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**COMPARAÇÃO DA RESISTÊNCIA DE UNIÃO DE  
SISTEMAS ADESIVOS EM DIFERENTES  
SUBSTRATOS DENTINÁRIOS**

**DISSERTAÇÃO DE MESTRADO**

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**Santa Maria, RS, Brasil  
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# **COMPARAÇÃO DA RESISTÊNCIA DE UNIÃO DE SISTEMAS ADESIVOS EM DIFERENTES SUBSTRATOS DENTINÁRIOS**

**Gabriel Ferreira Nicoloso**

Dissertação apresentada ao Curso de Mestrado do Programa de Pós-Graduação  
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requisito parcial para obtenção do grau de  
**Mestre em Ciências Odontológicas.**

**Orientadora: Prof. Dra. Rachel de Oliveira Rocha**

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**COMPARAÇÃO DA RESISTÊNCIA DE UNIÃO DE SISTEMAS  
ADESIVOS EM DIFERENTES SUBSTRATOS DENTINÁRIOS**

elaborada por  
**Gabriel Ferreira Nicoloso**

como requisito parcial para obtenção do grau de  
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...Aprende que não importa onde já chegou, mas onde está indo...

...Aprende que há mais dos seus pais em você do que você supunha...

...E você aprende que realmente pode suportar... que realmente é forte, e que pode ir muito mais longe depois de pensar que não se pode mais... ”

(William Shakespeare)

## **RESUMO**

Dissertação de Mestrado  
Programa de Pós-Graduação em Ciências Odontológicas  
Universidade Federal de Santa Maria

### **COMPARAÇÃO DA RESISTÊNCIA DE UNIÃO DE SISTEMAS ADESIVOS EM DIFERENTES SUBSTRATOS DENTINÁRIOS**

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Recentemente, uma nova categoria de sistemas adesivos foi proposta para a realização de procedimentos restauradores. Estes sistemas adesivos, intitulados “universais”, se propõem a diminuir a sensibilidade da técnica operatória, assim como aumentar a versatilidade na escolha do modo de aplicação a ser empregado sobre as estruturas dentárias. O objetivo deste trabalho foi apresentar dois artigos para avaliar o comportamento destes sistemas adesivos universais. O primeiro artigo se propõe a avaliar o sistema adesivo ‘universal’ (Single Bond Universal) aplicado nas duas estratégias de condicionamento à dentina desmineralizada de dentes decíduos e permanentes, em termos de resistência de união e comparar a dois sistemas de referência, Adper Single Bond 2 e Clearfil SE Bond. O segundo artigo comparou a resistência de união do Single Bond Universal em ambas estratégias de condicionamento aos mesmos sistemas adesivos de controle, porém à dentina desmineralizada e hígida de dentes permanentes. Quarenta e oito dentes permanentes e vinte e quatro dentes decíduos foram selecionados para realização de ambos estudos. Todos os dentes tiveram suas porções coronárias seccionadas para obtenção de superfícies planas em dentina (corpos de prova). A superfície dentinária dos dentes os quais representavam o substrato desmineralizado foram submetidas ao desenvolvimento de lesão de cárie por ciclagem de pH. Os demais dentes que representavam o substrato hígido foram mantidos intactos até os procedimentos adesivos. Os corpos de prova, de acordo com o tipo de dente, foram alocados aleatoriamente em grupos ( $n=6$ ) de acordo com o sistema adesivo/ estratégia de condicionamento. Após os procedimentos adesivos, blocos de resina composta foram confeccionados e os corpos de prova seccionados perpendicularmente a interface adesiva para obtenção de espécimes com área de seção de aproximadamente  $0,8\text{mm}^2$ . Os espécimes foram submetidos ao ensaio de microtração ( $1\text{mm/min}$ ). Os valores de resistência de união foram submetidos à análise de variância de dois-fatores (tipo de dente e sistema adesivo/ estratégia) ( $\alpha = 0,05$ ). No primeiro artigo, o sistema adesivo Single Bond Universal, em ambas estratégias de condicionamento, apresentou valores de resistência de união similares aos controles em ambos os substratos dentinários, assim como os diferentes substratos não apresentaram diferenças entre si. No segundo artigo, os valores de resistência de união à dentina hígida foram maiores comparados a dentina desmineralizada, porém nenhum sistema adesivo apresentou diferenças entre si. O sistema adesivo Single Bond Universal pode ser utilizado em ambas estratégias de condicionamento sem comprometer os procedimentos adesivos.

**Palavras-chave:** Dentina decídua. Dentina permanente. Microtração. Resistência de união. Sistemas adesivos universais.

## ABSTRACT

Master's Dissertation  
Post Graduate Program in Dental Science  
Federal University of Santa Maria

### COMPARISON OF THE BOND STRENGHT IN ADHESIVES SYSTEMS APPLIED TO DIFFERENT DENTIN SUSBTRATES

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Recently, a new category of adhesive systems has been proposed to perform restorative procedures. These adhesives, named as “universal” have been released in the market with the purpose to decrease the technique sensitivity, likewise they offer a great versatility in the procedure as can be applied to tooth structures in both application modes. The objective of this dissertation is to present two papers. The first article aimed to compare the bond strength of a “universal” adhesive (Scotchbond Universal Adhesive) applied in both application modes to its controls, Adper Single Bond 2 and Clearfil SE Bond, when bonded to caries-affected primary and permanent dentin. The second article compared the bond strength of the Scotchbond Universal Adhesive in both application modes to the same controls aforementioned to caries-affected and sound permanent dentin. Forty-eight permanent teeth and twenty-four primary teeth were selected to perform the experiments in both papers. All teeth had their mid-coronal dentin surfaces exposed by removing the occlusal third. The dentin surfaces, which represent caries-affected dentin substrate, were submitted to pH-cycling. The other teeth, sound dentin, remained intact until the bonding procedure. Specimens were randomly allocated ( $n=6$ ) according to the adhesives tested and conditions of the dentin. After bonding procedures, a composite core was build-up to a height of 4-5mm and then was sectioned perpendicular to the adhesive interface in order to obtain rectangular sticks ( $0.8 \text{ mm}^2$ ) that were submitted to microtensile tests (1mm/min). Two-way Analysis of Variance and post-hoc Tukey’s test ( $\alpha = 0.05$ ) were performed as statistical analysis. In the first paper, the universal adhesive, Scotchbond Universal Adhesive, presented statistically similar bond strength values to its controls in both dentin substrates, likewise the different substrates did not differ in bond strength values. In the second paper, adhesives bonded to sound dentin presented higher bond strength values compared with caries-affected dentin, but none of the adhesives tested presented statistically significant differences. The new “universal” adhesive, Scotchbond Universal Adhesive, can be applied in both application modes without the concern of compromise the bonding performance on resin composite restorations.

**Keywords:** Bond Strength. Microtensile. Permanent dentin. Primary dentin. Universal adhesives.

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## INTRODUÇÃO GERAL

Desde 1955, quando Michael G. Buonocore propôs o condicionamento ácido do esmalte dental para aumentar a adesão de materiais restauradores às estruturas dentais, fabricantes de diversas marcas comerciais tem disputado espaço no mercado odontológico na tentativa de lançar um material simples e universal para todas as situações restauradoras. Em 1982, Nakabayashi identificou que o condicionamento ácido seguido da aplicação de materiais resinosos à dentina formam uma zona de interdifusão entre monômeros resinosos e dentina inter e intratubular. Esta formação foi denominada camada híbrida e está relacionada ao aumento da adesão dos materiais resinosos à estrutura dentária.

Nas últimas décadas, um novo conceito denominado odontologia minimamente invasiva tem desafiado a adesão destes materiais resinosos, devido à preservação de uma dentina parcialmente desmineralizada - dentina afetada, na parede pulpar de cavidades submetidas a remoção parcial de tecido cariado (MALTZ et al., 2002; FALSTER et al., 2002; CASAGRANDE et al., 2009). Isto porque, inicialmente, os sistemas adesivos foram desenvolvidos para aplicação em substratos hígidos, os quais apresentam uma estrutura micro morfológica diferente da dentina afetada. Durante o processo carioso, des- e re-mineralização, há um aumento da porosidade da dentina intertubular, perda de cristalinidade da estrutura e alteração da matriz orgânica, o que promove um impacto significativo à adesão neste substrato (YOSHIYAMA et al., 2002). Devido a estas alterações na dentina afetada, uma incompleta infiltração de monômeros adesivos resulta na formação de camadas híbridas irregulares e defeituosas (ERHARDT et al., 2008), e assim, uma diminuição da performance adesiva pode ser esperada em relação à dentina sadia (ERHARDT et al., 2008, ZANCHI et al., 2010).

Existem duas principais categorias de sistemas adesivos, classificados de acordo com a interação destes com o substrato dental. Os primeiros sistemas adesivos comercialmente disponíveis no mercado odontológico foram classificados como “condicionamento ácido total”, que necessitavam da aplicação de um ácido, geralmente ácido fosfórico a 37%, nas estruturas dentais seguida da aplicação de *primer* e/ou adesivo (PASHLEY et al., 2011). Entretanto, esta estratégia de tratamento às estruturas dentais apresentava uma desvantagem, que em muitas vezes, resultava em falhas restauradoras. Através do condicionamento da dentina, grande parte de mineral era removido juntamente com a smear layer (lama dentinária) no procedimento de lavagem, expondo fibras colágenas que deveriam ser mantidas

idealmente úmidas para a correta formação da camada híbrida (TAY et al., 1996). Uma excessiva remoção de água durante o procedimento de secagem ocasionaria o colabamento das fibras colágenas impedindo uma adequada infiltração dos monômeros, e consequentemente, prejudicando a adesão dos materiais restauradores.

