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Bond strength of bulk fill composite to teeth prepared with Er:YAG laser

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Aim: The present *in vitro* study aimed to evaluate the bond strength of a bulk fill composite on dentin surfaces prepared with the Er: YAG laser. **Methods:** Twenty-four permanent third molars were selected and divided into 2 groups: CP - Conventional preparation with high-speed handpiece (control) and LA (laser) - Preparation with Er: YAG laser. The occlusal surface was removed to expose coronal dentin, which was subsequently prepared with a high-speed handpiece or Er: YAG laser (350mJ, 4Hz, 1.5 ml/min water flow). Both groups were restored with Filtek One Bulk Fill (3M ESPE) composite resin. After 24 hours, the samples were evaluated for microtensile bond strength (μTBS), fracture pattern, and scanning electron microscopy (SEM). **Results:** The data obtained in the μTBS test were submitted to *t*-test (α=0.05). The results showed no difference in μTBS when the different types of cavity preparation were compared (ρ=0.091). Fracture patterns revealed the prevalence of cohesive fracture in composite resin in CP (83.3%) and adhesive fracture in LA (92.1%). In the SEM analysis, the LA group demonstrated the presence of gaps between the composite resin and the irradiated dentin surface. The hybrid layer exhibited more regularity with the presence of longer and uniform resin tags in the CP group. **Conclusion:** The type of cavity preparation did not influence the values of bulk fill composite resin μTBS to dentin. Fracture patterns and scanning electron microscopy analyses suggested less interference at the adhesive interface in preparations performed using CP.

Keywords: Composite resins. Dental materials. Lasers. Adhesiveness.

Introduction

Composite resins are popular in dental clinics, due to their characteristics of mimicking dental structures, and their high physical[1] and mechanical[2] properties. However, despite all the technology and research applied to the development of composites, the knowledge about the tissues to which they must adhere, and improvement in restorative materials and techniques must be constantly evaluated[3.]

The search for faster and simpler clinical procedures, along with the attempt to reduce polymerization shrinkage, led to a new class of resin composites, known as bulk fill, which have gained increasing visibility in the market[4]. Whereas conventional composites are typically inserted in increments with a maximum thickness of two millimeters[5], the bulk fill resins allow a reduction in working time by decreasing the number of increments in the cavity to be restored, as it allows layers of up to four millimeters to be effectively polymerized[4,6].

Bulk fill composite can be inserted in a single increment since changes have been made either in the filler content or in their organic matrix. These approaches can result in lower viscosity monomers by the substitution or reduction of Bis-GMA and/or monomers with higher molecular weight, commonly based on TEGDMA, EBPDMA, Bis-EMA, and UDMA monomers. As a result, a more translucent material, with improved healing capacity and consequent decrease in polymerization shrinkage was obtained[6,7].

The type of composite resin and the filling technique can have a great impact on the resin composite bond to the tooth structure[8.] However, these are not the only factors to be considered. Preparation of the surface to be bonded is important for attaining clinical success and restoration durability[9]. The adhesion mechanism of the restorative material and dentin surface occurs through the interlock between the polymerized monomers and partially demineralized collagen fibrils[10].

The treatment of the dentin surface with Er: YAG laser is among the new methods studied. This is an alternative to the use of conventional rotary instruments, making it possible to eliminate noise during cavity preparation, decrease the pain sensation and perform a more conservative preparation[11-14].

Er: YAG laser is effective in ablating mineralized tissues because it emits a wavelength of 2.94 μm, which coincides with the absorption peaks of water and hydroxyapatite present in dental tissues. With the vaporization of water, there is an increase in the internal pressure of the molecules, generating micro explosions that lead to the eruption of the substrate in the form of microscopic particles, resulting in a micro-retentive pattern[15,16]. Thus, the changes in dentin morphology, resulting from the use of the Er: YAG laser, can promote a larger adhesive area because of surface micro retentions, opening of dentinal tubules, and absence of smear layer. This can influence the bonding quality of restorative materials when compared to cavity preparations performed with diamond burs used in high-speed handpiece[17-20].

