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Original research

Effect of filler and application mode on micro-shear bond strength of etch-and-rinse adhesive systems

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ABSTRACT

Objective: To evaluate the effect of the application modes and the presence of filler of etch-and-rinse adhesive systems on the micro-shear bond strength test (μSBS) to bovine dentin.
Methods: Twenty bovine teeth had the enamel removed and dentin surface exposed. Dentin was etched with 37% phosphoric acid and the following adhesive systems were applied: One Step and One Step Plus with two modes (no rubbing action and vigorous rubbing action). To each tooth six Tygon cylinders with internal 0.76 mm were filled with flowable composite resin. All cylinders were light polymerized for 40 s (Optilux 501). The μSBS was performed in a universal testing machine (Instron 5565), with specimens held in place by a wire (0.2 mm) loop, and force loaded to failure. The fracture mode was evaluated. The results in MPa were statistically analyzed by two-way ANOVA and Holm–Sidak tests ($\alpha = 0.05$).
Results: The two-way ANOVA detected no interactions between factors ($p = 0.865$), but only differences between the adhesive systems ($p = 0.042$) and application modes ($p = 0.014$). One Step Plus obtained a higher μSBS than One Step, and vigorous application increased bond strength of adhesives systems. The predominant failure modes of all groups were adhesive-mixed.
Conclusion: The adhesive systems showed the best μSBS results with presence of filler and active mode application.

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Efeito da carga e do modo de aplicação na resistência ao micro-cisalhamento de sistemas adesivos

Palavras-chave:
Adesivo
Técnica adesiva
Dentina
Resistência ao cisalhamento

RESUMO

Objetivo: Avaliar o efeito do modo de aplicação e da presença de partículas de carga de sistemas adesivos com condicionamento ácido total na resistência de união à dentina bovina.

Métodos: Vinte dentes bovinos tiveram o esmalte removido e a dentina exposta foi condicionada com ácido fosfórico a 37% onde os seguintes sistemas adesivos foram aplicados: One Step e One Step Plus aplicados de dois modos (sem agitação e com vigorosa agitação). Em cada dente foram confeccionados 6 cilindros de resina fluída através do preenchimento de microtubos plásticos (0,76 mm de diâmetro interno) que foram polimerizados por 40 s (Optilux 501). Os espécimes foram alocados em um dispositivo acoplado à uma máquina de ensaios mecânicos universal (Instron 5565), e um fio ortodôntico de 0,2 mm de diâmetro foi fixado à máquina e posicionado na interface adesiva ao redor do cilindro de resina e aplicada uma carga até o rompimento da união adesiva, para avaliação da resistência de união ao micro-cisalhamento. O modo de fratura foi analisado com lupa estereoscópica. Os resultados em MPa foram analisados estatisticamente pelo teste Anova de dois fatores e teste de Holm–Sidak para contraste de média (α=0,05).

Resultados: A análise estatística demonstrou que a interação dos fatores não foi significativa (p=0,865), havendo apenas diferença entre os adesivos (p=0,042) e modo de aplicação (p=0,014). O One Step Plus obteve os maiores valores de micro-cisalhamento quando comparado com o One Step, e o modo de aplicação vigoroso aumentou os valores de resistência adesiva dos materiais testados.

Conclusão: Os sistemas adesivos mostraram o melhor desempenho quando a carga estava presente e quando aplicados de forma vigorosa.

Introduction

In an effort to improve the mechanical properties of adhesives and minimize microleakage, discolorations and loss of retention, leading to restoration failure, some manufacturers have reinforced adhesives systems adding fillers to their formulation.[1] Therefore, this material could act as a stress absorbing layer due to its lower elastic modulus, allowing deflection between composite/dentin and improving marginal sealing. These features provide the advantage of better mechanical properties and high elasticity forces to compensate the resin polymerization contraction during restoration build-up and masticatory stresses.[2]

Unfilled adhesives provide lower mechanical properties and usually provide no radiopacity, which could mislead clinicians to interpret the adhesive radiotransparency as gap formation or recurrent caries at the restoration margin.[3]