Na tentativa de diminuir a sensibilidade da técnica operatória, outra categoria de sistemas adesivos foi proposta comercialmente, sendo intitulados “auto-condicionantes”. Esta categoria de sistemas adesivos incorporava na sua formulação monômeros acídicos que substituem o condicionamento com ácido fosfórico (VAN MEERBEEK et al., 2011). Durante a aplicação destes sistemas adesivos às estruturas dentais, a anteriormente removida smear layer, nos sistemas de “condicionamento ácido total”, passaria a ser incorporada a camada híbrida, devido ao simultâneo “condicionamento” e infiltração dos monômeros acídicos a estrutura dental. Desta maneira, o procedimento adesivo seria menos sensível a técnica operatória, devido a menor subjetividade de aplicação.

Recentemente, os fabricantes propuseram outra categoria de sistemas adesivos, intitulados “universais”, devido a sua grande versatilidade de emprego nos procedimentos adesivos (HANABUSA et al., 2012; DE GOES et al., 2014). Estes sistemas adesivos, segundo os fabricantes, podem ser aplicados no esmalte e dentina em ambas estratégias de condicionamento, “condicionamento ácido total” ou “auto-condicionantes”. Estes sistemas adesivos possuem em sua formulação monômeros acídicos funcionais, os quais são responsáveis pelo condicionamento da estrutura e ligação química ao cálcio disponível. Dentre eles, destaca-se o monômero 10-metacriloloxidecil dihidrogenofosfato (MDP), mais comumente encontrado entre estes sistemas adesivos, e que além de promover uma ligação química, é responsável pela formação de nano-camadas, as quais aumentam a estabilidade da camada híbrida (YOSHIDA et al, 2012a). Diversos estudos laboratoriais têm sido realizados na tentativa de elucidar os mecanismos envolvidos na adesão destes sistemas adesivos. Recentemente, um ensaio clínico randomizado comparou a efetividade de um sistema adesivo universal em restaurações realizadas em lesões cervicais não-cariosas (PERDIGÃO et al., 2014). Os autores observaram que após 18 meses, ambas as estratégias de aplicação foram efetivas quando critérios relacionados a retenção, sensibilidade pós-operatória, descoloração marginal foram avaliadas. Entretanto, restaurações realizadas na estratégia “auto-condicionante” apresentaram um maior número de discrepâncias marginais quando comparadas as demais estratégias. Deve-se ressaltar que todos estes estudos foram realizados em dentina hígida, a qual apresenta diferenças importantes em relação a dentina sadia, conforme anteriormente citado.

Ademais, em procedimentos restauradores, não existe uma diferenciação entre o tratamento de substratos dentários permanentes e decíduos, condições que se diferenciam pela menor concentração de mineral e maior densidade tubular do último (SUMIKAWA et al., 1999; ANGKER et al, 2004). Apesar destas diferenças na micro-morfologia, resultados divergentes são encontrados na literatura entre sistemas adesivos aplicados em ambos os substratos. Alguns autores relatam menores valores de resistência de união aos dentes decíduos (SENAWONGSE et al., 2004), enquanto outros não encontram diferenças entre dentes decíduos e permanentes (RICCI et al., 2010). Entretanto, é consenso que apesar de os valores de resistência de união variarem entre os estudos, os dentes decíduos são mais reativos ao condicionamento dental, justamente por apresentarem menor concentração de cálcio e maior densidade tubular (NÖR et al., 1996; NÖR et al., 1997). Desta maneira, os novos sistemas adesivos universais, devem ser amplamente estudados nas duas condições, dentina afetada e dentina decídua, principalmente na estratégia de “condicionamento ácido total”, devido a presença de monômeros acídicos na formulação destes sistemas adesivos, que poderia promover um sobre-condicionamento, se aplicados nesta estratégia.

O objetivo deste trabalho foi avaliar o comportamento de um sistema adesivo universal, considerando como fator de variação principal o tipo e a condição da dentina. Desta forma, dois artigos são apresentados, o primeiro com a proposta de comparar a resistência de união do sistema adesivo ‘universal’ (Single Bond Universal), aplicado nas duas estratégias de condicionamento, comparado aos sistemas Adper Single Bond 2 e Clearfil SE Bond à dentina desmineralizada de dentes decíduos e permanentes; e, no segundo artigo, a resistência de união do sistema adesivo Single Bond Universal, em ambas estratégias de condicionamento, foi comparada aos mesmos controles, porém aplicados à dentina desmineralizada e hígida de dentes permanentes.

## 1. ARTIGOS

Esta dissertação está baseada nas normativas da Universidade Federal da Santa Maria. Por se tratar de pesquisa envolvendo seres humanos, o projeto de pesquisa deste trabalho foi submetido à apreciação do Comitê de Ética em Pesquisa da Universidade Federal de Santa Maria, tendo sido aprovado (ANEXO A). Sendo assim, esta dissertação é composta de dois artigos que serão enviados para publicação nas revistas “*Journal of Adhesive Dentistry*” (Artigo I) e “*Journal of Dentistry*” (Artigo II).

**1.1. ARTIGO I**

**IS THERE A BEST PROTOCOL FOR BONDING A  
UNIVERSAL ADHESIVE TO CARIES-AFFECTED PRIMARY  
AND PERMANENT DENTIN?**

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## **Is there a best protocol for bonding a universal adhesive to caries-affected primary or permanent dentin?**

**Nicoloso GF, Antoniazzi BF, Lenzi TL, Soares FZM, Rocha RO**

### **Abstract**

*Purpose:* To evaluate whether the etch-and-rinse or self-etching mode is the best protocol for bonding to primary and permanent caries-affected dentin by a universal adhesive.

*Materials and Methods:* Flat mid-coronal dentin surfaces of 24 primary and 24 permanent molars were submitted to pH-cycling for 14 days to induce artificial caries-affected dentin. The teeth of each substrate (primary and permanent) were randomly assigned according to the adhesive systems and bonding strategy: a universal adhesive, Scotchbond Universal Adhesive – self-etch and etch-and-rinse techniques; a two-step etch-and-rinse adhesive, Adper Single Bond 2; and two-step self-etch system, Clearfil SE Bond, as controls. After bonding and restorative procedures, specimens were sectioned to obtain rectangular sticks ( $0.8 \text{ mm}^2$ ) that were submitted to microtensile tests (1mm/min). The data were analyzed by two-way Analysis of Variance and Tukey post-hoc test ( $\alpha = 0.05$ ).

*Results:* The universal adhesive showed similar bond strength values compared to the control groups, irrespective of the bonding strategy. Likewise, the same bonding performance was observed for all adhesives to either caries-affected primary or permanent dentin.

*Conclusion:* The new ‘universal’ adhesive, Scotchbond Universal Adhesive, can be used in both application modes in caries-affected primary and permanent dentin, as the bonding performance was not influenced by these different substrates.

**Keywords:** caries-affected dentin, artificial caries, microtensile, bond strength, universal adhesives

## Introduction

Microtensile bond strength testing remains a useful way to assess adhesive effectiveness,<sup>20</sup> adding the possibility to consider different substrate conditions. Bonding to caries-affected dentin is of paramount importance since the concept of minimally invasive dentistry considers the maintenance of a partial demineralized dentin on the cavities pulpal floor.<sup>2,6,13,21</sup>

Sound and caries-affected dentin present different mineral concentrations<sup>1</sup> and in the latter, micromorphological alterations can be detected. In this sense, the performance of adhesive systems to caries-affected dentin is compromised due to formation of an irregular and defective hybrid layer, as consequence of inadequate infiltration of monomers after conditioning steps through this demineralized substrate.<sup>5</sup>

It should also be stated that primary and permanent teeth are different structures, and therefore, different patterns of acid conditioning<sup>17,18</sup> and bonding performance might be achieved.<sup>24,28</sup> Despite this, it is noteworthy that no distinction is made between dentitions regarding adhesive procedures.

New dental adhesives called ‘universal’ or ‘multi-mode’ were recently launched in the market with the concept that the clinician could choose what bonding strategy will be used in each clinical situation.<sup>4,7,19</sup> The manufacturers of these products also claim that their use does not rely upon the tooth structure being prepared to restoration.

Although a recent systematic review<sup>23</sup> has evidenced that the performance of the universal adhesives by *in vitro* studies is not dependent on the adhesive strategy to sound dentin, the literature still scarce with regard the bond strength of these materials on different substrates, as caries-affected dentin.<sup>12</sup> In this sense, it is imperative to evaluate the bonding performance of universal adhesives in both self-etch and etch-and-rinse modes to caries-affected dentin of primary and permanent teeth, which represent common substrates in most clinical situations.

Therefore, this study aimed to evaluate whether the etch-and-rinse or self-etching mode is the best protocol for bonding to primary and permanent caries-affected dentin by a universal adhesive. The null hypotheses tested were that 1) different substrates do not influence the bonding performance of adhesive systems; 2) the ‘universal’ adhesive in both application modes has similar bond strength values to the other etch-and-rinse and self-etch adhesives.

## **Materials and Methods**

### *Tooth Selection and Preparation*

Twenty-four human third molars and twenty-four human primary molars were obtained from a pool after approval of the study protocol by the Local Ethics Committee. The teeth were disinfected in 0.5% cloramine-T solution and stored in distilled water at 4°C for up to 3 months.

Flat mid-coronal dentin surfaces were exposed after removal of occlusal enamel using a slow-speed diamond saw (Labcut 1010, Extec Co, Connecticut, EUA) under water-cooling. The surrounding enamel was removed using a diamond bur in a high-speed handpiece (#3146, KG Sorensen, São Paulo, Brazil) under copious irrigation. The exposed dentin surfaces were further polished with 600-grit silicon carbide abrasive paper for 60s in order to obtain standardized smear layers.<sup>27</sup>

### *Artificial caries induction*

All surfaces of the teeth were then covered with two-layers of an acid-resistant nail varnish (Colorama Maybelline: São Paulo, Brazil) in order to leave only the occlusal dentin surface exposed.

