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Laboratory Research

The Effect of Prophylaxis Method on Microtensile Bond Strength of Indirect Restorations to Dentin

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Clinical Relevance

The use of cavity cleaners on dentin before indirect resin restoration cementation results in variation in microtensile bond strengths (μTBS). Prophylaxis with aluminum oxide air abrasion, pumice paste, or chlorhexidine produced significantly higher μTBS to dentin than were produced with hydrogen peroxide and a sodium bicarbonate jet.

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SUMMARY

The aim of this study was to evaluate the effect of different materials used for dentin prophylaxis on the microtensile bond strengths (μTBS) of adhesively cemented indirect composite restorations. Sixty bovine incisors had the buccal surface ground with wet #600-grit silicon carbide abrasive paper to obtain a flat exposed superficial dentin and were submitted to different prophylaxis protocols, as follows: 3% hydrogen peroxide (HydP); 0.12% chlorhexidine (Chlo); sodium bicarbonate jet (SodB); 50-μm aluminum oxide air abrasion (AirA); pumice paste (PumP), and control group–water spray (Cont). After prophylaxis protocols a resin composite block (3.0 mm × 5.0 mm × 5.0 mm) was adhesively cemented using dual resin cement (Rely X ARC). After 24 hours of water storage, specimens were serially sectioned perpendicular to the bonded interface into 1-mm-thick slices. Each specimen was trimmed with a diamond bur to an hourglass shape with a cross-sectional area of approximately 1.0 mm^2 at the bonded area. Specimens were

tested (μTBS) at 0.5 mm/min using a universal testing machine. Scanning electron microscopy was used to examine the effects of prophylaxis techniques on dentin. Bond strength data (MPa) were analyzed by one-way analysis of variance and failure mode by Fisher test (α=0.05). μTBS data, means (SD), were (different superscripted letters indicate statistically significant differences): AirA, 25.2 (7.2)[a]; PumP, 24.1 (7.8)[a]; Chlo, 21.5 (5.6)[a]; Cont, 20.6 (8.1)[a]; HydP, 15.5 (7.6)[b]; and SodB, 11.5 (4.4)[c]. The use of aluminum oxide air abrasion, pumice paste, and chlorhexidine before acid etching did not significantly affect μTBS to dentin; however, the use of hydrogen peroxide and sodium bicarbonate jet significantly reduced μTBS.

INTRODUCTION

Complete intertubular resin infiltration and hybrid layer formation are critical factors for successful dentin bonding.[1] Oral fluids such as saliva, blood, and crevicular fluid can cause chemical incompatibility with dental materials.[2] Adhesive wetting of the tooth substrate can be inhibited by the presence of blood,[3] saliva,[2] hard-tissue particles from operative procedures, and remnants of temporary cement.[4,5] Several agents are recommended for cleansing of the dentin substrate to optimize resin monomer penetration into the collagen network: these include chlorhexidine,[6,7] hydrogen peroxide,[8] pumice paste,[9,10] sodium bicarbonate jet,[9] and aluminum oxide air abrasion.[5,11]

Applying a mixture of flour of pumice and water results in partial removal of the smear layer and surface erosion as a result of the mechanical abrasion of the pumice particles.[12] Pumice paste has been shown to adequately clean oil-contaminated dentin for etch-and-rinse adhesives.[10] Aluminum oxide air abrasion used as a cavity cleanser produces a rough irregular surface with increased surface area that increases the wettability of adhesive systems to tooth structure to provide additional mechanical retention, similar to that associated with etched enamel.[11] Additionally, a mechanical cleansing protocol, utilized prior to definitive cementation, increases the bond strength of luting cement to dentin following eugenol-containing temporary cements.[4,5]

