

UNIVERSIDADE FEDERAL DE SANTA MARIA  
CENTRO DE CIÊNCIAS DA SAÚDE  
PROGRAMA DE PÓS-GRADUAÇÃO EM CIÊNCIAS  
ODONTOLÓGICAS

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**EFEITOS DO PRÉ-TRATAMENTO DENTINÁRIO E  
TEMPERATURA DO ADESIVO NA RESISTÊNCIA ADESIVA DE UM  
SISTEMA ADESIVO UNIVERSAL**

Santa Maria, RS  
2016

**Bruna Gabrielle da Silva Sutil**

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NA RESISTÊNCIA ADESIVA DE UM SISTEMA ADESIVO UNIVERSAL**

Dissertação apresentada ao Curso de Mestrado do Programa de Pós-Graduação em Ciências Odontológicas, Área de Concentração em Odontologia, ênfase em Dentística, da Universidade Federal de Santa Maria (UFSM, RS), como requisito parcial para obtenção do grau de **Mestre em Ciências Odontológicas**.

Orientador: Prof<sup>o</sup>. Dr. Alexandre Henrique Susin

Santa Maria, RS

2016

Ficha catalográfica elaborada através do Programa de Geração Automática da Biblioteca Central da UFSM, com os dados fornecidos pelo(a) autor(a).

Sutil, Bruna Gabrielle da Silva  
EFEITOS DO PRÉ-TRATAMENTO DENTINÁRIO E TEMPERATURA DO  
ADESIVO NA RESISTÊNCIA ADESIVA DE UM SISTEMA ADESIVO  
UNIVERSAL / Bruna Gabrielle da Silva Sutil.- 2016.  
48 p.; 30 cm

Orientador: Alexandre Henrique Susin  
Dissertação (mestrado) - Universidade Federal de Santa  
Maria, Centro de Ciências da Saúde, Programa de Pós-  
Graduação em Ciências Odontológicas, RS, 2016

1. Sistemas Adesivos I. Susin, Alexandre Henrique  
II. Título.

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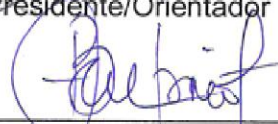
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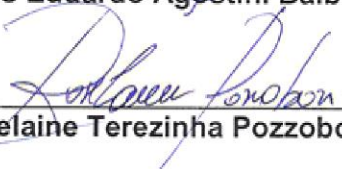
Aprovada em 28 de julho de 2016:



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Santa Maria, RS

2016

## DEDICATÓRIA

*Dedico este trabalho a meu esposo Silvano que apesar das intempéries da vida, sempre esteve ao meu lado, incentivando-me em todos os momentos. Sem o seu apoio, não seria possível alcançar esse objetivo. A você, minha eterna gratidão.*

## AGRADECIMENTOS

*Agradeço a Deus por permitir que eu chegasse até aqui, mantendo-me em condições necessárias para alcançar esse objetivo. Sem Ele, essa jornada não poderia ser cumprida.*

*Aos meus pais, Beatriz Pires da Silva e Alair Quadros Sutil, por me ensinarem que o estudo é o melhor caminho a seguir e por terem formado a pessoa que hoje eu sou. Obrigada por tudo.*

*Aos meus sogros Mari Maia e Gilmar Amarante, por terem me acolhido nos momentos que mais precisei e também pelo presente que me foi concedido: meu marido.*

*Ao meu marido Silvano Maia de Amarante, pelo apoio, carinho, paciência, dedicação e amor incondicional; se não fosse por você, esse objetivo não seria alcançado. Entretanto, por mais que tente escrever as mais belas e sinceras palavras, elas não seriam suficientes para expressar meu amor, admiração e gratidão.*

*Ao meu orientador, prof.<sup>o</sup> Alexandre Henrique Susin pela oportunidade de dividir comigo seus conhecimentos e pelo exemplo de profissional e pessoa humana. Obrigada pela paciência, incentivo e confiança em mim depositada.*

*À minha amiga Luana Brondani Costa, que desde a graduação até o início do mestrado se fez presente, me ajudando e compartilhando dos mesmos ideais.*

*Ao meu colega da disciplina de Dentística, Marcos Paulo Marchiori Carvalho, por toda a ajuda que me foi dada, e pelas dúvidas solucionadas a respeito da pesquisa e do funcionamento dos equipamentos.*

*Aos meus colegas de pós-graduação pelo convívio e ajuda nos momentos que precisei e, especialmente à Ângela Dalla Nora, Camila Rodrigues, João Luiz Pozzobon e Taiane Missau, que tive o prazer de conviver durante esses dois anos de mestrado. Obrigada pelos momentos de descontração, bem como pelas dúvidas e temores compartilhados.*

*A todos os meus amigos e familiares que, de alguma forma estiveram presentes comigo nessa trajetória, acompanhando-me.*

*À Universidade Federal de Santa Maria e ao curso de Odontologia por todo o suporte e estrutura fornecida, possibilitando minha formação.*

*Aos professores do Programa de Pós-Graduação em Ciências Odontológicas por todo o conhecimento compartilhado.*

*Ao Banco de Dentes Humanos da Universidade Federal de Santa Maria pela doação dos dentes utilizados nesta pesquisa.*

*À Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) pela concessão da minha bolsa, a qual viabilizou a execução deste trabalho.*

*A todos, os meus sinceros agradecimentos!*

*A verdadeira viagem de descobrimento não consiste  
em procurar novas paisagens, mas em ter novos olhos.*  
(Marcel Proust)



## RESUMO

### EFEITOS DO PRÉ-TRATAMENTO DENTINÁRIO E TEMPERATURA DO ADESIVO NA RESISTÊNCIA ADESIVA DE UM SISTEMA ADESIVO UNIVERSAL

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ORIENTADOR: Alexandre Henrique Susin

O presente estudo avaliou os efeitos do jateamento de partículas abrasivas e da temperatura sobre a resistência adesiva de um sistema adesivo universal à dentina. Utilizou-se 96 terceiros molares humanos hígidos e extraídos, divididos aleatoriamente em 12 grupos (n=8) de acordo com o adesivo Scotchbond Universal (SBU) aplicado no modo autocondicionante (SE) ou condicionamento ácido total (TE), temperatura do adesivo (20°C ou 37°C) e jateamento da dentina com partículas de óxido de alumínio ou bicarbonato de sódio. Após a realização das restaurações, foram obtidos espécimes em forma de palito com área seccional de 1 mm<sup>2</sup> para avaliar a resistência adesiva através do teste de microtração. O teste foi realizado em uma máquina de ensaio universal, a uma velocidade de 0,5 mm/min até a fratura ocorrer. As fraturas foram analisadas sob estereomicroscopia para classificar as falhas em adesiva, coesiva (resina ou dentina) e mista. Os dados foram submetidos ao teste ANOVA a dois fatores e ao teste de Tukey. A interação entre tratamento e temperatura foi estatisticamente significativa (p<0,01) para SBU SE. Ambos os tratamentos da dentina resultaram em valores maiores de resistência adesiva para SBU no modo TE, independente da temperatura do adesivo. Já para SBU aplicado na técnica autocondicionante, apenas o jateamento com bicarbonato de sódio foi significativamente diferente quando comparado ao grupo que não recebeu tratamento. O adesivo aquecido não teve influência significativa sobre a resistência adesiva à microtração, para todos os grupos testados. As fraturas adesivas foram predominantes para todos os grupos. Dessa forma, o pré-tratamento dentinário pode ser uma alternativa para melhorar as propriedades adesivas do sistema adesivo Scotchbond Universal.