Specimens were then submitted to pH-cycling to induce artificial caries-affected dentin. All specimens were individually immersed for 14 days in demineralizing solution (2.2 mM CaCl<sub>2</sub>, 2.2 mM NaH<sub>2</sub>PO<sub>4</sub>, 0.05 M acetic acid with adjusted pH of 4.5) for 8h and remineralizing solution (1.5 mM CaCl<sub>2</sub>, 0.9 mM NaH<sub>2</sub>PO<sub>4</sub>, 0.15 mM KCL adjusted pH of 7.0) for 16h at room temperature without agitation.<sup>11</sup> After every cycle, solutions were changed and specimens rinsed with deionized water and blotted dry. Solutions were periodically measured under a pH-meter.

### *Experimental design*

The teeth were randomly allocated into eight groups (n=6) according to type of teeth - caries-affected primary or permanent, and adhesive system - Scotchbond Universal Adhesive (SBU, 3M ESPE) in both application modes (etch-and-rinse – E&R and self-etch - SE), Adper Single Bond 2 (ASB, 3M ESPE) and Clearfil SE Bond (CSE, Kuraray).

### *Bonding Procedures*

A single trained operator applied all adhesive systems according to their respective manufacturer's instructions (Table 1). A resin composite core (Z100, 3M ESPE) was built up to a height of 4-5 mm in three to four increments that were individually light cured for 40s using a light-emitting diode curing unit (EMITTER C, SCHUSTER; Rio Grande do Sul,

Brazil) with an output of 600mW/cm<sup>2</sup> monitored with a radiometer. All specimens were stored in distilled water at 37°C for 24h prior to be prepared for microtensile testing.

#### *Microtensile Bond Strength ( $\mu$ TBS) Testing*

Specimens were sectioned perpendicular to the adhesive interface using a water-cooled diamond saw (Labcut 1010, Extec Co, Connecticut, EUA) in order to obtain rectangular sticks with a cross-sectional area of approximately 0.8mm<sup>2</sup>, measured with a digital caliper (Carbografite, Equipamentos Industriais Ltda., Rio de Janeiro, Brazil).

The bonded sticks were then fixed to a testing jig with cyanoacrylate glue and stressed at a crosshead speed of 1mm/min until failure using a universal testing machine (EMIC DL 1000, Equipment and Systems Ltda., Paraná, Brazil) with a load cell of 50N. The  $\mu$ TBS values (MPa) were determined dividing the measure force (N) at the time of fracture by the bonded area (mm<sup>2</sup>).

#### *Failure Mode*

The failure mode was determined to each debonded specimen under a stereomicroscope at 40X magnification and classified as adhesive/mixed (failure at the resin-dentin interface or mixed with cohesive failure of the neighboring substrate) or cohesive (failure exclusively within the dentin or resin composite). Representative specimens from each group were gold sputtered and then analyzed with scanning electron microscopy (SEM, Jeol JSM-T330A, Jeol Ltda., Tokyo, Japan) operated in the secondary electron mode with 20 kV to confirm the failure mode.

#### *Statistical analysis*

This study considered the tooth as experimental unit. Therefore, the mean value of all sticks from each tooth was calculated and then, the average  $\mu$ TBS of the six teeth was calculated per group. The sample size of 6 teeth per group was estimated previously considering an 80% power, a coefficient of variation of 20% and assuming a two-sided 5% significance level for comparisons. Premature failures (ptf) that occurred during preparation of the specimens (before testing) were excluded from statistical analysis.

The normal distribution of the data was confirmed using Kolmogorov-Smirnov test. The mean bond strength values were analyzed by two-way Analysis of Variance (ANOVA), considering the type of teeth and adhesive systems as main factors, and the cross-interaction between them. Statistical analysis was performed using the Minitab software (Minitab Inc., State College, Pennsylvania, USA) at a significance level of 5%.

## Results

Descriptive statistics including means of  $\mu$ TBS, standard deviation, total number of tested specimens and number of premature failures (ptf) are shown in Table 2.

The main factors type of teeth ( $p=0.557$ ) and adhesive systems ( $p=0.506$ ), as well as the cross-interaction factors “type of teeth vs. adhesive system” ( $p=0.729$ ), were not statistically significant. The bond strength of the Scotchbond Universal Adhesive was not influenced neither by application mode nor type of teeth (permanent or primary). Specimens of all experimental groups presented adhesive/mixed failures when evaluated under a stereomicroscope. This pattern was further confirmed in SEM images (Figure 1).

## Discussion

Universal adhesives were introduced in the market to simplify even more the bonding procedures. The manufacturers claimed that their bonding performance do not rely on the strategy used to perform resin composite restorations. Moreover, it is suggested that either etch-and-rinse or self-etch modes could be applied upon tooth structures, irrespective of the substrate. The results of our study confirm this statement, as both application modes did not compromise the bonding performance of the Scotchbond Universal Adhesive applied to caries-affected dentin of primary and permanent teeth.

The use of natural caries-affected dentin present several difficulties as a substrate to bond strength evaluations. The great variability among caries lesion, e.g., size, shape and depth, demand a lot of time to obtain natural lesions with satisfactory dentin thickness, especially in primary teeth. A recent study<sup>9</sup> compared natural caries-affected dentin and artificial caries-affected dentin and demonstrated that both substrates when bonded presented similar  $\mu$ TBS values. Thus, as artificially-created caries-affected dentin is less labor and time consuming, it was the substrate opted in our study to evaluate the performance of adhesive systems.

Previous studies<sup>3,14,29</sup> also showed similar bond strength values when the Scotchbond Universal Adhesive was applied in permanent dentin in both application modes. Nevertheless, all the aforementioned studies evaluated  $\mu$ TBS values only in sound dentin, and consequently, different results could be expected on caries-affected dentin. A recent study<sup>12</sup> verified that the Scotchbond Universal Adhesive bonded to caries-affected primary dentin following both strategies presented similar bond strength values to the control adhesives, Adper Single Bond Plus and Clearfil SE Bond. Although, it is noteworthy that the authors<sup>12</sup> found a drop of

approximately 50% in bond strength when the Scotchbond Universal Adhesive was applied in the self-etch mode compared to the etch-and-rinse strategy in sound primary dentin.

Primary and permanent teeth are different substrates. Chemical and micromorphological characteristics, such as lower calcium concentration and greater tubule density observed in primary teeth,<sup>1,26</sup> may impact the bonding performance of adhesive systems. Nevertheless, these data are still controversial, since some authors found lower bond strength values to sound primary dentin when compared to permanent ones,<sup>10,24,28</sup> while others showed similar results.<sup>22,25</sup>

In this sense, it was expected that the universal adhesive bonded to caries-affected dentin of primary teeth following etch-and-rinse strategy would result in lower bond strength values in comparison with caries-affected permanent dentin, by the association of preliminary phosphoric acid and the acidic primer of the adhesive. However, it was not observed in our study, which is possible related to the ultra-mild etching aggressiveness (pH = 2.7) of the Scotchbond Universal Adhesive. Our results suggest that considering caries-affected dentin substrate, the type of teeth does not influence the bond strength values of the adhesives evaluated, accepting the first null hypothesis. Further studies evaluating bonding degradation on universal adhesives to caries-affected dentin of both primary and permanent teeth should be conducted to confirm this finding.

It appears that the bond strength of the Scotchbond Universal Adhesive to caries-affected dentin does not rely neither on the application mode nor type of teeth. Noteworthy, it presented similar results compared to adhesive systems considered as controls, which are considered ‘gold-standards’ in each strategy. Thus, the second null hypothesis was also accepted. This could be due to either the acid resin monomer 10-MDP (methacryloyloxydecyl dihydrogen phosphate), which is able to establish a chemical bond to apatite crystallites and self-assembled nano-layers,<sup>8,30,31</sup> or the polyalkenoic acid copolymers, as both could bond chemically to tooth structures being observed in the adhesives evaluated. It is noteworthy that some state that polyalkenoic acid copolymers present in the Scotchbond Universal Adhesive could compete with MDP monomers for Ca-bonding sites and also prevent monomer approximation during polymerization due to its high molecular weight.<sup>15</sup> Nevertheless, these findings might be contradictory and not well explained, as a recent study confirmed that the Scotchbond Universal Adhesive did not present a drop in bond strength values over a period of 6 months of water storage.<sup>16</sup>

At this moment, there are no clinical trials available regarding the performance of universal adhesives in minimally invasive cavity preparations that preconize partial caries

removal in permanent or primary dentitions. Therefore, in vitro studies are essential to compare adhesive systems or different etching strategies, and choose ones that are best suitable for each substrate. Our findings suggest that as the universal adhesive system did not differ from the controls it can be used in both strategies making the procedure less technique sensitive when restoring carious primary or permanent teeth. Consequently, the practitioners could choose any application mode that best fits the clinical situation without the concern of compromise the restorative procedure.

In conclusion, the universal adhesive tested can be used in both application modes in caries-affected primary and permanent dentin, as the bonding performance was not influenced by these different substrates.

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**Clinical relevance:** There is no best protocol for bonding universal adhesives that contain MDP to caries-affected dentin in primary or permanent dentitions, which ensure a restorative procedure more user-friendly.