Different dentin-cleaning agents containing chlorhexidine digluconate isolated or associated with glass particles have been marketed with good performance.[7] The use of chlorhexidine as a cavity cleanser is based on its antimicrobial properties.[7] As a result of the cationic properties of the chlorhexidine, it easily binds to the tooth hydroxyapatite, the pellicle on the tooth surface, salivary proteins, bacteria, and phosphate groups.[6] Several studies have shown that the bond strength of some adhesives was compromised by the use of hydrogen peroxide.[13] The exact mechanism by which hydrogen peroxide affects dentin has yet to be fully understood. Hydrogen peroxide is capable of generating a hydroxyl radical, an oxygen-derived free radical that is known to accumulate in dentin.[14] Residual hydrogen peroxide solution in the collagen matrix and dentinal tubules may eventually break down into oxygen and water.[13] Liberation of oxygen could either interfere with resin infiltration into etched dentin or inhibit polymerization of resins that cure *via* a free-radical mechanism.[13]

The aim of this study was to analyze the influence of prophylaxis method (hydrogen peroxide, sodium bicarbonate jet, chlorhexidine, hydrogen peroxide, pumice paste, and air abrasion) on the microtensile bond strengths (μTBS) of indirect composite restorations fixed on bovine dentin. The null hypothesis was that the dental prophylaxis methods do not affect the μTBS of resin-based composite luted to bovine dentin.

MATERIALS AND METHODS

Sixty bovine teeth were extracted and stored in 0.2% thymol solution (Biopharma, Uberlândia, Brazil). The roots were cut off and the pulp was removed under water irrigation. Flat dentinal surfaces with standardized smear layers were created by grinding buccal surfaces with #600 silicon carbide papers (Norton, Campinas, Brazil). Specimens were randomly divided into six prophylaxis groups, as follows: 1) Cont, no cavity cleaner application, 15-second water-spray, and conventional etching (control group). 2) PumP, rubber cup application of pumice paste for 10 seconds, rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching, 3M-ESPE, St Paul, MN, USA) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 3) Chlo, 10-mL application of 0.12% chlorhexidine solution (Biopharma) for 10 seconds with rubber cup application, rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 4) HydP, 10-mL application of 3% hydrogen peroxide solution (Biopharma) for 10 seconds with rubber cup application, rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 5) SodB, sodium bicarbonate jet at 60-pound pressure for 10 seconds from 5 mm perpendicular to the surface (Profi II, Dabi Atlante, São Paulo, Brazil); rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds. 6) AirA, air abrasion with 50-μm aluminum oxide particles at four bars of pressure for 10 seconds from 5 mm perpendicular to the surface (Microjato Plus, Bio-Art, São Paulo, Brazil); rinsed with water spray for 10 seconds, etching with 37% phosphoric acid (Adper Etching) for 15 seconds, and the surface rinsed with water spray for 15 seconds.

Resin composite blocks (3.0 mm × 5.0 mm × 5.0 mm) were incrementally prepared in a silicon matrix with a microhybrid composite resin (Filtek Z250, 3M-ESPE). Each increment was light-cured with a quartz-tungsten-halogen curing lamp (XL 3000, 3M-ESPE) at 600 mW/cm^2 for 40 seconds, as measured by a radiometer (Radiometro, FANEM, São Paulo, SP, Brazil). The intaglio surface of the indirect resin restorations were air abraded with 50 μm aluminum oxide particles (Microjato Plus, Bio-Art) for 10 seconds with four bars of pressure at 10.0 mm of source-to-sample distance and silanized with a pre-hydrolyzed silane solution (Rely X Ceramic Primer, 3M-ESPE) for one minute.

