

Influence of dentin surface pre-treatment on microtensile bond strength of a single-bottle adhesive

Influência do pré-tratamento superficial da dentina na resistência de união à microtração de um sistema adesivo de frasco único

Abstract

Purpose: This study evaluated the influence of different methods of dentin surface pre-treatment on microtensile bond strength and failure mode of a single-bottle adhesive.

Methods: Twenty-four third molars were embedded in acrylic resin and ground until exposure of occlusal dentin. Six groups were tested according to the dentin surface pre-treatment: G1) 35% phosphoric acid (pa) (positive control); G2) slurry of pumice + pa; G3) air-abrasion with 25 μ m-aluminum oxide + pa; G4) air abrasion with 50 μ m-aluminum oxide + pa; G5) air abrasion with sodium carbonate + pa; G6) no treatment (negative control). The adhesive Single Bond (3M-ESPE) was applied on the dentin surface and photoactivated before a 6mm-height composite restoration was built (Z250, 3M-ESPE). After storage in distilled water at 37°C for 24h, all specimens (sectional area of 1 mm²) were subjected to a microtensile bond strength test at a crosshead speed of 0.5mm/min.

Results: Bond strength mean values (MPa) were: G1=29.28, G2=21.04, G3=21.66, G4=18.94, G5=19.90, and G6=19.03. Only G1 was statistically different from the other groups (ANOVA and Tukey's test, $\alpha=0.05$). SEM images of the adhesive interface after rupture showed prevalence of mixed failure type (adhesive fracture between dentin and adhesive agent plus partial cohesive fracture in the composite restoration or dentin) for all groups but G6.

Conclusion: The use of cleaning agents on dentin surface might not be necessary for adhesive procedures with dentin total acid-etching.

Key words: Dentin; dentin-bonding agents; tensile strength

Resumo

Objetivo: Este estudo avaliou a influência de diferentes métodos de pré-tratamento da superfície dentinária sobre a resistência de união à microtração e modo de falha de um sistema adesivo de frasco único.

Metodologia: Vinte e quatro terceiros molares foram incluídos em resina acrílica e desgastados até exposição da dentina oclusal. Seis grupos foram testados de acordo com o pré-tratamento da dentina: G1) ácido fosfórico a 35% (pa) (controle positivo); G2) pasta de pedrapomes + pa; G3) jateamento com óxido de alumínio 25 μ m + pa; G4) jateamento com óxido de alumínio 50 μ m + pa; G5) jateamento com carbonato de sódio + pa; G6) sem tratamento (controle negativo). O sistema adesivo Single Bond (3M-ESPE) foi aplicado na superfície dentinária e fotoativado antes da simulação de uma restauração de resina de 6mm de altura (Z250, 3M-ESPE). Após armazenamento em água destilada a 37°C por 24h, todos os espécimes (área transversal de 1 mm²) foram submetidos ao teste de microtração à velocidade de 0,5mm/min.

Resultados: Os valores médios de resistência de união (MPa) foram: G1=29,28; G2=21,04; G3=21,66; G4=18,94; G5=19,90; e G6=19,03. Somente o G1 foi estatisticamente diferente dos demais grupos (ANOVA e teste de Tukey, $\alpha=0,05$). A observação em MEV da interface adesiva após ruptura mostrou prevalência de falha do tipo mista (fratura adesiva entre dentina e agente adesivo associada à fratura coesiva parcial na restauração de resina ou dentina) para todos os grupos, exceto G6.

Conclusão: O uso de agentes de limpeza na superfície dentinária pode não ser necessário para procedimentos adesivos com condicionamento ácido total da dentina.

Palavras-chave: Adesivos dentinários; dentina; resistência à tração

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Introduction

The concept of total acid etching changed the clinical preparatory steps for adhesion procedures in dentin, allowing the formation of a resin-reinforced hybrid layer (1). Acid etching removes the smear layer and hydroxyapatite and modifies the dentin microstructure morphology, creating micromechanical retention (2). Furthermore, both surface energy and dentin permeability are altered, which increases the adhesive penetration into the demineralized collagen matrix and improves the sealing of exposed dentin tubules (3,4).

