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MICROTENSILE BOND STRENGTH OF ADHESIVE SYSTEMS OF SINGLE AND MULTIPLE STEPS

Resistência à microtração dos sistemas adesivos de única e múltipla etapas

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Abstract

OBJECTIVE: To evaluate the microtensile bond strength of ten dentin adhesive systems. **MATERIAL AND METHOD**: Sixty human molars were cut to the dentine level and restored with a hybrid composite and one of the adeshive systems. Each teeth were sectioned to obtain sticks measuring 1.0 ± 0.2 mm^2, which were then stressed at a crosshead speed of 1mm/min in a universal testing machine until failure. The failure modes were verified using optical microscopy. **RESULTS**: One Up Bond F™, All Bond 2™, One Step Plus™, Adper Prompt L-Pop™, One Step™, Single Bond™ and Clearfil SE Bond™ presented no statistically significant differences ($p>0.05$); the lowest bond strength values were obtained with Scotchbond MP™, Prime&Bond NT™ and Excite™ ($p<0.05$). **CONCLUSION**: The self etch primer systems and conventional systems exhibited the highest bond strength to dentin, except for Scotchbond MP™, Prime&Bond NT™ and Excite™. Adhesive fractures accounted for 69% of the total fractures.

Keywords: Adhesive systems. Bond strength. Restorative dentistry.

Resumo

***OBJETIVO**: Avaliar a resistência adesiva de dez sistemas adesivos. **MATERIAIS E MÉTODOS**: Os dentes foram seccionados em forma de palitos com tamanho de 1.0 ± 0,2 mm^2 e tracionados com velocidade de 1mm/min. Os tipos de fraturas foram verificados utilizando o microscópio óptico. **RESULTADOS**: Os sistemas adesivos (One Up Bond F™, All Bond 2™, One Step Plus™, Adper Prompt L-Pop™, One Step™, Single Bond™ e Clearfil SE Bond™ apresentaram os maiores valores de resistência adesiva em dentina. Os sistemas Scotchbond MP™, Prime&Bond NT™ e Excite™ apresentaram menor resistência. **CONCLUSÃO**: Os* *sistemas autocondicionantes e os convencionais apresentaram maior resistência adesiva em dentina. Os sistemas Scotchbond MP™, Prime&Bond NT™ e Excite™ apresentaram menor resistência adesiva. As fraturas adesivas representaram 69% do total das fraturas.*

Palavras-chave: *Adesivos. Resistência de união. Dentística.*

INTRODUCTION

The constant development in adhesive restorative dentistry has caused profound changes in dental practice, and technological advances such as new adhesive systems and products (1, 2).

Dentin bonding depends not only on the adhesive system, but also on the dentin substrate, and even now this bond is unpredictable (3). The wet tubular microstructure and the high organic content are factors that make it more difficult to perform bonding to dentin (4). The results are influenced by the type of dentin, the amount of remaining humidity in the substrate and the application technique inherent to the adhesive system itself, chemical composition, type of diluents and enamel and dentin treatment (5). Other possible variables are intimate contact with dentinal tubules and lateral branches, thickness (6) and mechanical properties of the bonding agents (7).

Adhesive systems can be divided into two categories**:** total-etching and self-etching. The total etching technique is based on removing the smear layer and demineralizing the dentin by acid etching and self-etching systems containing an acidic primer to demineralize the smear layer and subsurface dentin (8). These two adhesive systems demonstrate differences in bond strength to tooth substrate (5). Van Meerbeek et al. (9) proposed a classification of contemporary adhesives based on the adhesion strategy and application procedure. The adhesive systems could be categorized as: 3-step etch&rinse adhesives, 2-step etch&rinse adhesives, 2-step self-etch adhesives, and 1-step self-etch adhesives (10).

The one bottle or 2-step etch&rinse adhesive systems appeared on the market as an alternative to the 3-step etch&rinse adhesives (9-12). This type of adhesive system reduces the number of clinical steps and is less technique sensitive. Another modification was introduced: filler particles were added to increase viscosity, resulting in thicker layers (13). The self-etching system concept is based on the use of a polymerized acidic monomer that simultaneously etches and prepares the dentine with a primer. The advantages of these systems include complete infiltration of the bonding agent into the demineralized dentin and reduced number of clinical procedure (1). Most self-etching adhesive systems were applied in 2-steps, the acid etching and primer simultaneously and then the bonding agent (9-11). Recently, adhesive systems that associate the three steps in one application were marketed as one step self-etching products (9). The microtensile method plays an important role in bond strength testing since it produces considerably fewer cohesive fractures. It also allows the regional bond strengths within the teeth to be measured, which can be better related to clinical conditions (14). The microtensile bond test offers the opportunity to test more than one specimen from a single tooth; the results are relatively unaffected by specimen defects; and a high frequency of bond failures occur at the adhesive interface (14, 15).