A one-bottle adhesive system (Adper Single Bond 2, 3M-ESPE) was applied, air-dried, and light-cured for 20 seconds at 600 mW/cm^2. Restorations were cemented with a dual cured resin cement (RelyX ARC, 3M-ESPE), according to the manufacturer's instructions. A 500*g* load was applied for five minutes to standardize the luting cement thickness. Visible-light activation of superior and all lateral surfaces on the bonded blocks was performed for 40 seconds for each surface (XL3000, 3M-ESPE). Specimens were stored in distilled water at 37°C for 24 hours. Samples were positioned in a precision diamond cutting machine (1000 Isomet, Buehler Ltd, Lake Bluff, IL, USA) and serially sectioned perpendicular to the bonded interface to obtain four slices of approximately 1 mm in thickness per sample. Each slab was trimmed with a cylindrical diamond bur (#1090, Kg Sorensen, Barueri, SP, Brazil), resulting in an hourglass-shaped specimen with a cross-sectional bonded area of approximately 1.04 ± 0.05 mm^2. Each specimen was fixed to the microtensile testing device (Ciucchi device) with cyanoacrylate glue (Loctite Super Bonder, Henkel Loctite Corporation, Rocky Hill, CT, USA) and tested to failure in tension at 0.5 mm/min in a mechanical testing machine (EMIC DL-2000, São José dos Pinhais, PR, Brazil) to determine μTBS. After fracture, the specimen was removed from the testing apparatus and the cross-sectioned area measured at the site of fracture with a digital caliper (S235, Sylvac, Switzerland). A mean μTBS in MPa was reported for each tooth, with pretest failures ignored in the data analyses. All groups presented a normal and homogeneous distribution; therefore, one-way analysis of variance (ANOVA) with *post hoc* Tukey honestly significantly different (HSD) was used to determine whether there was a significant difference in the bond strengths among the prophylaxis methods.

All fractured specimens were observed under a stereomicroscope (Leika MZ12, Tokyo, Japan) and the failure mode was determined. The failure modes were classified as follows: apparently interfacial (within the adhesive joint), cohesive in dentin, cohesive in the resin cement or resin-based composite restoration, or mixed failures, in which the failures were recorded as the surfaces comprising the dominance of failure of each substrate. Differences in failure modes between groups were tested by Fisher exact test. A *p*-value of less than 0.05 was used as a criterion for statistical significance.

Additionally, to observe the effects of products used in dentin prophylaxis, specimens were wet-abraded to expose superficial dentin and submitted to the techniques used in this study. The specimens were allowed to dry in an oven at 37°C; next they were gold sputter-coated (MED 010, Balzers, Balzer, Liechtenstein) and observed under a scanning electron microscope (SEM; LEO 435 VP, LEO Electron Microscopy, Cambridge, UK). Representative areas of dentin-treated surfaces were photographed at 5000× magnification.

RESULTS

μTBS data are shown in Table 1. One-way ANOVA revealed a significant effect of prophylaxis methods ($p<0.001$). Tukey HSD test showed that the AirA, PumP, Chlo, and Cont groups presented significantly higher μTBS values than did the HydP and SodB groups, while HydP was significantly higher than SodB.

With regard to failure modes, the HydP (80.0%) and SodB (82.5%) were significantly more likely to produce interfacial failures than were the other three prophylaxis methods (67.5% for PumP, 62.5%

Table 1: *Microtensile Bond Strength Values (MPa) and Statistical Categories*

Prophylaxis Method	μTBS[a] Mean (SD)
AirA, Al_2O_3 air abrasion	25.2 (7.2) A
PumP, pumice paste	24.1 (7.8) A
Chlo, 0.12% chlorhexidine	21.5 (5.6) A
Cont, water-spray (control)	20.6 (8.1) A
HydP, 3% hydrogen peroxide	15.5 (7.6) B
SodB, sodium bicarbonate jet	11.5 (4.4) C

[a] *Different letters indicate significant differences by Tukey honestly significantly different test ($p<0.05$). μTBS, microtensile bond strengths; SD, standard deviation.*

for Chol, and 60% for AirA) or the control group (65%). Additionally, the SodB (15%) and HydP (12.5%) groups were more likely to produce pre-tested failure than were the other three prophylaxis methods (2.5% for PumP, Chol, and AirA) or the control group (5%). On the other hand, the AirA (15%), PumP (15%), Chol (12.5%), and Cont (10%) groups were more likely to have cohesive failure in the dentin mode than were the HydP (5%) and SodB (0%) groups. The same failure distribution was found with regard to mixed failures: AirA (15%), PumP (15%), Chol (12.5%), and Cont (10%) groups were more likely to have cohesive failure in the dentin mode than were the HydP (5%) and SodB (0%) groups (Table 2).