Besides acid etching, other pre-treatments of dentin surface before application of adhesive systems have been proposed, such as air abrasion with aluminum oxide, irrigation with sodium hypochlorite, and mechanical cleaning with slurry of pumice (5-10). However, the effectiveness of these methods to improve adhesion is contradictory and seems to be material-dependent (11). More recently marketed materials with no acid etching (and, therefore, no total removal of the smear layer) altered the clinical bonding procedure, and their adhesion may be affected by dentin pre-treatment.

This study compared the effect of four surface pre-treatment procedures on dentin microtensile bond strength of a one bottle adhesive system. The *a priori* hypothesis was that dentin pre-treatments other than the manufacturer's directions do not have a negative impact on bond strength. The failure mode after the microtensile test was observed on debonded specimens by using scanning electron microscopy (SEM).

Methods

Specimen preparation

Twenty-four extracted caries-free human third molars were cleaned with Robson brush and slurry of pumice and immersed in 0.2% chloramine solution for disinfection. The teeth were embedded in cylinders of self-curing acrylic resin, and their occlusal portion was ground with 120- and 600-grit silicon carbide paper in a water-cooled polishing machine (Struers DPU-10, Panambra, São Paulo, SP, Brazil) until exposure of occlusal dentin. After 5min-ultrasonication in distilled water to remove any debris, teeth were randomly assigned to six groups according to surface pre-treatments. In Group G1, dentin was etched with 35% phosphoric acid gel (3M-ESPE, St Louis, MN, USA) for 15s and rinsed with water spray for 15s. For G2, dentin surface was cleaned with Robson brush and slurry of pumice for 10s in low speed, and rinsed with water spray for 10s. In G3, dentin surface was air-abraded with 25 μ m-aluminum oxide for 10s, with pressure of 60psi, at a distance of 2mm from the equipment tip (Microjato Plus, Bio-Art, São Carlos, SP, Brazil), and rinsed with water spray for 10s. Specimens in G4 received the same pre-treatment used in G3 but with 50 μ m-aluminum oxide. In G5, the dentin surface was air-abraded with sodium bicarbonate (Profi Ceramic, DabiAtlante, São Paulo, SP, Brazil) for 10s, at a distance of 2mm from the equipment

tip, and rinsed with water spray for 10s. Groups G2, G3, G4, and G5 were additionally treated with 35% phosphoric acid for 15s and rinsed with water spray for 15s. Group G6 comprised specimens with no dentin pre-treatment or adhesive procedure, which was used as a control group to determine the dentin cohesive strength. Figure 1 displays the surface characteristics of dentin pre-treatments groups in SEM images.