Thus, this study aimed to evaluate the microtensile bond strength (μTBS) of an adhesive protocol associating the characteristics of an Er: YAG laser-prepared dentin surface and the advantages of bulk fill composite and the develop a faster restorative procedure with less polymerization shrinkage. The null hypothesis would be that the Er: YAG laser does not promote differences in µTBS than the conventional protocol using diamond bur.

Materials and methods

Experimental Design

This is a randomized study and the sample consisted of 30 dentin blocks, of which 24 blocks were used in the bond strength test (n= 12) and 6 in the morphological analysis (SEM) of the adhesive interface between the bulk fill composite and the dentin surface (n= 3). The groups were determined as follows: CP- Conventional preparation using high-speed handpiece (control) and LA - Preparation with Er: YAG laser. The quantitative variable response was µTBS (MPa). Qualitative analysis of fracture patterns (adhesive, cohesive or mixed) and scanning electron microscopy (SEM) were also performed to assess the adhesive interface.

Specimens preparation

After approval by the ethics committee (CAAE: 25790619.7.0000.5385), 30 healthy third molars (absence of carious lesions or fractures) were selected and cleaned with periodontal curettes (Millenium, Golgran, São Caetano do Sul, SP, Brazil), pumice paste/water (SSWhite Produtos Odontológico, São Cristovão, RJ, Brazil) and rubber cups (Soft, American Burrs, Palhoça, SC, Brazil). The teeth were stored in distilled water and kept in an oven at 37°C until the beginning of the experiment. The clinical crown was horizontally sectioned with the aid of a diamond-cutting disc mounted on a cooled cutting machine (Isomet 1000, model 11-2180) to remove 1/3 of the occlusal surface, thereby obtaining exposure to all the dentin. Subsequently, the coronal dentin was flattened with abrasive papers #600 and #1200, using a metallographic polisher (DP-9U2; Struers S/A, Copenhagen, Denmark)[21].

Preparation Technique

For samples that received Er: YAG laser preparation (Kavo Key Laser II – Kavo-Corp. Biberach, Germany) the non-contact mode was used, perpendicularly to the surface and focused at a distance of 12 mm from the sample[22] with scanning of the entire surface. This laser has a fiber diameter of 0.63mm; irradiation was performed using the energy of 350mJ and frequency of 4Hz[17], with a constant flow of 1.5 mL/min of water[23].

For the samples that received cavity preparation with a high-speed handpiece (Dabi Atlante, Ribeirão Preto, Brazil), a diamond tip #2096 (KG Sorensen, Alphaville, SP, Brazil) was used, perpendicularly to the surface, under constant cooling with distilled water[23] and treating the entire dentin surface.

Restorative Procedure

Immediately after performing the cavity preparation (conventional with high-speed handpiece or Er: YAG laser), the samples in all groups received the restorative material.

35% Phosphoric acid was applied (3M, ESPE St. Paul, MN, USA) for 15 seconds on the dentin surface, followed by washing with distilled water for 30 seconds and drying with absorbent paper. Subsequently, with the aid of a disposable brush, two layers of the Single Bond 2 adhesive system (3M, ESPE St. Paul, MN, USA) were applied with gentle jets of air between them to allow volatilization of the solvent. The adhesive system was light-cured for 10 seconds as recommended by the manufacturer (Radii-Cal SDI, Victoria, Australia).

Dentin surfaces were restored with Filtek One Bulk Fill resin (3M ESPE St. Paul, MN, USA), by the single increment technique, in a single layer 4mm high. The polymerization was carried out for 40 seconds, at a distance of 1 cm.

The samples were kept in distilled water and stored in an oven at 37°C for 24 hours for performing the μTBS test.

Microtensile Test

After storage, the specimens (n=12) were placed in the water-cooled diamond saw and sectioned to obtain four sticks of each tooth, measuring approximately 1.0mm^2 (+-0.2mm^2). The sticks were measured, identified and fixe,d in the device used for the μTBS test by using cyanoacrylate gel glue (Super Bonder gel, Henkel Ltda., São Paulo, SP, Brazil). Then, the μTBS test was performed in a Universal Testing Machine (EZ Test - Shimadzu, Tokyo, Japan) at a speed of 0.5 mm/min until failure occurred.

