

Impact of Intracoronal Dentin Treatment Prior to Bleaching on Bond Strength of Restorative Materials

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Surface treatment of dentin before the bleaching procedure may affect its permeability and influence the bond strength of restorative materials. This study evaluated the influence of surface treatment before the bleaching on shear bond strength (SBT) of restorative materials to intracoronal dentin. Dentin slabs were subjected to surface treatment: no bleaching (control - CON), no surface treatment + bleaching (HP), 37% phosphoric acid + bleaching (PA) and Er:YAG laser + bleaching (L). After the bleaching procedure, specimens (n=10) were restored with: microhybrid composite resin (MH), flowable composite resin (F), and resin-modified glass-ionomer cement (RMGIC). The shear test was carried out. ANOVA and Tukey's test ($\alpha=0.05$) showed significant difference for surface treatment and restorative materials ($p<0.05$). CON presented higher STB and was statistically different from HP ($p<0.05$). PA and L showed intermediate values and were statistically similar to CON and HP ($p>0.05$). STB for MH and F were higher than RMGIC ($p<0.05$), and did not differ from each other ($p>0.05$). The surface treatments with phosphoric acid and Er:YAG laser before the bleaching procedure provided shear bond strength at the same level of unbleached dentin and the composite resins presented superior bond strength to the intracoronal dentin.

Key Words: bleaching, dentin, Er:YAG laser, phosphoric acid, bond strength.

Introduction

The bleaching of nonvital discolored teeth has been advised for achieving aesthetically desirable appearance (1). Recovery of the chromatic alteration of the dental tissues is reached by penetration of the external and internal bleaching agents through the dentin (2).

When internal bleaching treatment is performed, filling material placed to seal the access cavity must be removed. At this time, smear layer is produced and this may reduce the dentinal diffusion of the bleaching agent inserted in the pulp chamber (3). Acid etching of tooth surfaces and laser treatment have been evaluated to prepare the intracoronal dentin prior to bleaching (4), removing the formed smear layer and exposing the dentinal tubules entrance (5).

Dentin etching with 37% phosphoric acid promotes dissolution of peritubular dentin, exposes the collagen fibers of the intertubular dentin and opens the dentinal tubules (3). On the other hand, Er:YAG laser irradiation of the dentin makes the surface irregular, with absence or modification of the smear layer and exposed tubules, changing the dentin permeability (6).

Although tooth whitening presents satisfactory cosmetic results, bleaching agents have been associated with undesirable effects in the dental hard tissues, including reduced microhardness (7), changes in modulus of elasticity (8) and dental substrate morphology (7). These aspects could interfere with the adhesion of restorative materials to the

tooth bleached surface (9). Additionally, the adhesive strength may also be compromised by residual oxygen remaining within the dentinal tubules, which inhibits the polymerization of restorative materials (8-11).

After bleaching procedure, different restorative materials, such as resin composite and glass-ionomer cements (GICs), may be employed to remake the damaged structure and to seal the access to the pulp chamber in anterior endodontically treated teeth (7,9). Besides the ability to adhere to tooth structure by means of adhesive systems, microhybrid composite resins exhibit satisfactory mechanical properties (12) and the flowable composite resins have greater flow and dissipation of tensions (13). The resin-modified glass-ionomer cements (RMGIC) combine the properties of conventional GICs, such as chemical adhesion to dental tissues and linear thermal expansion coefficient similar to the tooth and the mechanical properties of resins. These characteristics provide the RMGIC intermediate physical properties when compared with GICs and composite resins (14).

Given the divergent protocols for non-vital teeth bleaching, varying pretreatment of the surface and the diversity of restorative materials to restore bleached dentin, it becomes relevant to consider the impact of the surface treatment before the bleaching procedure on bond strength of different restorative materials to the intracoronal dentin.

