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Effect of caries-affected dentin on one-step universal and multi-step etch-and-rinse adhesives' bond strength

Efeito da dentina cariada na resistência adesiva de um sistema adesivo universal e um de condicionamento ácido total

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Resumo

Objetivo: Avaliar a influência da dentina afetada na resistência de união de um sistema adesivo universal e de um adesivo de condicionamento ácido total acetonado. **Material e método:** As faces oclusais de 60 terceiros molares humanos, hígidos e cariados, foram removidas a fim de expor o substrato dentinário. Os dentes foram divididos aleatoriamente em 6 grupos: Single Bond Universal (3M Dental Products, Seefeld, Germany), nos protocolos de condicionamento total e autocondicionante, e Prime&BondNT (DentsplyDeTrey, Konstanz, Germany), em dentina hígida e afetada. A *smearlayer* de 30 espécimes de dentina hígida foi padronizada com lixas de granulação 600. A dentina residual infectada de 30 espécimes foi removida com uma broca carbide número 4 até que nada fosse detectável por uma inspeção clínica. Pinos de resina composta (Filtek Z350 XT, 3M ESPE, St. Paul, MN) foram construídos usando tubos de amido como matriz. O teste de microcisalhamento foi realizado em uma máquina universal de testes até que houvesse a fratura. **Resultado:** Diferenças significantes foram observadas na resistência de união apenas para os espécimes de dentina hígida, mas diminuiu para os espécimes de dentina afetada, enquanto que o desempenho da adesão do Prime&BondNT não foi influenciada pela condição dentinária. **Conclusão:** A dentina afetada reduziu a resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em comparação à dentina hígida. A resistência de união do Single Bond Universal, em

Descritores: Dentina afetada por cárie; substrato dentinário; sistema adesivo multimodo.

Abstract

Objective: To evaluate the influence of caries-affected dentin on bond strength of a universal one-step and a multi-step etch-and-rinse adhesive system. **Material and method:** Enamel of 60 third human molars with and without caries was removed to expose dentin. The teeth were randomly assigned to six groups: Single Bond Universal (3M ESPE, St. Paul, MN, USA) in etch-and-rinse and in self-etch mode and Prime & Bond NT (Dentsply Co, Konstanz, Germany), all on sound and caries-affected dentin. Smear layer of the 30 sound dentin specimens was standardized by polishing with 600-grit SiC paper under water cooling. Residual infected dentin of the 30 caries-affected specimens was removed with a number 4 CA carbide bur until no caries smooth tissue was detectable by tactile-visual inspection. Cylinders of a light cured composite resin (Filtek Z350 XT, 3M ESPE) were built up using starch tubes and microshear test was performed until failure. The data was analyzed by one-way ANOVA and Tukey's post hoc test. **Result:** Significant differences in microshear bond strength (μ SBS) were observed for the caries-affected groups, but not for sound dentin. The μ SBS of Single Bond Universal were not influenced by the application protocol on sound dentin, however they were lower in the caries-affected group with both application protocols. The μ SBS for Prime & Bond NT was not influenced by the dentin conditions. **Conclusion:** Caries-affected dentin decrease in bond strength of Single Bond Universal in comparison to sound dentin. The bond strength of Prime & Bond NT was not altered by tset conditions.

Descriptors: Caries-affected dentin; dentinal substrate; multimode adhesive system.

INTRODUCTION

Interfacial integrity influences the efficacy and longevity of composite resin restorations¹. The researchers and manufacturers have dedicated efforts to obtain adequate hybridization of enamel and dentin introducing modifications on the adhesive systems and protocols through the last years. Some of these alterations resulted in better bond performances concerning to hybrid layer formation and mechanical properties^{2,3}.

Due to the non-uniform permeability and moisture of the dentin, adhesion in this substrate is still considered a challenge in clinical practice. Based on this, in order to facilitate clinical procedure, the new generation of simplified adhesive systems, also known as universal or multimode, can be used as self-etching or etch-and-rise. In addition, they would also be effective in both wet and dry demineralized dentin, thus overturning the critical wash and drying phase of the acid conditioning step⁴.

Studies involving bonding to normal dentin are still predominant in restorative dentistry⁵⁻⁷. Although major efforts concerning caries-affected dentin have been observed lately, since selective caries removal has been widely advocated to preserve tooth structure and avoid unnecessary pulp tissue exposure. Thereby, resin composite is bonded into a prepared cavity after removal of infected dentin, in which the cavity floor frequently consists of caries affected dentin⁸.