The values obtained were recorded in Newton (N). The average of each tooth (four sticks) was calculated and, finally, the mean values were from each tooth were obtained. The dimension of the fractured area was recorded with a digital caliper (King Tools 150mm/6", São Paulo, SP, Brazil) and subsequently, the microtensile bond strength values were converted into Megapascal (MPa)[21].

Fracture Pattern Analysis

After the specimen ruptured, the surfaces were evaluated with the aid of a clinical microscope (model ALL 03 - EL. Commercial Alliance of São Carlos Ltda. - ME, São Carlos, SP, Brazil) to identify the type of fracture. Specimens were evaluated at 16X magnification. Failures were classified as adhesive (fracture between the substrate/restorative material interface), cohesive in dentin (fracture in dentin), cohesive in resin (fracture in the restorative material) or mixed (combined adhesive and cohesive fracture).

Scanning Electron Microscopy of Bond Interface

Three specimens were prepared for each group, following the same preparation and restoration protocols performed for the microtensile strength test. After the restoration, the specimens were sectioned longitudinally with a double-faced diamond disk, finished with water abrasives paper with decreasing grain (#600 and 1200) on the inner portion of the adhesive interface and polished with pastes containing aluminum in suspension, with a granulation of 0.3 μm (Arotec, Cotia, SP, Brazil). The specimens were washed in an ultrasound bath for 10 minutes to remove possible residues on the surface.

After obtaining the specimens, they were immersed for 12 hours, in a glutaraldehyde solution (2.5%) in 0.1M sodium cacodylate buffer, pH7.4. After this period, the specimens were washed with distilled water. The sections were then dehydrated with ascending grades of ethanol: 25% (20 min), 50% (20 min), 75% (20 min), 95% (30 min) and 100% (60 min).

Later, they were fixed on metal stubs, sputter coated with gold and analyzed by scanning electron microscopy (EVO 50; Carl Zeiss, Cambridge, England). Representative areas were photographed at 1500X, aiming to verify the quality of hybrid layer restoration, the presence of irregularities and gaps[21].

Data Analysis

The data were assessed for normality with the Shapiro-Wilk test. The results were submitted to *t*-test at the significance level of α=0.05. All statistical analyses were carried out using SPSS for Windows software (IBM SPSS Statistics 21).

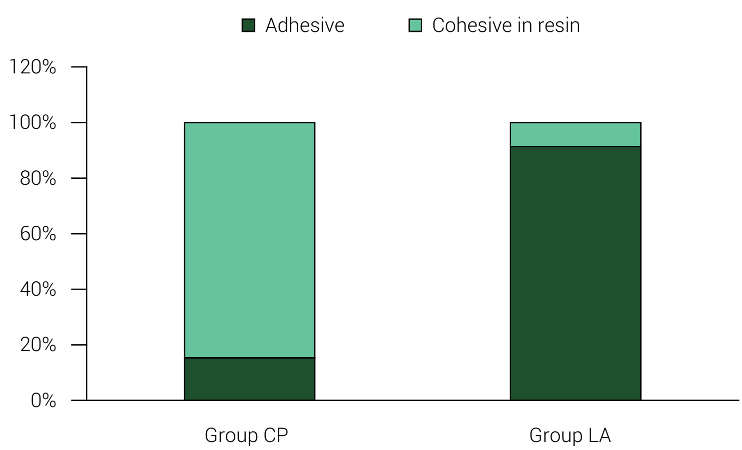
Results

The results showed no difference in μTBS between the different types of cavity preparation (ρ=0.091) (Table 1).

Table 1. Mean (±SD) of μTBS values (MPa) of the specimens that received preparations performed with high-speed handpiece or Er: YAG laser

Groups	μTBS
CP (Conventional preparation)	27.81 (7.68)
LA (Er:YAG laser)	34.68 (12.66)

Relative to fracture patterns, the prevalence of cohesive fracture in composite resin in CP (83.3%) and adhesive fracture in LA (92.1%) were observed (Graph 1).



Graph 1. Percentage of fracture patterns for each group studied

Scanning Electron Microscopy analysis of the bond interface of group LA demonstrated the formation of irregular hybrid layer, with presence of cracks between the resin composite and irradiated dentin surface, with accumulation of adhesive in the regions of valleys and a thin layer in the regions of peaks. Short and small quantity of resin tags were observed. In Group CP, formation of regular hybrid layer was observed, with presence of numerous, longer and uniform resin tags (Figure 1).