The present study compared the dentin bond strength of 10 differents adhesive systems: 3-step etch&rinse adhesives, 2-step etch&rinse adhesives, 2-step self-etch adhesives, and 1-step self-etch adhesive. The null hypothesis tested was that there is no difference between any of these materials.

MATERIAL AND METHOD

Specimens

Sixty intact, non-carious, extracted human third molars stored at 4^{o}C in 0.5% chloramine solution. All teeth were obtained in accordance with a protocol revised and approved by the Research Ethics Comitee of UNESP-FOAr.

Method

The occlusal enamel was removed perpendicular to the long axis of each tooth using a low speed saw (Isomet 1000 ™, Buehler, IL,USA) and a diamond wafering blade (15LC T=1/2", 06"dia/0.020", #114276), under water cooling.

The dentinal surfaces were ground flat with 320-grit silicon carbide paper while mounted in a polishing machine under adequate water cooling. Complete enamel elimation was confirmed by optical microscope at 30 X magnification. In order to standardize the smear layer, teeth were abraded with 600-grit silicon carbide paper in a polishing machine under abundant cooling for 60s.

The teeth were randomly assigned in accordance with the type of adhesive used, as summarized in Table 1. Next, a 5 mm thick layer of hybrid resin composite (Z100, 3M/ESPE, Dental Products, St. Paul, MN, USA) was placed on the treated dentin surface and photopolymerized for 20s by a light-curing unit XL 3000 (3M/ESPE, Dental Products, St. Paul, MN, USA) with a light output of 600 mW/cm^2, followed by storage in distilled water at 37°C for 24h.

The bonded specimens were sectioned into 1.0 mm-thick slabs using a low-speed diamond saw, and then the tooth was rotated 90 degrees and again sectioned lengthwise, resulting in sticks with a cross-sectional area of 1.0±0.1 mm^2. A total of 30 dentin-composite specimens were obtained from each group of five teeth. Before testing the samples for tensile stress, the specimens were evaluated under optical microscope at 40X magnification to check for flaws and enamel in the adhesion area. The cross-sectional areas and remaning dentin thickness of the selected specimens were measured using a digital caliper (Digimatic Caliper ™, #BB071467, Mitutoyo, Tokyo, Japan).

The specimens were attached to a universal testing machine (EMIC-DL 500 ™, São José dos Pinhais, PR, Brazil), using a cyanoacrylate glue (Superbonder Gel ™, Henkel Loctite Adesivos, Itapevi, SP, Brazil) plus an accelerator (Zapit, DVA, Corona, CA, USA). The specimens were stressed under tension until failure at a crosshead speed of 1mm/min. Bond strength data were analyzed by one-way ANOVA. Group comparisons were made by the Tukey HSD test. All statistical tests were applied at a confidence level of 5%.

The fractured surfaces were observed under optical microscope at 60X magnification to verify the failure modes. Fracture mode was classified into one of three types: adhesive, cohesive and mixed.

One non-carious third molar was restored with each bonding system and the hybrid composite. A mesial-distal cut was made and the halves were immersed in glutaraldehyde for two minutes, washed with a air/water spray and placed in a 37% phosphoric acid solution for 10 s, and finally for 20 s in a 1% HCl solution. The specimens were coated with gold, and observed by SEM (JSM T330A, JEOL ™, Tokyo, Japan), 20 kV and 3500 X.