SEM revealed a rough irregular surface on the specimens treated with AirA (Figure 1A), crystalline deposits on the surface of specimens treated with SodB (Figure 1B), and the presence of surface grooves on PumP samples (Figure 1C). However, all of the specimens demonstrated the presence of a remnant smear layer prior to the etching acid (Figure 1A-E), which was removed after using phosphoric acid for all groups (Figure 1F).

DISCUSSION

The null hypothesis was rejected. This *in vitro* study showed that prophylaxis with 3% hydrogen peroxide solution and sodium bicarbonate jet negatively influenced the μTBS of the indirect resin restoration fixed to dentin with resinous cement, as compared to the control group. During bond procedures, isolation of the working field is crucial, as any contamination of a prepared tooth might have a detrimental effect on adhesion and retention of restorative materials.[5] In addition, any dentin prophylaxis method should be efficient while leaving no detrimental remnants of the cleaning agent.

Prophylaxis with 0.12% chlorhexidine solution applied prior to acid etching resulted in no significant effect on μTBS values of a luting procedure realized with dual-cure resinous cement and a one-bottle adhesive system (Table 1). Previous studies[6,15] have demonstrated that chlorhexidine application

Table 2: *Mode of Failure of Results Using Different Prophylaxis Methods*

Prophylaxis Method	Apparently Interfacial	Cohesive in Dentin	Cohesive in Resin Restoration or Resin Cement	Mixed	Premature Failure	Total
Cont	26	4	1	7	2	40
AirA	24	6	2	7	1	40
HydP	32	2	0	1	5	40
SodB	33	0	0	1	6	40
PumpP	27	6	1	5	1	40
Chlo	25	5	3	6	1	40

Abbreviations: Cont, water-spray (control); AirA, Al_2O_3 air abrasion; HydP, 3% hydrogen peroxide; SodB, sodium bicarbonate jet; PumP, pumice paste; Chlo, 0.12% chlorhexidine.