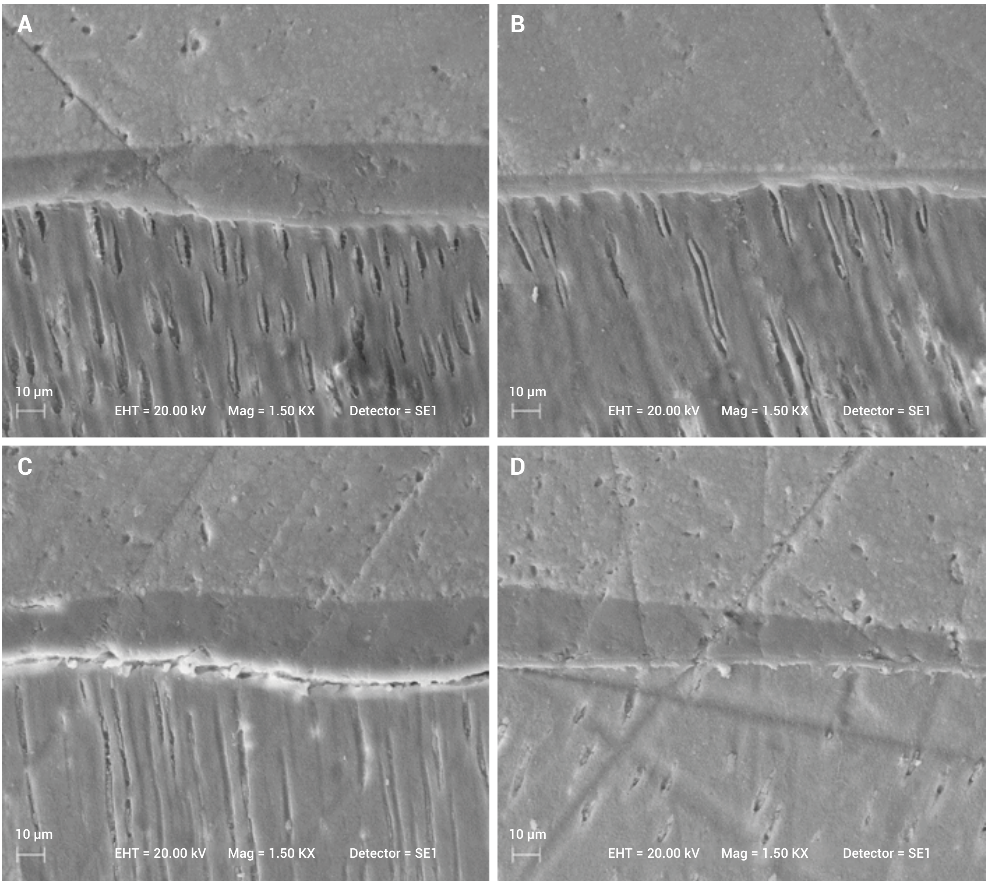


Figure 1. Images representative of scanning electron microscopy in the different groups. A and B represent samples of Group CP (conventional preparation). C and D represent samples of Group LA (Er: YAG laser).

Discussion

An effective bond between the restorative material and the tooth structure is an essential factor for successful procedures in restorative dentistry. A failure in the adhesive protocol can damage tooth/restoration interface, which may lead to marginal discolorations, recurrence of caries lesions, postoperative hypersensitivity and other harmful impacts on the pulp[24].

The null hypothesis was accepted since the results showed that there was no significant difference in the µTBS of bulk fill composite to surfaces conventionally prepared with high-speed handpiece or with Er: YAG laser. The results obtained in this in vitro study suggest that the adhesive protocol is efficient and can be a good alternative for restorative procedures cases like in pediatric dentistry, geriatric dentistry, and special patients by reducing clinical time and facilitating care.

