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Shear Bond Strength of Total-Etch and Self-Etching Adhesive Systems to Bovine Dentin

Alessandra Bühler BORGES[a], *Ana Lucia MARSILIO*[b], *José Roberto RODRIGUES*[a]

[a]*Departamento de Odontologia Restauradora, Faculdade de Odontologia, UNESP*
12245-000 São José dos Campos - SP
[b]*Departamento de Odontologia Social e Clínica Infantil, Faculdade de Odontologia, UNESP*
12245-000 São José dos Campos - SP

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Abstract: The aim of this study was to investigate the shear bond strength of two adhesive systems: a total-etch technique Scotchbond Multi-Purpose Plus (3M/Espe, St. Paul, MN, USA) and a self-etching technique Etch & Prime 3.0 (Degussa AG, Hanau, Germany) to bovine dentin, before and after thermo cycling. Bovine incisors were embedded in self-cured resin and labial dentin surfaces were ground with sequential abrasive sand papers. The specimens were divided into two groups to receive the adhesive systems and a composite resin cylinder (Z100 - 3M/Espe, St. Paul, MN, USA) was bonded to the dentinal surface. Each group was subdivided into two subgroups (A and B). The subgroup A was shear tested after 24 hours and the subgroup B was subjected to 500 thermal cycles, between 5 °C and 55 °C and, then, shear tested. The bond strength was performed on a Universal Instron machine, with a speed of 0.5 mm/min. The failure mode was classified in adhesive, cohesive and mixed. The 2-way ANOVA test ($\alpha = 0.05$) revealed that there were no significant differences between the bond strength of the two adhesive systems, and that the thermal cycling did not significantly influence the shear bond strength proportioned by the two adhesive systems tested. The Scotchbond Multi-Purpose Plus groups showed predominantly adhesive failures and the Etch & Prime 3.0 groups showed predominantly mixed fractures.

Keywords: *Dentin bonding agents; shear bond strength; thermal cycling*.

Resumo: O objetivo deste estudo foi comparar a efetividade da união de dois sistemas adesivos: um de condicionamento total Scotchbond Multi-Uso Plus (3M/Espe, St. Paul, MN, USA) e outro autocondicionante Etch & Prime 3.0 (Degussa AG, Hanau, Germany) à dentina bovina antes e após a termociclagem. Incisivos bovinos foram embutidos em resina acrílica e suas superfícies vestibulares foram desgastadas até a exposição da dentina. Os espécimes foram divididos em dois grupos, nos quais aplicaram-se os sistemas adesivos e, posteriormente, a resina composta Z100 (3M/Espe, St. Paul, MN, USA) por meio de uma matriz de teflon. Esses grupos foram subdivididos em dois subgrupos (A e B). O subgrupo A foi submetido ao teste de cisalhamento após 24 horas e o subgrupo B recebeu a ciclagem térmica (500 ciclos entre 5 °C e 55 °C), para depois ser submetido ao teste de cisalhamento. O ensaio mecânico foi realizado em máquina Instron Universal à velocidade de 0,5 mm/min e as interfaces adesivas rompidas foram analisadas em estereomicroscópio. O teste estatístico ANOVA com dois fatores ($\alpha = 0,05$) revelou que os valores de resistência adesiva não apresentaram diferença estatisticamente significante e que a termociclagem não influenciou a resistência adesiva proporcionada pelos materiais testados. Os grupos Scotchbond Multi-Uso Plus apresentaram predominância de falhas adesivas e os grupos Etch & Prime 3.0 apresentaram maior quantidade de fraturas mistas.

Palavras-chave: *Adesivos dentinários; resistência adesiva; termociclagem.*

Introduction

The formation of an effective hybrid layer is achieved by the diffusion of monomers in the collagen fibers, and this is the main bonding mechanism of the total etch adhesive systems. However, this surface rich in collagen can collapse during rinsing and drying of the acid, interfering in the monomer diffusion. In addition, if dentin is overetched, the monomers can be unable to fully penetrate the exposed collagen, forming a porous zone at the base of the hybrid layer[22,25].

Due to the technique-complexity and sensitivity, the innovations of the adhesive systems were directed toward a simplified application process. To avoid the collapse of the collagen network and to simplify the clinical technique, the self-etching adhesive systems were developed. With these systems, etching and priming occurs simultaneously by infiltrating the smear layer-covered dentin with acid resins[10,25,26].