**Palavras-chave:** Dentina. Jateamento. Resistência à microtração. Sistemas adesivos. Temperatura.

## ABSTRACT

### EFFECTS OF DENTIN PRETREATMENT AND ADHESIVE TEMPERATURE ON BOND STRENGTH OF A UNIVERSAL ADHESIVE SYSTEM

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ADVISOR: ALEXANDRE HENRIQUE SUSIN

This study aimed to evaluate the effects of dentin pretreatment and temperature on the bond strength of a universal adhesive system to dentin. Ninety-six extracted non-carious human third molars were randomly divided into 12 groups (n=8) according to Scotchbond Universal Adhesive (SbU) applied in self-etch (SE) and total-etch (TE) mode, adhesive temperature (20°C or 37°C) and sodium bicarbonate or aluminum oxide air abrasion. After composite build up, bonded sticks with cross-sectional area of 1 mm<sup>2</sup> were obtained to evaluate the microtensile bond strength ( $\mu$ TBS). The specimens were tested at a crosshead speed of 0.5 mm/min on a universal testing machine until failure. Fractured specimens were analyzed under stereomicroscope to determine the failure patterns in adhesive, cohesive (dentin or resin) and mixed fractures. The microtensile bond strength data was analyzed using two-way ANOVA and Tukey's test ( $p < 0.01$ ). Interaction between treatment and temperature was statistically significant to SbU applied in SE technique. Both dentin treatments showed higher bond strength to TE mode, regardless of adhesive temperature. When compared to control group, sodium bicarbonate increased bond strength of SbU in SE mode. Adhesive temperature did not significantly affect the  $\mu$ TBS of tested groups. Predominantly, adhesive failure was observed for all groups. Thus, the dentin pretreatment may be an alternative to improve the adhesive properties of the Scotchbond Universal adhesive system.

**Keywords:** Adhesive systems. Air abrasion. Dentin. Microtensile bond strength. Temperature.

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## 1 INTRODUÇÃO

As restaurações estéticas e minimamente invasivas tem se tornado amplamente populares entre os pacientes que procuram pelo tratamento odontológico. Dessa forma, a Odontologia adesiva cada vez mais tem desenvolvido materiais e técnicas que permitem a confecção, bem como a longevidade dessas restaurações.

Com a finalidade de reduzir os passos de aplicação clínicos e também diminuir a sensibilidade da técnica (CHEN et al., 2015; FREEMAN et al., 2012), foram desenvolvidos os sistemas adesivos universais ou multi-mode que, devido a sua versatilidade, podem ser aplicados tanto na técnica etch-and-rinse, quanto no modo self-etch. (CHEN et al., 2015; HANABUSA et al., 2012; MARCHESI et al., 2014; ROSA; PIVA; SILVA, 2015; WAGNER et al., 2014). O modo de aplicação etch-and-rinse preconiza realizar o condicionamento ácido da dentina previamente à aplicação do sistema adesivo, a fim de remover a smear layer da superfície dentinária e permitir a penetração do adesivo no substrato; já a técnica self-etch consiste apenas na aplicação do adesivo, dispensando o passo de ataque ácido; nessa técnica, a smear layer não é removida mas incorporada na camada adesiva. (LUQUE-MARTINEZ et al., 2014; SUYAMA et al., 2013; TAKAMIZAWA et al., 2015; ZHANG et al., 2016).

Embora novos sistemas adesivos sejam desenvolvidos para melhorar seu desempenho clínico, pode-se utilizar diferentes técnicas de tratamento da dentina e modificações do adesivo para aumentar a interação entre eles e, conseqüentemente, o sucesso clínico a longo prazo. Sabe-se que uma adesão efetiva depende da quantidade de substrato disponível, visto que o embricamento mecânico ocorre por meio do contato interfacial entre adesivo e substrato. Assim uma abordagem alternativa para aumentar a área disponível para adesão são os procedimentos de pré-tratamento da dentina, através de técnicas de jateamento, as quais promovem uma alteração mecânica do substrato para receber os procedimentos adesivos (FREEMAN et al., 2012).

O jateamento com partículas de óxido de alumínio já foi indicado para aumentar a adesão de superfícies metálicas aos materiais resinosos, para confecção e limpeza do preparo cavitário, remoção de tecido cariado e restaurações defeituosas (FRANÇA; SANTOS; LOVADINO, 2007; MANHART et al., 1999;

SENGUN et al.; 2008). Já o jateamento com bicarbonato de sódio tem sido indicado para profilaxia da superfície dentinária, removendo placa e detritos provenientes do preparo cavitário, como a smear layer, e tem demonstrado ser superior a outros métodos de limpeza como as taças de borracha, principalmente em áreas de difícil acesso, como preparos cavitário mais profundos (ARMAS-VEGA et al., 2007; ROSIN et al., 2005). Entretanto, o jateamento também tem sido indicado como um pré-tratamento da dentina, pois aumenta a rugosidade superficial e a área disponível para a adesão (BARABA et al., 2015; COLI et al., 1999; FRANÇA; SANTOS; LOVADINO, 2007; FREEMAN et al., 2012). A remoção da smear layer que pode ser obtida pela jateamento com partículas abrasivas, pode melhorar a infiltração do adesivo na dentina, aumentando a resistência adesiva (CHAVES; GIANNINI; AMBROSANO, 2002).

Além da quantidade de substrato, a efetividade adesiva também depende das propriedades mecânicas do adesivo polimerizado. Em virtude disso, idealmente os solventes presentes no adesivo deveriam ser removidos completamente antes da polimerização, visto que solventes residuais podem inibir a polimerização e afetar as propriedades adesivas (LUQUE-MARTINEZ et al., 2014; OGURA et al., 2012; SHIRATSUCHI et al., 2013). Assim, para facilitar a evaporação do solvente e aumentar a velocidade de penetração do adesivo, pode-se optar pela utilização de adesivo pré-aquecidos (LOGUERCIO et al., 2011; SHARAFEDDIN; NOURI; KOOHPEIMA, 2015; VALE et al., 2014). Por meio do estímulo de suas reações químicas, algumas propriedades das soluções monoméricas podem ser alteradas pelo aumento da temperatura, dentre elas, o grau de conversão e a viscosidade, influenciando, dessa forma, a qualidade e estabilidade da camada adesiva. Assim, a temperatura pode aumentar a mobilidade dos radicais livres pelo aumento do grau de conversão, beneficiando a polimerização; bem como diminuir a viscosidade e melhorar a velocidade de espalhamento do adesivo (REIS et al., 2009; ALEXANDRE et al., 2008; SADR et al., 2007; VALE et al., 2014).

Assim, esse estudo se justifica por interagir os pré-tratamentos dentinários com a temperatura do adesivo, na busca de uma técnica adesiva que resulte em melhor desempenho na resistência de união de um sistema adesivo universal à dentina.

## **2 ARTIGO – DENTIN PRETREATMENT AND ADHESIVE TEMPERATURE: EFFECTS ON BOND STRENGTH OF A UNIVERSAL ADHESIVE SYSTEM**

Este artigo será submetido à publicação no periódico *Journal of Dentistry*, ISSN: 0300-5712, Fator de Impacto: 2.749, Qualis: A1. As normas para publicação estão descritas no Anexo A.