This study sought to evaluate *in vitro* the influence of

the surface treatment of intracoronal dentin with 37% phosphoric acid and Er:YAG laser prior to bleaching with 38% hydrogen peroxide on dentin bond strength of three restorative materials: microhybrid composite resin, flowable composite resin and RMGIC. The null hypothesis tested was that the surface treatments of intracoronal dentin before bleaching would not affect the bond strength of restorative materials to dentin.

Material and Methods

This study was approved by the Ethics Committee of the University of Ribeirão Preto (Protocol #070/11).

Experimental Design

The factors under study were surface treatment at four levels (I- no treatment and unbleached, II- no treatment + bleaching with 38% hydrogen peroxide, III- acid etching with 37% phosphoric acid + bleaching with 38% hydrogen peroxide and IV- Er:YAG laser irradiation + bleaching with 38% hydrogen peroxide) and restorative materials at three levels [a. Microhybrid composite resin (MH), b. Flowable composite resin (F) and c. Resin-modified glass-ionomer cement (RMGIC)]. Experimental units were 120 intracoronal human dentin slabs, which were randomly assigned into 12 groups (n=10). The response variable was shear bond strength in MPa.

Selection and Preparation of Dental Slabs

Sixty sound human permanent maxillary canines, stored in thymol (0.1%) at 9 °C were selected and examined on stereomicroscopy (Leica Microsystems, Wetzlar, Germany) under 20x magnification to discard those with fractures or cracks in the crown.

Teeth were sectioned on cementoenamel junction with a diamond saw (KG Sorensen, Barueri, SP, Brazil) on a low-speed handpiece (Dabi Atlante, Ribeirão Preto, SP, Brazil). Dental crowns were sectioned in mesiodistal direction, and then each slab was again sectioned on incisal, mesial, distal and cervical surfaces. From each crown, two quadrangular dentin slabs measuring 5x5 mm were obtained, totaling

120 dental specimens.

The sections were individually embedded in polyester resin (JET; Clássico, São Paulo, SP, Brazil) using polyvinyl chloride (PVC) cylinders (1.5 cm diameter and 1.5 cm high), with the dentin surfaces facing upwards. After polymerization, PVC cylinders were removed and specimens were ground under water irrigation using 400- and 600-grit SiC paper. Finishing was accomplished with 1200-grit SiC paper for 60 cycles to standardize the surface smoothness. After polishing, specimens were washed with 10 mL of 1% sodium hypochlorite for 10 min, to simulate the irrigation during biomechanical preparation of radicular canal.

Surface Treatment

The specimens were randomly assigned and subjected to one of the four following surface treatments: I- no treatment and unbleached, II- no treatment + bleaching with 38% hydrogen peroxide, III- acid etching with 37% phosphoric acid + bleaching with 38% hydrogen peroxide and IV- Er:YAG laser irradiation + bleaching with 38% hydrogen peroxide. A brief description of the surface treatment applied in each group is presented in Table 1.

For acid etching with 37% phosphoric acid (Super Etch; 3M ESPE, St. Paul, MN, USA), the product was applied on dentin surface for 30 s, rinsed with water and dried with absorbing paper.

For surface irradiation, the Er:YAG laser (Opus 20; OpusDent Ltd., Tel-Aviv, Israel) used at a 2,940 nm wavelength, 200 mJ, frequency of 10 Hz, an energy density of 3.5 J/cm² and spot diameter of 1.0 mm. The laser beam was delivered under constant water cooling in non-contact and focused mode (at a 12-mm standardized distance), using an automatic custom-designed apparatus. The specimens were placed in a semi-adjustable base which was automatically moved in both right-to-left and forward-to-back directions. Each specimen was irradiated during approximately 10 s.