On the other hand, adhesives with fillers have high viscosity, that make it difficult for the monomers to penetrate into the collagen fibers until it infiltrates into the dental tubules, and this correct infiltration is an essential factor to form the hybrid layer.[4–6] It has been shown that when unfilled adhesives are vigorously rubbed onto dentin surfaces, high immediate and long-term bond strengths to demineralized dentin can be obtained.[7,8] The application mode, particularly for systems containing fillers, could play an important role in the bonding process, if they are agitated on dentin during application in order to improve their properties.[2]

This application mode increases the bond strength because the diffusion rate of the monomer is a function of both penetrability into dentinal substrate and the diffusibility of the adhesive solution itself.[9,10]

For instance, it was demonstrated that application significantly improved the bond strength values for adhesive system,[11] however, unfortunately, the influences of bonding performance of adhesive with filler are unclear in the literature.

Therefore, the aim of the present study was to evaluate the effect of application mode (with and without agitation) on the microshear bond strength to bovine dentin of filled and unfilled two-step etch-and-rinse adhesive systems. The null hypothesis was that the application mode could not interfere in the bond strength of adhesives with and without filler.

Materials and methods

The roots of 20 bovine teeth kept in 0.5% chloramine T at 4 °C were sectioned using a diamond disk (Isomet 1000, Buehler; Lake Bluff, IL, USA) and the coronal pulpal tissue removed. The crowns portions were mounted in plastic rings with acrylic resin (Jet Clássico Ltda, São Paulo, SP, Brazil). The enamel was removed from the labial surface using 120-grit sand paper (Norton, São Paulo, SP, Brazil) under running water. The flat dentin surface was wet ground with 600-grit SiC abrasive paper (Norton, São Paulo, SP, Brazil) for 60 s to obtain a standard smear layer. After this, the teeth were randomly

Table 1 – Adhesive systems, composition and application mode (information supplied in the safety data sheets and material instructions).

Adhesive systems	Composition	Adhesive application
One Step (Bisco, Schaumburg, IL, USA)	Bis-GMA, BPDM, HEMA, initiator and acetone	1. Acid-etch (15 s), rinsing (15 s) and air-dry (10 s) leaving dentin moist; 2. Application of two coats of the adhesive; 3. Air dry for 5 s at 20 cm; 4. Light activation for 10 s – 600 mW/cm^2.
One Step Plus (Bisco, Schaumburg, IL, USA)	Bis-GMA, BPDM, HEMA, dental glass (8.5%) and acetone	1. Acid-etch (15 s), rinsing (15 s) and air-dry (10 s) leaving dentin moist; 2. Application of two coats of the adhesive; 3. Air dry for 5 s at 20 cm; 4. Light activation for 10 s – 600 mW/cm^2.

Bis-GMA: bisphenol A-diglycidyl, ester dimethacrylate; BPDM: biphenyl dimethacrylate; HEMA: 2-hydroxyethyl methacrylate.

divided into four groups ($n = 5$), according to the combination of adhesive (2 levels) and application mode (2 levels).

The adhesive systems were applied as described in Table 1. After applying the adhesive on dentin surface, six vinyl Tygon tubes (Tygon-tubing, S-54-HL, Saint Gobain Performance Plastic, Maime Lakes, FL, USA) 0.75 mm in diameter and 0.5 mm high were placed on each dentin surface (6 Tygon tubes per tooth) and the adhesive system was light-cured (Optilux 501, Demetron, Kerr, Orange, CA, USA) for 10 s at 600 mW/cm^2. The Tygon tube cylinders were filled up with Filtek Flow A2 (3M/ESPE, St. Paul, MN, USA), and light activated, using the same light unit at 600 mW/cm^2 for 60 s. This intensity was monitored using a radiometer (Curing Radiometer, Demetron, Kerr, Orange, CA, USA). After light curing, the matrices were removed by cutting the Tygon tube along their long axis using a scalpel blade. Specimens were stored in distilled water at 37 °C for 24 h prior to testing.

All resin cylinders were checked under an optical microscope (10×) in order to discard any evident defects at the interface. No defects were observed and all specimens were deemed testable.