Comparisons between normal and caries-affected dentin have shown the influence of the dentinal condition on adhesive systems in mechanical tests⁹. Studies indicate that bonding to caries-affected dentin results in lower bond strengths as related to sound dentin. This is due to the weaker structure of the demineralized caries-affected dentin that limits adhesive infiltration because of the tubules filled with acid-resistant mineral deposits and to the unusual conformation of the hybrid layer, which is commonly thicker. Costa et al.¹⁰, showed low resin tags formation with short penetration into demineralized dentin regions and the increasing water content and the more permeable condition of caries-affected dentin are concerns that may compromise bonding quality/stability over time when using hydrophilic adhesives systems¹⁰⁻¹².

The aim of this study was to evaluate the influence of caries-affected dentin on bond strengths of a multimode and an acetone-based etch-and-rinse adhesive systems by a microshear test. The null hypothesis tested was that caries-affected dentin negatively influences the microshear bond strength of the multimode adhesive system.

MATERIAL AND METHOD

The *in vitro* study was approved by the local Committee of Ethics in Research (protocol n. 556.453). Thirty caries-affected and thirty caries-free extracted human third molars were selected. Before the experiments, they were disinfected in 0.5% chloramine T and stored in saline solution at 5°C, until the use in this study.

The enamel of all teeth was transversally sectioned at the mid coronal third by using a 22mm-diameter thin KG 7010 double-surface diamond saw (Medical Burs, Ind. & Com. Ltda., Cotia, Brazil) under copious water irrigation. In the caries-free dentin teeth, 600-grit silicon carbide paper was used for 60s under running water to produce a standardized smear layer. The caries-affected teeth had the infected dentin removed with a number 4 CA carbide bur (Medical Burs, Ind. & Com. Ltda.) until no caries smooth tissue was detectable by tactile-visual inspection.

A one-step universal adhesive system (Single Bond Universal, 3M ESPE, St. Paul – MN, USA) and a multi-step, acetone-based etch-and-rinse adhesive system (Prime&BondNT, DentsplyDeTrey, Konstanz, Germany) were used according to the manufacturer instructions. The prepared teeth were randomly assigned to six equal groups (n = 10) according to dentin conditions and adhesive systems:

- Group 1 sound dentin and Single Bond Universal applied in etch-and-rinse mode;
- Group 2: sound dentin and Single Bond Universal applied in self-etch mode;
- Group 3: sound dentin and Prime & Bond NT;
- Group 4: caries-affected dentin and Single Bond Universal in etch-and-rinse mode;
- Group 5: caries-affected dentin and Single Bond Universal applied in self-etch mode and;
- Group 6: caries-affected dentin and Prime & Bond NT.

Application mode, chemical composition and batch number of the adhesive systems tested are shown in Table 1.

After the adhesive procedures, as recommended for manufacturer, cylindrical restorations with a light-cured composite resin (Filtek Z350 XT shade A2, 3M ESPE, St. Paul, MN, USA) were built up on dentin using starch tubes of 0.96 mm inner diameter and 2.0 mm

Table 1. Adhesive Systems with approaching, chemical composition and batch number

Adhesive System	approaching	Chemical composition	Batch number
Single Bond Universal - 3M Dental Products, Seefeld, Germany	Etch and Rinse and Self-etching (SBU ER) (SBU SE)	methacryloyloxydecyl dihydrogen phosphate, phosphate monomer, dimethacrylate resins, hydroxyethyl methacrylate, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	1320500617
Prime & Bond NT - Dentsply De Trey, Konstanz, Germany	Etch and Rinse (PBNT)	Di and Tri methacrylate resins, silica, Monophosphate Dipentatriol penthacrylate (PENTA), nanofiller, initiators, stabilizer, cetylamine hydrofluoride, acetone	496153OP

height¹³. One tube per sample was positioned on the caries-affected dentin and another one on the sound dentin surface. After light curing of adhesive for 20 s - light-emitting diode (LED Schuster Ind. Ltda, Santa Maria, Brazil, at 800 mW/cm² - the tubes were filled in with two increments of composite resin and light-cured for 40 s. All the procedures were performed by a previously trained operator at room temperature of 20°C.