Specimens from negative control group (no treatment plus no bleaching) were kept in relative humidity at 37 °C. For bleaching, the specimens were exposed to 38%

Table 1. Description of the surface treatments applied in each group

Surface treatment	Description
No treatment + no bleaching	Storage in relative humidity at 37 °C.
No treatment + bleaching	Bleaching with 38% hydrogen peroxide performed in a session with three applications of 10 min and intervals of 5 min between each one.
Acid etching + bleaching	Etching with 37% phosphoric acid for 30 s followed by rinsing and drying. Bleaching with 38% hydrogen peroxide performed in a session with three applications of 10 min and intervals of 5 min between each one.
Laser irradiation + bleaching	Er:YAG laser irradiation (200 mJ/10 Hz) for 10 s. Bleaching with 38% hydrogen peroxide performed in a session with three applications of 10 min and intervals of 5 min between each one.

hydrogen peroxide (Opalescence X-tra Boost; Ultradent Products, Inc., South Jordan, UT, USA) in gel form, which was mixed with red dye at the moment of use, according to the manufacturer's instruction. A session of bleaching was performed applying a thin layer of gel (2-mm thick) on dentin surface for 10 min. This procedure was repeated three times during the session, with 5 min waiting time between each application.

All specimens were sealed with cotton ball and provisional restorative material (Coltosol; Vigodent AS Ind. Com., Rio de Janeiro, RJ, Brazil). Slabs were stored in relative humidity with artificial saliva at 37 °C for 10 days (15).

Specimen Restoration

The samples were restored with the chosen materials for each experimental group (n=10): microhybrid composite resin (Filtek Z250, 3M ESPE), flowable composite resin (FilteK Z350 Flow, 3M ESPE) and RMGIC (Vitremar, 3M ESPE).

Specimens were restored using a split polytetrafluoroethylene mold (3 mm inner diameter, 4 mm high) stabilized with vinyl polysiloxane (Perfil Denso; Vigodent), in order to obtain restorative material cylinders with the above described measurements.

On specimens restored with composite resin, the surface was etched with 37% phosphoric acid for 15 s, washed with distilled water during 15 s and dried with absorbing paper. Two layers of Single Bond adhesive (3M, ESPE) were applied, followed by an air spray, and then light-cured for 10 s (1500 mW/cm², Raddi Plus, SDI Ltd, Victoria, Australia) according to the manufacturer's instructions.

For groups restored with microhybrid and flowable composite resin, the material was inserted in 3 increments, light-activated by LED for 20 s at each increment, maintaining the end of the optic fiber 10 cm from the resin surface using a device developed for this purpose.

The RMGIC was prepared incorporating all powder in liquid (1:1; powder/liquid), inserted in a single increment with Centrix syringe (DFL Ind. SA, Rio de Janeiro, RJ, Brazil) and light-activated for 40 s, as recommended by the manufacturer. After removing the split teflon mold,

all restored specimens were stored in relative humidity at 37 °C during 24 h.

Shear Bond Strength Test

The specimens were subjected to shear bond test in a universal testing machine (Instron 4444; Instron Corporation, Canton, MA, USA) at a 0.5 mm/min cross-head speed and a 2 kN load cell until the restoration displacement. Shear bond strength values were registered in kN and transformed into MPa.

Failure modes at the resin/dentin interface were analyzed under a stereoscopic microscope at 40x magnification (Leica Microsystems), and displayed in percent. Failure was considered adhesive if it occurred at the dentin/adhesive interface, cohesive if it occurred in the material or the substrate and mixed if it involved both the interface and the material.

Statistical Analysis

After checking homoscedasticity (Levene's test) and normality (Kolmogorov-Smirnov's test), two-way ANOVA and Tukey's test were used. The statistical analysis was performed with the 17 SPSS software (SPSS Inc., Chicago, IL, USA) at a 5% significance level.

Results

Two-way ANOVA showed statistically significant difference for the factor surface treatment ($p=0.000$) and restorative material ($p=0.001$).

The specimens without surface treatment and unbleached showed higher bond strength than those without surface treatment and bleached ($p<0.05$), which had the lowest mean bond strength. The acid-etched and bleached specimens and those irradiated with Er:YAG laser before bleaching showed intermediate values, sometimes similar to the control group ($p>0.05$) and sometimes to the group without surface treatment and bleached ($p>0.05$) (Table 2).