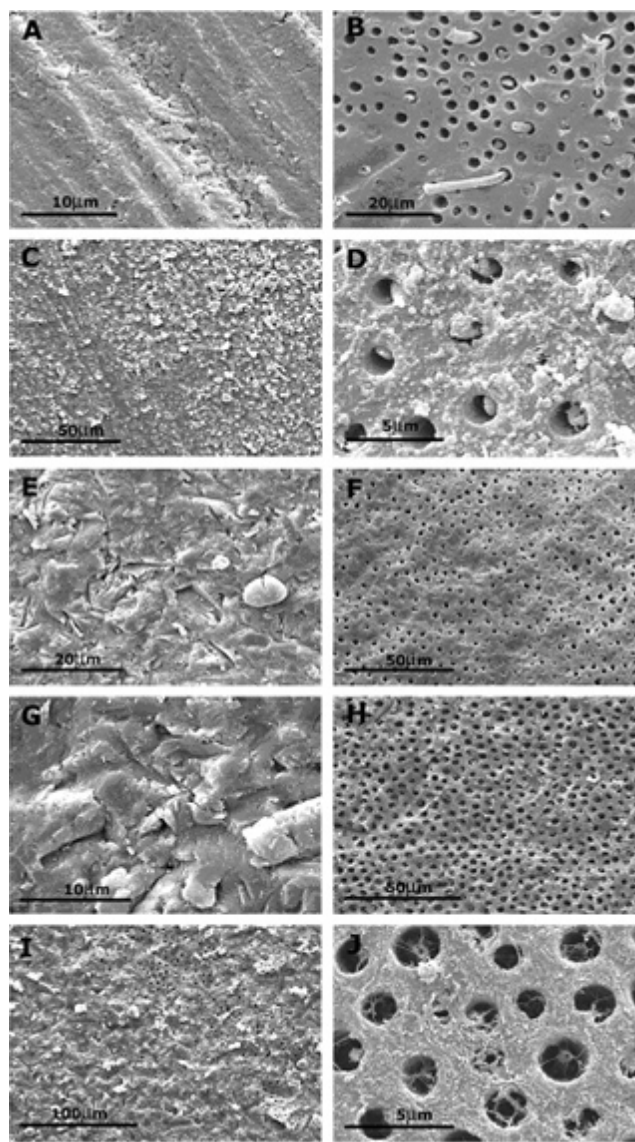


Fig. 1. SEM images of dentin treated with: (A) diamond rotary instrument (presence of smear layer); (B) 35% phosphoric acid; (C) pumice slurry; (D) pumice slurry followed by 35% phosphoric acid etching (presence of contaminants); (E) 25 μ m-aluminum oxide air abrasion; presence of particles after water spray; (F) 25 μ m-aluminum oxide air abrasion followed by 35% phosphoric acid etching; (G) 50 μ m-aluminum oxide air abrasion; presence of particles after water spray; (H) 50 μ m-aluminum oxide air abrasion followed by 35% phosphoric acid etching; (I) sodium bicarbonate air abrasion; presence of particles after water spray; (J) sodium bicarbonate air abrasion followed by 35% phosphoric acid etching.

After dentin pre-treatment, the adhesive system Single Bond (3M-ESPE, St Louis, MN, USA) was applied to the dentin surface according to the manufacturer's directions and photoactivated by halogen light (Optilux 501, Kerr USA, Orange, CA, USA). Light intensity output (520mW/cm²) was monitored with a radiometer unit (Demetron, Danbury, CT, USA). To simulate a 6mm-height flat restoration, three 2mm-increments of composite resin Z250 (color B2, 3M-ESPE, St Louis, MN, USA) were sequentially packed and photoactivated for 20s. All specimens were then stored in distilled water at 37°C for 24h before mechanical testing.

Microtensile bond strength

Specimens were sectioned perpendicularly to the adhesive interface using a water cooled diamond-impregnated saw (Labcut 1010, Extec, London, England) at 400rpm to result test specimens (resin-dentin sticks) with a cross-sectional area of 1.0mm² (n=20/group). The sticks were observed with stereoscopic magnifying lens (Olympus, SZ80G, Tokyo, Japan) with 30X magnification to screen for defects or voids in the adhesive layer, and specimens with such defects were discarded. For G6, test specimens consisted of dentin sticks with cross-sectional area of 1.0mm².

The test specimens were individually fixed to the microtensile strength test device with cyanoacrylate adhesive (Loctite 416, Loctite, São Paulo, SP, Brazil) and accelerator (ZIP Kicker, Pacer, Rancho Cucamonga, CA, USA) to position the adhesive area perpendicularly to the long axis of the tensile force. The microtensile strength test was performed using the universal testing machine EMIC DL-2000 with the software MTest (EMIC, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5mm/min. The maximum values of tension were recorded in Newtons (N) and then transformed in MPa using the following formula: MPa=N/mm². Data

were statistically analyzed by ANOVA and Tukey's test at the 0.05 level of significance.

Evaluation of failure mode

After rupture in the microtensile strength test, the debonded specimens were observed in SEM to classify the failure mode into: Type 1: adhesive fracture between adhesive agent and dentin; Type 2: adhesive fracture between dentin and adhesive agent plus partial cohesive fracture in the composite restoration or dentin; Type 3: cohesive fracture in dentin; Type 4: cohesive fracture in the composite restoration.