The specimens were fixed in an Instron Machine (Model 5565, Canton, MA, USA) by tying a thin wire, 0.2 mm in diameter (Morelli Ortodontia, São Paulo, SP, Brazil) around each resin cylinder over the bonded interface. The force was loaded to failure, at a crosshead speed of 0.5 mm/min.

The fractured surface of each resin cylinder was evaluated under a stereoscopic microscope at 10× magnification. Failures were considered: adhesive/mixed when the adhesive residue partially covered the bonded area; cohesive in resin cylinder when residues of the resin remained covering the tooth surface, corresponding to the entire diameter of the bonded area.

The micro-shear bond strength was calculated and expressed in MPa. Five teeth were used for each experimental condition ($n = 5$). The bond strength values of all specimens (6 resin cylinders) from the same tooth were averaged for statistical purposes. Resin cylinders with premature and cohesive failures were not included in the mean value of the tooth.

Statistical analysis was performed using the SigmaPlot 12 software (SigmaPlot v. 12.3, Systat Software Inc., San Jose, USA). Before submitting the data to analysis using the appropriate statistical test, the Kolmogorov–Smirnov test was

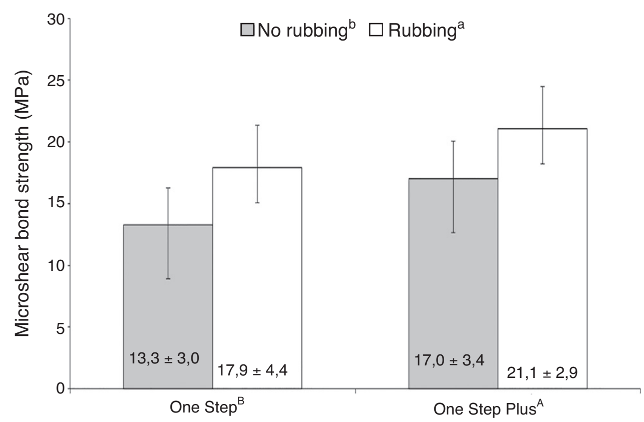

Fig. 1 – Bond strength averages (in MPa) of the adhesive systems according to the application mode. Capital letters show statistical differences among adhesive systems. Lower-case letters indicate differences between the application modes.

performed. After observing the normality of the data distribution ($P = 0.806$) the statistical analysis was performed.

The μSBS means were analyzed by two-way ANOVA (adhesive vs. application mode) and Holm–Sidak tests for pair-wise comparisons ($\alpha = 0.05$).

Results

The overall microshear bond strength (μSBS) values of adhesives are shown in Fig. 1 and fracture modes are shown in Table 2. Only the main factors were statistically significant ($p = 0.865$). The vigorous application mode showed higher μSBS to dentin than no rubbing action ($p = 0.014$). For the factor adhesive, the highest μSBS was found for adhesive One Step Plus ($p < 0.042$). The type of failure found most frequently for both materials was adhesive/mixed.

Discussion

The current study revealed that the action of vigorously rubbing the adhesives is essential to provide high immediate bond

Table 2 – Number of specimens according to fracture pattern mode from each experimental condition.[a]

Adhesive	One Step				One Step Plus			
Application mode	No rubbing action		Vigorous rubbing action		No rubbing action		Vigorous rubbing action	
Total number of resin cylinder	30		30		30		30	
Fracture mode (AM/C)*	Adhesive/mixed	Cohesive	Adhesive/mixed	Cohesive	Adhesive/mixed	Cohesive	Adhesive/mixed	Cohesive
	26	0	28	1	28	0	27	1
Pre-test failure resin cylinder	4		1		2		2	

[a] AM – adhesive–mixed fracture mode; C – cohesive fracture mode (dentin or resin).
* Information supplied by the manufacturer.

