hybrid layer, these layers may function as dampen tensions, preserving the integrity of the adhesive interface and its durability [19,20]. The study of these properties is also important in the research of fractures at the interface, since cracks can be generated and propagated in the presence of critical stress in the region [21].

Table 4 - Results of the Tukey test for the Modulus of Elasticity values of the adhesive layer

Laser	Adhesive	Mean (GPa)	Homogeneous Groups		
With Laser	SB	12.9	A		
Without Laser	CSEB	11.1	A	B	
With Laser	CSEB	9.8		B	C
Without Laser	SB	8.4			C

* Equal letters indicate groups that are homogeneous.

The parameters of Nd:YAG laser used in this study were based in previous studies [9,10] that used high energy densities for irradiation on dentin impregnated with non-polymerized self-etch and total-etch adhesives, and that observed favorable results of dentin bond strength. Therefore, it was used 140 mJ, 10 Hz and 174.16 J/cm2 energy density of Nd: YAG laser with the purpose to find favorable results in the nanohardness and modulus of elasticity on the bond interface using this technique.

Application of Nd:YAG laser was not found to influence the nanohardness of the hybrid layer, irrespective of the type of adhesive system used, therefore, the first hypothesis was accepted. The group that was irradiated with laser did not present a distinct behavior for the variable hardness in the hybrid layer in comparison with the non irradiated group. Nevertheless, the CSEB presented a lower nanohardness value in the adhesive layer, without laser application, when compared with the other groups (Table 3). Batista et al. [22] observed that the use of Nd:YAG Laser (140 mJ, 10 Hz, 174.16 J/cm2) on the non polymerized SB promoted a significant increase in the rate of solvent evaporation, probably due photothermal effect of the Nd:YAG Laser, increasing the surface temperature at the irradiation site, and consequently increasing the degree of solvent evaporation. Also, a maintenance of the collagen network may occur by non polymerized adhesives and control of intrinsic water because Nd:YAG laser irradiation acts on the dentin substrate optimizing the evaporation of the solvents [22]. Consequently, this factor can minimize the problem of the amount of water in the dentin structures during the adhesive application, which can increase conversion of the adhesive monomers and optimize the longevity of adhesive restorations [22].

Sandr et al. [23] observed higher nanohardness values when increasing the degree of solvent evaporation. Other studies have shown that removal of residual water or solvent enhance the mechanical properties of the resin within the hybrid layer [24,25]. Maybe Nd:YAG Laser irradiation on the non polymerized CSEB can improved the degree of solvent evaporation of this adhesive, and, consequently, increased the nanohardness value in the adhesive layer compared to Group CSEB/Control.

Also, the Group CSEB/Control presented a lower nanohardness value in the adhesive layer when compared with Groups SB/Control and SB/Laser. This result can be due the differences of the two adhesives tested regarding the type of monomeric system, the type and amount of inorganic filler particles, the particles silanization quality, among other aspects [26].

The second null hypothesis was rejected because, application of Nd:YAG laser influenced the modulus of elasticity of the hybrid layer. The highest mean for the hybrid layer occurred in the samples that received SB followed by Nd:YAG laser irradiation (13.98 ± 2.7 GPa). Alternatively, the lowest modulus of elasticity value of the hybrid layer occurred in the Group SB/Control (without laser) (11.22 ± 2.07 GPa). According to some authors, the probable increase in the modulus of elasticity of the hybrid layer may be due to dehydration of the collagen [27,28]. In the present study, the laser was applied on the adhesive surface before it was light polymerized, a fact that could cause dehydration of the collagen. Another factor in the increased modulus of elasticity could be the reduction in the presence of water within the hybrid layer due to the possible evaporation caused by the laser, which would facilitate the conversion of monomers into polymers [22,29].

Higher modulus of elasticity in the hybrid layer can hypothesize greater penetration of the adhesive, ensuring a higher concentration of monomers within the partially demineralized dentin [30-32]. The Nd:YAG Laser irradiation on the non polymerized SB increases the degree of solvent evaporation of this adhesive [22], and, consequently, can maybe increased the penetration of the adhesive within the partially demineralized dentin. Consequently, after the polymerization, a polymer tougher maybe formed with higher elastic modulus in the hybrid layer.

The third null hypothesis was rejected, because the present study proved this superiority of the hybrid layer when compared with the adhesive layer with regards to the harness values, corroborating the findings of other authors [14,33,34]. The hardness and modulus of elasticity of a material mainly depend on its density and chemical composition, characteristics that may be altered when changes in its structure occur, or due to heat or mechanical treatments to which the material is submitted [35]. It is believed that the collagen network present within the hybrid layer increases in rigidity, and consequently in hardness values in relation to the adhesive layer [3].

In the adhesive layer, the modulus of elasticity of the SB, which received irradiation, presented a higher mean value when compared with the CSEB. Initially, one could suppose that this may have happened because the SB has water and ethanol as solvent, whereas the CSEB contains only water in the composition of the acid primer [36]. According to Batista et al. [22], as the adhesive system received laser treatment before light polymerization, greater evaporation of the solvent present in SB occurred. Solvent evaporation provides adhesive systems with better mechanical properties [19,37].

Also, authors [8-10] believe that this technique can 'promote the development of a new substrate, in which dentin substrate and adhesive would be fused by the action of laser' [8,9]. This new adhesive substrate is composed of the fusion and recrystallization of dentinal hydroxyapatite in the presence of resin monomers, which can result in a 'more numerous tags and improved adhesive infiltration' [8,9], probably producing a more resistant substrate contributing to better bond strength [8-10].

For the Group SB/Control, a lower modulus of elasticity value was observed. This result may indicate that, during the application of this adhesive, the total removal of the solvents and water did not occur, and according to some authors, this would provide lower modulus of elasticity values [19,38].

In addition, the modulus of elasticity of the interface continued to provide lower values than those of dentin and resin composite, even after the application of laser, which demonstrated maintenance of the characteristic of "elastic cavity" [39]. Therefore, the increase of the modulus of elasticity with characteristic of "elastic cavity" might decrease stress generated in the hybrid layer during the polymerization shrinkage, preserving the integrity of the adhesive interface and its durability.

No clinical studies were found in the literature using the technique developed by Gonçalves et al. [8], which could predict the influence of Nd:YAG Laser irradiation on dentin impregnated with non polymerized adhesive on the longevity of the restoration.

The results obtained in this study cannot be directly extrapolated to a clinical situation, therefore, further "in vitro" studies are necessary to observe the behavior of the bond interface, using the irradiation technique with Nd:YAG Laser with others Nd:YAG Laser parameters, higher power levels and lower frequencies associated with simulate the pulp pressure.

Our results may be suggested that the mechanism of action of the Nd:YAG laser can increase the modulus of elasticity of adhesive layer, modifying the interaction of this layer with the dentin substrate during the bonding process, a factor that may contribute to greater longevity of composite restorations.

CONCLUSION

Based on the current methodology, the application of Nd:YAG laser on the adhesive systems before light curing increased the modulus of elasticity of the hybrid layer, irrespective of the type of adhesive system used.

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Date submitted: 2015 Sep 14

Accept submission: 2015 Dec 02