Although the conventional three-step adhesive systems are more time-consuming and technique-sensitive, they may be more durable compared to the simplified systems, because the self-priming and self-etching adhesives are more hydrophilic and hence more permeable to water derived from the underlying dentin, which is undesirable, because this permeability can lead to premature degradation of resin-dentin bonds over the time[26].

Thermal cycling is the *in vitro* process of subjecting a restoration or a tooth to temperature extremes compatible with the oral cavity[10]. This procedure aids to understand the stress caused by long time storage and thermal changes. Carracho et al.[4] (1991) asserted that thermal cycling is an important parameter to determine the stability of adhesive bonding agents.

Thermal cycling stresses the bond between resin and the tooth and is considered as an adjunct to *in vivo* simulated testing[10]. Previous studies showed different results of bond strengths after thermal cycling methods, depending on the adhesive system used[2,5,10,14,18].

The aim of this study was to compare the shear bond strength of a conventional total etch and a self-etching adhesive system to dentin before and after thermal cycling. The null hypotheses tested are that the self-etching adhesive system produces bond strengths comparable to the total etch adhesive system and that the thermal cycling does not affect bond strength to dentine.

Material and method

The materials used for this study are outlined in Table 1.

Forty eight 3-year-old bovine incisors, erupted and intact, were extracted immediately after the animals have been sacrificed. The teeth were cleaned, immersed in distilled water and frozen at –18 °C. The roots were sectioned at the middle level with a low speed diamond saw and the pulp was removed using endodontic instruments.

By means of a round diamond bur, in a high-speed handpiece with copious water spray, a coronal access in the lingual face of teeth was made, until the pulp chamber exposition. The pulp chamber was filled with utility wax to avoid penetration of embedding media. The teeth were embedded in self-curing acrylic resin (Classico Artigos Odontológicos, São Paulo, SP, Brazil) to allow the exposition of the buccal area and placed into tap water to reduce the temperature rise from the exothermic polymerization of the acrylic resin[14].

The pulp chamber access was open allowing the measurement of the remaining dentin thickness, with a thickness spring caliper (Oto Armingen CIA Ltda – RS Brasil). The dentin thickness was standardized in 2 mm. The dentin surface was exposed using a wet 80-grit silicon carbide and the dentin was polished with sequential 320, 400 and 600-grit sandpaper on a polishing machine (Politriz DP 10, Struers, São Paulo, SP, Brazil). To limit the area for the application of the adhesive system, a special Scotchtape Mold with a standard central hole, 4 mm in diameter, was placed on each specimen.

Specimens were divided into two groups of 24 teeth each. Group 1, received the adhesive Scotchbond Multi-Purpose Plus (SMPP) and Group 2, the adhesive Etch & Prime 3.0 (E&P) according to the manufacturers instructions. A split

Table 1. Materials used with respective batch numbers and manufacturers

Material	Composition	Batch No.	Manufacturer
Scotchbond Multi-Purpose Plus	Primer: HEMA, polyalkenoik acid copolymer, water Adhesive: Bis-GMA, HEMA, amines	08039	3M/ESPE, St. Paul, MN, USA
Etch & Prime 3.0	Catalyst: HEMA, photoinitiators, stabilizers, tetrameth-acryloxuethylpyrophosphate Universal:HEMA, distilled water, ethanol, stabilizers	43020060	Degussa AG, Hanau, Germany
Z100	Bis-GMA, TEGDMA, zirconia-silica	08039	3M/ESPE, St. Paul, MN, USA

teflon mold was adapted to the specimen to insert the composite resin Z100. The insertion was done in two layers, each layer was light cured for 40 seconds (Degulux, Degussa Hülls, Hanau, Germany; 600 mW/cm²) and the composite resin cylinders with 4 mm in diameter by 4 mm in height were bonded to dentin surface. After that, the specimens were stored in distilled water at 37 °C for 24 hours.

The two groups were divided into two subgroups (A and B). The subgroup A (n = 12) was shear tested after 24 hours and the subgroup B (n = 12) was subjected to 500 thermal cycles, between 5 °C and 55 °C, with a dwell time of 30 seconds (Etica Equipamentos Científicos, São Paulo, SP, Brazil) and then, shear tested. The specimens underwent laboratorial testing using a universal testing machine (Model 4301, Instron Corp., Buckinghamshire, England) at a crosshead speed of 0.5 mm/min.