## Dentin pretreatment and adhesive temperature: Effects on bond strength of a universal adhesive system

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**Keywords:** Adhesive systems

Dentin

Temperature

Air abrasion

Microtensile bond strength

## Dentin pretreatment and adhesive temperature: Effects on bond strength of a universal adhesive system

### ABSTRACT

**Objectives:** To evaluate the effects of dentin pretreatment and temperature on the bond strength of a universal adhesive system to dentin. **Methods:** Ninety-six extracted non-carious human third molars were randomly divided into 12 groups (n=8) according to Scotchbond Universal Adhesive (SbU) applied in self-etch (SE) and etch-and-rinse (ER) mode, adhesive temperature (20°C or 37°C) and sodium bicarbonate or aluminum oxide air abrasion. After composite build up, bonded sticks with cross-sectional area of 1 mm<sup>2</sup> were obtained to evaluate the microtensile bond strength ( $\mu$ TBS). The specimens were tested at a crosshead speed of 0.5 mm/min on a testing machine until failure. Fractured specimens were analyzed under stereomicroscope to determine the failure patterns in adhesive, cohesive (dentin or resin) and mixed fractures. The microtensile bond strength data was analyzed using two-way ANOVA and Tukey's test ( $p < 0.05$ ). **Results:** Interaction between treatment and temperature was statistically significant to SbU applied in self-etch technique. Both dentin treatments showed higher bond strength to ER mode, regardless of adhesive temperature. When compared to control group, sodium bicarbonate increased bond strength of SbU in SE technique. Adhesive temperature did not significantly affect the  $\mu$ TBS of tested groups. Predominantly, adhesive failure was observed for all groups. **Conclusions:** Dentin surface treatment with sodium bicarbonate air abrasion improves bond strength of SbU, irrespective of adhesive application mode.

**Clinical significance:** Improved bond strength was observed when dentin air abraded with sodium bicarbonate, regardless of adhesive technique and temperature, which makes this approach an alternative to increase adhesive performance of Scotchbond Universal Adhesive

### INTRODUCTION

With the development and improvement of aesthetic restorative materials, adhesive systems have become essential elements in various clinical applications. Adhesive systems are responsible for the bonding of restorative material to dental structures. Thus, the longevity of adhesive restoration is directly associated with the effectiveness of adhesive systems.<sup>1</sup>

The adhesion of composite resins to the dentin substrate is based on the effect of conditioners and interaction with substrate. While some adhesive systems require the conditioning of dentin with phosphoric acid, as etch-and-rinse systems (ER), others preserve the smear layer by incorporating it into the adhesive layer, they

are the self-etch systems (SE). Self-etch systems use an acidic primer, which can be separated from the adhesive or can be all-in-one, in which the primer and adhesive are present in a single bottle.<sup>2</sup> New universal or multi-mode adhesives are single-step self-etch adhesives that can be applied both to the ER and SE techniques.<sup>3,4</sup>

The technique etch-and-rinse is more sensitive, as it is susceptible to operator error and can be affected by the condition of the substrate and by the local environment. Thus, self-etching primer systems have been developed to minimize the sensitivity of the technique and reduce the time of clinical application.<sup>5,6</sup> However, in study that evaluated the effectiveness of the self-etching primer, there was a major failure of restoration using this type of adhesive when compared to adhesives with a three-step prior acid conditioning.<sup>7</sup> This failure is associated with the inability of the adhesive to penetrate correctly into the smear layer and reach the underlying dentin.<sup>8,9</sup>

Dentin adhesion depends not only on the successful penetration of the adhesive into the dental substrate, but also on the mechanical properties of the polymerized adhesive. Thus, the solvents and the water present in the adhesive should ideally be removed to a sufficient degree because residual solvents can inhibit the polymerization and negatively affect the mechanical properties of the adhesive.<sup>10,11</sup> However, it is widely known that complete evaporation of the solvent should not occur, as it is also essential for monomers to penetrate deep regions of the conditioned dentin. Hence, a clinical approach to improve the adhesive properties indicates the use of warm air<sup>10-17</sup> to facilitate the evaporation of the solvent as well as the use of pre-heated adhesive systems<sup>18-21</sup> in order to increase the speed of penetration of the adhesive and the evaporation of the monomeric content. The quality and the stability of the adhesive layer can be influenced by the heated adhesive through the stimulation of their chemical reactions.<sup>22</sup> Hence, some properties of the monomeric solutions, such as viscosity and degree of monomer conversion, can be changed by temperature, which influences effectiveness and adhesive stability.<sup>19-21,23</sup> Temperature can increase the mobility of free radicals, thus benefiting the polymerization of the adhesive by increasing the degree of conversion.<sup>23</sup>

Although new adhesive systems should be developed in order to improve performance, we can use different dentin clinical manipulation techniques to increase the bonding and, consequently, long-term clinical success. An alternative approach is

pretreatment of the dentin through air abrasion techniques, which promote a mechanical modification of the substrate to receive the adhesive procedures.<sup>6,24</sup>

Abrasion with aluminum oxide particles has been used to increase the bonding of metallic surfaces to resinous materials, to prepare and clean the cavity, and to remove decayed tissue and faulty restorations.<sup>1,25-27</sup> Meanwhile, abrasion with sodium bicarbonate has been used for the prophylaxis of the dentinal surface and removal of plaque and debris during cavitory preparation. Sodium bicarbonate abrasion is superior to other cleaning methods, such as the rubber cups, mainly in areas that are difficult to access, such as during deeper cavitory preparations.<sup>28,29</sup> However, air abrasion has also been used in dentin pretreatment, because it increases the surface roughness and the area available for adhesion and thus improves the interfacial contact between the dentin and the adhesive.<sup>1,6,30-33</sup> The removal of the smear layer, by abrasion with aluminum oxide particles, can improve adhesive infiltration into the dentin, increasing the bond strength.<sup>2</sup> Other studies also demonstrated that the abrasion associated with dentin conditioning yields similar results to conditioned-only samples.<sup>27,30,32,33</sup>

Thus, the aim of this *in vitro* study was to evaluate the effect of pretreatment of the dentin surface on the bond strength of a universal adhesive system to dentin and the impact of temperature on adhesion. The tested hypotheses were: 1) The surface pretreatments increases the bond strength in both adhesive techniques; 2) Pre-heating of the adhesive system improves the adhesion.

## **MATERIALS AND METHODS**

### **Selection and preparation of teeth**

This study was previously submitted to the Institutional Ethics Committee in Research. Ninety-six intact human third molars (n=8) were used. Sample size was calculated according expected means, 80% statistical power and 5% significance level, using the OriginPro 2015 software (OriginLab Co, Northampton, USA). They were stored in a chloramine T 0.5% solution at a temperature of 4 °C for one month. The teeth were then removed from the disinfectant solution, washed abundantly, stored in distilled water of the same temperature, and used within six months. The

root portion of the teeth was removed and the occlusal third sectioned with a diamond disk at low speed and cooled with water, using a cutting machine (Labcut® 1010, Extec; Enfield, EUA). The sectioned root portions were then embedded in PVC tubes (Tigre S.A., Joinville, Brazil) with sticky wax (New wax, Technew, São Paulo, Brazil). The enamel surfaces were abraded with #180 grit SiC paper under running water until exposure of a flat dentin surface. To standardize the smear layer, dentinal surfaces were polished under running water with #600 grit SiC paper for 60 seconds using a polishing machine (Arotec PL 4; São Paulo, Brazil). The teeth were randomly divided into 12 experimental groups by *www.random.org* site (Randomness and Integrity Services Ltd, Dublin, Ireland) according to the adhesive system method used (self-etch or etch-and-rinse techniques), surface treatments (sodium bicarbonate or aluminum oxide air abrasion) and temperature of the adhesive (20 °C or 37 °C) (Table 1 and Figure 1).

#### Abrasive and restoration procedures

In the groups that received surface treatments, the dentin was air abraded with sodium bicarbonate for 15 seconds, at a distance of 5 mm, a pressure of 60 psi, and with an angle of 90° between the jet and dentin (Jet Laxis Sonic BP, Schuster, São Paulo, Brazil). The dentinal surface received an air/water spray jet for 30 seconds and was dried with absorbent paper. In another treatment, the dentinal surface was abraded with aluminum oxide particles (50 µm) with an angle of 90° between the jet and dentin, for 10 seconds, at a distance of 5 mm and a pressure of 60 psi (Micro-jato, Bio Art; São Carlos, Brazil). The dentinal surface was then washed with an air/water spray for 15 seconds and dried with absorbent paper.