TABLE 1 - Adhesive systems, manufacturers, application procedures and compositions

Groups	Lot	Application	Composition
All Bond 2 Bisco Inc.,IL,USA	0100008068	3-step etch&rinse	Bis-GMA, UDMA, BPDM, Ethanol, Water
One Step Bisco Inc., IL, USA	0100008080	2-step etch&rinse	Acetone, HEMA, Bis-GMA, BPDM
One Step Plus Bisco Inc., IL, USA	0200004676	2-step etch&rinse	Acetone, HEMA, Bis-GMA, BPDM, fillers, *p*-dimethylaminobenzoic acid.
Excite Ivoclar, Liechtenstein	B29610	2-step etch&rinse	HEMA, TEGDMA, phosphoric acid acrylate, silicon dioxide, ethanol
Prime&Bond NT Dentsply, DE, USA	0112000092	2-step etch&rinse	PENTA, UDMA, Nanofiller, Initiators, acetone,Cetylaminehydrofluoride
Scotchbond MP 3M/ESPE, MN, USA	7543	3-step etch&rinse	HEMA, polyalkenoic acid copolymer. Bis-GMA and dimethacrylates
Single Bond 3M/ESPE, MN, USA	9EA	2-step etch&rinse	Bis-GMA, HEMA, water, ethanol, initiator dimethacrylates, polyalkenoic acid copolymer
Adper Prompt L-Pop 3M/ESPE, MN, USA	15/2Y2	1-step self-etch	Bis-GMA, CQ stabilizers, water, HEMA, polyalkenoic acid, stabilizers
One Up Bond F J Morita Inc., CA USA	000231E	2-step self-etch	Mac-10, phosphate monomer, water; fluoro-aluminosilicate glass
Clearfil SE Bond Kuraray, Japan	51135	2-step self-etch	MDP; HEMA; water, dimethacrylate, Bis-GMA, HEMA; colloidal silica

RESULTS

Mean bond strength, standard deviation (SD) and percentage failure modes obtained with each adhesive systems are shown in Table 2. No statistical significant differences were detected between the adhesive systems Scotchbond MP™, Prime&Bond NT™ and Excite™ (p>0.05), which exhibited the lowest bond strength. The self etching primer systems and conventional systems (All Bond 2™, One Step Plus™, One Step™ and Single Bond™) exhibited higher bond strength values, for which no statistical significant differences were detected (p>0.05).

The adhesive fractures accounted for 69% of the total number of fractures. No cohesive failures were observed in dentin. In almost all-adhesives systems, most of the failures were adhesive, except for Excite™, which showed a higher percentage of mixed failures.

The SEM photomicrographs of each experimental group are shown in Figure 1. All systems were able to penetrate the demineralized dentin.

TABLE 2 - Bond strength mean (SD) of ten adhesives systems and distribution (percentage) of failure mode: A – adhesive, M - mixed

Adhesive Systems	Mean ± SD	Failure Mode	
		A %	M %
One Up Bond F	36.73±10.52[a]	70.00	30.00
All Bond 2	34.29±12.63[a]	56.67	43.33
One Step Plus	33.18±11.33[a]	93.33	6.67
Adper Prompt L-Pop	31.76±11.30[a]	70.00	30.00
One Step	29.02±11.28[a]	70.00	30.00
Single Bond	28.82±14.74[a]	66.67	33.33
Clearfil SE Bond	28.08±12.09[a]	66.67	33.33
Scotchbond MP	26.31±9.09[b]	53.33	46.67
Prime&Bond NT	21.35±10.22[b]	53.33	46.67
Excite	19.52±10.08[b]	50.00	50.00

Same letters connect groups that are not statistically significant different (p<0.05).