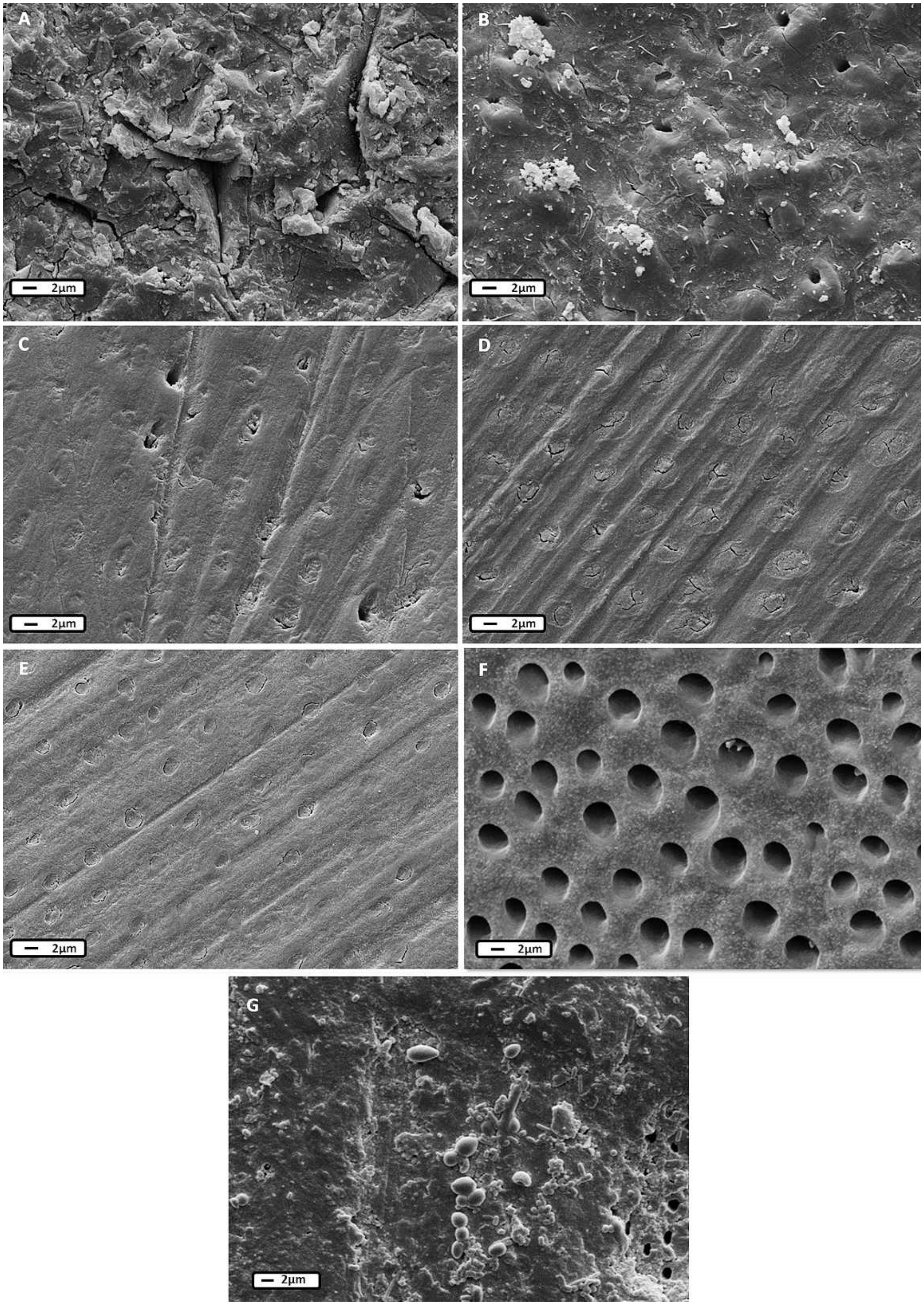

Figure 1. *A. Irregular dentin surface created by air abrasion with 50-µm aluminum oxide particles; B. deposition of crystals and granular residues over dentin surface treated with sodium bicarbonate jet; C. presence of surface grooves and partial removal of smear layer produced by pumice paste; D. dentin surface treated with hydrogen peroxide showing no apparent alteration; E. dentin surface treated with chlorhexidine showing no alteration on surface; F. representative image for all groups of the dentin surface without smear layer completely removed after phosphoric acid etching; G. dentin surface untreated produced by the lab technique. (original magnification 5000×)*

prior to acid etching had no adverse effects on immediate composite-adhesive bonds to dentin. Chlorhexidine applied after acid etching and before use of an adhesive system might be useful for the preservation of dentin bond strength.[16] Chlorhexidine remains the gold standard as an antiplaque and antigingivitis agent.[17] It has been shown that chlorhexidine has an affinity to bacteria, probably because of an interaction between the positively charged chlorhexidine molecule and negatively charged groups on the bacterial cell wall. This interaction increases the permeability of the bacterial cell wall and thus permits the agent to penetrate into the cytoplasm and cause death of the microorganism.[17] Since 0.12% chlorhexidine solution did not produce apparent morphological alteration (Figure 1E) and, additionally, did not produce a negative effect on bond strength to dentin,[6,15] and because it has an antibacterial effect, the use of this product may be a potentially good option for cleaning of cavity preparations before adhesive restorative procedures. It is important to emphasize that the 0.12% chlorhexidine solution used in this study differs from 0.12% mouthwash formulations, which would be contraindicated because they contain glycerin.