The specimens were stored in distilled water at 37° C for 24 h. The starch tubes and residues were removed with air-water spray. They were examined under $10 \times$ magnification by using a stereomicroscope (Discovery V20, Carl Zeiss AG, Oberkochen, Germany) to identify bonding defects.

The specimens were placed in a jig attached to a universal testing machine (EMIC DL 1000, Instron Brazil, São José dos Pinhais, Brazil). A 0.5mm diameter stainless steel wire loop was adapted precisely to the dentin–adhesive interface and shear load was applied at a crosshead speed of 0.5 mm/min until failure. The fracture pattern was examined at $70 \times$ magnification under a stereomicroscope and classified as adhesive, mixed and cohesive.

Means of microshear bond strengths were compared by two-way ANOVA followed by Tukey's test at a significance level of 5%.

RESULT

The dentin condition significantly affected microshear bond strength values of Single Bond Universal (p < 0.001). The adhesive system significantly affected the microshear bond strength values as well (p < 0.001) (Tables 2 and 3).

Figure 1 (Boxplot) shows that the bond strength values of Single Bond Universal were not influenced by the application mode, (i.e. etch-and-rinse or self-etch), but in both application modes it decreased in the caries-affected dentin group. The bond performance of Prime & Bond NT was not significantly influenced by the dentinal condition.

Mainly adhesive failures occurred in all groups. The dentinal condition did not influence the failure pattern. Mixed and cohesive failures were well distributed among the groups.

DISCUSSION

Mechanical tests are used to evaluate bond strength of adhesives on dental hard tissues. The microshear test allows bond strength measurement at standardized regions, preserving uniformity of the tested areas^{11,12}. Tedesco et al.¹³ introduced a starch tube matrix as a substitute of polyethylene matrix to build up composite specimens for this test. The main advantage of this matrix is that it is may easily be removed by water immersion.

On sound dentin, Single Bond Universal presented superior results than Prime & Bond NT regardless its application protocol. This can be explained by the presence of MDP in the composition of the Single Bond Universal, which provides a chemical bond to dentin⁶. Yoshida et al.¹⁴ showed an effective chemical interaction between MDP and hydroxyapatite forming a stable nano-layer, thus increasing the mechanical strength of the adhesive interface. In addition, the deposition of stable salts of MDP-Ca and nano-layers may explain the high binding stability, which has already been proven

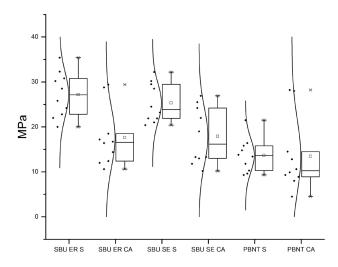


Figure 1. Boxplot-Wiskers of the μ SBS in MPa for the groups (Weibull distribution curves). SBU ER sound dentin; SBU ER caries-affected; SBU SE sound dentin; SBU SE caries-affected; 5: PBNT sound dentin, and PBNT caries-affected.

Table 2. Means of bond strength a	nd (standard deviation)	- Failure according groups tested (%)

	SBU ER	A M C	SBU SE	A M C	PBNT	A M C
Normal dentin	27.2 (5.7) ^{aA}	80 10 10	25.3 (4.5) ^{aA}	80 20 -	13.7 (3.3) ^{aB}	90 - 10
Caries-affected dentin	17.6 (7.1) ^{bA}	80 10 10	17.9 (5.7) ^{bA}	80 10 10	13.5 (6.3) ^{aA}	90 10 -

Tukey's test. Means value with the same uppercase letter are not significantly different to line and means value with the same lowercase letter are not significantly different to column (p>0.05).

Table 3. Critical values F distribution

	F-crit	F	p-value
Dentin Substrate (2)	7.12	14.33	<0.001
Adhesive System-approaching (3)	5.02	13.84	<0.001
(2) x (3)	3.16	3.50	<0.0372

The factors of analysis: dentin substrate (2), adhesive system (approaching) (3) and interaction (2)x(3).

both in laboratory and clinical research on both substrates studied^{6,14}. Complete penetration of resin monomers surrounding collagen fibrils is essential for strong adhesion and a perfect preservation of the collagen architecture and to seal the dentin surface. Since carious process disorganizes and degrades collagen fibrils and modifies dentin tubule distribution, caries-affected dentin does not permit uniform penetration of adhesive monomers and, consequently, regular hybridization¹⁵.