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Table 1. Adhesive systems (batch number), composition and application modes\*

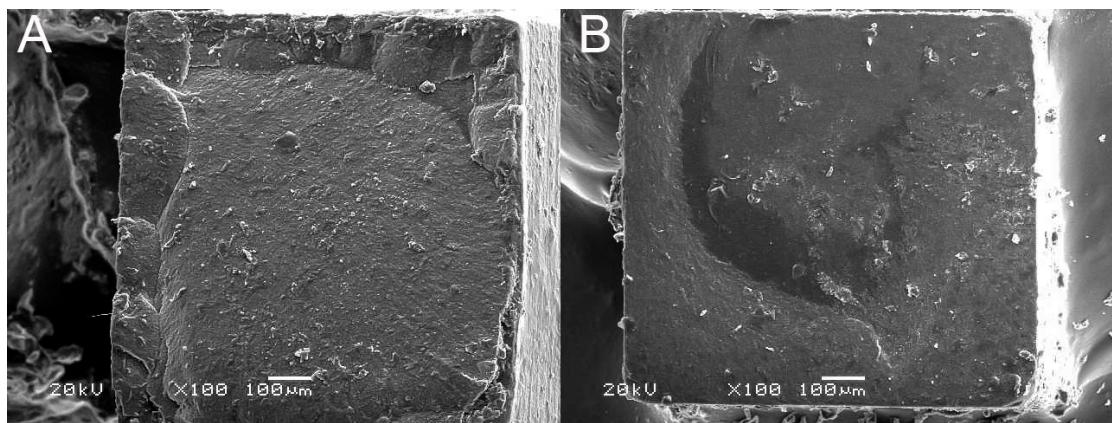
Adhesive System/ Batch Number	Composition**	Mode of application	
		Self-etch	Etch-and-rinse
Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA)  Batch number: (N508311)	Etchant: phosphoric acid 37% Bond: bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly(itaconic) acids, 10% by of 5 nm-diameter spherical silica particles	N.A.	1. Apply etchant for 15s 2. Rinse for 10s 3. Blot excess of water 4. Apply actively 2 consecutives layers of adhesive for 15s 5. Gently air dry for 5s 6. Light cure for 10s
Clearfil SE Bond (Kuraray Dental Inc., Okayama, Japan)  Batch number: Primer (012333A) Bond (01865A)	Primer: MDP, HEMA, hydrophobic dimethacrylate, camphorquinone, water, N,N- diethanol toluidine Bond: MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, camphorquinone, N,N- diethanol p-toluidine, colloidal silica	1. Apply actively primer to tooth surface and leave in place for 20s 2. Dry with air stream to evaporate the volatile solvents 3. Apply actively bond to tooth surface and then create a uniform film using a gentle air stream 4. Light cure for 10s	N.A.
Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA)  Batch number: (509806)	Etchant: phosphoric acid 37% Bond: MDP, HEMA, dimethacrylate resins, methacrylate-modified polyalkenoic acid copolymer ethanol, water, filler, initiator, silica	1. Apply actively the the adhesive to the entire surface for 20s. If necessary, rewet the disposable applicator 2. Direct a gentle air stream over the adhesive for 5s or until it no longer moves and the solvent completely evaporated 3. Light cure for 10s	1. Apply etchant for 15s 2. Rinse thoroughly 3. Blot excess of water 4. Apply adhesive as for the self-etch mode

\* According to manufacturers. \*\* Abbreviations: Bis-GMA: bisphenol A glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; N.A.: not applicable

Table 2. Means of bond strength (MPa) and standard deviations (SD) [number of tested specimens/premature failures specimens] for all experimental groups.

<b>Adhesive system</b>				
<b>Type of teeth</b>	SBU (E&R)	SBU (SE)	ASB	CSE
Caries-affected primary dentin	22.65(8.92) [53/3]	19.57(4.71) [61/1]	22.55(2.89) [48/0]	19.70(5.66) [65/5]
Caries-affected permanent dentin	23.88(2.57) [59/10]	22.74(5.67) [84/2]	20.93(4.79) [67/6]	20.47(3.11) [75/4]

Figure 1. SEM photograph of fractured specimens representative of mixed failure pattern from A) Adper Single Bond 2 and B) Scotchbond Universal Adhesive – etch-and-rinse strategy.



## 1.2. ARTIGO II

### THE BONDING PERFORMANCE OF A UNIVERSAL ADHESIVE TO CARIES-AFFECTED DENTIN

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## The bonding performance of a universal adhesive to caries-affected dentin

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### ABSTRACT

**Objectives:** To evaluate the bonding performance of a universal adhesive to sound and caries-affected dentin using either an etch-and-rinse or self-etch strategy.

**Methods:** Flat mid-coronal dentin surfaces from 48 third molars were randomly assigned to 8 groups according to substrate (sound dentin – SD and caries-affected dentin – CAD: pH-cycling for 14 days) and to adhesive and application mode, Scotchbond Universal Adhesive (self-etch or etch-and-rinse strategies); Adper Single Bond 2 (two-step etch-and-rinse adhesive) and Clearfil SE Bond (two-step self-etch system), as controls. After restorations were constructed, specimens were stored in water for 24 h and then resin-dentin sticks were prepared ( $0.8 \text{ mm}^2$ ). The sticks were tested under tension at 1.0 mm/min. Data were analyzed with two-way ANOVA and Tukey's test ( $\alpha = 0.05$ ).

**Results:** Caries-affected dentin presented lower  $\mu\text{TBS}$  values compared to sound dentin, irrespective of the adhesive system or application mode ( $p=0.000$ ). The universal adhesive applied following both etch-and-rinse and self-etch strategies showed similar bond strength compared with control adhesive systems ( $p=0.07$ ).

**Conclusions:** Caries-affected dentin compromises the bond strength of a universal adhesive, irrespective of the application mode. However, the bonding performance of the universal adhesive is not influenced by etch-and-rinse or self-etch strategies.

**Clinical Significance:** Universal adhesive systems can be used in different application modes when bonded to dentin. It should be highlighted that even this new category cannot maintain their bonding performance to caries-affected dentin.

Keywords: dentin, pH-cycling, caries-affected dentin, microtensile, universal adhesives

## 1. Introduction

The universal adhesive systems have recently been proposed to be used in both etch-and-rinse or self-etch strategies, irrespective of tooth structure, giving to practitioners the choice of the convenient strategy, depending on which best fits the clinical conditions, making the bonding procedure more user-friendly.

Few studies have assessed these universal systems, but a recent systematic review<sup>1</sup> pointed out that universal adhesives do not present differences regarding application modes when bonded to sound dentin. Conversely, the enamel bond strength of universal adhesives is improved with prior phosphoric acid etching. The authors also suggested that studies performed in different substrates, such as carious dentin, are desirable to assess the performance of these new universal adhesives.

Caries-affected dentin is a substrate commonly observed on clinical restorations, that presents important differences in comparison with sound dentin. More porous intertubular dentin, loss of crystallinity and alteration of the organic matrix are some of the characteristics from caries-affected dentin that can negatively impact on the adhesion to this substrate.<sup>2</sup> Moreover, an incomplete infiltration of the adhesive monomers, formation of irregular and defective hybrid layers,<sup>3</sup> and consequently, a drop in the bonding performance might be expected.<sup>3,4</sup>

Since these new “universal” adhesives were not yet evaluated on caries-affected permanent dentin, a laboratory study must be conducted to compare the bonding performance of these systems with previous adhesives’ categories that are already consolidated in the literature. It can be expected that universal adhesives applied in etch-and-rinse mode tend to produce deeper dentin demineralization,<sup>5,6</sup> resulting in thicker hybrid layers<sup>7</sup> and incomplete impregnation areas beneath the hybrid layer, compromising immediate and long-term bond strength to caries-affected dentin. To extent of our knowledge, this is the first investigation that attempts to elucidate the bonding performance of universal adhesives on caries-affected permanent dentin.

Thus, the aim of this study was to evaluate the bonding performance of a universal adhesive to sound and artificial caries-affected dentin applied in etch-and-rinse and self-etching strategies compared to correspondent adhesives. The null hypotheses tested were: (1) the bond strength is not affected by substrate condition and (2) the universal adhesive has similar bond strength to the controls, regardless of the application mode.

## **2. Materials and Methods**

### **2.1. Tooth selection and preparation**

Forty-eight sound third molars were selected from a pool after the review and approval of the study protocol by Institutional Ethics Committee. Teeth were disinfected in 0.5% aqueous cloramine and stored in distilled water at 4°C for up to 3 months.

Mid-coronal flat dentin surfaces were exposed after removal of occlusal enamel using a diamond saw (Labcut 1010, Extec Co, Connecticut, EUA) under water-cooling. The surrounding enamel was then removed using a diamond bur in a high-speed handpiece (#3146, KG Sorensen, São Paulo, Brazil) under copious irrigation. The exposed dentin surfaces were further polished with 600-grit silicon carbide paper under running water for 60s to create a standardized smear layer.<sup>8</sup>

### **2.2. Experimental design**

Teeth were randomly allocated into eight groups (n=6) according to the adhesive systems tested, Scotchbond Universal Adhesive (SBU, 3M ESPE) in the both application modes (etch-and-rinse and self-etch), Adper Single Bond 2 (ASB, 3M ESPE) and Clearfil SE Bond (CSE, Kuraray); and substrate condition, sound and caries-affected dentin. Twenty-four molars were subjected to artificial caries-affected dentin induction using a pH-cycling model.<sup>9,10</sup> The other half of the teeth remained intact, stored in distilled water, until the bonding and restorative procedures.

### **2.3. Artificial caries induction by pH-cycling**

Specimens were covered with two layers of acid resistant nail varnish, except the exposed dentin surfaces. Then, specimens were immersed individually in a demineralizing solution (2.2 mM CaCl<sub>2</sub>, 2.2 mM NaH<sub>2</sub>PO<sub>4</sub>, 0.05 M acetic acid with adjusted pH of 4.5) for 8 hours and in a remineralizing solution (1.5 mM CaCl<sub>2</sub>, 0.9 mM NaH<sub>2</sub>PO<sub>4</sub>, 0.15 mM KCL with adjusted pH of 7.0) for 16 hours.<sup>9,10</sup> After every cycle, solutions were changed and specimens rinsed with deionized water and blotted dry. This procedure was carried out for 14 days. Solutions were periodically measured under a pH-meter.