The Scotchbond Universal (SbU) adhesive (3M ESPE, St Paul, USA) was used in etch-and-rinse (ER) and self-etch (SE) techniques at a temperature of 20 °C or heated in the specific device to 37 °C. Application mode was performed according to manufacturer's instructions (Table 2). After the adhesive procedures, a composite resin restoration was built on the dentinal surface at increments of 2 mm thick, using the nanofilled dental resin Z350 (3M ESPE; St Paul, USA) in the shade A2. The resin layers were photocured individually for 20 seconds using RADII (SDI, São Paulo, Brazil) with an intensity of 800 mW/cm<sup>2</sup>. Thereafter, the specimens were stored in

distilled water for 24 hours in order to be prepared for the microtensile bond strength test.

#### Microtensile bond strength test ( $\mu$ TBS)

A diamond disk was used at low speed and under refrigeration in order to prepare the specimens for the microtensile test. These specimens were mounted on a cutting machine (Labcut  $\text{\textcircled{R}}$  1010, Extec; Enfield, USA). The teeth were sectioned into slices parallel to the long axis and then cut again perpendicular to the first sections. This was done in order to obtain specimens in the form of sticks with an adhesive area of 1 mm<sup>2</sup>, measured through a digital pachymeter (Kingtools, São Paulo, Brazil). The specimens were examined through a stereomicroscope (Discovery V20, Carl-Zeiss, Oberkochen, Germany), with an amplification of 10 x. Thus, sticks with bubble inclusions and adhesive failures were excluded. Each specimen was attached to a  $\mu$ TBS testing device with cyanoacrylate-based adhesive gel (Super Bonder, Loctite Ltda, São Paulo, Brazil) and subjected to a tensile force in a universal test machine (EMIC DL-2000, São José dos Pinhais, Brazil), with a 50 KgF load cell at a speed of 0.5 mm/min.

#### Analysis of failure mode

To determine if the failure that occurred was adhesive (fracture at the interface between the resin and dentin), cohesive (fracture within the body of the resin or dentin), or mixed (adhesive fracture combined with cohesive fracture), the specimens were analyzed by stereomicroscopy (Discovery V20, Carl-Zeiss, Oberkochen, Germany) with 40 x amplification.

#### Statistical analysis

The Shapiro-Wilk test was employed to verify a normal distribution. Bond strength data were analysed separately using two-way ANOVA (air abrasion vs. temperature) for etch-and-rinse and self-etch strategies. The Tukey test was used for

multiple comparisons, with a significance level of 5%. All statistical analyses were performed in the OriginPro 2015 software (OriginLab Co, Northampton, USA).

## RESULTS

Means and standard deviations of the tested adhesive techniques are presented according to surface treatment and temperature in Table 3. A two-way ANOVA test showed that factor temperature ( $p= 0.689$ ) was not statistically significant for SbU in SE mode, while the dentinal treatment ( $p< 0.001$ ) and the interaction of factors ( $p= 0.012$ ) had a significant effect on the SE technique. For SbU in mode ER, temperature ( $p=0.002$ ) and treatment ( $p< 0.001$ ) were significant.

Both surface pretreatments showed higher values of microtensile bond strength when the SbU adhesive was used in the ER technique, regardless of the temperature of the adhesive. For the application in SE mode only, the sodium bicarbonate air abrasion increased the bond strength when compared to control group.

Analysis of the fracture pattern presented predominantly adhesive failures for all groups (Figure 2). However, the four types of failures were observed in all groups (Figures 2, 3, 4 and 5). Descriptive results are presented in Table 4.

## DISCUSSION

Universal dental adhesives were developed mainly with regard to dentin; with the aim of simplifying the clinical steps and reducing the sensitivity of clinical techniques. Due to their versatility, they may be used with the etch-and-rinse and self-etch techniques.<sup>34,35</sup> As they are a class of recent and little studied adhesives, alternative approaches, such as dentinal pre-treatment<sup>2,6</sup> and the pre-heating of the adhesive<sup>20,21</sup> can be carried out to improve adhesive properties.

Cleaning of the dentinal surface with sodium bicarbonate air-powder polishing before adhesives procedures is a very common technique and aims to remove plaque and debris present in the cavity, which may influence bond strength.<sup>28,36</sup> In this study, the treatment of dentin with a sodium bicarbonate significantly increased the bond strength of the two techniques when compared to control group. This

finding contradicts the results of Frankenberger et al., which reported that a sodium bicarbonate air-polishing considerably decreased the bond strength, regardless of the adhesive and adhesive technique.<sup>37</sup> Other studies have also demonstrated that adhesion is affected when a sodium bicarbonate was applied to the dentin<sup>36</sup> and the enamel.<sup>28</sup> Rosin et al. found no significant difference in bond strength of dentin when it was abraded with sodium bicarbonate, both for self-etch and etch-and-rinse mode.<sup>29</sup> On the other hand, Nikaido et al. demonstrated that bond strength was adversely affected only when the sodium bicarbonate air-abrasion was used with a self-etching adhesive and recommended that the abraded dentin be conditioned with acid before applying the adhesive.<sup>38</sup> These findings differ from ours, but one should take into account that we used a different adhesive to evaluate the bonding of dentin treated with sodium bicarbonate.

In the present study, the high bond strength values obtained with dentin abraded with sodium bicarbonate may be explained by the modification of the abrasion technique used, as well as the type of adhesive applied. Regarding the technique, the dentin was rinsed with an air/water jet for twice the time of abrasion with sodium bicarbonate. This step could have removed the sodium bicarbonate particles more effectively. These particles can act as a contaminant and hinder the close contact between the adhesive and the dentin, resulting in the reduction of bond strength that was observed in previous studies.<sup>36-38</sup> Moreover, the Scotchbond Universal adhesive system contains the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (MDP) which has a higher bond strength than some universal adhesives.<sup>34,39</sup>

The pretreatment of dentin with aluminum oxide air abrasion improves the dentinal bond strength due to increased surface roughness and contact between the dentin and the adhesive.<sup>1,6,30</sup> In addition, the superficial removal of the smear layer by abrasion could increase the infiltration of the resin monomers in the dentin and thus increase adhesion.<sup>2</sup> In this study, when the adhesive was used in the etch-and-rinse mode, the bond strength increased significantly when the dentin abraded with aluminum oxide. On the other hand, there were no differences in bond strength when the SbU was used in self-etch mode. In partial agreement with these results, previous studies have shown that the abrasion of dentin with aluminum oxide does not interfere with bond strength in both self-etch and etch-and-rinse modes.<sup>1,2,6,8,30</sup> The increase in bond strength observed for the SbU in etch-and-rinse strategy may



be due to a change in the dentinal surface caused by abrasion with aluminum oxide, which increases the area available for adhesion.<sup>6</sup> In addition, acid conditioning removes both the smear layer and any aluminum oxide particles left on the dentinal surface, thus exposing the dentinal tubules and improving the infiltration of the adhesive in the dentin. Because of this, our first hypothesis was partially accepted.