In the carious process, dentin shows two well-defined layers: the outer infected layer, composed of soft and degraded tissue infected by bacteria, and the inner affected layer, containing harder, sclerotic tissue¹⁶. The intense processes of demineralization and remineralization in caries-affected dentin promotes the formation of β -tricalcium phosphate (β TP) inside dentinal tubules. β TP is less soluble than hydroxyapatite and might induce defects and discontinuity in the demineralized layer^{9,15,17}. Phosphoric acid etching for 10–20 s cannot remove mineral deposits in dentinal tubules. However, conditioning for longer time (45s) is said to allow partial dissolution of calcifications, consequently forming funnel-shaped resin tags and allowing infiltration of adhesive monomers into the tubular anastomoses, increasing the bond strength of adhesive systems¹⁷.

The lower results obtained with Single Bond Universal on caries-affected dentin prove the null hypothesis of this study. The findings reinforce the view that acidic conditioners may not be sufficiently strong to etch sclerotic dentin, so bonding to this type of dentin may require a different pre treatment^{3,9,15}. For instance, use of hypochloric acid before mild self-etch adhesive application can significantly improve microtensile bond strength on caries-affected dentin and the increased bond strength is associated with more regular monomeric infiltration into caries-affected dentin¹⁸. Application of phosphoric acid before the use of a self-etch adhesive on dentin has also been reported, but no influence of the pretreatment on the immediate bond strength was determined¹⁷, contradicting previous results which showed that conditioning with phosphoric acid before application of self-etch adhesives decreases bond strength¹⁹.

A study comparing the microshear bond strengths of three adhesives selected according to their pH as strong self-etch adhesive (Adper Prompt L-Pop), intermediate self-etch adhesive (OptiBond Solo Plus), and mild self-etch adhesive (Clearfil SE Bond) showed better results on normal dentin than on caries-affected dentin, corroborating the finds of this study. The chemical composition and number of bottles rather than the acidity might thus be the primary reason of the lower bond strength on caries-affected dentin. However, these results are not supported by findings obtained with two mild self-etch adhesives (Aqua Bond and Tyrian SPE), which showed higher results on caries-affected dentin⁹ Although Single Bond Universal in the self-etch approach was not compared with another self-etch adhesive, the present results imply that bond strength depends on dentinal condition. Use of a hydrophobic resin coat before composite resin placement may increase bond strength of self-etch adhesives on normal dentin³. In this perspective hydrophobic resin coats might not be efficient on caries-affected dentin because of the fragility of the subsuperficial layer, which is poorly impregnated by adhesive monomers^{1,15,18}.

In the present study, Prime & Bond NT did not show significant differences between the substrates and its microshear bond strength tended to be lower than that of Single Bond Universal in the sound dentin specimens. This finding is similar to a previous report where acetone-based etch-and-rinse adhesives have lower bond strength than self-etch adhesives on normal dentin and caries-affected dentin²⁰. The low vapor pressure of acetone promotes alterations in the chemical composition of adhesives after application on dentin. The delicate balance of the components might be altered may be reflected in the bond performance^{21,22}. In particular, prolonged application time may improve the bond performance of acetone-based adhesives on normal dentin. In a previous study, modification of the application protocol led to higher bond strengths both immediately and on the long term²³. This result contradicts the findings of Elkassas et al.²⁴, who modified application protocols to test the bond efficiency of acetone-based etch-and-rinse and acetone-based self-etch adhesives. The authors reported that double application decreased bond strength of Prime&BondNT but increased that of G-Bond.

Prime&BondNT presented no significant differences to Single Bond Universal on caries-affected dentin. Differences in the histologic pattern of caries-affected dentin might influence the bond performance of all the studied adhesives²⁵. Therefore, altered dentin might be a decisive barrier to improve bond strength, independent of physicochemical characteristics, chemical compositions, and application protocols.

Given the peculiarities of the microshear test, predominantly adhesive failures were expected^{11,13}. Accordingly, the most common failure pattern was adhesive failure (80-90% of the specimens).

CONCLUSION

Within the conditions of the present study, caries-affected dentin lead to a decrease in bond strength of Single Bond Universal in comparison to sound dentin, and bond strength of Prime & Bond NT was lower than that of Single Bond Universal, but was not altered by substrate conditions.

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CONFLICTS OF INTERESTS

The authors declare no conflicts of interest.

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