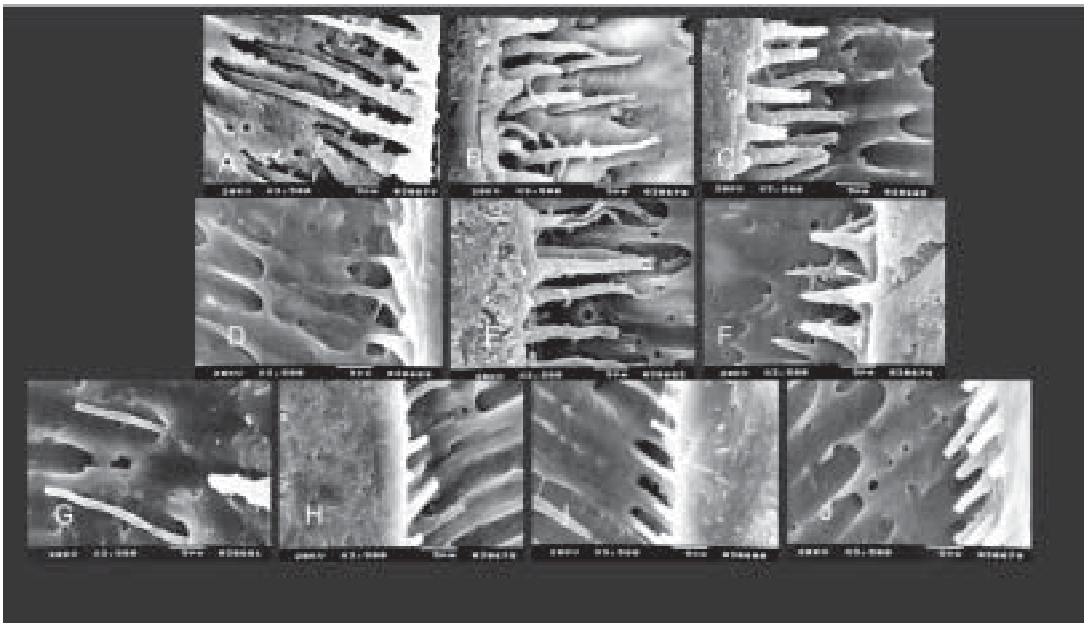

FIGURE 1 - Scaning electron micrographs illustrating interfacial morphology of all adhesive systems: (A) One Up Bond F™, (B) All Bond 2™, (C) One Step Plus™, (D) Adper Prompt L-Pop™, (E) One Step™, (F) Single Bond™, (G) Clearfil SE Bond™, (H) Scotchbond MP™, (I) Prime&Bond NT™ and (J) Excite™

DISCUSSION

The microtensile test demonstrates a trend towards increasing bond strength values and is considered more consistent than conventional test methods (16, 17). The bond strength values obtained by the shear and tensile tests present lower values when compared with the microtensile test (18, 19).

Measuring bond strength by using shear bond tests causes an irregular distribution of the force at the interface. Therefore, the microtensile test provides advantages, such as: better stress distribution; more adhesive failures than cohesive failures, the possibility of testing bond strength in different areas of the same tooth (20), easier analysis of the bonding failures by SEM (21). This was confirmed in the present study, by trend towards increased adhesive layer thickness resulting in a decreased number of specimens with cohesive fracture. Adhesive fractures were classified as: 65% adhesive and 35% mixed. The group that presented the highest number of adhesive fractures was the One Step Plus™ (28/30), followed by the Adper Prompt L-Pop™ (21/30), One Up Bond F™ (21/30), One Step™ (21/30), Clearfil SE Bond™ (20/30), Single Bond™ (20/30), All Bond 2™ (17/30), Scotchbond MP™ (16/30), Prime&Bond NT™ (16/30) and Excite™ (15/30).

Reported dentin bond strengths have showed great variations due to the complexity of the dentin substrate, technique sensitivity of the bonding agent itself, and the differences in measuring bonding techniques (22). Obtaining of etched dentin hybridization with bonding quality is due to the humidity of the adherent substrate, which can be specific and dependent on the type of adhesive system used (23).

Organic solvents can be water, alcohol and acetone based, with adhesive systems containing acetone and ethanol, demanding higher humidity when compared with systems that present water in their composition (4). In this study, the different types of solvents might not affected the bond strength to dentine. Obtainment of dentin humidity compatible with the solvent present in each adhesive system used, could have been favored by the conditions of the substrate (flattened dentin) and the protocol followed, allowing one to work with a pre-determined quantity of water (23), which in some way could have led to the statistical similarity in the bond strength values attained by the different adhesive systems (Table 2).

In this study, it was not possible to detect the influence of the number of stages on bond strength. This is probably due to the similarity of the bonding approach in each system, as well as the use of effective "water displacing" solvents, which helped to draw the adhesives deeper into the superficial demineralized moist dentin (24).

The results of this study cannot be compared with other findings in the literature, as each author demonstrates different bond strength values, irrespective of the adhesive system used. These conflicting results could be due to the variety of methodologies used, types and depths of dentin, composition of adhesive systems and mainly the variability of operators. In addition, further tests should be developed to ratify the bond strengths of adhesive systems, including *in vivo* tests.

This study demonstrated that self etching primer and conventional systems had similar bond strengths, except for three conventional systems (Scotchbond MP™, Prime&Bond NT™ and Excite™). The fractures that occurred were predominantly of the adhesive type and all of the groups presented resinous tags inside the dentinal tubules.

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