Temperature influences some properties of monomers by decreasing the viscosity and increasing the speed of spreading of the adhesive and deeper penetration in dentin, besides increasing the degree of conversion of monomers, which influences the adhesive effectiveness.<sup>18-21</sup> In this study, however, the pre-heating of the adhesive did not significantly alter the bond strength, except when associated with aluminum oxide groups; thus, the second tested hypothesis was partially accepted. This finding corroborates some studies, which concluded that the increase in temperature had no significant effect on an adhesive based on ethanol/water, like the SbU tested in this study.<sup>14,20</sup> Other studies showed that higher immediate bond strength was associated with the highest temperatures for enamel (40 °C)<sup>23</sup> and dentin (50 °C),<sup>21</sup> however, the adhesive systems that presented these results are classified as etch-and-rinse adhesives, thus being different from the SbU. Another study revealed that the heated adhesive (40 °C) increased the bond strength for the etch-and-rinse Adper Single Bond adhesive, but did not influence the Clearfil SE Bond, considered a self-etching adhesive.<sup>18</sup>

The stereomicroscopy analysis revealed a predominance of adhesive failures for all experimental groups. This finding corroborates some studies, which showed an adhesive fracture pattern.<sup>4,20,34</sup> However, other studies have revealed a pattern of different fractures, predominantly cohesive<sup>1</sup> and mixed failures.<sup>29</sup> While adhesive failures microscopically represent a rupture in the interface between the resin and dentin characterized by an opening of the dentinal tubules, cohesive failures indicate that the hybrid layer is intact.<sup>1</sup>

Within the limitations of an *in vitro* study, our findings suggest that the dentinal surface can undergo alternative approaches prior to adhesive procedures, for example pretreatment with sodium bicarbonate air abrasion, in order to improve the performance of the Scotchbond Universal adhesive system.

## **CONCLUSION**

Pretreatment of the dentin with sodium bicarbonate air abrasion increases the bond strength of universal adhesive, regardless whether the etch-and-rinse or the self-etch technique is used. Treatment of dentin using abrasion with aluminum oxide particles influences adhesion only when using the etch-and-rinse application mode. Pre-heating of the adhesive does not significantly influence bond strength. More studies are needed to evaluate the influence of these approaches on the adhesive effectiveness of universal adhesive systems in the short and long term.

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## TABLES

Table 1: Experimental groups

Groups	Adhesive technique	Air abrasion	Adhesive temperature
1	SbU SE	No	20°C
2	SbU SE	No	37°C
3	SbU SE	Sodium bicarbonate	20°C
4	SbU SE	Sodium bicarbonate	37°C
5	SbU SE	Aluminum oxide	20°C
6	SbU SE	Aluminum oxide	37°C
7	SbU ER	No	20°C
8	SbU ER	No	37°C
9	SbU ER	Sodium bicarbonate	20°C
10	SbU ER	Sodium bicarbonate	37°C
11	SbU ER	Aluminum oxide	20°C
12	SbU ER	Aluminum oxide	37°C

*Abbreviations - SbU: Scotchbond Universal adhesive (3M ESPE, St. Paul, MN, US); SE: self-etch mode; ER: etch-and-rinse mode.*

Table 2: Material (batch number), composition and application mode according to the manufacturer's instructions

Material	Composition	Application Mode
Scotchbond Universal Adhesive – SbU (610586) 3M ESPE, St. Paul, MN, USA	10-MDP, HEMA, silane, dimethacrylate resins, Vitrebond™ copolymer, filler, ethanol, water, initiators	Self-etch technique: 1. Apply the adhesive to the entire prepared tooth and rub it in for 20 s. 2. Gently air-dry the adhesive for approximately 5 s for the solvent to evaporate. 3. Light cure for 10 s.
		Etch-and-rinse technique: 1. Apply etchant for 15 s 2. Rinse for 10 s 3. Air dry for 5 s 4. Apply adhesive as in the self-etch mode
Filtek Z350 (187396) 3M ESPE, St. Paul, MN, USA	Bis-GMA, UDMA, TEGDMA, Bis- EMA, silica particles, zirconia, silica/zirconia clusters	1. Increments of 2 mm thickness 2. Light cure each increment for 20 s

*Abbreviations – 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol A glycidyl methacrylate; UDMA: urethane dimethacrylate or 1,6-di(methacryloyloxyethylcarbamoyl)-3,3,0,5-trimethylhexaan; TEGDMA: triethylene glycol dimethacrylate; Bis-EMA, epoxyated bisphenol A dimethacrylate.*



Table 3: Microtensile Bond Strength (MPa) values (means and standard deviations) of the different experimental groups (\*)

Surface treatment	SbU Self-etch		SbU Etch-and-rinse	
	20°C	37°C	20°C	37°C
No (control)	36.14 (6.63) <sup>A</sup>	32.92 (6.19) <sup>A</sup>	30.10 (5.93) <sup>a</sup>	33.78 (4.08) <sup>a</sup>
Sodium bicarbonate	65.45 (4.46) <sup>B</sup>	59.53 (11.48) <sup>B</sup>	49.65 (2.38) <sup>b,c</sup>	51.44 (7.62) <sup>c,d</sup>
Aluminum oxide	37.46 (13.42) <sup>A</sup>	49.67 (7.2) <sup>C</sup>	44.26 (8.62) <sup>b</sup>	55.08 (3.74) <sup>d</sup>

(\*) Similar capital (self-etch) and lower (etch-and-rinse) are not statistically significant ( $p < 0.05$ ).

Table 4: Number and percentage of specimens (%) according to the fracture pattern mode

Application mode	Surface treatment - adhesive temperature	Fracture pattern			
		A	Cr	Cd	M
Self-etch	Control - 20°C	90 (84,11)	12 (11,21)	3 (2,81)	2 (1,87)
	Control - 37°C	97 (85,09)	11 (9,65)	4 (3,51)	2 (1,75)
	Sodium bicarbonate - 20°C	77 (60,16)	36 (28,12)	9 (7,03)	6 (4,69)
	Sodium bicarbonate - 37°C	70 (73,68)	19 (20,00)	4 (4,21)	2 (2,11)
	Aluminum oxide - 20°C	75 (70,75)	23 (21,70)	2 (1,89)	6 (5,66)
	Aluminum oxide - 37°C	75 (86,21)	8 (9,19)	3 (3,45)	1 (1,15)
Etch-and-rinse	Control - 20°C	87 (83,65)	8 (7,69)	5 (4,81)	4 (3,85)
	Control - 37°C	92 (83,64)	10 (9,09)	5 (4,54)	3 (2,73)
	Sodium bicarbonate - 20°C	94 (73,44)	22 (17,18)	6 (4,69)	6 (4,69)
	Sodium bicarbonate - 37°C	100 (87,72)	9 (7,89)	4 (3,51)	1 (0,88)
	Aluminum oxide - 20°C	87 (72,11)	13 (10,92)	16 (13,45)	3 (2,52)
	Aluminum oxide - 37°C	95 (66,90)	31 (21,83)	12 (8,45)	4 (2,82)

Abbreviations - A: adhesive fracture mode; Cr: cohesive in resin fracture mode; Cd: cohesive in dentin fracture mode; M: mixed fracture mode.