Bulk fill composite resin is still a new material on the market and is being scientifically tested[6,25,26]. As for the adhesion to dentin substrate, regardless of the adhesive used[25] and the type of cavity[27,28], its bond strength showed high μTBS values, validating the results found in the CP group. No studies in the literature have evaluated the bond strength between bulk fill composite and Er: YAG laser-treated dentin. However, a study reported by Tekce et al.[29] (2018) evaluated, qualitatively and quantitatively, the microleakage of bulk fill in enamel prepared with diamond bur or laser through SEM analysis and like in the LA group of this study, also found irregular hybrid layer and gaps at the adhesive interface, even though it was statistically similar to the conventional preparation.

The adhesive system used in this study was the conventional two-step system (Single Bond 2 - 3M) and the μTBS results found did not corroborate the study by Ramos et al.[30] (2014), which states that the self-etching adhesive system showed better results than the etch-and-rinse for Er: YAG laser preparation. Future studies can be developed to evaluate different adhesives systems under these same conditions, however the results found in a literature review by Lopes et al.[31] (2015) are contradictory, because there is no defined standard protocol and laser data vary from the type of adhesive, parameters, restorative material and even the bond strength methodology used.

The laser parameters used in this study were based on the study by Corona et al.[17] (2007) who used 350 mJ energy and a pulse repetition rate of 4 Hz, thereby achieving greater depth of the ablation and slight dentinal tubules enlargement when compared with smaller parameters. As regards water flow, the samples were irradiated with 1.5 mL/min of water. This factor is extremely important during surface preparation, as the laser produces less thermal injury to the pulp when it is associated with water-cooling[32,33].

In this study, SEM analysis of the bond interface showed an irregular hybrid layer, with the presence of cracks and gap formation between the composite resin and the dentin irradiated with Er: YAG laser, suggesting changes in collagen fibrils and reaffirming the studies by He et al.[34] (2017) and Aranha et al.[35] (2007). The irregular hybrid layer could have occurred due to the micro retentive surface created by laser irradiation, with the accumulation of adhesive in regions called valleys, where higher pulse energy was used, and a thinner layer of adhesive filling the regions called peaks, as reported in the literature[21,36]. Short and small amounts of resin tags were observed in the group irradiated with the laser, differing from the studies by Aranha et al.[35] (2007) and Galafassi et al.[21] (2014). As the parameters they used were lower, there may have been less potential for ablation of the dentin structure and less influence on the collagen content. As regards the preparations performed with high-speed handpiece, SEM revealed the formation of a regular hybrid layer, with the presence of numerous, longer and more even resin tags.

Although no statistically significant difference values were observed in the microtensile bond strength test, when the fracture patterns were evaluated, it was possible to note a difference between the groups. The prevalence of adhesive fractures found in the LA group corroborated the findings in the study by Comba et al.[37] (2019) who used a conventional resin composite. Irrespective of the adhesive protocol, they observed the same fracture pattern predominance, suggesting that failure in hybrid layer formation might have negatively affected bonding. A possible explanation for these adhesive bond failures was reported in a study that evaluated the chemical and mechanical modifications of dentin irradiated with Er: YAG laser. He et al.[34] (2017) observed that irradiation negatively affected the nanomechanical properties in the subsurface layer of dentin (< 15 μm in depth), with a decrease in organic and mineral components and a higher degree of crystallinity, due to phosphate and carbonate ions being recrystallized during irradiation with the laser. In addition, they observed changes in collagen content, with denaturation of fibrils and a consequent reduction in the interfibrillar space, which limits composite resin diffusion, causing poor hybridization and affecting its bond to the dentin structure.

Despite the limitations of this study, the restorative technique and surface preparation method chosen can directly influence the bond quality[9,19] Further research showing the longevity of these restorations is needed, in order to find out whether the interferences found in this study will harm long-term bond strength. Furthermore, investigations into the interaction of bulk fill resin composites on dentin surfaces prepared with different parameters of Er: YAG laser are also of fundamental importance, with the aim of suggesting an effective protocol for performing direct restorations in posterior teeth, with less invasive preparations and use of restorative materials in procedures that require less time to perform.

In conclusion, the type of cavity preparation did not influence the values of bulk fill composite μTBS to dentin, but fracture patterns and Scanning Electron Microscopy analysis suggested less interference at the adhesive interface in preparations performed at high-speed handpiece.

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