The fractured teeth were analyzed by a stereomicroscope (Stemi 2000C, Carl Zeiss, Jena, Germany – 20X) to determine the mode of failure. The fracture modes were classified into three groups: adhesive failure between bonding resin and dentin, cohesive failure in dentin and mixed failure, a combination of adhesive and cohesive failures.

Statistical Analysis – The results were analyzed by calculating the mean SBS (MPa) and standard deviation for each group. The data were subjected to two-way ANOVA, at the 0.05 significance level, to found significant differences between the bond strength of the groups.

Result

The means and standard deviations of the SBS for the evaluated materials are presented in Table 2. Significant differences between the bond strength of the two materials evaluated could not be demonstrated, before and after thermal cycling, by using two-way ANOVA test ($p > 0.05$).

Figure 1 represents the mean bond strength values and standard deviation of the different conditions tested.

Fracture Pattern – The results of fracture pattern observed after the shear test are listed in Table 3, divided as adhesive (A), cohesive in dentin (CD) and mixed (M).

Discussion

The adhesive systems SMPP and E&P were chosen since they utilize different smear layer treatments. The first one is a conventional three-step adhesive system that removes the smear layer completely by acid etching and demineralizes the subsurface intact dentin. Following rinsing, the primer and the adhesive are applied to the conditioned substrate. The second, a single-step self-etching adhesive system, has the tetramethacryloxyethylpyrophosphate component present in the catalyst bottle which ionizes when in contact with the water of the universal bottle, originating a solution with pH = 1.4[3]. This adhesive system dissolves the smear layer with acidic resins while, simultaneously, promotes monomer impregnation into the underlying dentin.

Table 2. Mean SBS and standard deviation (MPa) for the tested materials before and after thermal cycling (subgroups A and B, respectively)*

Scotchbond Multi-Purpose Plus		Etch & Prime 3.0	
Subgroup A	Subgroup B	Subgroup A	Subgroup B
11.2 (3.0)[a]	11.8 (2.8)[a]	11.3 (2.6)[a]	11.8 (2.1)[a]

*same lower case letter means no significant difference (5%)

Table 3. Numeric distribution of the fracture patterns observed according to the tested group

Scotchbond Multi-Purpose Plus						Etch & Prime 3.0					
Subgroup A			Subgroup B			Subgroup A			Subgroup B		
A	CD	M	A	CD	M	A	CD	M	A	CD	M
11	-	1	11	-	1	5	-	7	5	1	6

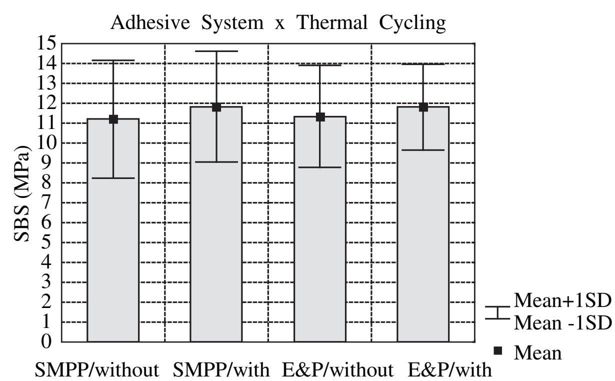

Figure 1. Mean bond strength values of the four groups tested.

Bovine dentin was used in this study to facilitate the standardization of teeth age, storage and acquisition. According to Nakamichi et al.[15] (1983), Saunders[23] (1988), Tagami et al.[24] (1990), Fowler et al.[8] (1992) and Helvatjoglu-Antoniades et al.[10] (2004), bovine dentin can be used as substitute of human dentin in bond strength tests. Bovine dentin is similar to the human substrate in respect to radiodensity, which reflects similarity in the atomic composition and physical structure[7].

The SBS means of the two materials tested, a conventional adhesive system (SMPP) and a self-etching adhesive system (E&P) were statistically similar (Table 2). Cardoso et al.[3] (1998) compared the same adhesive systems using microtensile and shear bond strength tests and obtained statistic similar results between the groups. Konno et al.[12] (2003) also found statistic similar results of SBS mean values when using SMPP and E&P adhesive systems.