### **2.4. Bonding and restorative procedures**

All adhesives were applied on dentin surfaces according to their respective manufacturer's instructions by a single trained operator (Table 1). After the bonding procedures, all teeth received a hybrid resin composite (Z100, 3M ESPE, Minnesota, USA) in four increments of 1 mm each. Each resin composite layer was individually light-cured for 40s using a light-emitting diode curing unit (EMITTER C, SCHUSTER; Rio Grande do Sul,

Brazil) with an output of 600mW/cm<sup>2</sup> monitored with a radiometer. All bonded specimens were stored in distilled water at 37°C for 24h prior to be prepared for microtensile testing.

## **2.5. Microtensile bond strength ( $\mu$ TBS) testing**

Each bonded specimen was longitudinally sectioned in the mesiodistal and buccolingual directions across the bonded interface using a water-cooled diamond disk in a cutting machine (Labcut 1010, Extec Co, Connecticut, EUA). This procedure resulted in rectangular sticks with a cross-sectional area of approximately 0.8mm<sup>2</sup>. Each stick had its cross-sectional area measured with a digital caliper (Carbografite, Equipamentos Industriais Ltda., Rio de Janeiro, Brazil).

Each stick was fixed with cyanoacrylate glue to a testing jig and stressed till failure under tension at a crosshead speed of 1mm/min using a universal testing machine (EMIC DL 1000, Equipment and Systems Ltda., Paraná, Brazil) with a load cell of 50N. A single experienced operator performed the test.

## **2.6. Failure Mode**

The failure mode of each stick was analyzed using a stereomicroscope at a magnification of 40x. Failures were classified as adhesive/mixed (failure at the resin-dentin interface or mixed with cohesive failure of the neighboring substrate) or cohesive (failure exclusively within the dentin or resin composite). Representative specimens from each group were gold sputtered and analyzed with scanning electron microscopy (SEM, Jeol-JSM-T330A, Jeol Ltda., Tokyo, Japan) operated in the secondary electron mode with 20 kV to confirm the failure mode.

To confirm the effect of artificial caries induction, an additional tooth was sectioned into two halves and prepared exactly as described elsewhere, with the exception of bonding procedures. One-half was subjected to pH-cycling to artificial caries induction using the same protocol described previously. The other one was maintained with sound dentine. Sections were immersed in EDTA solution (0.7 M, pH = 7.4) for 5 min followed by immersion in sodium hypochlorite (0.34 M, pH = 12.3) for 3 min.<sup>11</sup> Then, specimens were dehydrated in ascending degrees of ethanol (30, 50, 70 and 80% for 5 min each, and 90, 95 and 100% for 10 min each). Subsequently, it was gold sputtered and analyzed with SEM operated in the secondary electron mode with 15 kV.

## **2.7. Statistical analysis**

The experimental unit in this study was the tooth. Thus, the mean of the  $\mu$ TBS values of all of the sticks from the same tooth were averaged for statistical analysis. The  $\mu$ TBS means for every test group was expressed as the mean of the six teeth used per group. The

sample size of 6 teeth per group was estimated previously considering an 80% power, a coefficient of variation of 20% and assuming a two-sided 5% significance level for comparisons. Only specimens with adhesive/mixed failures were considered. Pre-test failures (ptf) that occurred during specimens' preparation before testing were not included in the statistical analysis, but rather are presented descriptively from each group in an independent analysis.

The normal distribution of the data was confirmed using Kolmogorov-Smirnov test. The  $\mu$ TBS values were analyzed by two-way Analysis of Variance (ANOVA) (adhesives systems *vs.* substrate condition) and post-hoc Tukey test at a significance level of 0.05. Statistical analysis was performed using the Minitab software (Minitab Inc., State College, Pennsylvania, USA).

### **3. Results**

Representative images of sound and caries-affected dentin can be seen in Figure 1. Sound dentin presented a regular surface with wide-open dentin tubules, while an irregular surface clearing indicating demineralization on intertubular dentine and extensive opening of dentinal tubules can be seen in caries-affected dentin.

Bond strength means and standard deviations for all experimental groups are shown in Table 2. There was a statistically significant difference considering the main factor substrate condition ( $p=0.000$ ). The  $\mu$ TBS to caries-affected dentin were lower than those obtained for sound dentin. The difference among adhesives was not statistically significant ( $p=0.070$ ). The cross-product interaction “substrate condition *vs.* adhesive” was also not statistically significant ( $p=0.091$ ).

A higher number of pre-test failures were observed in caries-affected dentin. In these groups, all failures were adhesive/mixed (Figure 2). Adhesives bonded to sound dentin produced either adhesive/mixed or cohesive failures (Figure 3). Pre-test failures were observed in sound dentin only when using Clearfil SE Bond.

### **4. Discussion**

Considering that in dental practice different substrates are frequently encountered in the cavity preparation, it is important to evaluate the bonding performance of recently launched universal adhesives to sound and caries-affected dentin. The main idea from this study was to assess the bonding performance to sound and caries-affected dentin using a universal adhesive in etch-and-rinse or self-etch strategy and compare to the adhesives

considered as ‘gold-standard’ in each category. All adhesives assessed in our study had a decrease in bond strength values to caries-affected dentin; rejecting our first null hypothesis.

Our findings are in accordance to previous studies that found lower values to caries-affected dentin compared to sound one,<sup>4,12</sup> however, no studies have evaluated universal systems in this substrate and as a result, comparisons are difficult to be made. The carious process creates porous zones within intertubular dentin due to the mineral loss. In this sense, the porosity of this substrate permits deeper etching,<sup>5</sup> and consequently, thicker hybrid layers are found when adhesives are bonded to caries-affected dentin.<sup>3,13-15</sup> It is noteworthy that although thicker hybrid layers are formed, this does not represent an advantage, as an irregular and defective hybrid layer can be observed,<sup>3</sup> as consequence of inadequate infiltration of monomers after conditioning steps through this demineralized substrate. Moreover, mineral content seems to be the main reason for the decreased bond strength values observed in caries-affected dentin when compared to sound dentin, irrespective of the adhesive system (etch-and-rinse or self-etch).<sup>16,17</sup>

Recently, the same universal system (Scotchbond Universal Adhesive) was evaluated in caries-affected dentin, though in primary teeth. The  $\mu$ TBS values to caries-affected dentin were also lower than those obtained for sound dentin, except when this system was used as self-etch mode. Despite this, it is known that permanent and primary dentin are different substrates,<sup>18,19</sup> and therefore, it is crucial to also investigate how this adhesive will perform on caries-affected dentin of permanent teeth.

Furthermore, a higher number of pre-test failures were observed in the caries-affected dentin, which may be attributed to the lower cohesive strength of the substrate.<sup>2</sup> Cohesive failures did not occur when adhesives were bonded to caries-affected dentin. Conversely, greater frequency of cohesive failures was found in sound dentin, probably due to the higher bond strength values observed.

Similar  $\mu$ TBS values were observed among evaluated adhesive systems, irrespective of the application mode of the universal adhesive. Thus, our second null hypothesis must be accepted, as the Scotchbond Universal Adhesive did not differ from its control materials. Our findings corroborate results reported in the literature,<sup>20-22</sup> in which some authors found no differences in bond strength to sound dentin between self-etch and etch-and-rinse strategies using Scotchbond Universal Adhesive. It is valid to assume that, the composition of Scotchbond Universal Adhesive seems to be a ‘hybrid’ of the other systems, including the polyalkenoic acid copolymer (Vitrebond Copolymer) and the functional monomer MDP that are capable to establish a chemical bond to hydroxyapatite, and thus, the similar bond strength

among evaluated systems might be expected. Nevertheless, different results can also be found on the literature when compared it to the same controls.<sup>23</sup>

Artificial caries induction by pH-cycling was used in our study in order to obtain a standardized flat caries-affected dentin surfaces for bond strength evaluation. Although there are some morphologically differences between natural and artificial caries-affected dentin, e.g., tubules obliteration and microhardness (KHN) values,<sup>9</sup> the bond strength values seem not be influenced by the type of caries,<sup>16</sup> permitting the use of laboratorial models in attempt to overcome the great variability of natural caries-affected dentin.

Immediate microtensile tests, despite some intrinsic limitations and inter-laboratories differences, are helpful to rank adhesives and effort to compare new materials to already extensive studied ones, and therefore, choose the adhesives that should undergo clinical investigations. Our results suggest that despite a significant drop on bond strength to caries-affected dentin had been found, the Scotchbond Universal Adhesive can be used in both application modes, independently of dentin condition. Therefore, it might represent that these new category of bonding materials are less technique sensitive.

It has been verified that use of the self-etch approach for bonding the universal adhesive that contain MDP to sound dentin improves stability *in vitro*.<sup>20</sup> From now on, we expect that the etch-and-rinse strategy might be more critical over time when using carious substrate. Studies evaluating the long-term performance of the universal adhesives to caries-affected dentin are necessary to consolidate the recommendation of the new adhesives, since this substrate is more prone to deterioration over time and contain less calcium available to the chemical bonding.

## 5. Conclusion

The use of a universal adhesive system following both etch-and-rinse and self-etch strategies does not influence the bond strength to dentin. Nevertheless, the universal adhesive was not able to ensure the same bonding performance to caries-affected dentin, as a significant drop in bond strength values were observed compared to sound dentin.