The two ends of each specimen were glued together so that the fractured surfaces were arranged side by side. The specimens were immersed in 2.5% glutaraldehyde for 12h at 4°C for fixation, washed with 20mL buffer solution of sodium cacodilate 0.2M with pH 7.4 for 1h, and washed in distilled water three times for 1min. For dehydration, the specimens were sequentially immersed in ethyl alcohol (25% for 20min, 50% for 20min, 75% for 20min, and 95% for 20min), and dried at 37°C for 48h. The prepared specimens were mounted on stubs for sputtering with gold at 10mA for 1min and observed in SEM (Philips XL 30, Philips Electronic Instruments Inc, Mahwah, NJ, USA).

Results

Mean bond strength values of the experimental groups are listed in Table 1. Group G1 (35% phosphoric acid etching), showed the highest bond strength compared with the other groups, which were not statistically different ($P>0.05$).

Figure 2 displays the results of the failure mode analysis at the adhesive interface. The distribution of failure mode was relatively uniform among all groups. Type 2 failure (adhesive fracture between dentin and adhesive agent plus partial cohesive fracture in the dentin or composite restoration) was the most prevalent failure mode (Figure 3).

Table 1. Microtensile bond strength (MPa) of the experimental groups (n=20).

Experimental Groups	Mean* (MPa)	Standard deviation	Coefficient of variation (%)
G1) 35% acid phosphoric (pa)	29.28 ^a	4.50	28.77
G2) Pumice slurry + pa	21.04 ^b	3.59	22.59
G3) Air abrasion with 25µm – aluminum oxide + pa	21.66 ^b	1.76	19.01
G4) Air abrasion with 50µm – aluminum oxide + pa	18.94 ^b	2.16	20.86
G5) Air abrasion with sodium bicarbonate + pa	19.90 ^b	1.47	17.19
G6) Dentin	19.03 ^b	1.72	18.93

* Means followed by different letters are statistically different (ANOVA and Tukey's test, $\alpha=0.05$).

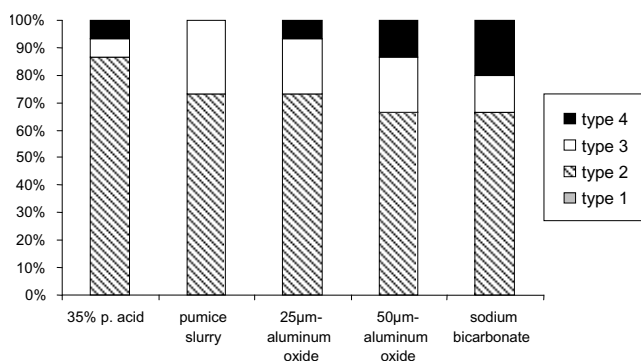


Fig. 2. Distribution (%) of failure mode by treatment group after the microtensile strength test. Type 1: adhesive fracture between adhesive agent and dentin; type 2: adhesive fracture between dentin and adhesive agent plus partial cohesive fracture in the adhesive agent; type 3: cohesive fracture in dentin; type 4: cohesive fracture in the composite restoration.

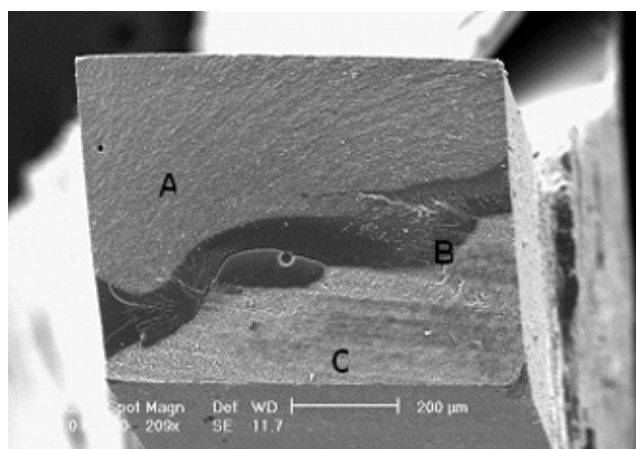


Fig. 3. Type 2 failure (mixed type: adhesive fracture between dentin and adhesive agent plus partial cohesive fracture in the adhesive agent). A – composite resin. B – adhesive. C – dentin.