Previous studies[4,5] showed that aluminum oxide abrasion was effective in removing cement residues, resulting in better dentin wettability and, consequently, facilitating the infiltration of the adhesive system into the dentin after acid etching. However, another study[18] reported similar bond strengths when acid etching was associated with aluminum oxide abrasion or when it was performed in isolation. As the particles collide with a solid target, in this case the dentin surface, the kinetic energy of the particles is transferred, resulting in microscopic fractures of the target (Figure 1A).[5] As a result, contaminants may be removed from the surface, avoiding the negative effect on the μTBS of the indirect resin restoration.

Cleaning with pumice and water paste is widely considered to be necessary as the last step in cavity preparation or as the first step in the restorative process. Pumice paste does not result in residue deposition on dentin (Figure 1C). In addition to the elimination of residue, pumice paste constitutes an adequate cleaning technique that eliminates bacterial plaque and may produce a thinner smear layer, thus facilitating acid conditioning on dentin.

It is well known that hydrogen peroxide is a strong oxidant and is also weakly acidic, which could increase the wettability of the dentin surface.[19] Hydrogen peroxide affects the inorganic components of dentin through acidic demineralization and attacks the organic-rich intertubular dentin by collagen denaturation.[19] The reduction in bond strength is probably due to these properties of peroxide and their action on dental tissues. Hydrogen peroxide degradation results in the release of water and free radicals from oxygen; these products, however, show no apparent morphological alteration (Figure 1D). The reduction in bond strength of resin-based composite to dentin may also have been due to the presence of free radicals from oxygen that interfered with polymerization of the adhesive resin or adhesive resin cement.[20]

Residues of sodium bicarbonate precipitate on the dentin surface of specimens treated with sodium bicarbonate jet are clearly visible (Figure 1B). The residues of sodium bicarbonate and changes in superficial pH probably interfered with the action of the phosphoric acid, affecting adhesive system and dentin interaction.[21] Despite its efficiency in cleaning pits and fissures as a result of its high degree of penetration in these areas,[9] the sodium bicarbonate jet also has drawbacks, as its residue or pH surface alterations may be responsible for altering phosphoric acid action and for its poor performance on smooth surfaces. Failure analysis demonstrated that the failures for the sodium bicarbonate group were consistently within the interfacial region. The particle deposition resulted in a thin amorphous layer of carbonates and sodium over the dentin surface, which was difficult to remove, resulting in predominantly adhesive failures and six out of 40 pre-test failures.

The hydrogen peroxide solution and sodium bicarbonate jet resulted in more interfacial failure modes and pre-test failures than did the other prophylaxis methods. All other groups resulted in more mixed and cohesive failure than did HydP and SodB. This may be because 1) stress concentration at the interfacial region was not sufficient to create rupture of the bonded joint in more efficacious prophylaxis groups, and 2) hourglass specimen designs concentrate stress in the neck of the dentin or resin, therefore tending to produce cohesive or mixed failures.

There are limitations to the current study. The study results only pertain to the materials used in this study, since only one conventional adhesive system associated with only one dual-cure resin cement was tested. Additional studies are required to determine the effect of the prophylaxis methods using different types of self-adhesive resin cements and with previously contaminated dentin. Additionally, the combination of pumice paste and chlorhexidine may be tested to evaluate for potential synergistic effects on dentin bond strength. The present study tested only 24-hour bond strength and did not include a durability challenge through water storage, thermocycling, cyclic loading, or their combination. Additionally, no temporary cement was used before the definitive indirect restoration cementation.

In a clinical situation, before the adhesive luting procedure the cavity preparation should be cleaned effectively; therefore, the use of mechanical prophylaxis methods that use aluminum oxide air abrasion, pumice paste, or chlorhexidine might be considered before the adhesive integration of the restoration, whereas the use of hydroxide peroxide solution and sodium bicarbonate jet should be avoided as prophylaxis methods before adhesive procedures.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. The use of products as a cavity cleanser before etching dentin can influence μTBS;
2. The use of hydrogen peroxide and sodium bicarbonate jet significantly reduced μTBS values to dentin; and
3. Aluminum oxide air abrasion, chlorhexidine, and pumice paste before acid etching produced no negative effect on μTBS to dentin.

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Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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