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Table 1. Adhesive systems (batch number), composition and application modes\*

Adhesive System/ Batch Number	Composition**	Mode of application	
		Self-etch	Etch-and-rinse
Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA)  Batch number: (N508311)	Etchant: phosphoric acid 37% Bond: bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly(itaconic) acids, 10% by of 5 nm-diameter spherical silica particles	N.A.	1. Apply etchant for 15s 2. Rinse for 10s 3. Blot excess of water 4. Apply actively 2 consecutives layers of adhesive for 15s 5. Gently air dry for 5s 6. Light cure for 10s
Clearfil SE Bond (Kuraray Dental Inc., Okayama, Japan)  Batch number: Primer (012333A) Bond (01865A)	Primer: MDP, HEMA, hydrophobic dimethacrylate, camphorquinone, water, N,N- diethanol toluidine Bond: MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, camphorquinone, N,N- diethanol p-toluidine, colloidal silica	1. Apply actively primer to tooth surface and leave in place for 20s 2. Dry with air stream to evaporate the volatile solvents 3. Apply actively bond to tooth surface and then create a uniform film using a gentle air stream 4. Light cure for 10s	N.A.
Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA)  Batch number: (509806)	Etchant: phosphoric acid 37% Bond: MDP, HEMA, dimethacrylate resins, methacrylate-modified polyalkenoic acid copolymer ethanol, water, filler, initiator, silica	1. Apply actively the the adhesive to the entire surface for 20s. If necessary, rewet the disposable applicator 2. Direct a gentle air stream over the adhesive for 5s or until it no longer moves and the solvent completely evaporated 3. Light cure for 10s	1. Apply etchant for 15s 2. Rinse thoroughly 3. Blot excess of water 4. Apply adhesive as for the self-etch mode

\* According to manufacturers. \*\* Abbreviations: Bis-GMA: bisphenol A glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; N.A.: not applicable

Table 2. Microtensile bond strength (MPa) means (standard deviation) for all adhesives and substrates\*.

Substrate condition	Adhesive**			
	SBU (E&R)	SBU (SE)	ASB	CSE
Sound dentin	54.99(9.55) <sup>A</sup>	41.66(7.70) <sup>A</sup>	52.75(9.57) <sup>A</sup>	48.41(7.23) <sup>A</sup>
Caries-affected dentin	23.88(2.57) <sup>B</sup>	22.74(5.67) <sup>B</sup>	20.93(4.79) <sup>B</sup>	20.47(3.11) <sup>B</sup>

\*Different letters means differences statistically significant between groups (Tukey test, p < 0.05).

\*\* SBU application mode: E&R – etch-and-rinse; SE – self-etching.

Figure 1. (A) Specimen from sound dentin with regular surface. (B) Specimen from pH-cycling showing intertubular porous zones followed by an irregular surface and extensive opening of dentinal tubules as consequence of demineralization.

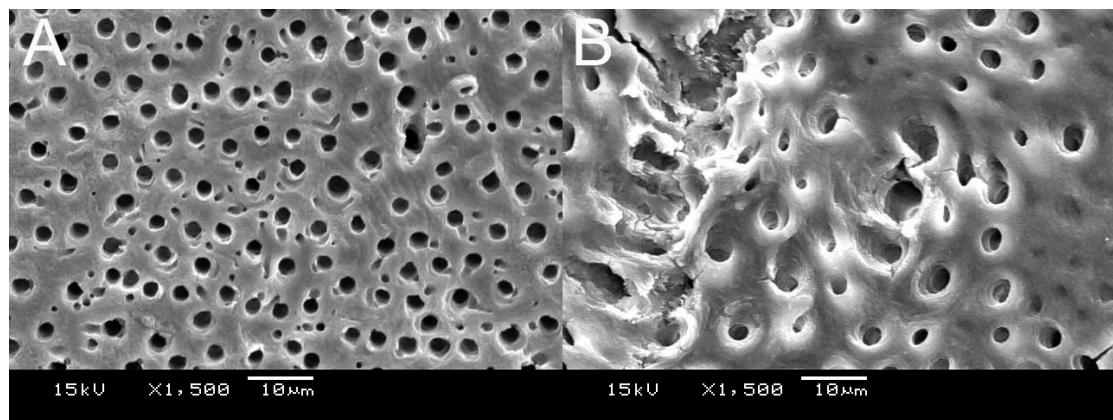


Figure 2. Fracture pattern distribution (%), considering the total number of specimens tested/pre-testing failures (right numbers in each line) for each experimental group in caries-affected dentin.

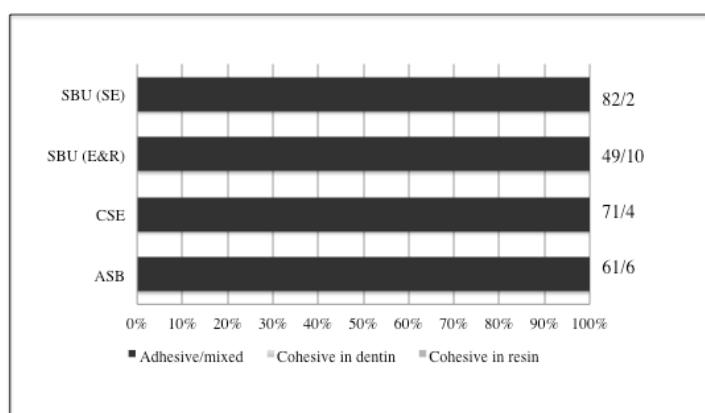
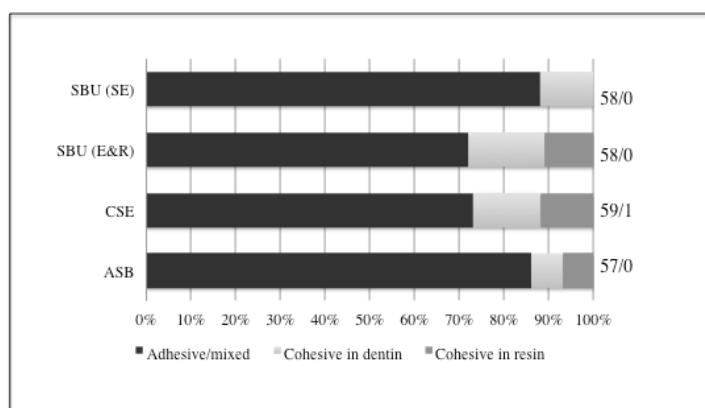


Figure 3. Fracture pattern distribution (%), considering the total number of specimens tested/pre-testing failures (right numbers in each line) for each experimental group in sound dentin.



## 2. DISCUSSÃO GERAL

Recentemente um novo conceito sobre sistemas adesivos foi proposto. Estes sistemas adesivos, intitulados “universais”, foram desenvolvidos em busca de procedimentos mais simples e menos sensíveis a técnica operatória. Juntamente com a evolução dos materiais adesivos, uma mudança de paradigma em procedimentos restauradores, odontologia minimamente invasiva, vem sendo aplicada com maior frequência entre clínicos. Nesta abordagem restauradora, diferentes substratos dentinários são observados durante o preparo cavitário. Desta maneira, os procedimentos adesivos são realizados sobre dentina hígida e dentina afetada sem diferenciação nas etapas adesivas. Portanto, frente ao recente lançamento de sistemas adesivos universais, que não dependem da estratégia de condicionamento, torna-se essencial avaliar laboratorialmente o seu comportamento entre os diferentes substratos dentinários.

Um decréscimo nos valores de resistência de união foi observado quando os sistemas adesivos foram aplicados sobre dentina desmineralizada em relação a dentina hígida. Nossos resultados vão ao encontro de estudos prévios que compararam a performance de sistemas adesivos à dentina afetada e hígida (ERHARDT et al., 2014; ZANCHI et al., 2010). Entretanto, diferentes resultados são observados na literatura, principalmente em relação ao sistema adesivo Clearfil SE Bond, e assim, alguns autores encontraram valores de resistência de união similares em ambos os substratos (OMAR et al., 2007). O processo carioso promove uma perda de mineral aumentando a porosidade da dentina intertubular, o que proporciona uma maior desmineralização frente ao condicionamento ácido, assim como a formação de uma camada híbrida mais espessa (PEREIRA et al., 2006; ERHARDT et al., 2008). Este aumento na espessura da camada híbrida não está relacionado a uma vantagem na adesão, mas com a formação de uma camada defeituosa pela inadequada infiltração dos monômeros (ERHARDT et al., 2008). Desta maneira, é esperado um decréscimo nos valores de resistência de união, mesmo em sistemas adesivos autocondicionantes.

Menores valores de resistência de união foram observados em dentes com dentina desmineralizada, o que foi acompanhado de um maior número de falhas pré-testes (ptf), em ambos substratos, dentina desmineralizada decídua e permanente. Assim como, somente falhas adesivas/mistas foram observadas nestes substratos. Isto pode estar relacionado a formação de uma camada híbrida defeituosa e irregular (ERHARDT et al., 2008). Por outro

lado, quando os sistemas adesivos foram empregados em substrato hígido, somente uma falha pré-teste (ptf) foi observada entre todos adesivos avaliados, assim como um grande número de falhas coesivas em dentina e resina foi constatado, o que provavelmente está relacionado a maiores valores de resistência de união pela formação de uma camada híbrida adequada.

Dentes decíduos e permanentes possuem diferentes concentrações de cálcio e densidade tubular, e portanto diferentes resultados no comportamento de sistemas adesivos são encontrados em alguns estudos (UEKASA et al., 2006; LENZI et al., 2010). Entretanto, não existe consenso na literatura se estes diferentes padrões micro-morfológicos são capazes de resultarem em menores valores de resistência de união. Desta maneira, alguns autores encontraram resultados similares quando compararam sistemas adesivos em dentes permanentes e decíduos (SOARES et al., 2005; RICCI et al., 2010), o que vai ao encontro dos nossos resultados.