Discussion

For the adhesive system used in this study (Single Bond-3M/ESPE), dentin etching with 35% phosphoric acid yielded higher bond strength than the tested alternative surface pre-treatments. These results suggest that these methods would not be recommended as substitute or additional procedures for phosphoric acid etching in similar clinical conditions involving adhesive restorations on intact dentin. However, when complete removal of surface contaminants is necessary, such as presence of eugenol, biofilm, or residues of restorative material, extrapolation of these results should be conservative.

Groups with alternative pre-treatments followed by 35% phosphoric acid etching had similar bond strength, which was lower than the control group. SEM images showed that air abrasion with aluminum oxide (25µm and 50µm)

using low air pressure (60 psi) resulted in an irregular and retentive dentin surface. Previous studies suggested the use of air abrasion with aluminum oxide to replace acid etching (5-10), but their materials and procedures were different from those in the present study. Manhart et al. (8) found satisfactory bonding to dentin using aluminum oxide abrasion with high air pressure, but low air pressure did not modify the dentin substrate like acid etching. The alteration of dentin morphology and partial removal of smear layer, therefore, may be dependent on the pressure used for particles acceleration. Higher pressures would promote greater modification of dentin surface and higher bond strength. In the present study, the size of aluminum oxide particle had no detectable effect. Also, it may be difficult to completely remove aluminum oxide particles with air-water spray or even acid etching as depicted in Figure 1. These residues may act as weakening sites for bonding between adhesive and dentin.

Similarly, mechanical cleaning of the dentin substrate with slurry of pumice or sodium bicarbonate produced small modifications of dentin morphology, which was not significantly different from the non-treated dentin. The presence of contaminants from pre-treatments before acid etching may compromise bonding stability. This may help to explain the lower bond strength found for these procedures.

Although bond strength was different for the phosphoric acid group, all groups basically had type 2 failure (mixed failure in the adhesive agent and cohesive in dentin or composite restoration). To some extent, these results were expected because the fractured areas are the weakest components of adhesive interfaces composed of composite resin, adhesive agent, hybrid layer, and dentin (12,13).

However, when analyzing the failure mode per group, the groups with low bond strength had high frequency of failure in the adherent substrate (composite and dentin). This type of failure is expected when bond values are high. All groups but G1 (phosphoric acid etching) had bond strength similar to dentin cohesive strength (G6), justifying the presence of dentin cohesive fracture in type 2 failures. Interpretation of failure mode at the adhesive interface should be cautious because it seems too simplistic to classify the complex dynamic mechanics of bonding failures into absolute and fixed categories, although this classification has been largely used (12,14-16). A more detailed classification based on measurements of individual areas and expressed as a percentage of the total number of failures has been proposed to overcome these deficiencies (17).

Direct extrapolation of this study to the clinics is limited because many others factors can influence the microtensile strength (18), for instance, the microleakage (19) that can vary as a function of adhesive type, *e.g.*, total-etching *versus* self-etching systems, and acid conditioning with different acids which do not seem to prevent marginal gap in composite restorations (20). Furthermore, dentin adhesives including the single-bottle adhesive used in the present study are very sensitive to handling and saliva contamination

that significantly reduces the quality of bonding to dentin and enamel (21-23). For these cases, additional dentin pretreatment besides regular acid etching may be beneficial to bonding. Therefore, further studies should explore the possibility of improving adhesion to dentin using combined dentin pretreatment and different types of adhesive systems for adverse and challenging clinical situations.

Conclusions

In summary, the results suggest that the tested alternative pre-treatments do not improve bond strength to dentin for Single Bond. For this adhesive agent it is not necessary to perform a more complex or multi-step procedures to prepare the dentin substrate.

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