strength to dentin. The highest bond strength was obtained when the adhesive system with filler (One Step Plus) content was scrubbed on the dentin surface. The null hypothesis of the study was thus denied. The results might suggest that the vigorous application to the demineralized dentin surface during vigorous rubbing might compress the collapsed collagen network like a sponge.[12–14] As the pressure is relieved, the compressed collagen expands and the adhesive solution may be drawn into the collapsed collagen mesh.[8,11] The vigorous rubbing action provides infiltration into collagen and adhesive resin replaces all the water within the demineralized matrix, which was previously occupied by mineral.[7]

In the present study, an unfilled adhesive One Step and a filled adhesive One Step Plus, which consists of 8.5% glass fillers with an average particle size of 1 μm,[15] were used. The presence of filler was determinant to achieve higher microshear bond strength values. Several simplified etch-and-rinse adhesive systems available on the market contain filler particles in their composition, e.g., Adper Single Bond 2 (3M/ESPE), Prime & Bond NT (Dentsply), Optibond Solo Plus (Kerr), Excite (Ivoclar Vivadent), One Step Plus (Bisco) to improve their clinical performance.[16,17]

The nanoparticles may infiltrate into the demineralized tubules and intertubular dentin, especially into the interfibrilar spaces, and reinforce the adhesive interface layer.[18] The addition of fillers in dental adhesives is able to increase the mechanical strength of the material and help in the function of stress relief during mastication, working as an elastic buffer.[19,20]

On the other hand, a high filler concentration in adhesive systems may increase the viscosity of the material, making it difficult for it to penetrate into the collagen network.[16,21,22] Incomplete penetration of adhesive system into the demineralized dentin may lead to exposure of the collagen fibers,[23] and leave the dentin unprotected against exogenous substances, thus reducing clinical performance.[24] Filler particles were found around the tubular orifices and in some dentin tubules;[25] however, they were not capable of penetrating into the spaces between collagen fibers because the width of interfibrillar spaces is about 20 nm.[15]

Maybe, a vigorous application of the adhesive system can contribute to the penetration of filler particles in the adhesive interface and increase the mircoshear bond strength. Thus, adhesives containing fillers should be applied vigorously to improve the penetration of particles within the hybrid layer. The particles in an adhesive system provide obstacles at the crack front, changes in crack trajectory, consume energy during crack extension and increase the debonding energy.[26] Some studies have also shown that incorporation of nanoparticle fillers has the potential to promote strengthening of the adhesive resin and the adhesive interface, with improvements in the degree of conversion and polymerization efficacy.[27,28] Mechanical properties (ultimate tensile strength and microhardness) as well as water sorption and solubility of the experimental filled adhesives were either improved or remained unchanged in comparison with those of the unfilled experimental adhesive.[17]

On the other hand, some studies have demonstrated that the presence of fillers in adhesive systems did not show any improvement in their mechanical properties[6] and bond strength to dentin.[15,20] In spite of the presence of filler in the adhesive systems having shown controversial results in the literature, the addition of fillers in these materials may make their radiographic visualization possible in situations in which there is a suspicion of caries, thereby improving the diagnostic accuracy.[17,29]

In this study, the failure mode was mainly of adhesive/mixed type for both application protocols and adhesive materials. In fact, the adhesive layer is the weakest region of the tooth/restoration interface, which has a greater concentration of stress, caused by load.[30,31] Although the mode of application under agitation and the adhesive containing filler show the high values of microshear it did not influence the fracture mode. This may be due to a correct load applied exactly at the bonded interfaces.

Despite of wire loop method shows a better stress distribution, due wire position closer to the interface stresses. But the microshear test can produce a tensile stress[32,33] in the resin cylinder base on which the wire is placed by causing a lever.

The addition of particles could increase the mechanical properties and radiopacity, but also should be given some bioactivity to the material in an attempt to compensate the hybrid layer degradation.[34–37]

Conclusion

The result of the present study showed that the application mode was significant and the best bond strength results were obtained when systems with filler content were agitated on the dentin surface.

Ethical disclosures

Protection of human and animal subjects. The authors declare that no experiments were performed on humans or animals for this study.

Confidentiality of data. The authors declare that no patient data appear in this article.

Right to privacy and informed consent. The authors declare that no patient data appear in this article.

Conflicts of interest

The authors have no conflicts of interest to declare.

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