Os resultados deste trabalho, confirmam que o sistema adesivo Single Bond Universal pode ser utilizado em ambas estratégias de aplicação e independentemente do substrato dentinário, sem comprometimento da eficácia adesiva. Nossos resultados vão ao encontro de outros estudos (WAGNER et al., 2014; CHEN et al., 2015) que avaliaram o comportamento de sistemas adesivos universais em dentina hígida e não encontraram diferenças entre os modos de aplicação. Os resultados deste trabalho ainda afirmam que o sistema adesivo Single Bond Universal não apresenta diferença nos valores de resistência de união quando comparado aos controles, Adper Single Bond 2 e Clearfil SE Bond, considerados padrões-ouro em ambas estratégias. Alguns autores (MUÑOZ et al., 2013) encontraram menores valores de resistência de união quando compararam o Single Bond Universal aos controles em dentes permanentes hígidos, enquanto outros autores (LENZI et al., 2015) encontram resultados similares, quando compararam este sistema adesivo em substrato dentinário desmineralizado.

O sistema adesivo Adper Single Bond 2 possui em sua formulação, o copolímero do ácido polialquenóico que promove uma ligação química ao cálcio disponível na zona de interdifusão. O sistema adesivo Clearfil SE Bond, possui o monômero funcional 10-metacriloiloxidecile dihidrogenofosfato (MDP) que além de promover uma ligação química a estrutura dental, promove a formação de nano-camadas responsáveis pela maior estabilidade da interface adesiva (YOSHIDA et al., 2012a). Na formulação do sistema adesivo Single Bond Universal, ambos componentes estão presentes, no qual o processo de adesão não foi completamente elucidado, pois alguns autores demonstram que ambos componentes podem

competir pelo mesmo sítio de ligação de cálcio, assim como o copolímero do ácido polialquenóico poderia prevenir a aproximação dos monômeros durante a polimerização devido ao seu alto peso molecular (MUÑOZ et al., 2013). Além disso, o Single Bond Universal possui monômeros HEMA, os quais são capazes de inibir uma adequada formação das nano-camadas quando presente juntamente ao monômero 10-MDP (YOSHIDA et al., 2012b). Entretanto, um recente estudo (MUÑOZ et al., 2015) avaliou a estabilidade deste adesivo e não encontrou nenhuma diferença nos valores de resistência de união após 6 meses de envelhecimento em água. Ademais, sistemas adesivos universais que apresentam somente o monômero 10-MDP em sua formulação quando aplicados em substratos hígidos, resultam em valores de resistência de união inferiores aos demais (WAGNER et al., 2014). Desta maneira, é provável que outros fatores produto-relacionados sejam importantes para uma adequada performance adesiva, além da adesão química (SCHOLTANUS et al., 2010).

Uma recente revisão sistemática avaliou a performance de sistemas adesivos universais *in vitro* aplicados em dentina e esmalte (ROSA et al., 2015). Os autores encontraram que, independentemente do modo de aplicação, a resistência de união destes sistemas adesivos não foi prejudicada quando aplicados a dentina, entretanto, valores inferiores foram encontrados no modo autocondicionante quando estes sistemas foram aplicados ao esmalte dental. Da mesma maneira, o sistema adesivo Single Bond Universal apresentou resultados similares em ambos modos de aplicação quando utilizado em restaurações sobre lesões cervicais não-cariosas durante um acompanhamento de 18 meses (PERDIGÃO et al., 2014). Conforme anteriormente citado, o substrato hígido e cariado apresentam diferenças micro-morfológicas, e deste modo é necessário a avaliação destes adesivos universais também em substratos cariados. Os resultados deste estudo comprovam a versatilidade destes sistemas adesivos universais. Portanto, estes sistemas adesivos apresentam uma excelente performance adesiva, possibilitando a sua aplicação em ambos modos e em diferentes substratos sem prejuízo da adesão, tornando os procedimentos adesivos menos sensíveis a técnica.

### **3. CONSIDERAÇÕES FINAIS**

Esta dissertação avaliou o comportamento do sistema adesivo Single Bond Universal em diferentes substratos dentinários. Este sistema adesivo, independente da estratégia de aplicação, apresentou valores de resistência de união similares aos controles, Adper Single Bond 2 e Clearfil SE Bond, quando comparado a dentina desmineralizada decídua e permanente. Nenhum sistema adesivo avaliado apresentou valores de resistência de união diferentes quando os substratos decíduos e permanentes desmineralizados foram comparados. Assim como, quando o sistema adesivo Single Bond Universal foi comparado entre dentes permanentes, porém à dentina hígida e desmineralizada, valores de resistência de união similares, independente da estratégia de aplicação, foram obtidos quando comparado aos seus controles. Entretanto, a dentina desmineralizada resultou em valores de resistência de união inferiores em todos os sistemas adesivos quando comparada a dentina hígida. Desta maneira, os sistemas adesivos recentemente disponíveis no mercado, intitulados universais, devido a sua versatilidade em procedimentos restauradores adesivos, é uma excelente alternativa aos “antigos” sistemas adesivos. Esta grande versatilidade dos sistemas adesivos universais proporciona procedimentos restauradores mais simples e menos sensíveis a técnica operatória, facilitando o emprego destes em diversas situações clínicas.

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## **ANEXO**

Anexo A - Carta de aprovação do Comitê de Ética em Pesquisa da UFSM



UNIVERSIDADE FEDERAL DE  
SANTA MARIA/ PRÓ-REITORIA  
DE PÓS-GRADUAÇÃO E



## **PARECER CONSUBSTANCIADO DO CEP**

## DADOS DO PROJETO DE PESQUISA

## **Título da Pesquisa:** Comparação da resistência de união de sistemas adesivos

**Pesquisador:** Rachel de Oliveira Rocha

## **Área Temática:**

Versão: 1

CNAE: 36765714 3.0000 5346

Instituição Proponente: Universidade Federal de Santa Maria/ Pró-Reitoria de Pós-Graduação e Pesquisa

#### **Retrocededor Principal: Financiamento Próximo**

## DADOS DO PARECER

Número do Parecer: 832.891

Data da Relatório: 14/10/2014

## **Apresentação do Projeto:**

Estudo laboratorial com o uso de dentes humanos fornecidos por Banco de Dentes

Com a constante busca pela realização de procedimentos conservadores e simplificados, diferentes materiais restauradores que permitem adequada adesão aos substratos dentários hígidos e cariados tem sido estudados. Apesar da crescente incorporação de novos sistemas adesivos ao mercado, existem poucos estudos que avaliam o comportamento destes materiais nos diferentes substratos de dentes deciduos e permanentes.

Desta maneira, o objetivo deste projeto é avaliar in vitro a resistência de união de 3 diferentes sistemas adesivos (Adper Single Bond 2, Clearfil SE Bond e Single Bond Universal) aos substratos esmalte e dentina de dentes deciduos e permanentes, nas condições hígidos ou alterados por lesões de cárie, por meio dos testes de microcislhamento e microtracção.

*Numa próxima submissão convém explicar como se delimita o n.*

#### **Objetivo da Pesquisa:**

Objetivo: avaliar a resistência de união do sistema adesivo Single Bond Universal ao esmalte e dentina, hígidos ou alterados por lesão de cárie, decidua e permanente quando comparado a

**Endereço:** Av. Roraima, 1000 - prédio da Reitoria - 2º andar

#### **Bairro: Gamboa**

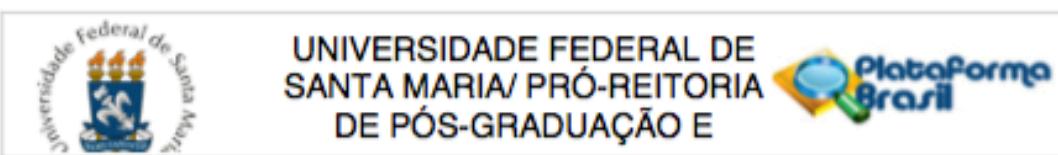
CEP: 97-105-970

UF: RS

Municipio: SANTA MARIA

Teleton

E-mail: [con-utem@gmail.com](mailto:con-utem@gmail.com)



Continuação do Parecer: 832.891

sistemas adesivos considerados como controle.

**Avaliação dos Riscos e Benefícios:**

Riscos: não estão previstos riscos aos doadores dos dentes salvo se houver quebra do sigilo das informações fornecidas previamente aos Bancos de Dentes fornecedores dos dentes para a pesquisa.

Benefícios: não estão previstos benefícios diretos aos doadores dos dentes mas sim aqueles decorrentes do conhecimento gerado pela pesquisa.

**Comentários e Considerações sobre a Pesquisa:**

-

**Considerações sobre os Termos de apresentação obrigatória:**

Presentes e adequados.

**Recomendações:**

Acesse ao novo site do CEP - <http://coral.ufsm.br/cep> - e, na aba "Orientações gerais", encontre modelos para apresentação de documentos.

**Conclusões ou Pendências e Lista de Inadequações:**

-

**Situação do Parecer:**

Aprovado

**Necessita Apreciação da CONEP:**

Não

Endereço:	Av. Roraima, 1000 - prédio da Reitoria - 2º andar	CEP:	97.105-970
Bairro:	Camobi	UF:	RS
Município:	SANTA MARIA		
Telefone:	(55)3220-9362	E-mail:	cep.ufsm@gmail.com

## **Anexo B - Normas para publicação no periódico Journal of Adhesive Dentistry**

### **GUIDELINES FOR AUTHORS**

The Journal of Adhesive Dentistry is a bi-monthly journal that publishes scientifically sound articles of interest to practitioners and researchers in the field of adhesion to hard and soft dental tissues. The Journal publishes several types of peer-reviewed original articles:

1. Clinical and basic science research reports – based on original research in adhesive dentistry and related topics.
2. Reviews topics – on topics related to adhesive dentistry.
3. Short communications – of original research in adhesive dentistry and related topics. Max. 4 printed pages, including figures and references (max. characters 18,000). High priority will be given to the review of these papers to speed publication.
- 4a. Invited focus articles – presenting a position or hypothesis on a basic science or clinical subject of relevant related topics. These articles are not intended for the presentation of original results, and the authors of the articles are selected by the Editorial Board.
- 4b. Invited commentaries – critiquing a focus article by addressing the strong and weak points of the focus article. These are selected by the Editorial Board in consultation with the focus article author, and the focus article and the commentaries on it are published in sequence in the same issue of the Journal.
5. Invited guest editorials – may periodically be solicited by the Editorial Board.
6. Proceedings of symposia, workshops, or conferences – covering topics of relevance to adhesive dentistry and related topics.
7. Letters to the Editor – may be submitted to the editor-in-chief; these should normally be no more than 500 words in length.