## FIGURES

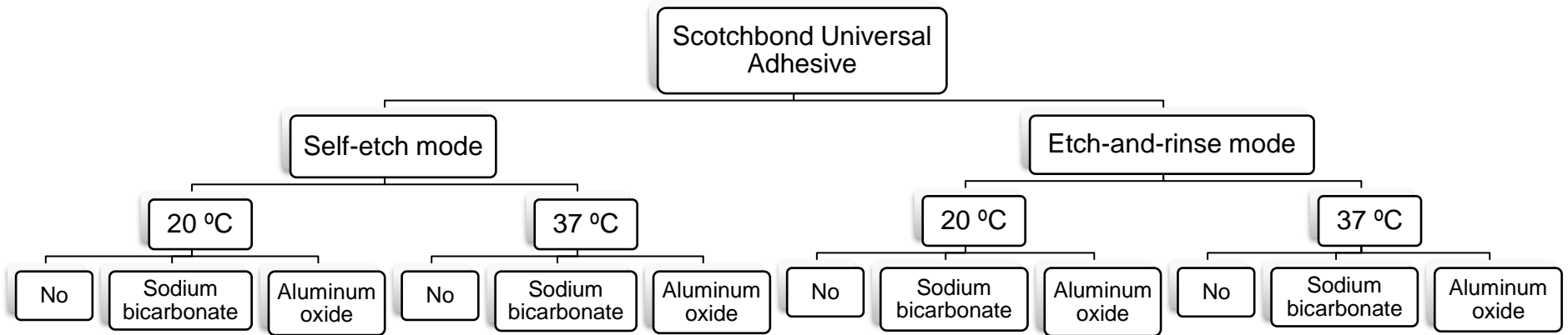


Figure 1: Experimental groups

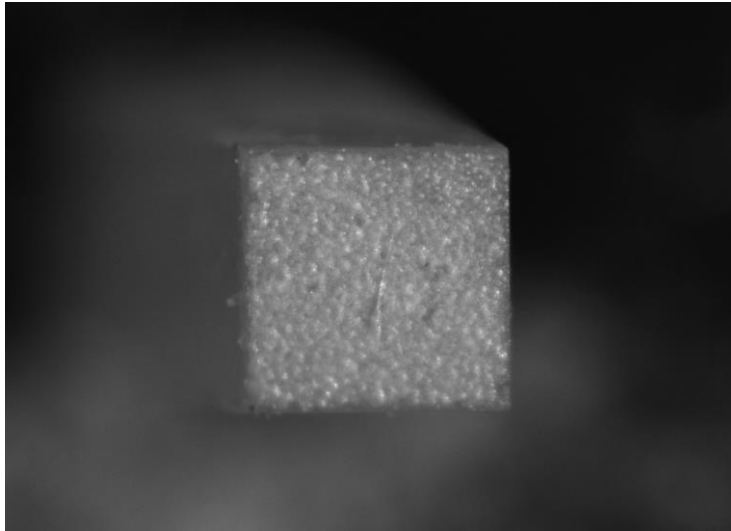


Figure 2: Representative image of adhesive fracture (40x magnification)

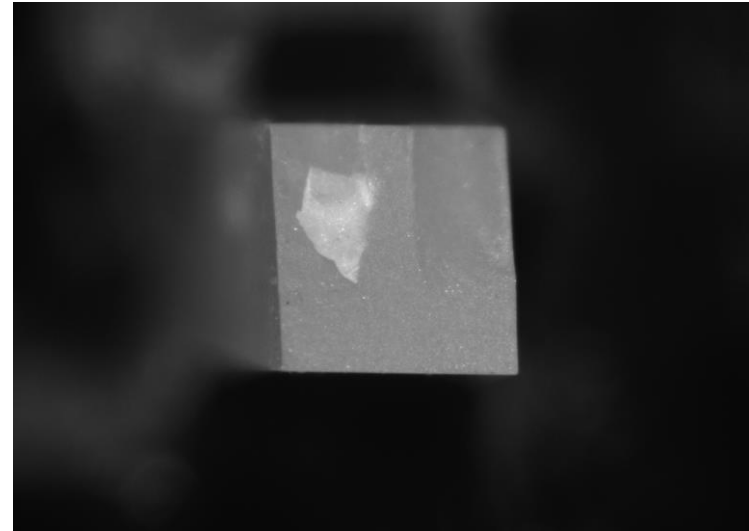


Figure 3: Representative image of cohesive in resin fracture (40x magnification)

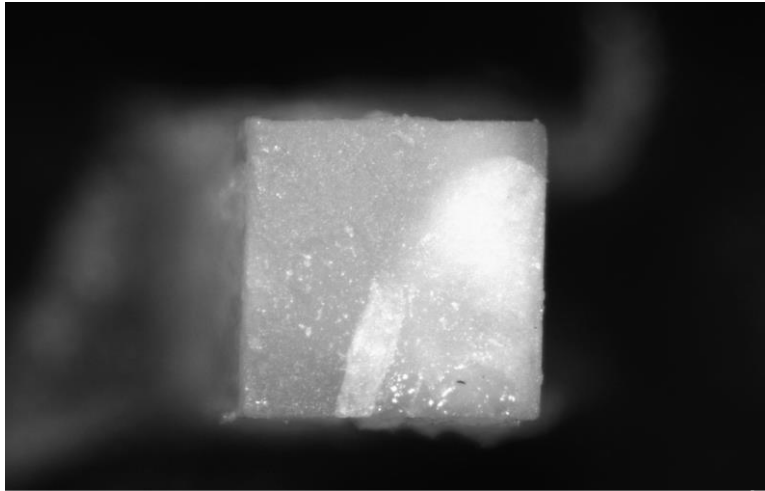


Figure 4: Representative image of cohesive in dentin fracture (40x magnification)

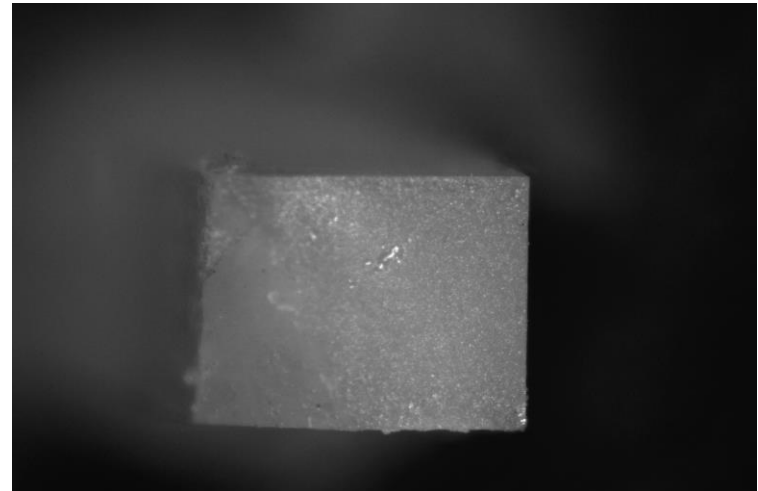


Figure 5: Representative image of mixed fracture (40x magnification)

### 3 CONCLUSÃO

O pré-tratamento da dentina com jato de bicarbonato de sódio aumenta a resistência adesiva do adesivo universal, independente da técnica etch-and-rinse ou self-etch. O tratamento dentinário com jateamento de partículas de óxido de alumínio influencia a adesão apenas na técnica etch-and-rinse. O pré-aquecimento do adesivo parece não alterar significativamente a resistência adesiva.

Portanto, dentro das limitações de um estudo *in vitro*, nossos achados sugerem que a superfície dentinária pode receber algumas abordagens alternativas antes dos procedimentos adesivos, como o pré-tratamento com partículas abrasivas de bicarbonato de sódio, com o intuito de melhorar o desempenho do sistema adesivo Scotchbond Universal. Entretanto, novos estudos são necessários para avaliar a influência dessas abordagens sobre a efetividade adesiva, a curto e longo prazo, dos sistemas adesivos universais.

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WAGNER. A. et al. Bonding performance of universal adhesives in different etching modes. **Journal of Dentistry**, v. 42, n. 7, p. 800-807, 2014.

ZHANG. Z-Y. et al. Defying ageing: an expectation for dentine bonding with universal adhesives? **Journal of Dentistry**, v. 45, p. 43-52, 2016.