The microhybrid and flowable composite resins provided higher bond strength values, similar between them ($p>0.05$) and different from the RMGIC ($p<0.05$) (Table 3).

In the failure analysis, adhesive failures prevailed in all groups, except for the untreated group restored with

Table 2. Shear bond strength of restorative materials to dentin subjected to different surface treatment

Surface Treatment	Means±SD (MPa)
No treatment/unbleached	3.40±1.06 a
No treatment/bleaching	2.17±0.92 b
Phosphoric acid/bleaching	2.76±0.80 ab
Laser/bleaching	2.77±0.93 ab

Different letters indicates statistically significant difference ($p<0.05$).

Table 3. Bond strength of restorative materials to intracoronal dentin

Restorative Material	Means± SD (MPa)
Microhybrid composite resin	3.12±1.36 a
Flowable composite resin	3.44±1.90 a
Resin-modified glass-ionomer cement	1.77±0.75 b

*Similar letters indicate statistical difference ($p<0.05$).

flowable composite resin, which presented higher amount of cohesive failures within the substrate. The percentage of failures is displayed in Figure 1.

Discussion

The null hypothesis tested in this study, that there would be no significant difference in bond strength of restorative materials to dentin whether prior to bleaching exposes or not the surface to treatments, was rejected.

In the current study, the bond strength of specimens without surface treatment and without bleaching was greater than specimens without surface treatment and bleaching. This fact may be attributed to the mechanism of action of the bleaching agent on the dentin, with presence of the OH⁻ radical (16) in the dentin (1). The OH⁻ radical is a strong oxidizing agent that has high penetrating power, acts in intertubular and peritubular dentin by degradation of the organic portion, breaking the polypeptide chain by the destruction of amino-acids of the matrix (16). The presence of residual oxygen on the dentin surface may impair the penetration of resin into dentin tubules, creating an adhesive interface more susceptible to fracture (17). This fact may explain the prevalence of the adhesive failures in all bleached specimens, regardless of the restorative material.

In this study, there was an interval of 10 days (15) between bleaching procedure and restoration in order to minimize the effect of residual oxygen in dentin tubules. However, to simulate the clinical situation, the surface was sealed with temporary cement that could compromise complete release of the oxygen from the dentin. The oxygen in the tubules inhibits the polymerization of the adhesive system and the resin composite, hence it reduces the strength of the interface between the dentin and restorative material (9,15).

When the dentin was subjected to acid etching or

Er:YAG irradiation prior to bleaching, the bond strength was similar to untreated and unbleached specimens. The surface treatment with phosphoric acid promotes dentin demineralization, exposes collagen fibers, intensifies the infiltration of resin monomers, increasing the adhesion (3). The Er:YAG laser modifies the smear layer, promotes dentinal tubules' opening (total or partial) and produces an irregular surface (6). This ultrastructural aspect favors the adhesion of restorative materials, as previously reported (18,19).

Regarding restorative materials, microhybrid and flowable composite resins demonstrated higher bond strength when compared with RMGICs. The adhesion process of GICs is by chemical bonding between the carboxylic polyacids from material, which are chelating agents, and calcium ions from tooth (20). Nevertheless, GIC's bond strength is considered low, since chemical adhesion is lower than mechanical adhesion to tooth structure (21) by means of the hybrid layer, as occurs in resin materials. Although RMGIC presents chemical and also micromechanical adhesion to dentin structure (22), it is speculated that the mechanical component is lower than the one found in the composites, and this aspect seems to be insufficient to provide similar adhesion among them (21). Another aspect that may have contributed to the lower bond strength of the RMGIC is the possible presence of bubbles, which is very frequent (20). Bubbles along the interface between dentine and RMGIC allow an uneven stress concentration that may lead to failure in adhesion (21).

Composite resins adhere to dental substrate by mechanical imbrication within the tubules as well penetration of resin material inside the intertubular dentin collagen matrix (23). This mechanism explains the superior adhesion of microhybrid and flowable composite resins to intracoronal dentin, as demonstrated in the present study.