### **SUBMISSION INSTRUCTIONS**

Submission of manuscripts in order of preference:

1. Submission via online submission service ([www.manuscriptmanager.com/jadd](http://www.manuscriptmanager.com/jadd)). Manuscript texts should be uploaded as PC-word files with tables and figures preferably embedded within the PC-word document. A broad range of file formats are acceptable. No paper version required but high resolution photographs or illustrations should be sent to the editorial office (see below). Online submissions are automatically uploaded into the editorial office's reviewer assignment schedule and are therefore processed immediately upon upload.
2. Submission via e-mail as a PC-word document ([wintonowycz@quintessenz.de](mailto:wintonowycz@quintessenz.de)). Illustrations can be attached in any format that can be opened using Adobe Photoshop, (TIF, GIF, JPG, PSD, EPS etc.) or as Microsoft PowerPoint Documents (ppt). No paper version required but high resolution photographs or illustrations should be sent to the editorial office.
3. One paper copy of the manuscript plus a floppy diskette or CD-ROM (mandatory) containing a PC-word file of the manuscript text, tables and legends. Figures should be included on the disk if possible in any format that can be opened using Adobe Photoshop, (Tif, Gif, JPG, PSD, EPS etc.) or as a Microsoft PowerPoint Document (ppt).

Mailing address:

Quintessenz Verlags-GmbH, Karin Wintonowycz  
The Journal of Adhesive Dentistry, Ifenpfad 2-4, D-12107 Berlin, Germany.

Illustrations that cannot be sent electronically will be scanned at the editorial office so that they can be sent to reviewers via e-mail along with the manuscript to expedite the evaluation process. Resubmitted manuscripts should also be submitted in the above manner. Please note that supplying electronic versions of your tables and illustrations upon resubmission will assure a faster publication time if the manuscript is accepted. Review/editing of manuscripts. Manuscripts will be reviewed by the editor-in-chief and at least two reviewers with expertise within the scope of the article. The publisher reserves the right to edit accepted manuscripts to fit the space available and to ensure conciseness, clarity, and stylistic consistency, subject to the author's final approval. Adherence to guidelines. Manuscripts that are not prepared in accordance with these guidelines will be returned to the author before review.

## MANUSCRIPT PREPARATION

- The Journal will follow as much as possible the recommendations of the International Committee of Medical Journal Editors (Vancouver Group) in regard to preparation of manuscripts and authorship (Uniform requirements for manuscripts submitted to biomedical journals. Ann Intern Med 1997;126: 36-47).
- Title page. The first page should include the title of the article (descriptive but as concise as possible) and the name, degrees, job title, professional affiliation, contribution to the paper (e.g., idea, hypothesis, experimental design, performed the experiments in partial fulfillment of requirements for a degree, wrote the manuscript, proofread the manuscript, performed a certain test, consulted on and performed statistical evaluation, contributed substantially to discussion, etc.) and full address of all authors. Phone, fax, and e-mail address must also be provided for the corresponding author, who will be assumed to be the first listed author unless otherwise noted. If the paper was presented before an organized group, the name of the organization, location, and date should be included.
- 3-8 keywords.
- Structured abstract. Include a maximum 250-word structured abstract (with headings Purpose, Materials and Methods, Results, Conclusion).
- Introduction. Summarize the rationale and purpose of the study, giving only pertinent references. Clearly state the working hypothesis.
- Materials and Methods. Present materials and methods in sufficient detail to allow confirmation of the observations. Published methods should be referenced and discussed only briefly, unless modifications have been made. Indicate the statistical methods used, if applicable.
- Results. Present results in a logical sequence in the text, tables, and illustrations. Do not repeat in the text all the data in the tables or illustrations; emphasize only important observations.

- Discussion. Emphasize the new and important aspects of the study and the conclusions that follow from them. Do not repeat in detail data or other material given in the Introduction or Results section. Relate observations to other relevant studies and point out the implications of the findings and their limitations.
- Acknowledgments. Acknowledge persons who have made substantive contributions to the study. Specify grant or other financial support, citing the name of the supporting organization and grant number.
- Abbreviations. The full term for which an abbreviation stands should precede its first use in the text unless it is a standard unit of measurement.
- Trade names. Generic terms are to be used whenever possible, but trade names and manufacturer should be included parenthetically at first mention.
- Clinical Relevance. Please include a very brief (2 sentences or 3 lines) clinical relevance statement.

## REFERENCES

- All references must be cited in the text, according to the alphabetical and numerical reference list.
- The reference list should appear at the end of the article, in alphabetical and numerical sequence.
- Do not include unpublished data or personal communications in the reference list. Cite such references parenthetically in the text and include a date.
- Avoid using abstracts as references.
- Provide complete information for each reference, including names of all authors. If the reference is part of a book, also include title of the chapter and names of the book's editor(s).

Journal reference style:

1. Turp JC, Kowalski CJ, Stohler CS. Treatment-seeking patterns of facial pain patients: Many possibilities, limited satisfaction. *J Orofacial Pain* 1998;12:61-66.

Book reference style:

1. Hannam AG, Langenbach GEJ, Peck CC. Computer simulations of jaw biomechanics. In: McNeill C (ed). *Science and Practice of Occlusion*. Chicago: Quintessence, 1997:187-194.

## ILLUSTRATIONS

- All illustrations must be numbered and cited in the text in order of appearance.
- Submitted figures should meet the following minimum requirements:
  - High-resolution images should have a width of 83 mm and 300 dpi (for column size).

- Graphics (bar diagrams, schematic representations, drawings) wherever possible should be produced in Adobe Illustrator and saved as AI or EPS files.
- All figures and graphics should be separate files – not embedded in Word or Power Point documents.

Upon article acceptance, high-resolution digital image files must be sent via one of the following ways:

1. As an e-mail attachment, if the files are not excessively large (not more than 10 MB), to our production department: Steinbrueck@quintessenz.de
2. Online File Exchange Tool: Please send your figures with our Online File Exchange Tool. This web tool allows you to upload large files (< 350.0 MB) to our server. Please archive your figures with a maximum size of 350 MB first. Then upload these archives with the following link: <http://files.qvnet.de/JAD/>, password: IAAD.

Please name the archive with your name and article number so we can identify the figures. Line drawings—Figures, charts, and graphs should be professionally drawn and lettered large enough to be read after reduction. Good-quality computer-generated laser prints are acceptable (no photocopies); also provide electronic files (eps, ai) if possible. Lines within graphs should be of a single weight unless special emphasis is needed.

#### **Legends**

–Figure legends should be grouped on a separate sheet and typed double-spaced.

#### **TABLES**

- Each table should be logically organized, on a separate sheet, and numbered consecutively.
- The title and footnotes should be typed on the same sheet as the table.

#### **MANDATORY SUBMISSION FORM**

The Mandatory Submission Form, signed by all authors, must accompany all submitted manuscripts before they can be reviewed for publication. Electronic submission: scan the signed form and submit as JPG or TIF file.

#### **PERMISSIONS & WAIVERS**

- Permission of author and publisher must be obtained for the direct use of material (text, photos, drawings) under copyright that does not belong to the author.
- Waivers must be obtained for photographs showing persons. When such waivers are not supplied, faces will be masked to prevent identification. For clinical studies the approval of the ethics committee must be presented.

#### **PAGE CHARGE**

The first 8 printed pages in an article are free of charge. For excess pages, the charge is €140 per printed page. The approximate number of characters on a printed page is approximately 6,800. Please also consider the number and size of illustrations.

## **Anexo C - Normas para publicação no periódico Journal of Dentistry**

### **GUIDE FOR AUTHORS**

The Journal of Dentistry is the leading international dental journal within the field of Restorative Dentistry. Placing an emphasis on publishing novel and high-quality research papers, the Journal aims to influence the practice of dentistry at clinician, research, industry and policy-maker level on an international basis. Topics covered include the management of dental disease, periodontology, endodontontology, operative dentistry, fixed and removable prosthodontics, and dental biomaterials science, long-term clinical trials including epidemiology and oral health, dental education, technology transfer of new scientific instrumentation or procedures, as well clinically relevant oral biology and translational research. Submissions are welcomed from other clinically relevant areas, however, the Journal places an emphasis on publishing high-quality and novel research. Queries in relation to manuscript content should be directed to the Journal Editorial Office in the first instance.

#### **Submissions**

Authors are requested to submit their original manuscript and figures via the online submission and editorial system for Journal of Dentistry. Using this online system, authors may submit manuscripts and track their progress through the system to publication. Reviewers can download manuscripts and submit their opinions to the editor. Editors can manage the whole submission/review/review/publish process. Please register at: <http://ees.elsevier.com/jjod>

#### **Types of paper**

Contributions falling into the following categories will be considered for publication:

- Original Research Reports: maximum length 6 printed pages approximately 20 typescript pages, including illustrations and tables.
- Review articles: maximum length 10 printed pages, approximately 33 typescript pages, including illustrations and tables.
- Short communication for rapid publication: maximum length 2 printed pages, approximately 7 typescript pages, including illustrations.
- Letters providing informed comment and constructive criticism of material previously published in the Journal.

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