## **ANEXO A – NORMAS PARA PUBLICAÇÃO NO PERIÓDICO JOURNAL OF DENTISTRY**

### **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/revise/publish process. Please register at: <http://ees.elsevier.com/jjod>

### **Double-blind review**

This journal uses double-blind review, which means that both the reviewer and author name(s) are not allowed to be revealed to one another for a manuscript under review. The identities of the authors are concealed from the reviewers, and vice versa. More information is available on our website. To facilitate this, please include the following separately:

Title page (with author details): This should include the title, authors' names and affiliations, and a complete address for the corresponding author including an e-mail address.

Blinded manuscript (no author details): The main body of the paper (including the references, figures, tables and any Acknowledgements) should not include any identifying information, such as the authors' names or affiliations.

### **Use of word processing software**

It is important that the file be saved in the native format of the word processor used. The text should be in single-column format. Keep the layout of the text as simple as possible. Most formatting codes will be removed and replaced on processing the article. In particular, do not use the word processor's options to justify text or to hyphenate words. However, do use bold face, italics, subscripts, superscripts etc. When preparing tables, if you are using a table grid, use only one grid for each individual table and not a grid for each row. If no grid is used, use tabs, not spaces, to align columns. The electronic text should be prepared in a way very similar to that of conventional manuscripts (see also the Guide to Publishing with Elsevier). Note that source files of figures, tables and text graphics will be required whether or not you embed your figures in the text. See also the section on Electronic artwork.

To avoid unnecessary errors you are strongly advised to use the 'spell-check' and 'grammar-check' functions of your word processor.

### **Introduction**

The introduction must be presented in a structured format, covering the following subjects, although not under subheadings: succinct statements of the issue in question, and the essence of existing knowledge and understanding pertinent to the issue. In keeping with the house style of Journal of Dentistry, the final paragraph of the introduction should clearly state the aims and/or objective of the work being reported. Prospective authors may find the following form of words to be helpful: "The aim of this paper is to ..." Where appropriate, a hypothesis (e.g. null or a priori) should then be stated.

### **Essential title page information**

- Title. Concise and informative. Titles are often used in information-retrieval systems. Avoid abbreviations and formulae where possible.
- Author names and affiliations. Please clearly indicate the given name(s) and family name(s) of each author and check that all names are accurately spelled. Present the authors' affiliation addresses (where the actual work was done) below the names. Indicate all affiliations with a lower-case superscript letter immediately after the author's name and in front of the appropriate address. Provide the full postal address of each affiliation, including the country name and, if available, the e-mail address of each author.
- Corresponding author. Clearly indicate who will handle correspondence at all stages of refereeing and publication, also post-publication. Ensure that the e-mail address is given and that contact details are kept up to date by the corresponding author.
- Present/permanent address. If an author has moved since the work described in the article was done, or was visiting at the time, a 'Present address' (or 'Permanent address') may be indicated as a footnote to that author's name. The address at which the author actually did the work must be retained as the main, affiliation address. Superscript Arabic numerals are used for such footnotes.

The title page should contain the following information:

- Title of paper
- Short title
- Name(s), job titles and address(es) of author(s) (no academic degrees necessary)
- Name, address, telephone, fax and e-mail of the corresponding author
- Up to 6 keywords

The house style of Journal of Dentistry requires that articles should be arranged in the following order: Title, Abstract, Introduction, Materials and Methods, Results, Discussion, Conclusions, Acknowledgements, References, Tables, Figures. A cover letter should accompany the new manuscript submission, within which the authors should indicate the significance of the work being submitted in a statement no more than 100 words. A signed permission note (details below) must also be included.

**Abstract:** should not exceed 250 words and should be presented under the following subheadings: Objectives. Methods; Results; Conclusions (For Reviews: Objectives; Data; Sources; Study selection; Conclusions). A 50 word 'Clinical Significance' statement should appear at the end of the abstract advising readers of the clinical importance and relevance of their work. These subheadings should appear in the text of the abstract. Please repeat the title of the article at the top of the abstract page.

**Introduction:** must be presented in a structured format. covering the following subjects. although not under subheadings: succinct statements of the issue in question. and the essence of existing knowledge and understanding pertinent to the issue. In keeping with the house style of Journal of Dentistry. the final paragraph of the introduction should clearly state the aims and/or objective of the work being reported. Prospective authors may find the following form of words to be helpful: "The aim of this paper is to ..." Where appropriate. a hypothesis (e.g. null or a priori) should then be stated.

**Keywords:** up to 6 keywords should be supplied.

### **Statistics**

Statistical methods should be described with enough detail to enable a knowledgeable reader with access to the original data to verify the reported results. When possible. findings should be quantified and appropriate measures of error or uncertainty (such as confidence intervals) given. Details about eligibility criteria for subjects. randomization and the number of observations should be included. The computer software and the statistical method(s) used should be specified with references to standard works when possible (with pages specified). See [http://www.icmje.org/manuscript\\_1prepare.html](http://www.icmje.org/manuscript_1prepare.html) for more detailed guidelines.

**References:** These should appear in the text in numerical order and should follow a modified form of the Vancouver Reference system (details may be found at <http://www.icmje.org/index.html#reference>). Please note that the house style of the Journal of Dentistry is different from the standard Vancouver reference style in that it includes a requirement:

- to refer to the name of the Journal in full
- to put the name of the Journal in Italics
- to put the volume number in bold

### **Color artwork**

Please make sure that artwork files are in an acceptable format (TIFF (or JPEG). EPS (or PDF). or MS Office files) and with the correct resolution. If. together with your accepted article. you submit usable color figures then Elsevier will ensure. at no additional charge. that these figures will appear in color online (e.g.. ScienceDirect

and other sites) regardless of whether or not these illustrations are reproduced in color in the printed version. For color reproduction in print, you will receive information regarding the costs from Elsevier after receipt of your accepted article. Please indicate your preference for color: in print or online only. Further information on the preparation of electronic artwork.

### **Illustration services**

Elsevier's WebShop offers Illustration Services to authors preparing to submit a manuscript but concerned about the quality of the images accompanying their article. Elsevier's expert illustrators can produce scientific, technical and medical-style images, as well as a full range of charts, tables and graphs. Image 'polishing' is also available, where our illustrators take your image(s) and improve them to a professional standard. Please visit the website to find out more.

### **Figure captions**

Ensure that each illustration has a caption. Supply captions separately, not attached to the figure. A caption should comprise a brief title (not on the figure itself) and a description of the illustration. Keep text in the illustrations themselves to a minimum but explain all symbols and abbreviations used.

### **Tables**

Please submit tables as editable text and not as images. Tables can be placed either next to the relevant text in the article, or on separate page(s) at the end. Number tables consecutively in accordance with their appearance in the text and place any table notes below the table body. Be sparing in the use of tables and ensure that the data presented in them do not duplicate results described elsewhere in the article. Please avoid using vertical rules.

### **Reference management software**

Most Elsevier journals have their reference template available in many of the most popular reference management software products. These include all products that support Citation Style Language styles, such as Mendeley and Zotero, as well as

EndNote. Using the word processor plug-ins from these products, authors only need to select the appropriate journal template when preparing their article, after which citations and bibliographies will be automatically formatted in the journal's style. If no template is yet available for this journal, please follow the format of the sample references and citations as shown in this Guide.

Users of Mendeley Desktop can easily install the reference style for this journal by clicking the following link:

<http://open.mendeley.com/use-citation-style/journal-of-dentistry>

When preparing your manuscript, you will then be able to select this style using the Mendeley plug-ins for Microsoft Word or LibreOffice.