Despite the differences in composition and properties

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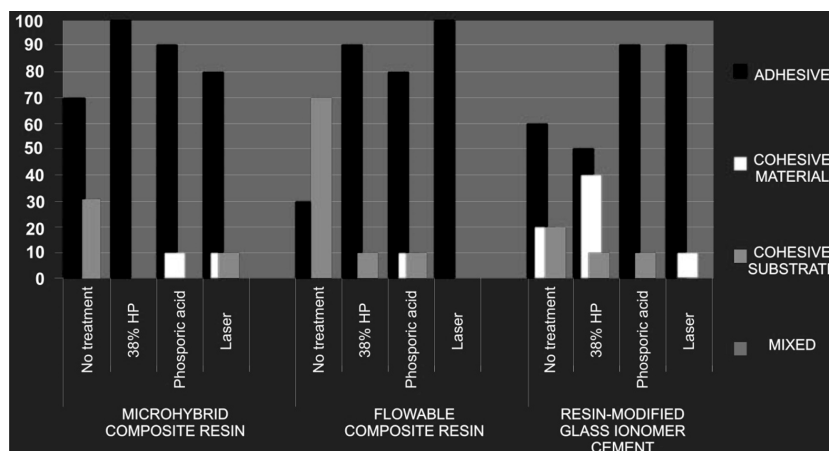


Figure 1. Failure modes observed in each group after the shear test (%).

of resins, both have characteristics that favor the adhesion process. The flowable composite resin presents reduced content of fillers and viscosity, with a high runoff (24), which contributes to its infiltration into dentin. The microhybrid composite resin used in this study has a high amount of crosslinking in chemical structure that increases the modulus of elasticity and enhances the mechanical strength (12). For both composite resins, polymerization shrinkage in the adhesive interface was reduced by the incremental restoration technique.

Within the limitations of an *in vitro* study, it may be concluded that the surface treatment with 37% phosphoric acid or Er:YAG laser irradiation before the intracoronal bleaching provides similar bond strength to unbleached dentin. The microhybrid and flowable resins had superior adhesion to dentin than the RMGIC. Even though both etching with 37% phosphoric acid and Er:YAG laser irradiation have been efficient to increase bond strength to bleached dentin, it is necessary to consider that increased dentin permeability may enhance the risk of adverse effects (25). Further researches are needed to confirm the obtained results, as well as to indicate or to contraindicate the surface treatment before a bleaching procedure.

Resumo

O tratamento superficial da dentina previamente ao clareamento pode afetar a sua permeabilidade e influenciar a resistência de união de materiais restaurados. Este estudo avaliou a influência do tratamento superficial antes do clareamento na resistência ao cisalhamento (RC) de materiais restauradores à dentina intracorônária. Fragmentos de dentina foram submetidos ao tratamento de superfície: não clareadas (controle - CON), sem tratamento de superfície + clareamento (HP), ácido fosfórico 37% + clareamento (AF), e laser Er:YAG + clareamento (L). Após o procedimento clareador, os espécimes foram restaurados com (n=10): resina composta micro-híbrida (MH), resina composta fluida (F), e cimento de ionômero de vidro modificado por resina (CIVMR). O teste de cisalhamento foi realizado. ANOVA e teste de Tukey ($\alpha=0,05$) mostraram diferença significativa para tratamento superficial e material restaurador ($p<0,05$). O grupo controle apresentou maior resistência de união e foi estatisticamente diferente de HP ($p<0,05$). AF e L mostraram valores intermediários e foram similares ao CON e HP ($p>0,05$). A resistência de união para MH e F foi maior que CIVMR ($p<0,05$), e não diferiram entre si ($p>0,05$). O tratamento da superfície dentinária com ácido fosfórico e laser Er:YAG previamente ao clareamento promoveu resistência de união ao nível da dentina não clareada e a adesão à dentina intracorônária foi superior com as resinas compostas.

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