Although the SBS values obtained by the two adhesive systems tested were similar, when the longevity of the adhesive systems was investigated, it was observed that E&P was significantly affected by the storage time, but SMPP was not influenced by 6 months of water storage[12]. This may be explained by the fact that the self-etching adhesives are more hydrophilic than the conventional adhesive systems and more permeable to water originated from dentine and, therefore more susceptible to degradation of resin-dentin bonds[26].

Since the self-etching adhesive systems should diffuse and infiltrate through the smear layer to reach the underlying dentin, the thickness of the smear layer might compromise the quality of the bond[25]. In the present study, the 600-grit abrasive paper was used to simulate the creation of smear layer on dentin surfaces, nevertheless, when thicker smear layers were created with diamond burs or with coarser sandpapers, it was reported lower values of bond strength results with self-etching systems[13,17,19]. Therefore, the method used to create the smear layer could also influence the results obtained in this study.

Thermal cycling is one method of simulation of the long-term effects of temperature change in the oral environment[6,14] and is considered an important parameter to determine the stability of adhesive bonding agents[4]. Thermal cycling regimen of 500 cycles between 5 °C and 55 °C, with a dwell time of 30 seconds, was used because this is the regimen recommended by International Standard Organization (ISO[11], 1994). In literature reviews performed by Al-Salehi, Burke[1] (1997) and Gale, Darvell[9] (1999), this was the most frequently used regimen.

The effect of thermal cycling on SBS of adhesive systems is controversial. In this study, the thermal cycling did not significantly influence the SBS of the two materials tested (Figure 1). This result agrees with those of Chan et al.[5] (1985), that did not find significant difference in bond strengths before and after thermal cycling and those of Bedran-de-Castro et al.[2] (2004), that also found no statistically significant influence of thermal cycling on microleakage and SBS to bovine dentin using 2000 cycles.

Miyazaki et al.[14] (1998), tested the SBS to bovine dentin of self-etching primer systems and self-priming adhesive systems after 3000, 10000 and 30000 cycles, and found significant decrease on SBS only with self-priming adhesive systems, after 30000 cycles. In fact, Titley et al.[27] (2003) asserted that the effects of thermal cycling in dentin bond strength are variable and dependent upon the adhesive system.

Al-Salehi, Burke[1] (1997) asserted that there is no evidence to suggest that thermal cycling has a significant effect on bond strength values. Titley et al.[27] (2003) affirmed that it is unlikely that thermal cycling parameters used in laboratorial studies represent the conditions found in the oral cavity. Therefore, they believe that the thermal cycling has little relevance with respect to the performance of restorative resins in the clinical oral environment.

Nevertheless, Helvatjoglu-Antoniades et al.[10] (2004) reported that thermal cycling significantly reduced the SBS of total etch and self-etching adhesive systems, using 5000 cycles. The authors attributed this result to the possible effect of hydrolysis at the interfaces of bonding resin and hybrid layer.

Nikaido et al.[16] (2002) and Price et al.[21] (2003) observed that cavity configuration factor could influence the results of bond strength of the adhesive systems when the specimens are subjected to thermal cycling regimens. In their studies, the thermal cycling did not have a significant effect on bond strength for specimens made in molds with low *C*-factor, but the thermal stress adversely affected the bond between the resin and the tooth in the specimens made in a mold with a high *C*-factor. In the present study, the *C*-factor was low, because the resin composite was bonded to the dentin at the bottom of the mold only and not to the sides, as it occurs clinically in cavities.

As well as observed by Titley et al.[27] (2003), the thermal cycling had little influence on the type of failures produced after the shear test. Most of the fractures with SMPP were adhesive and E&P showed predominantly mixed adhesive/cohesive fractures, followed by adhesive fractures. The failure modes observed in this study may be a consequence of the methodology used. One specimen produced a cohesive fracture of the dentin at 16.34 MPa. According to Pashley et al.[20] (1995), the presence of cohesive fractures in dentin can be due to abnormal stress distributions during shear testing. Konno et al.[12] (2003) observed 100% of adhesive failures with E&P 3.0 due to incomplete infiltration of the acidic monomer and dissolution of the smear layer observed by scanning electron microscopy. With SMPP, these authors observed adhesive and mixed failures with good penetration and retention of the adhesive in dentin tubules.

Conclusion

Based on the present study experimental conditions, it follows that there were no significant differences among the SBS of the two adhesive systems and that thermal cycling did not significantly influence bond strength of the two adhesive systems tested, therefore the null hypotheses tested in this study were accepted.

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