### **Submission checklist**

The following list will be useful during the final checking of an article prior to sending it to the journal for review. Please consult this Guide for Authors for further details of any item. Ensure that the following items are present:

One author has been designated as the corresponding author with contact details:

- E-mail address
- Full postal address

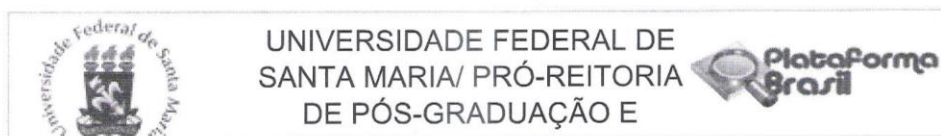
All necessary files have been uploaded, and contain:

- Keywords
- All figure captions
- All tables (including title, description, footnotes)

Further considerations

- Manuscript has been 'spell-checked' and 'grammar-checked'
- References are in the correct format for this journal
- All references mentioned in the Reference list are cited in the text, and vice versa

## ANEXO B - PARECER CONSUBSTANCIADO DO COMITÊ DE ÉTICA EM PESQUISA DA UNIVERSIDADE FEDERAL DE SANTA MARIA



### PARECER CONSUBSTANCIADO DO CEP

#### DADOS DO PROJETO DE PESQUISA

**Título da Pesquisa:** Resistência ao microcissalhamento de um sistema adesivo universal: Efeitos do pré-tratamento da superfície dentinária, da temperatura do adesivo e de diferentes tempos de armazenamento

**Pesquisador:** ALEXANDRE HENRIQUE SUSIN

**Área Temática:**

**Versão:** 2

**CAAE:** 47522515.7.0000.5346

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

**Patrocinador Principal:** Financiamento Próprio

#### DADOS DO PARECER

**Número do Parecer:** 1.195.439

#### Apresentação do Projeto:

O projeto corresponde a dissertação de mestrado vinculada ao Programa de Pós-Graduação em Ciências Odontológicas.

Trata-se de estudo laboratorial que irá avaliar o efeito de diferentes tratamentos sobre a resistência adesiva de um sistema adesivo universal a dentina. Serão utilizados 96 dentes (n=8) divididos aleatoriamente em 12 grupos, de acordo com: a técnica adesiva a ser aplicada (autocondicionante ou com condicionamento ácido prévio), temperatura do adesivo (ambiente ou a 37°C), com e sem jateamento com partículas de óxido de alumínio e armazenamento (1 ou 180 dias). Serão realizados o teste de microcissalhamento, análise do modo de falha em estereomicroscopia e MEV e os dados submetidos a análise estatística.

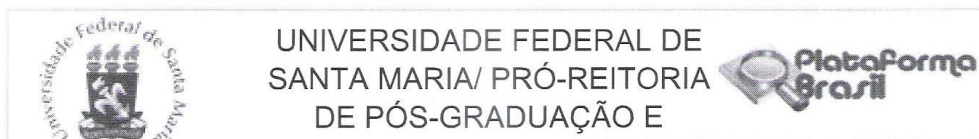
Pesquisadores apresentam cronograma e orçamento.

#### Objetivo da Pesquisa:

O presente trabalho se propõe a avaliar:

- O efeito do pré-tratamento da superfície dentinária sobre a resistência ao microcissalhamento da dentina;

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**UF:** RS **Município:** SANTA MARIA  
**Telefone:** (55)3220-9362 **E-mail:** cep.ufsm@gmail.com



Continuação do Parecer: 1.195.439

- O impacto da temperatura sobre as propriedades adesivas de um sistema adesivo universal;
- A influência da armazenagem/envelhecimento na durabilidade de união entre dentina e resina.

**Avaliação dos Riscos e Benefícios:**

Sobre os riscos cita-se: "Os riscos relacionados à pesquisa serão praticamente inexistentes, visto que não haverá captação de sujeitos, já que se trata de um estudo laboratorial, na qual serão utilizados dentes extraídos doados pelo Banco de Dentes Humanos da UFSM. A manipulação dos dentes será feita utilizando luvas de procedimento e somente após a desinfecção dos mesmos em solução de cloramina T a 0,5%."

Riscos estão descritos de maneira adequada e consistente no projeto e na Plataforma Brasil.

Sobre os benefícios cita-se: "Através dos resultados obtidos, se favoráveis, os tratamentos dentinários propostos poderão auxiliar o cirurgião-dentista a obter restaurações adesivas mais estáveis, beneficiando seus pacientes."

Benefícios estão adequadamente descritos.

**Comentários e Considerações sobre a Pesquisa:**

A pesquisa é relevante pois objetiva uma melhor performance das restaurações adesivas realizadas nos pacientes.

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

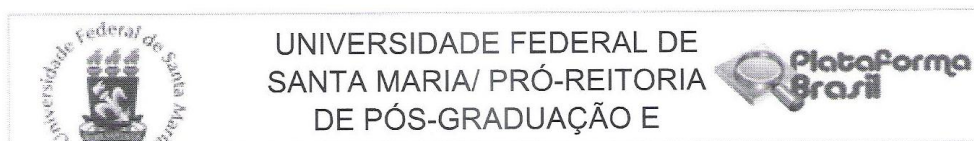
Folha de rosto, registro no GAP, Autorização institucional e Termo de Confidencialidade estão apresentados de maneira adequada.

São apresentados dois Termos de Confidencialidade. É necessário apenas um termo. Sugere-se utilizar o modelo apresentado no site do CEP, o que foi feito pelos pesquisadores em um dos documentos apresentados.

Os pesquisadores propõe dispensa de TCLE e justificam da seguinte forma: "A pesquisa usará dentes extraídos doados pelo banco de dentes permanentes humanos da UFSM."

A justificativa é aceitável e os pesquisadores apresentaram termo de doação de dentes assinada pelo

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Continuação do Parecer: 1.195.439

responsável pelo Banco de Dentes Humanos da UFSM comprometendo-se a doar os dentes para a referida pesquisa após a aprovação pelo Comitê de Ética.

**Recomendações:**

Veja no site do CEP - <http://w3.ufsm.br/nucleodecomites/index.php/cep> - na aba "orientações gerais", modelos e orientações para apresentação dos documentos. Acompanhe as orientações disponíveis, evite pendências e agilize a tramitação do seu projeto.

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

O projeto não apresenta pendências e pode ser aprovado.

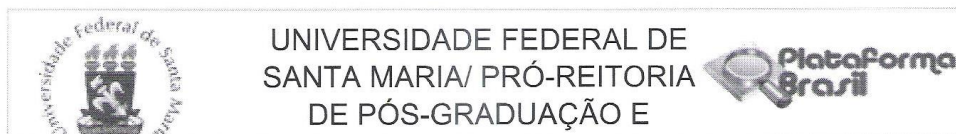
**Considerações Finais a critério do CEP:**

Este parecer foi elaborado baseado nos documentos abaixo relacionados:

Tipo Documento	Arquivo	Postagem	Autor	Situação
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Declaração de Pesquisadores	GAP.jpg	13/04/2015 21:16:43		Aceito
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Informações Básicas do Projeto	PB_INFORMAÇÕES_BÁSICAS_DO_PROJETO_496256.pdf	13/04/2015 21:18:28		Aceito
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Outros	PB_XML_INTERFACE_REBEC.xml	13/04/2015	ALEXANDRE	Aceito

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Continuação do Parecer: 1.195.439

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Outros	BANCO DE DENTES.jpg	12/08/2015 17:01:00		Aceito
Projeto Detalhado / Brochura Investigador	Projeto de Pesquisa.docx	12/08/2015 17:13:31		Aceito
Projeto Detalhado / Brochura Investigador	Projeto de Pesquisa.docx	12/08/2015 17:13:31		Aceito
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**Situação do Parecer:**

Aprovado

**Necessita Apreciação da CONEP:**

Não

SANTA MARIA, 24 de Agosto de 2015

Assinado por:  
**CLAUDEMIR DE QUADROS**  
(Coordenador)

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